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14 November 1991

University of California
Los Alamos National Laboratory
Attention: Dr. Kenneth Rea
Environmental Restoration Program
Technical Support Office
P.O. Box 1663, MS K-485
Los Alamos, NM 87545

Re: ER Program, Mound Plant
Operable Unit 3, Miscellaneous Sites
Quality Assurance Project Plan, Final

RFW WO# 2744-40-01

Dear Dr. Rea:

The Operable Unit 3, Miscellaneous Sites, Quality Assurance Project Plan (QAPP) has been approved by US EPA and Ohio EPA. The Draft and Draft Final revisions were annotated by strike-through and double-underline text to show revisions; therefore, it is necessary to issue a Final QAPP that contains only clean copy. Distribution of the Final QAPP is as follows:

Diana Mally (US EPA)	5
Martha Hatcher (OEPA)	3
Art Kleinrath (DOE DAO)	1
Dick Neff (EG&G)	5
John Sands (DOE EM-40)	1
Dave Meyer (DOE AL)	1
Tom Farmer (ER TSO)	1
WESTON Internal	6
WESTON/IT Analytics	3

In addition to the removal of strike-throughs and underlines, minor editing corrections were made to correct a misspelled word on page 1-1, correct page numbers on the table of contents, and insert Figure 10.1 after its first call-out in text.

If you have any questions, please contact John Price or Cemantha Davisson at (513) 772-3444 or Lyn Lawlor at (215) 430-7331.

Sincerely,

ROY F. WESTON, INC.

for William M. Little

Michael P. Mauzy, P.E.
ER Program, TSO Subcontractor
Project Manager

JBP/shm

Enclosure

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23-01 -01 -02 - -9407140018

ENVIRONMENTAL RESTORATION PROGRAM

MISCELLANEOUS SITES OPERABLE UNIT 3, SITE-WIDE QUALITY ASSURANCE PROJECT PLAN

MOUND PLANT
MIAMISBURG, OHIO

October 1991

DEPARTMENT OF ENERGY

ALBUQUERQUE OPERATIONS OFFICE

ENVIRONMENTAL RESTORATION PROGRAM

TECHNICAL SUPPORT OFFICE

LOS ALAMOS NATIONAL LABORATORY

FINAL

ENVIRONMENTAL RESTORATION PROGRAM

MISCELLANEOUS SITES

OPERABLE UNIT 3

LIMITED FIELD INVESTIGATION ACTIVITIES AT MOUND PLANT
QUALITY ASSURANCE PROJECT PLAN

MOUND PLANT
MIAMISBURG, OHIO

October 1991

DEPARTMENT OF ENERGY

ALBUQUERQUE OPERATIONS OFFICE

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TECHNICAL SUPPORT OFFICE

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30 January 1992

University of California
Los Alamos National Laboratory
Attention: Dr. Kenneth Rea
Environmental Restoration Program
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P.O. Box 1633, MS K-485
Los Alamos, New Mexico 87545

RE: ER Program, Mound Plant
Miscellaneous Sites, Operable Unit 3
QAPP Addendum, Revision 3
RFW WO #2744-40-01

Dear Dr. Rea:

Please find enclosed a replacement page 6-1 to the Operable Unit 3, Miscellaneous Sites, Quality Assurance Project Plan (QAPP) Addendum. This page replaces the one distributed on January 24, 1992.

This revision is being transmitted directly to U.S. EPA and Ohio EPA at the request of Art Kleinrath (DOE DAO).

Should you have any questions, please contact John Price or Cemantha Davisson at (513) 772-3444, or Lyn Lawlor at (215) 430-7331.

Sincerely,

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24 January 1992

University of California
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RE: ER Program, Mound Plant
Miscellaneous Sites, Operable Unit 3
QAPP Addendum, Revision 3
RFW WO #2744-40-01
REVISED, replaces 1/9/92 version

Dear Dr. Rea:

Please find enclosed modification pages to the Operable Unit 3, Miscellaneous Sites, Quality Assurance Project Plan (QAPP) Addendum. These modifications address the following changes to the scope of work:

Expanded Field Quality Control

Equipment (rinsate) blanks have been added to the field quality control requirements. This data will be used to evaluate the sampling equipment decontamination technique. With this addition, analytical methodology and laboratory quality control for analysis of water samples have been included. The IT Analytical Services-Technology Development Laboratory SOP for the preparation and analysis of water samples has been included for U.S. EPA and Ohio EPA review.

Addition of IT St. Louis Laboratory

Sediment samples collected in the glass melter room potentially have radioactivity levels above that which can be handled at the IT Technology Development Laboratory in Knoxville, TN. The IT Analytical Services Laboratory in Earth City, MO (i.e., St. Louis Laboratory) is capable of receiving these samples and can provide dioxin/furan analyses in accordance with the conditions of the newest revision to the QAPP addendum. We will advise you if, upon collection, these samples are radioactive enough to be shipped to St. Louis.

These revisions are being transmitted directly to U.S. EPA and Ohio EPA at the request of Art Kleinrath (DOE DAO).

Should you have any questions, please contact John Price or Cemantha Davisson at (513) 772-3444, or Lyn Lawlor at (215) 430-7331.

Sincerely,

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for William M. Little

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	D. Root, IT-TDL	
	ER TSO File	

OPERABLE UNIT 3

LIMITED FIELD INVESTIGATION

QUALITY ASSURANCE PROJECT PLAN

DEPARTMENT OF ENERGY, MOUND PLANT

MIAMISBURG, OHIO

Approved by _____ Date 10/22/91
U.S. EPA Region V Remedial Project Manager

Approved by _____ Date 10/18/91
U.S. EPA Region V Quality Assurance Manager

Approved by _____ Date 10/24/91
Ohio EPA Remedial Project Manager

Approved by _____ Date 10/24/91
DOE Dayton Area Office Remedial Project Manager

Approved by _____ Date 10/24/91
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Approved by _____ Date 10/24/91
ER Program Manager, Mound Plant

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APPENDIX C IT ANALYTICAL SERVICES QUALITY ASSURANCE MANUAL - IT RADIOLOGICAL SCIENCES LABORATORY (OAK RIDGE, TENNESSEE)

APPENDIX D IT ANALYTICAL SERVICES QUALITY ASSURANCE MANUAL - IT ANALYTICAL LABORATORY KNOXVILLE (MIDDLEBROOK PIKE), TENNESSEE

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ACRONYMS

AA	atomic absorption
ASTM	American Society for Testing and Materials
CCV	continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CGI	combustible gas indicator
CLP	Contract Laboratory Program
CRDL	contract-required detection limit
CRQL	contract-required quantitation limit
DOE	U.S. Department of Energy
EPA	U.S. Environmental Protection Agency
ER	Environmental Restoration
EPH	extractable petroleum hydrocarbons
FIDLER	field instrument for the detection of low-energy radiation
GC	gas chromatography
GC/MS	gas chromatography/mass spectrometry
GPR	ground-penetrating radar
HMX	octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine cyclotetramethylenetetranitramine
HPLC	high pressure liquid chromatography
ICB	initial calibration blank
ICP	inductively coupled plasma
ICS	interference check sample
ICV	initial calibration verification
IDL	instrument detection limit
IT	International Technology Corporation
LCS	laboratory control sample
LIMS	Laboratory Information Management System
LLD	lower limit of detection
LRA	linear range analysis
MSA	method of standard addition
MSD	matrix spike duplicate
NB	nitrobenzene
PCBs	polychlorinated biphenyls
PID	photoionization detector
PVC	polyvinyl chloride
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
RCRA	Resource Conservation and Recovery Act
RDX	hexahydro-1,3,5-trinitro-s-triazine cyclotetratmethylenetetranitramine
RI/FS	remedial investigation/feasibility study
RPD	relative percent difference
RRF	relative response factor
RSD	relative standard deviation
SOP	Standard Operating Procedure
SOW	Statement of Work
SA	amount of spike added
SD	standard deviation
SR	sample results
SSR	spiked sample result
TAL	Target Analyte List
TCL	Target Compound List

TNT	2,4,6-trinitrotoluene
TSO	Technical Support Office
Tetryl	A-methyl-N,2,4,6-tetranitroanaline
USATHAMA	U.S. Army Toxic and Hazardous Materials Agency
VOC	volatile organic compound
1,3-DNB	1,3-dinitrobenzene
1,3,5-TNB	1,3,5-trinitrobenzene
2,4-DNT	2,4-dinitrotoluene
2,6-DNT	2,6-dinitrotoluene

1. INTRODUCTION

1.1. OVERVIEW

Quality assurance is a system of measures taken to ensure that a desired product meets a defined level of quality. This Quality Assurance Project Plan (QAPP) presents the system of quality assurance to be implemented for the Operable Unit 3 Limited Field Investigation at the Mound Plant. The plan provides specific procedures that delineate how field and laboratory data of known and accepted quality will be generated.

A system of quality assurance consists of two elements: quality control and quality assessment. Quality control is a system of procedures performed to control the quality of the product, usually with defined standards of performance for those procedures. Quality assessment is a program of activities to evaluate the performance of implemented quality control procedures and the quality of the product.

This QAPP describes the standards of performance, defined as acceptance criteria, for quality control checks as presented in section 3. This QAPP also describes the quality control procedures for sampling activities (sampling procedures in section 4 and sample custody in section 5), for field screening and field measurements (section 6), and for laboratory analyses (section 6). Quality control procedures for calibration of field and laboratory instrumentation are outlined in section 7. Specific quality control steps, defined as quality control checks, for these activities are discussed in section 8. The procedures for data reduction, validation, and reporting are included in section 9. As part of the quality control program, preventive maintenance procedures for equipment and instrumentation are summarized in section 11. Corrective actions for the planned field and laboratory activities are necessary for a quality control program in order to keep the quality of generated data under control. The corrective actions for these activities are provided in section 13.

Quality assessment activities for this investigation include evaluation of field and laboratory quality control data, performance and system audits, and issuing of quality assurance reports to management. Procedures for these activities are described in sections 10, 12, and 14, respectively.

In fulfilling its role of ensuring that the goals of the project are met, the quality assurance program relies on the structure of the project organization and on the effectiveness of key individuals in carrying out their responsibilities. This report describes the project organization and identifies the individuals who are responsible for assessing the collection and generation of data and for ensuring that this data is of defined quality. This organizational structure and the defined responsibilities are provided in section 2.

1.2. PROJECT DESCRIPTION

Operable Unit 3 includes 22 potential release sites at Mound Plant for which there are little or no data currently available. The objectives of the Operable Unit 3 Limited Field Investigation are to develop a conceptual site model, enhance the scoping effort, and provide adequate data to write the work plan (EPA 1988a). Furthermore, the Federal Facility Agreement between the U.S. Department of Energy (DOE) and the U.S. Environmental Protection Agency (EPA) states the following:

> Based on the results of these studies, potential release sites will either be eliminated from further investigation or carried forward into the Remedial Investigation/Feasibility Study (RI/FS).

Therefore, an additional objective of this limited field investigation is to collect sufficient data to eliminate any of the Miscellaneous Sites from further consideration, collect sufficient data to combine any site with another appropriate Mound Plant Operable Unit, or determine that an addendum will be prepared to the Operable Unit 3 Work Plan to address additional site characterization of any Mound Plant Miscellaneous Site.

This is a limited field investigation; therefore, it focuses primarily on source identification and characterization and does not include all elements of an RI/FS. For example, characterization of hydrogeology and groundwater contamination is generally not addressed by the work proposed in this plan. Such elements would be included in a follow-up RI/FS work plan if further investigation is deemed necessary.

The following sections present summaries of the background information concerning the 22 potential release sites included in Operable Unit 3. It should be noted that little or no past data are available for most of these sites. The data that are available are discussed in the Operable Unit 3 Limited Field Investigation Work Plan, which also presents the proposed sampling for this investigation. Figure 1.1 shows the locations and approximate sizes of the Operable Unit 3 potential release sites. More detailed figures are presented in the Operable Unit 3 Limited Field Investigation Work Plan.

1.2.1. Paint Shop Area

The paint shop area is located on the Mound Plant Main Hill, southeast of Building M (Figure 1.1). This site covers approximately 3,600 ft^2 of surface area, most of which is covered by asphalt. Historically, paint shop wastes were placed at the Area B landfill (DOE 1989) prior to offsite disposal by a contractor (DOE

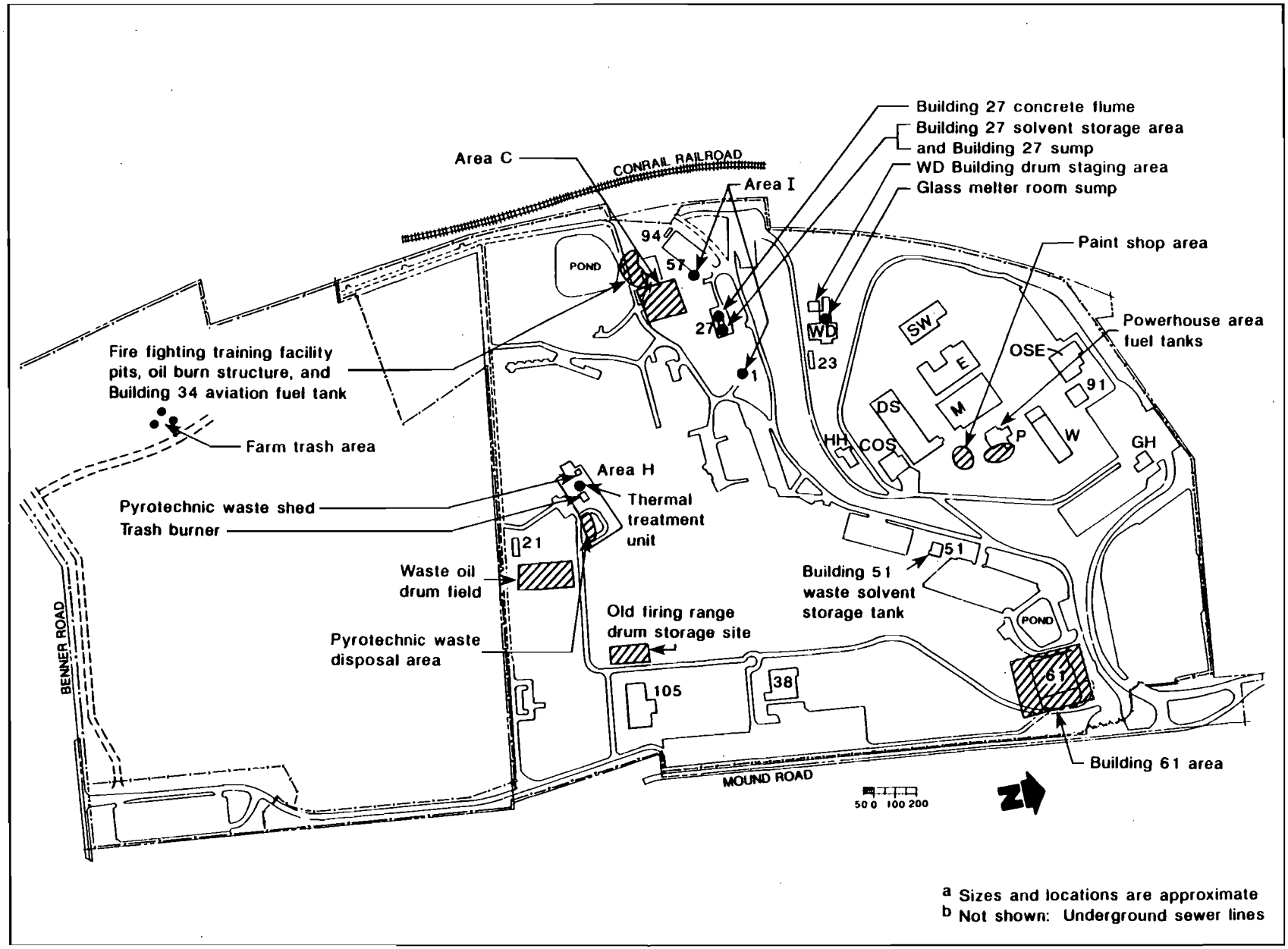


Figure 1.1. Operable Unit 3 potential release sites.[a,b]

1986). However, it is possible that leaks or spills could have contaminated the local area around the paint shop (DOE 1986). Currently, at least one 55-gallon drum of materials (potentially consisting of waste) is located at the paint shop building. The type or quantity of contaminants that may have been released onto soils in this area is not known. No environmental samples have been collected at the paint shop area.

1.2.2. Powerhouse Area

The powerhouse fuel tank area is located on the Main Hill, immediately east of Building P (the powerhouse) and northeast of the paint shop area (Figure 1.1). Stained soils are present in the powerhouse area, indicating that spills have occurred. A spill of No. 2 fuel oil (approximately 20 ft^2 in size) has been reported, but a search of available documents has not substantiated this information. Fuel oil contamination of the area may have resulted from leakage of fuel from four 25,000-gallon tanks located at Building P. No environmental samples have been collected at the powerhouse area.

1.2.3. WD Building Drum Staging Area

The WD Building drum staging area is located at the southwest corner of the WD Building (Figure 1.1). Both empty and partially filled 55-gallon drums are staged in this area. The drum staging area is under lain by a a concrete pad, which slopes to the south to drain precipitation runoff. There is no curbing on the pad to prevent runoff from discharging to the south.

The radioactive/mixed waste storage area, Limited Action Sites, Operable Unit 7 (not part of this site, but adjacent to it inside the WD Building) stores radioactive/mixed wastes including scintillation vials and acid/base waste, polychlorinated biphenyls (PCBs), lead, uranium, thorium, mercury, ethylene glycol, oils, paint thinner, and mixed unknown chemicals. Materials at the WD Building drum staging area may not include all of these materials and no inventory is available. But in 1988 it was reported that some of the open drums in the drum staging area contain small pieces of glass from the glass melter furnace. The waste in these drums are exposed to precipitation. Stains have been observed extending from the concrete pad to the adjacent soil. However, no environmental samples have been collected at the WD Building drum staging area.

1.2.4. Glass Melter Room Sump

The glass melter room sump is a concrete unit located in the WD Building annex, beneath the glass melter furnace (Figure 1.1). The furnace is proposed for use as an incinerator for various hazardous wastes and radioactive mixed wastes generated by onsite activities. Mound Plant is currently in the process of

acquiring a Resource Conservation and Recovery Act (RCRA) Part B permit that covers operation of the glass melter furnace. To date, only test burns have been conducted in this furnace. Floor drains discharge wash water from the floor beneath the furnace to the sump. The sump is approximately 4 ft in diameter and 8 ft deep and is covered. The sump was also used to collect wastewater from several drains originating from the glass melter's off-gas treatment system and wash water from the floor of the glass melter room. Mound Plant personnel describe the sump as being plugged and therefore not capable of being operated at top efficiency. Potential contamination of the sump may be associated with wastewater being treated by the off-gas treatment system and floor wash water (RFA 1988). No samples have been taken of the liquid in the sump.

1.2.5. Building 51 Waste Solvent Tank

The Building 51 waste solvent tank is located adjacent to Building 51 in the valley between Main Hill and SM/PP Hill (Figure 1.1). The tank was used from 1970 until the waste solvent incinerator was dismantled in 1972 (RFA 1988). The 1,000-gallon tank was used to store waste organic solvents from various Mound Plant processes and may have included some metals, radioactive contaminants, or PCBs, depending on how the solvents were used.

The Building 51 solvent storage tank was removed as part of the Mound Plant underground tank program in December 1990. Prior to the removal, notice was provided to the Ohio EPA Bureau of Underground Storage Tank Regulation. In addition, notice was provided to the Ohio EPA RCRA Section that the Building 51 tank was being removed as an RCRA closure. A closure plan for the tanks was included as part of the notice.

The closure plan for the tanks included provision for sampling and analysis of soils, and samples were collected. As of January 1991, a closure report, including discussion of sampling and analysis, was in preparation. The sampling and analysis plan for soil samples collected during the tank removals was not reviewed by the EPA Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) group nor was it reviewed by the Ohio EPA Office of Corrective Actions, and the acceptability of the data to those entities has not been determined. Also, the regulatory status of the tanks, i.e., whether they should be included in the CERCLA program, is under evaluation. Therefore, it is not clear whether additional sampling and analysis are needed to fulfill CERCLA objectives, and the proposed sampling and analysis specifications in the work plan has been included as a contingency.

1.2.6. Building 61 Former Heavy Equipment Area

Building 61, the Mound Plant shipping and receiving center, is located in the upper valley between the Main Hill and SM/PP Hill in the northeastern portion of Mound Plant (Figure 1.1). It was used as a heavy equipment staging area in the early decades of plant operations (40s and 50s) and predates the construction of Building 61. Because the Building 61 area was used for the staging of heavy equipment, the area could have been used for dumping waste oil; however, no spills or waste disposals have been documented (DOE 1986). No environmental samples have been collected in or around Building 61.

1.2.7. Building 27 Sites

There are three Building 27 sites: the solvent storage area, the concrete flume, and the sump. All three are located behind (south of) Building 27 (Figure 1.1). These sites are discussed together in the Operable Unit 3 Work Plan because they are adjacent to each other and received the same wastewater stream, which contained acetone, ethanol, and high explosives. The primary waste liquid was acetone. The concrete flume and the solvent storage area are still active and receive the wastewater. The sump is inactive, but would receive wastewater if the concrete flume overflowed. No evidence of leaks or spills has been observed or reported at the Building 27 sites. No environmental samples have been taken at any of these sites.

The Building 27 solvent storage area is currently in service. It consists of three or four 55-gallon covered drums on a concrete pad. The drums receive Building 27-generated wastewater that was formerly discharged into a sump or leach pit. Filled drums are transported from this area to the hazardous waste storage area once a week. The drums appeared to be in good condition and no evidence of releases was observed during a RCRA visual site inspection.

The concrete flume was formerly used to convey wastewater from inside Building 27 to the Building 27 sump. Since the sump was taken out of service, the wastewater in the concrete flume has been pumped into drums at the Building 27 solvent storage area. The flume is still in service and wastewater is pumped from it into the drums twice daily. If overflows occur, the wastewater will drain into the Building 27 sump located downstream of the flume. The flume is an in-ground structure measuring approximately 20 ft long, 10 inches wide, and 12 inches deep. The capacity of the flume is approximately 100 gallons. The bottom and sidewalls of the flume are constructed of concrete, and it is covered with a metal lid.

The Building 27 sump is an inactive pit. It is approximately 6 ft long, 3 ft wide, and 4 ft deep, lined with concrete and covered with a metal lid. The sump was used to collect wastewater from Building 27, to filter

it to remove suspended contaminants, and to discharge the effluent to the Building 27 leach pit. Sump effluent was discharged by gravity through an underground pipeline. The leach pit is located approximately 200 ft west of the sump. The sump was dredged every three to four years to remove accumulated sludge. This sludge and the spent filters were destroyed onsite by thermal treatment.

The sump has been out of service since 1985. Wastewater currently generated at Building 27 does not now reach the sump, but is held in the flume and drummed. No releases have been documented other than discharge to the leach pits and no evidence of releases was observed during a visual site inspection (RFA 1988). The sump was observed to contain only rainwater, and the concrete liner appeared to be in good condition.

1.2.8. Area I, Buildings 1 and 27 Leach Pits

Area I consists of two unlined leach pits (infiltration/evaporation basins): the Building 1 leach pit, located southwest of Building 1 near the plant drainage ditch, and the Building 27 leach pit, located southwest of Building 27 and behind (south of) Building 42 (Figure 1.1).

The Building 1 leach pit is approximately 5 ft deep and triangular in shape. Two sides of the pit are approximately 75 ft long and the third side is approximately 50 ft long. The Building 1 leach pit was put into use in the early 1960s. Wastewater entered the Building 1 leach pit through three pipes: one for routine discharge from Building 43; the second for overflow from Building 43; and the third from the Building 1 sump and storm drains. All three of these pipes are still in place; however, the Building 1 leach pit presently receives water from only the Building 1 storm drains.

The Building 27 leach pit is approximately 3 ft deep and square in shape with approximate measurements of 15 ft long by 15 ft wide. The Building 27 leach pit was used in the early 1960s. Wastewater entered the Building 27 leach pit through a single underground pipe from the Building 27 sump to the center of the pit. This pipe is still in place; however, the Building 27 leach pit was closed in 1985 and receives no wastewater through the pipe.

Because there were no drains in either leach pit, the liquid fraction of the wastewater discharged into the leach pits infiltrated the ground or evaporated (DOE 1987). The Building 1 leach pit, however, contained a standpipe through which excess liquids could be discharged to the plant drainage ditch. A series of historical aerial photographs show that the Building 27 leach pit was consistently full, while the Building 1 leach pit was consistently empty. The plant drainage ditch is being addressed under the Site-Wide investigation, Operable Unit 9.

The Area I leach pits received similar wastewater streams, which contained acetone, ethanol, and high explosives. The primary waste liquid in the wastewater was acetone. The leach pits are probable sources of contaminants to the environment because the wastewater had the potential to infiltrate the subsurface at these locations.

1.2.9. Building 34 Sites

Three Building 34 sites - the oil burn structure, the fire-fighting training facility pits, and the Building 34 aviation fuel tank - are located in the area bounded by the overflow pond to the south, the retention basins to the northwest, and Building 34 to the northeast (Figure 1.1). Petroleum products were used or stored at all of these sites (RFA 1988).

The oil burn structure is an open-topped, in-ground pit, formerly used to test shipping containers by subjecting them to a fuel fire for 15 minutes. The pit has brick-lined walls, approximately 10 ft on each side and 8 ft deep. The bottom of the pit is concrete (thickness of concrete unknown). The structure was taken out of service in 1979 (RFA 1988). An unknown liquid has been observed in the bottom of the structure, and vegetation has been observed growing through the seams in the concrete. Jet fuel (JP-4) used for the burns was stored in the Building 34 aviation fuel tank located approximately 50 ft from the oil burn structure.

The fire-fighting training facility consists of two concrete pits: one approximately 10 ft long by 10 ft wide by 1 ft deep, and the other approximately 20 ft long by 10 ft wide by 1 ft deep. Black residue and rainwater have been observed in the smaller pit and the soil surrounding this pit shows black staining. Black residue has been observed on the larger pit but standing rainwater has not. Three to five gallons of diesel fuel were used to create fires for fire-fighting training purposes. The fires were extinguished with water from a nearby hydrant.

The aviation fuel tank is a 350- to 400-gallon underground storage tank, which historically supplied fuel to the nearby oil burn structure. The tank was put into service in the early 1970s and was removed from service in 1979. In 1988, approximately 250 gallons of fuel remained in the tank.

Mound Plant has an ongoing program to remove underground storage tanks as a "best management practice." Many tanks are being removed years in advance of the time required by regulation. The Building 34 aviation fuel tank was removed as part of the Mound Plant underground tank program in December 1990. Prior to the removal, notice was provided to the Ohio EPA Bureau of Underground Storage Tank Regulation. In addition, notice was provided to the Ohio EPA RCRA Section that the Building 51 tank was being removed as an RCRA closure. A closure plan for the tank was included as part of the notice.

The closure plan for the tank included provision for sampling and analysis of soils, and samples were collected. As of January 1991, a closure report, including discussion of sampling and analysis, was in preparation. The sampling and analysis plan for soil samples collected during the tank removal was not reviewed by the EPA CERCLA group nor was it reviewed by the Ohio EPA Office of Corrective Actions, and the acceptability of the data to those entities has not been determined. Also, the regulatory status of the tank, i.e., whether it should be included in the CERCLA program, is under evaluation. Therefore, it is not clear whether additional sampling and analysis is needed to fulfill CERCLA objectives, and the proposed sampling and analysis specifications in the work plan have been included as a contingency.,

Aviation fuel was stored at the Building 34 aviation fuel tank and was used in test-burning operations at the oil burn structure. Components of aviation fuel include the VOCs benzene, toluene, ethylbenzene, and xylenes. Diesel fuel was used in the fire-fighting training pits to create demonstration fires. Approximately 300 gallons of diesel fuel were used at the facility in 1987 at a rate of 3 to 5 gallons per demonstration.

The VOCs benzene, toluene, ethylbenzene, and xylenes are only minor components of diesel fuel. Semivolatile organic compounds such as naphthalene and other polynuclear aromatic compounds are major components of diesel fuel.

1.2.10. Area C, Waste Storage Area

Area C is located in the lower central valley at Mound Plant. The area is estimated to have measured up to 200 ft by 150 ft and was low land located beside the plant drainage ditch. In the 1950s, Area C was used for disposal of lithium carbonate containers (RFA 1988). Subsequent to its use for disposal of the containers, fill was added to the natural topography at Area C. As a result, the exact location of Area C is under some question, but its approximate location is immediately east of Building 34 and northeast of the overflow pond (Figure 1.1).

The thickness of the fill added to Area C has been estimated using ground-penetrating radar (GPR) data. It is estimated to range from about 0 ft at the east end of the area, to a maximum of 9 ft in the middle, to a depth of 4 ft at the west end of the area. Currently, Area C is a grassy open field that is used as a contractors' equipment yard. Semi-trailers, rebar, and other non-hazardous materials are staged in the area.

The lithium containers placed in Area C during the 1950s potentially contain residual lithium carbonate. In addition, small quantities of other wastes could possibly have been placed in this area (DOE 1986), but the nature (volumes and types) of any other wastes is unknown.

Reportedly, metal debris from Area 13 was moved to Area C in 1950. The metal was originally placed in Area 13 with polonium-210-contaminated-wood. The wood was burned in 1950, and the remaining metal debris moved to Area C. Any residual polonium-210 in the debris moved to Area C would have decayed since 1950 because polonium-210 has a half-life of 138.4 days. Area 13 is being addressed under Radioactively Contaminated Soils (Operable Unit 5). Some of the anomalies detected during the GPR survey in Area C (described below) may represent the lithium containers or metal from Area 13.

1.2.11. Area H

Area H is located near Building 53, in the southwestern portion of the SM/PP Hill at Mound Plant (Figure 1.1) (DOE 1986). There are four separate sites within the boundaries of Area H: the pyrotechnic waste disposal area, the trash burner, the thermal treatment unit, and the pyrotechnic waste shed. These sites are grouped in one area because of the similar nature of the potential contaminants at the sites (pyrotechnic materials) and because the investigations of the sites overlap each other due to their close proximity. These sites are described below.

The pyrotechnic waste disposal area includes the area that was formerly used for disposal of pyrotechnic materials in solution (RFA 1988). The area is reported to be adjacent to the southern fence line of the area. Wastewater containing dissolved pyrotechnic materials was last disposed of in this area in 1985. The filtrate containing small amounts of dissolved pyrotechnic materials was poured on the ground.

The trash burner (crucible) is an inactive unit (dismantled in 1988) located in Area H, east of Building 90 on the SM/PP Hill (Figure 1.1). The trash burner was enclosed in a 4-ft by 4-ft by 4-ft metal box (cubicle) and a 10-ft by 10-ft by 12-ft high chain-link fence enclosure. A screen gate over the box prohibited releases of large particulate matter to the atmosphere. The box was underlain by concrete.

Waste burned or detonated at the trash burner included mild detonating fuses, pyrotechnic materials, small amounts of explosives, and cardboard contaminated with explosives, pentramine cobalt (III) perchlorate, and thermite powder; wastes may also contain organic solvents such as acetone and Freon™. The trash burner was used to burn up to 40 pounds of high explosives trash per event. The ash was collected in a 55-gallon drum adjacent to the trash burner.

The thermal treatment unit is located east of Building 90 in Area H (Figure 1.1). The thermal treatment unit is an active unit operated on a batch basis. The unit consists of a 55-gallon drum located inside a 10-ft by 10-ft by 10-ft concrete cubicle, covered with a metal screen roof. The concrete walls are lined with 4-inch-thick steel plate. The floor of the cubicle is covered with sand. Wastes are detonated remotely from an

adjacent building. The drum is mounted on fire brick and cooled during the burn by a surrounding base filled with water and antifreeze.

The pyrotechnic waste shed is a temporary storage area for pyrotechnic waste prior to thermal destruction. The shed is constructed on a concrete pad without a curb. The unit is still in service. The dimensions of the shed are 15 ft by 9 ft, with 7-ft-high chain-link fence side walls. The roof is constructed of 18-gauge galvanized corrugated steel. Contaminated trash and pyrotechnic wastes are saturated with mineral oil, double-bagged, and sealed in a metal can. Pyrotechnical components are stored in non-propagating trays inside a metal suitcase.

1.2.12. Waste Oil Drum Field Area

The waste oil drum field is an inactive site located northeast of Building 21 and southeast of Area H on SM/PP Hill (Figure 1.1). The area was approximately 50 ft by 100 ft (5,000 ft^2) and contained drums. With approval of the EPA, 55-gallon drums were temporarily stored on pallets above the soil during an interim period between dismantling of the previous hazardous waste storage facility (Old Building 72) and completion of the new building. The drums were moved to the hazardous waste storage area of the Limited Action Sites, Operable Unit 7, when it was completed.

Drums at the south end of the drum field contained waste oil; drums at the north end contained metal-plating shop waste, solvents, explosive/solvent waste, batteries, kitchen grease, herbicides, photographic solutions, epoxy resins, lab chemicals, ethylene glycol, scintillation vials (less than 50 Ci/liter), and other unknown chemical wastes. Stained soil has reportedly been observed at one location at the southern end of the area, but no stains were observed during a 1988 site inspection. No environmental samples have been collected in this area.

1.2.13. Old Firing Range Drum Storage Site

The old firing range drum storage site is located west of Building 105 on SM/PP Hill (Figure 1.1). The area is approximately 150 ft by 100 ft (15,000 ft^2) and was used for drum storage in the 1960s. The exact number of drums that were stored in this area and whether the drums leaked are unknown. In the 1970s, the drums were moved to the hazardous waste storage area of the Limited Action Sites, Operable Unit 7. No environmental samples have been taken in this area.

1.2.14. Farm Trash Area (Previous Owner)

The farm trash area is located near the southwest corner of the undeveloped Mound Plant property (Figure 1.1). It is the former location of a residence that was razed when the DOE purchased the property in 1981. The site is about 500 ft north of Benner Road and approximately 500 ft east of the western edge of the Mound Plant property.

The farm trash area covers less than an acre. It was used to receive trash, tires, and household debris when the farm was in use. Mound Plant has never used this area for trash disposal. It is characterized by a lack of vegetation and local dark stains on soil near the assumed eastern edge of the dump. The source of the stains is not known but they are probably related to farm vehicle maintenance. No environmental samples have been collected in this area.

1.2.15. Underground Sewer Lines

The underground sewer lines located throughout the Mound Plant are used to convey sanitary and industrial wastewater and storm runoff from plant sites to the sewage disposal building area (Building 57, of Operable Unit 5, Radioactively Contaminated Soils). The sewer lines are constructed of cast iron, ductile iron, vitrified clay, steel pipe, polyvinyl chloride (PVC) pipe, or concrete with diameters ranging from 4 to 12 inches.

The sludge currently produced from the wastewater treatment is known to contain plutonium-238 but it does not exhibit hazardous characteristics. Sampling of the sludge in August 1990 by the Mound Plant Waste Management Department confirmed the absence of leachable contaminants above regulatory levels (unpublished data). Sources of wastewater conveyed through the underground sewer lines include rest rooms, showers, laundry facilities, laboratory sinks, and rinses from a small metal-refinishing operation. Some of the laboratory sinks have reportedly received small amounts of solvents, photographic solution, acids, and bases.

A TV-screening survey was performed by Mound Plant personnel on the underground sewer lines to define sections of sewer lines that need to be repaired or replaced. If a sewer line was found to be damaged or broken and it was decided that the line would be repaired instead of replaced, a fiberglass-like material (insituform) was used to repair the line. For each repair, a sleeve of the fiberglass-like material was driven into the pipe by adding pressurized water to the line. This forced the material to conform and adhere to the pipe sidewall.

1.3. SUMMARY OF OPERABLE UNIT 3 ANALYTICAL PARAMETERS

The Limited Field Investigation Work Plan discusses the scope of work and rationale for sampling and analysis for Operable Unit 3. The parameters to be analyzed are designed to determine the presence of contaminants in representative samples, including the presence of unexpected contaminants. The selected laboratory methods of analysis for these parameters will be performed under the EPA's Contract Laboratory Program (CLP) where appropriate and using approved and known methodologies where available.

Objectives for data quality are discussed in the following sections of this QAPP. However, analytical levels, as defined by EPA's "Data Quality Objectives for Remedial Response Activities" (EPA 1987), may be assigned to the planned analyses. These analytical levels were designed by the EPA to serve as guidance for obtaining data of appropriate quality for its intended use. A general description of the typical types of analyses performed, the types of data uses, the limitations, and a generic description of data quality are provided for each analytical level (I, II, III, IV, and V) in the EPA guidance. These analytical levels have been applied to each set of parameters to be analyzed and are presented in Table I.1 for each task of the investigation. The purpose for data collection and the media to be investigated for each task are also included in this table. Project schedule is provided as Table I-2.

Table I.1. Analytical Levels for Operable Unit 3 Limited Field Investigation

Site	Purpose	Media	Field Parameters	Laboratory Parameters	Analytical Level
Paint shop area	To identify hazardous contaminants that may be present due to past surface spills of paint shop wastes. Results will be used to guide decisions for further investigation	surface and subsurface soils	FIDLER		I
			organic vapors		I
			soil classification		I
				Total TCL/TAL	IV
				TCL VOCs	IV
				tritium	V
				plutonium-238 and thorium-232 screen	III
Powerhouse area fuel tanks	To determine the extent of contamination from fuel oil spills	surface and subsurface soils	organic vapors		I
			soil classification		I
			FIDLER		I
				Total TCL/TAL	IV
				EPH (as no. 2 fuel oil)	V
				TCL VOCs	IV
				plutonium-238 and thorium-232 screen	III
				EPH (for characterization of No. 2 fuel oil)	III
WD Building drum staging area	To identify hazardous contaminants that may be present in surface and subsurface soils adjacent to pad due to leaks from drums; to identify if tritium has migrated to the soils adjacent to WD building	surface and subsurface soils	organic vapors		I
			soil classification		I
			FIDLER		I
				total TCL/TAL	IV
				TCL VOCs	IV
				trituim	V
				plutonium-238 and thorium-232 screen	III

Table I.1. (continued)

Site	Purpose	Media	Field Parameters	Laboratory Parameters	Analytical Level
Glass melter room sump	To determine whether hazardous constituents are present in sump waste water and sediment	sump liquid and sediment	organic vapors pH, conductance, temperature, and alkalinity for sump liquid		I I I
				total TCL/TAL	IV
				TCL VOCs	IV
				trituim	V
				EPH (as kerosene)	V
				acetonitrile and acrylonitrile	V
				plutonium-238 and thorium-232 screen	III
Building 51, Waste solvent storage tank	To identify hazardous contaminants that may be present where the tank was removed and to verify closure	surface soils in pit	FIDLER organic vapors soil classification		I I I
				total TCL/TAL	IV
				TCL VOCs	IV
				plutonium-238 and thorium-232 screen	III
Building 61, former heavy equipment area	To identify hazardous contaminants that may be present. Results will be used to guide the decision for further investigation	surface and subsurface soils	FIDLER organic vapors soil classification		I I I
				total TCL/TAL	IV
				TCL VOCs	IV
				EPH (as motor oil)	V
				plutonium-238 and thorium-232 screen	III

Table I.1. (continued)

Site	Purpose	Media	Field Parameters	Laboratory Parameters	Analytical Level
Building 27 sites: - solvent storage area - concrete flume - sump	To assess whether the identified sites are or have been sources of hazardous contaminants released to the environment through leaks or spills Results will be used to guide decisions on additional investigations	surface and subsurface soils	FIDLER Combustible gas organic vapors soil classification	 total TCL/TAL TCL VOCs explosives plutonium-238 and thorium-232 screen	I I I I IV IV V III
Area 1, Building 1 and Building 27 leach pits (1 each)	To identify hazardous contaminants that may be present in subsurface beneath Buildings 1 and 27 leach pits due to past releases of waste water to pits	subsurface soils	organic vapors FIDLER soil classification	 total TCL/TAL TCL VOCs explosives plutonium-238 and thorium-232 screen	I I I IV IV V III
Building 34 sites: Oil burn structure	To assess whether hazardous contaminants are present at the identified sites. Results will be used to guide the decision to conduct additional investigations	unfiltered water and sediment	pH, conductance, and temperature, and alkalinity for water organic vapors	 total TCL/TAL TCL VOCs EPH (as diesel) plutonium-238 and thorium-232 screen	I I IV IV V III

Table I.1. (continued)

Site	Purpose	Media	Field Parameters	Laboratory Parameters	Analytical Level
Building 34 sites: (continued)		subsurface soils	FIDLER		I
			organic vapors		I
			soil classification		I
				total TCL/TAL	IV
				EPH (as diesel)	V
				plutonium-238 and thorium-232 screen	III
fire fighting training facility		subsurface soils	FIDLER		I
			organic vapors		I
			soil classification		I
				total TCL/TAL	IV
				EPH (as diesel)	V
				plutonium-238 and thorium-232 screen	III
aviation fuel tank		surface soils	FIDLER		I
			organic vapors		I
			soil classification		I
				total TCL/TAL	IV
				TCL VOCs	IV
				EPH (as diesel)	V
				plutonium-238 and thorium-232 screen	III
		unfiltered water	organic vapors, pH, conductance, temperature and alkalinity		I
				total TCL/TAL	IV
				EPH (as diesel)	V

Table I.1. (continued)

Site	Purpose	Media	Field Parameters	Laboratory Parameters	Analytical Level
Area C Waste storage area	To investigate the presence of contamination in subsurface soils to use as a guide for the decision for further action	surface and subsurface soils	FIDLER organic vapors soil classification	 total TCL/TAL TCL VOCs lithium plutonium-238 and thorium-238 screen	I I I IV IV V III
Area H	To investigate the presence of hazardous contaminants in subsurface soils for selected locations: pyrotechnic waste disposal area, Building 53, Building 90, trash burner, thermal treatment unit, pyrotechnic waste shed, and drainage way	subsurface soils	FIDLER organic vapors soil classification	 total TCL/TAL TCL/VOCs explosives plutonium-238 and thorium-238 screen	I I I IV IV V III
Waste oil drum field	To identify hazardous contaminant that may be present due to leaks or spills	surface and subsurface soils	FIDLER organic vapors soil classification	 total TCL/TAL TCL VOCs explosives plutonium-238 and thorium-238 screen	I I I IV IV V III
Old firing range drum storage site	To identify hazardous contaminants that may be present in near surface and subsurface soils	Near surface, subsurface soils	FIDLER organic vapors soil classification	 total TCL/TAL TCL VOCs plutonium-238 and thorium-238 screen	I I I IV IV III

Table I.1. (concluded)

Site	Purpose	Media	Field Parameters	Laboratory Parameters	Analytical Level
Farm trash area	To identify hazardous contaminants that maybe present due to substances released to soils by previous property owner. Results will be used to guide the decisions for a more extensive investigation	surface and subsurface soils	FIDLER organic papers soil classification	 total TCL/TAL TCL VOCs plutonium-238 and thorium-238 screen	I I I IV IV III
Underground sewer line	To identify releases of hazardous contaminants that may have resulted from leakage. Results will be used to identify releases with the potential to contaminate groundwater and to identify sections of underground sewer lines that require additional investigation	subsurface and sediment samples	FIDLER organic vapors soil classification	 total TCL/TAL explosives nitrate-nitrite chloride plutonium-238 and thorium-232 screen	I I I IV V III III III

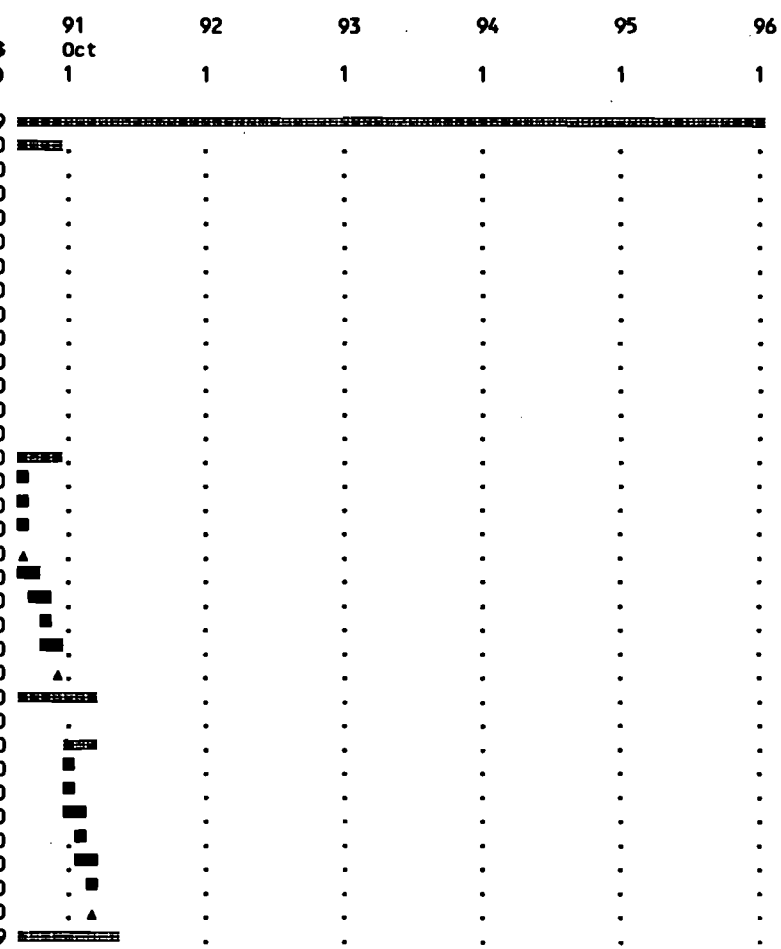
TCL - Target Compound List for volatile organic compounds, semivolatile organic compounds, and pesticides/PCBs.
TAL - Target Analyte List for metals and cyanide.

Table I.2. Operable Unit 3 Schedule

Schedule Name : MOUND MISC. SITES BASELINE SCHEDULE
Responsible : ER TSO/DOE DAO
As-of Date : 1-Oct-90 Schedule File : ALMD-3DM

TASK ID # AL-MD-3

Task Name	Start Date	Duratn (Days)	End Date	Total $ (EAC)
1184 AL-MD-3 MISC. SITES	1-Oct-90	2,194	2-Oct-96	3,879,943.19
RESPONSE ACTION (OPTIONAL)	1-Oct-90	336	1-Sep-91	350,953.80
EST SOW/COST FOR RESP	1-Oct-90	14	14-Oct-90	13,825.60
TM1 WORK PLAN/SAP	1-Dec-90	159	8-May-91	37,902.40
PREPARE TM1 WORK PLAN/SAP	1-Dec-90	73	11-Feb-91	22,582.00
PRODUCTION/REVIEW	12-Feb-91	8	19-Feb-91	4,800.00
DAO DISTRIBUTION/DRAFT	20-Feb-91	3	22-Feb-91	0.00
DELIVER DRAFT TM1	23-Feb-91	0	23-Feb-91	0.00
REGULATORY REVIEW	23-Feb-91	30	24-Mar-91	0.00
RESPOND TO COMMENTS	25-Mar-91	34	27-Apr-91	5,720.40
PRODUCTION/REVIEW	28-Apr-91	8	5-May-91	4,800.00
DAO DIST COMMENT RESP	6-May-91	3	8-May-91	0.00
TM1 COMMENT RESP COMPLETE	9-May-91	0	9-May-91	0.00
FIELDWORK	23-Feb-91	80	13-May-91	225,007.50
TM2 LETTER REPORT ON FIELDWORK	14-May-91	111	1-Sep-91	37,902.40
PREPARE TM2 LETTER REPORT	14-May-91	25	7-Jun-91	22,582.00
PRODUCTIONI/REVIEW	8-Jun-91	8	15-Jun-91	4,800.00
DAO DISTRIBUTION DRAFT	16-Jun-91	3	18-Jun-91	0.00
DELIVER DRAFT TM2	19-Jun-91	0	19-Jun-91	0.00
REGULATORY REVIEW	19-Jun-91	30	18-Jul-91	0.00
RESPOND TO COMMENTS	19-Jul-91	34	21-Aug-91	5,720.40
PRODUCTION/REVIEW	22-Aug-91	8	29-Aug-91	4,800.00
DAO DISTRIBUTION/COMMENT RESP	30-Aug-91	3	1-Sep-91	0.00
TM2 COMMENT RESPONSE COMPLETE	2-Sep-91	0	2-Sep-91	0.00
NEPA	1-Feb-91	326	23-Dec-91	10,204.00
TM1 NEPA ENV CKLIST (SEE OU1)	1-Feb-91	106	17-May-91	0.00
TM2 NEPA ENV CKLIST/CX RESP	1-Oct-91	84	23-Dec-91	9,336.00
PREPARE CKLIST FOR RESP	1-Oct-91	14	14-Oct-91	4,536.00
PRODUTION/REVIEW	15-Oct-91	8	22-Oct-91	4,800.00
DAO REVIEW AND DECISION	23-Oct-91	31	22-Nov-91	0.00
DAO DISTRIBUTION/DRAFT	23-Nov-91	3	25-Nov-91	0.00
DOE AL REVIEW	26-Nov-91	14	9-Dec-91	0.00
DOE HQ REVIEW	10-Dec-91	14	23-Dec-91	0.00
CX APPROVED	24-Dec-91	0	24-Dec-91	0.00
LIMITED FIELD INVESTIGATION	1-Oct-90	510	22-Feb-92	1,096,056.29



Detail Task — Summary Task — Baseline
(Progress) — (Progress) — Conflict
(Slack) — (Slack) — Resource delay
Progress shows Percent Achieved on Actual — Milestone
Scale: 30 days per character

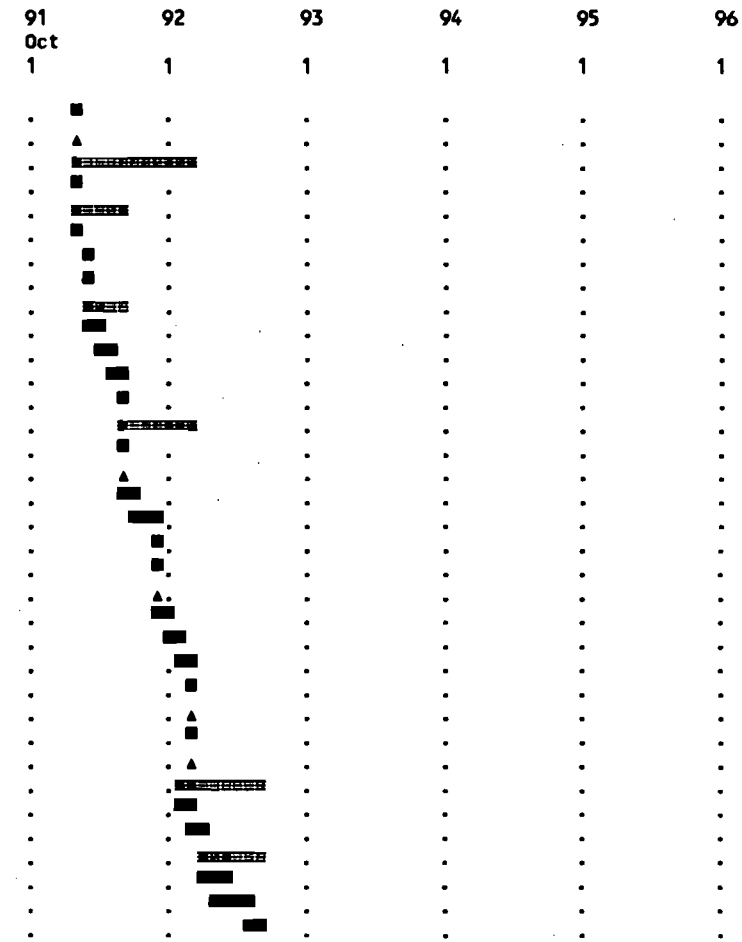
TIME LINE Gantt Chart Report, Strip 1, Page 1

Table I.2. (page 2 of 9)

Schedule Name : MOUND MISC. SITES BASELINE SCHEDULE
Responsible : ER TSO/DOE DAO
As-of Date : 1-Oct-90 Schedule File : ALMD-3DM

TASK ID # AL-MD-3

Task Name	Start Date	Duratn (Days)	End Date	Total $ (EAC)
DAO DISTRIBUTION/COMMENT RESP	20-Feb-92	3	22-Feb-92	0.00
COMMENT RESPONSE COMPLETE	23-Feb-92	0	23-Feb-92	0.00
RI WORK PLAN/SAP	9-Feb-92	308	12-Dec-92	126,615.05
EST. SOW & COST FOR WORK PLAN	9-Feb-92	14	22-Feb-92	17,105.40
DEVELOP WORK PLAN	23-Feb-92	121	22-Jun-92	49,654.05
SITE BACKGROUND	23-Feb-92	5	27-Feb-92	23,213.40
SCHEDULES	28-Feb-92	6	4-Mar-92	941.85
PROJECT MANAGEMENT PLAN	5-Mar-92	6	10-Mar-92	2,721.60
SAMPLING & ANALYSIS PLAN	11-Mar-92	90	8-Jun-92	12,877.20
DRAFT QA PROJECT PLAN	11-Mar-92	30	9-Apr-92	4,989.60
DRAFT FIELD SAMPLING PLAN	10-Apr-92	30	9-May-92	3,477.60
DRAFT HEALTH & SAFETY PLAN	10-May-92	30	8-Jun-92	4,410.00
PRODUCTION/REVIEW	9-Jun-92	14	22-Jun-92	9,900.00
REVIEW CYCLE	23-Jun-92	173	12-Dec-92	48,090.60
DAO DISTRIBUTION/WORK DRAFT	23-Jun-92	3	25-Jun-92	0.00
DOE WORK DRAFT COMPLETE	26-Jun-92	0	26-Jun-92	0.00
DOE REVIEW	26-Jun-92	30	25-Jul-92	0.00
RESPOND TO COMMENTS	26-Jul-92	34	28-Aug-92	21,231.60
PRODUCTION/REVIEW	29-Aug-92	14	11-Sep-92	9,900.00
DAO DISTRIBUTION/DRAFT	12-Sep-92	3	14-Sep-92	0.00
DELIVER DRAFT TO REGULATORS	15-Sep-92	0	15-Sep-92	0.00
REGULATORY REVIEW	15-Sep-92	30	14-Oct-92	0.00
RESPOND TO COMMENTS	15-Oct-92	34	17-Nov-92	12,159.00
PRODUCTION/REVIEW	18-Nov-92	8	25-Nov-92	4,800.00
DAO DISTRIBUTION/DRAFT FINAL	26-Nov-92	3	28-Nov-92	0.00
DELIVER DRAFT FINAL-REGULATORS	29-Nov-92	0	29-Nov-92	0.00
REGULATORY APPROVAL	29-Nov-92	14	12-Dec-92	0.00
REGULATORY APPROVAL RECEIV	13-Dec-92	0	13-Dec-92	0.00
RI FIELD WORK	15-Nov-92	195	28-May-93	1,155,331.50
EST SOW/COST - RI FIELD WRK	15-Nov-92	14	28-Nov-92	12,275.80
SELECT SUBCONTRACTORS	29-Nov-92	41	8-Jan-93	16,253.20
GATHER RI DATA	9-Jan-93	140	28-May-93	901,628.50
PRIME CONT. FIELD WORK	9-Jan-93	60	9-Mar-93	219,191.50
CONDUCT SAMPLE ANALYSIS	29-Jan-93	90	28-Apr-93	623,280.00
DATA VALIDATION	29-Apr-93	30	28-May-93	59,157.00



Detail Task — Summary Task — °°°°° Baseline
(Progress) — (Progress) — ▸▸▸ Conflict
(Slack) — (Slack) — Resource delay
Progress shows Percent Achieved on Actual — ▲ Milestone
---------- Scale: 30 days per character ----------

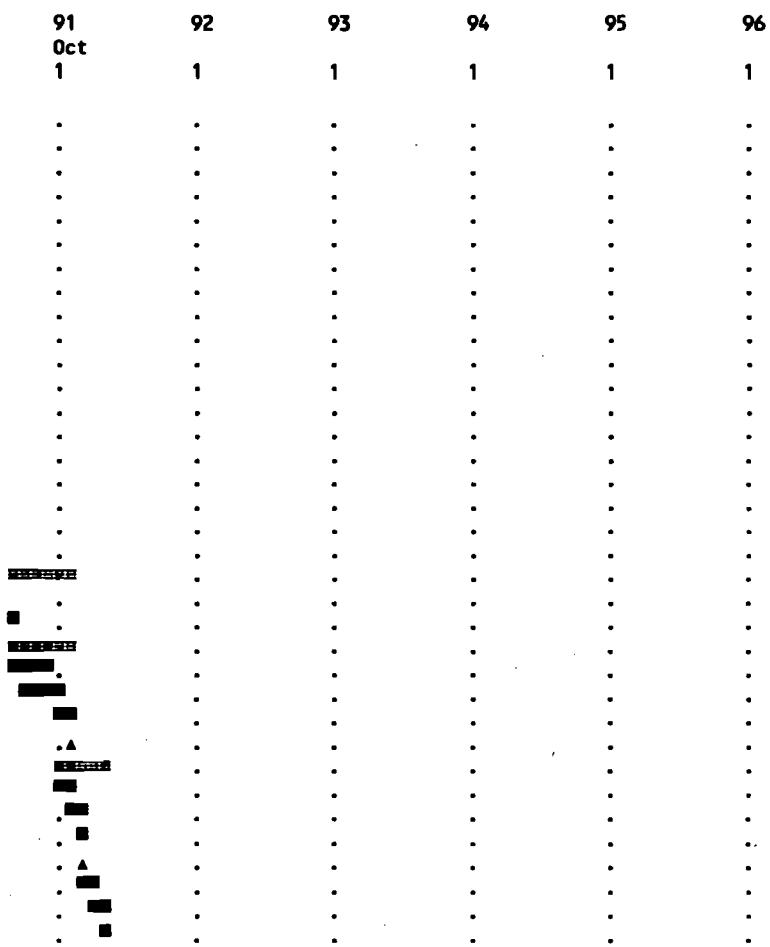
TIME LINE Gantt Chart Report, Strip 1, Page 3

Table I.2. (page 3 of 9)

Schedule Name : MOUND MISC. SITES BASELINE SCHEDULE
Responsible : ER TSO/DOE DAO
As-of Date : 1-Oct-90 Schedule File : ALMD-3DM

TASK ID # AL-MD-3

Task Name	Start Date	Duratn (Days)	End Date	Total $ (EAC)
LIMITED FIELD INV. WORK PLAN	1-Oct-90	241	29-May-91	119,359.20
PREPARE WORK DRAFT	1-Oct-90	24	24-Oct-90	40,000.00
PRODUCTION/REVIEW	25-Oct-90	14	7-Nov-90	9,900.00
DAO DISTRIBUTION/WORK DRAFT	8-Nov-90	3	10-Nov-90	0.00
DOE WORK DRAFT COMPLETE	11-Nov-90	0	11-Nov-90	0.00
DOE REVIEW	11-Nov-90	30	10-Dec-90	0.00
RESPOND TO COMMENTS	11-Dec-90	34	13-Jan-91	20,000.00
PRODUCTION/REVIEW	14-Jan-91	14	27-Jan-91	9,900.00
20 DAY EXTENSION REQUEST (DOE)	28-Jan-91	20	16-Feb-91	0.00
DAO DISTRIBUTION/DRAFT	17-Feb-91	3	19-Feb-91	27,501.60
DELIVER DRAFT TO REG	20-Feb-91	0	20-Feb-91	0.00
REGULATORY REVIEW	20-Feb-91	30	21-Mar-91	0.00
20 DAY EXTENSION (EPA)	22-Mar-91	10	31-Mar-91	0.00
RESPOND TO COMMENTS	1-Apr-91	34	4-May-91	7,257.60
PRODUCTION/REVIEW	5-May-91	8	12-May-91	4,800.00
DAO DISTRIBUTION/DRAFT FINAL	13-May-91	3	15-May-91	0.00
DELIVER DRAFT TO REGULATORS	16-May-91	0	16-May-91	0.00
REGULATORY APPROVAL	16-May-91	14	29-May-91	0.00
REGULATORY APPROVAL RECEIVED	30-May-91	0	30-May-91	0.00
LIMITED FIELD INVESTIGATION	16-May-91	176	7-Nov-91	911,781.89
EST SOW/COST - FIELD WRK	16-May-91	12	27-May-91	8,924.20
SELECT SUBCONTRACTORS	16-May-91	41	25-Jun-91	13,305.60
GATHER FIELD DATA	26-Jun-91	135	7-Nov-91	889,552.09
PRIME CONT. FIELD WORK	26-Jun-91	70	3-Sep-91	202,451.40
CONDUCT SAMPLE ANALYSIS	16-Jul-91	85	8-Oct-91	640,077.49
DATA VALIDATION	9-Oct-91	30	7-Nov-91	47,023.20
FIELD WORK COMPLETE	8-Nov-91	0	8-Nov-91	0.00
PREINVESTIGATION TM 2	9-Oct-91	137	22-Feb-92	19,579.20
PREPARE TM 2	9-Oct-91	51	28-Nov-91	2,721.60
PRODUCTION/REVIEW	29-Nov-91	8	6-Dec-91	4,800.00
DAO DISTRIBUTION/DRAFT	7-Dec-91	3	9-Dec-91	0.00
DELIVER DRAFT TM 1	9-Dec-91	0	9-Dec-91	0.00
REGULATORY REVIEW	10-Dec-91	30	8-Jan-92	0.00
RESPOND TO COMMENTS	9-Jan-92	34	11-Feb-92	7,257.60
PRODUCTION/REVIEW	12-Feb-92	8	19-Feb-92	4,800.00



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Scale: 30 days per character

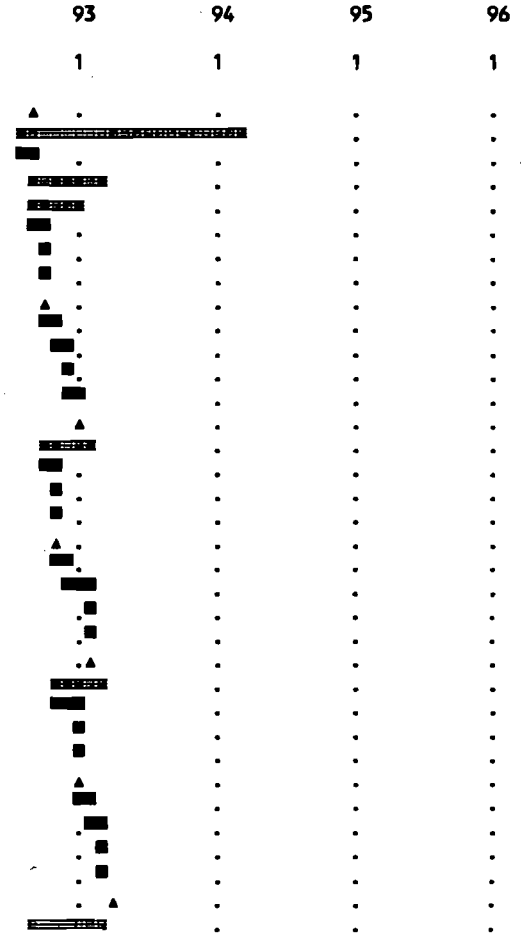
TIME LINE Gantt Chart Report, Strip 1, Page 2

Table I.2. (page 4 of 9)

Schedule Name : MOUND MISC. SITES BASELINE SCHEDULE
Responsible : ER TSO/DOE DAO
As-of Date : 1-Oct-90 Schedule File : ALMD-3DM

TASK ID # AL-MD-3

Task Name	Start Date	Duratn (Days)	End Date	Total $ (EAC)
RI FIELD WORK COMPLETE	29-May-93	0	29-May-93	0.00
REMEDIAL INVESTIGATION REPORT	15-May-93	557	22-Nov-94	539,452.10
ESTIMATE SOW & COST FOR RIR	15-May-93	14	28-May-93	19,421.30
TM DEVELOPMENT (4)	29-May-93	204	18-Dec-93	240,132.00
TM 1 SOURCE/SOIL	29-May-93	116	21-Sep-93	43,620.00
PREPARE TM 1	29-May-93	30	27-Jun-93	26,762.40
PRODUCTION/REVIEW	28-Jun-93	8	5-Jul-93	4,800.00
DAO DISTRIBUTION/DRAFT	6-Jul-93	3	8-Jul-93	0.00
DELIVER DRAFT TM1	9-Jul-93	0	9-Jul-93	0.00
REGULATORY REVIEW	9-Jul-93	30	7-Aug-93	0.00
RESPOND TO COMMENTS	8-Aug-93	34	10-Sep-93	7,257.60
PRODUCTION/REVIEW	11-Sep-93	8	18-Sep-93	4,800.00
DAO DISTRIBUTION/COMMENT RESP	19-Sep-93	3	21-Sep-93	0.00
COMMENT RESPONSE COMPLETE	22-Sep-93	0	22-Sep-93	0.00
TM 2 AIR/GAS	9-Jul-93	116	1-Nov-93	25,929.60
PREPARE TM2	9-Jul-93	30	7-Aug-93	9,072.00
PRODUCTION/REVIEW	8-Aug-93	8	15-Aug-93	4,800.00
DAO DISTRIBUTION/DRAFT	16-Aug-93	3	18-Aug-93	0.00
DELIVER DRAFT TM2	19-Aug-93	0	19-Aug-93	0.00
REGULATORY REVIEW	19-Aug-93	30	17-Sep-93	0.00
RESPOND TO COMMENTS	18-Sep-93	34	21-Oct-93	7,257.60
PRODUCTION/REVIEW	22-Oct-93	8	29-Oct-93	4,800.00
DAO DISTRIBUTION/COMMENT RESP	30-Oct-93	3	1-Nov-93	0.00
COMMENT RESPONSE COMPLETE	2-Nov-93	0	2-Nov-93	0.00
TM 3 HYDRO/GEO INVESTIGATION	19-Aug-93	122	18-Dec-93	51,331.20
PREPARE TM3	19-Aug-93	36	23-Sep-93	34,473.60
PRODUCTION/REVIEW	24-Sep-93	8	1-Oct-93	4,800.00
DAO DISTRIBUTION/DRAFT	2-Oct-93	3	4-Oct-93	0.00
DELIVER DRAFT TM3	5-Oct-93	0	5-Oct-93	0.00
REGULATORY REVIEW	5-Oct-93	30	3-Nov-93	0.00
RESPOND TO COMMENTS	4-Nov-93	34	7-Dec-93	7,257.60
PRODUCTION/REVIEW	8-Dec-93	8	15-Dec-93	4,800.00
DAO DISTRIBUTION/COMMENT RESP	16-Dec-93	3	18-Dec-93	0.00
COMMENT RESPONSE COMPLETE	19-Dec-93	0	19-Dec-93	0.00
DEVELOP TM4	8-Jun-93	194	18-Dec-93	119,251.20



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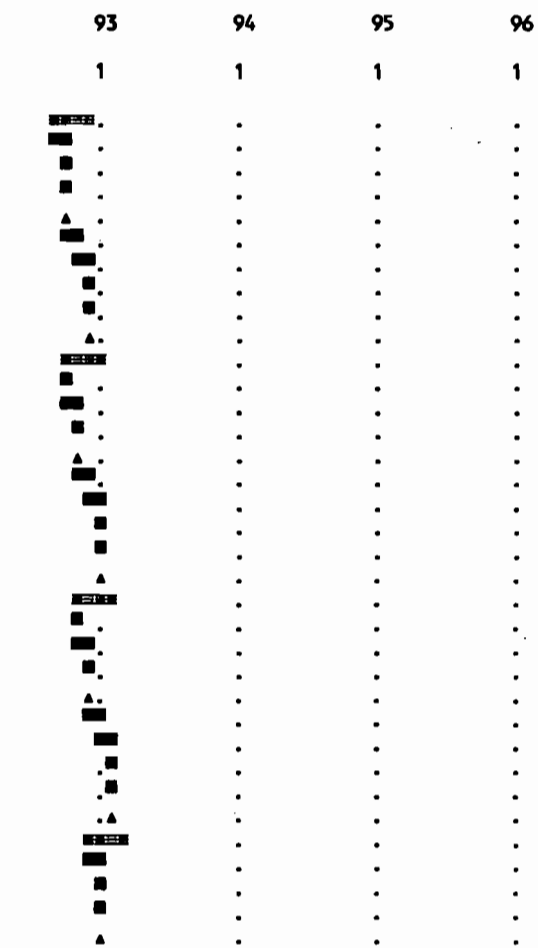
TIME LINE Gantt Chart Report, Strip 1, Page 4

Table I.2. (page 5 of 9)

Schedule Name : MOUND MISC. SITES BASELINE SCHEDULE
Responsible : ER TSO/DOE DAO
As-of Date : 1-Oct-90 Schedule File : ALMD-3DM

TASK ID # AL-MD-3

Task Name	Start Date	Duratn (Days)	End Date	Total $ (EAC)
TM4A OUTLN OF BRA RPT FORMAT	8-Jun-93	101	16-Sep-93	21,600.00
PREPARE TM4A	8-Jun-93	15	22-Jun-93	6,000.00
PRODUCTION/REVIEW	23-Jun-93	8	30-Jun-93	4,800.00
DAO DISTRIBUTION/DRAFT	1-Jul-93	3	3-Jul-93	0.00
DELIVER TM4A	4-Jul-93	0	4-Jul-93	0.00
REGULATORY REVIEW	4-Jul-93	30	2-Aug-93	0.00
RESPOND TO COMMENTS	3-Aug-93	34	5-Sep-93	6,000.00
PRODUCTION/REVIEW	6-Sep-93	8	13-Sep-93	4,800.00
DAO DISTRIBUTION/COMMENT RES	14-Sep-93	3	16-Sep-93	0.00
COMMENT RESPONSE COMPLETE	17-Sep-93	0	17-Sep-93	0.00
TM4B HAZ CHEM/INDICATORS	4-Jul-93	101	12-Oct-93	20,136.00
PREPARE TM4B	4-Jul-93	15	18-Jul-93	4,536.00
PRODUCTION/REVIEW	19-Jul-93	8	26-Jul-93	4,800.00
DAO DISTRIBUTION/DRAFT	27-Jul-93	3	29-Jul-93	0.00
DELIVER TM4B	30-Jul-93	0	30-Jul-93	0.00
REGULATORY REVIEW	30-Jul-93	30	28-Aug-93	0.00
RESPOND TO COMMENTS	29-Aug-93	34	1-Oct-93	6,000.00
PRODUCTION/REVIEW	2-Oct-93	8	9-Oct-93	4,800.00
DAO DISTRIBUTION/COMMENT RES	10-Oct-93	3	12-Oct-93	0.00
COMMENT RESPONSE COMPLETE	13-Oct-93	0	13-Oct-93	0.00
TM4C EXP ASSESS/TOXICITY	30-Jul-93	106	12-Nov-93	36,465.60
PREPARE TM4C	30-Jul-93	20	18-Aug-93	20,865.60
PRODUCTION/REVIEW	19-Aug-93	8	26-Aug-93	4,800.00
DAO DISTRIBUTION/DRAFT	27-Aug-93	3	29-Aug-93	0.00
DELIVER TM4C	30-Aug-93	0	30-Aug-93	0.00
REGULATORY REVIEW	30-Aug-93	30	28-Sep-93	0.00
RESPOND TO COMMENTS	29-Sep-93	34	1-Nov-93	6,000.00
PRODUCTION/REVIEW	2-Nov-93	8	9-Nov-93	4,800.00
DAO DISTRIBUTION/COMMENT RES	10-Nov-93	3	12-Nov-93	0.00
COMMENT RESPONSE COMPLETE	13-Nov-93	0	13-Nov-93	0.00
TM4D RISK CHARACTERIZATION	30-Aug-93	111	18-Dec-93	41,049.60
PREPARE TM4D	30-Aug-93	25	23-Sep-93	28,274.40
PRODUCTION/REVIEW	24-Sep-93	8	1-Oct-93	4,800.00
DAO DISTRIBUTION/DRAFT	2-Oct-93	3	4-Oct-93	0.00
DELIVER TM4D	5-Oct-93	0	5-Oct-93	0.00



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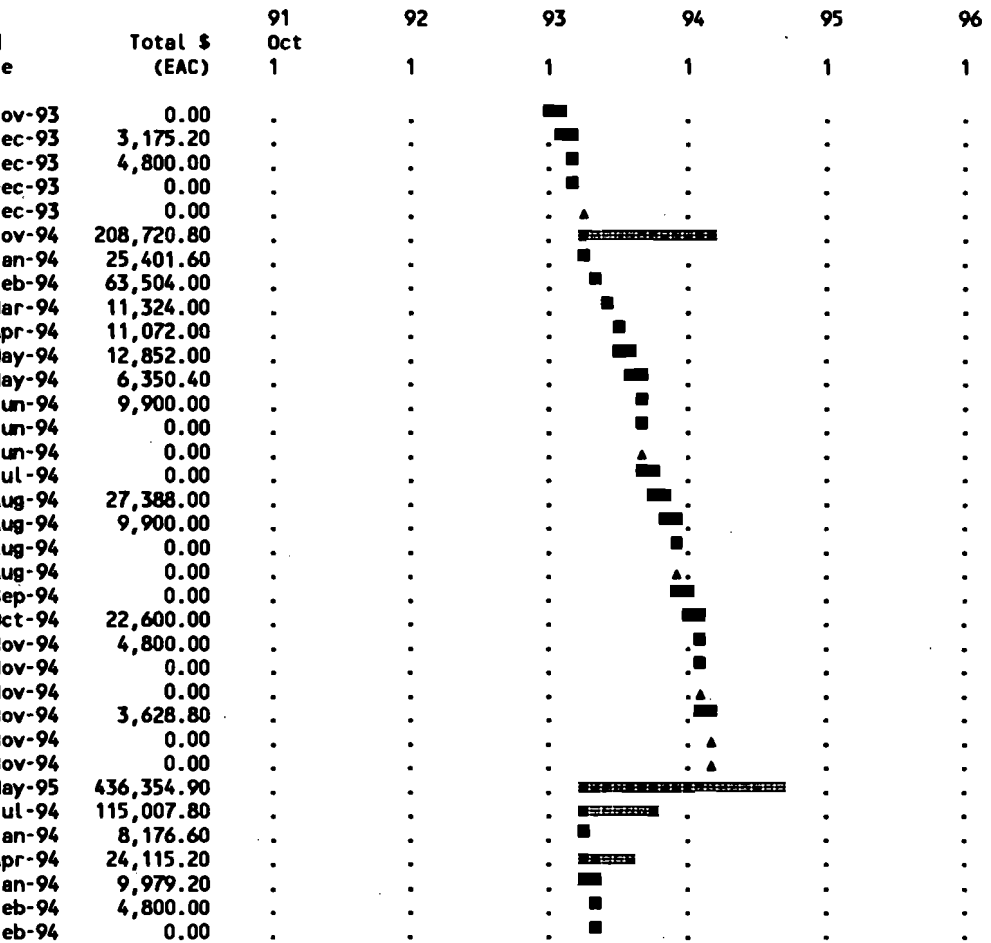
TIME LINE Gantt Chart Report, Strip 1, Page 5

Table I.2. (page 6 of 9)

Schedule Name : MOUND MISC. SITES BASELINE SCHEDULE
Responsible : ER TSO/DOE DAO
As-of Date : 1-Oct-90 Schedule File : MLMD-3DM

TASK ID # AL-MD-3

Task Name	Start Date	Duratn (Days)	End Date	Total $ (EAC)
REGULATORY REVIEW	5-Oct-93	30	3-Nov-93	0.00
RESPOND TO COMMENTS	4-Nov-93	34	7-Dec-93	3,175.20
PRODUCTION/REVIEW	8-Dec-93	8	15-Dec-93	4,800.00
DAO DISTRIBUTION/COMMENT RES	16-Dec-93	3	18-Dec-93	0.00
COMMENT RESPONSE COMPLETE	19-Dec-93	0	19-Dec-93	0.00
DEVELOP DRAFT RIR/EA	19-Dec-93	339	22-Nov-94	208,720.80
DRAFT BRA SECTION	19-Dec-93	30	17-Jan-94	25,401.60
EVALUATE RI/NEPA DATA	18-Jan-94	30	16-Feb-94	63,504.00
DRAFT AREA DESCRIP & CHARACTER	17-Feb-94	30	18-Mar-94	11,324.00
NATURE & EXTENT OF CONTAM	19-Mar-94	15	2-Apr-94	11,072.00
CONTAMINANT FATE & TRANSPORT	3-Apr-94	33	5-May-94	12,852.00
CONCLUSIONS & NEED FOR ADD. RI	6-May-94	14	19-May-94	6,350.40
PRODUCTION/REVIEW	20-May-94	14	2-Jun-94	9,900.00
DAO DISTRIBUTION/WORK DRAFT	3-Jun-94	3	5-Jun-94	0.00
DOE WORK DRAFT COMPLETE	6-Jun-94	0	6-Jun-94	0.00
DOE REVIEW	6-Jun-94	30	5-Jul-94	0.00
RESPOND TO COMMENTS	6-Jul-94	34	8-Aug-94	27,388.00
PRODUCTION/REVIEW	9-Aug-94	14	22-Aug-94	9,900.00
DAO DISTRIBUTION/DRAFT	23-Aug-94	3	25-Aug-94	0.00
DELIVER DRAFT RIR	26-Aug-94	0	26-Aug-94	0.00
REGULATORY REVIEW	26-Aug-94	30	24-Sep-94	0.00
RESPOND TO COMMENTS	25-Sep-94	34	28-Oct-94	22,600.00
PRODUCTION/REVIEW	29-Oct-94	8	5-Nov-94	4,800.00
DAO DISTRIBUTION/DRAFT FINAL	6-Nov-94	3	8-Nov-94	0.00
DELIVER DRAFT FINAL	9-Nov-94	0	9-Nov-94	0.00
REGULATORY APPROVAL	9-Nov-94	14	22-Nov-94	3,628.80
REGULATORY APPROVAL RECEIVED	23-Nov-94	0	23-Nov-94	0.00
INTEGRATE TO OU 9	23-Nov-94	0	23-Nov-94	0.00
FEASIBILITY STUDY/EA	19-Dec-93	529	31-May-95	436,354.90
TECH MEMO DEVELOPMENT (4)	19-Dec-93	196	2-Jul-94	115,007.80
ESTIMATE SOW & COST FOR TM	19-Dec-93	14	1-Jan-94	8,176.60
TM1 SCREEN TECHNOLOGY	2-Jan-94	116	27-Apr-94	24,115.20
PREPARE TM1	2-Jan-94	30	31-Jan-94	9,979.20
PRODUCTION/REVIEW	1-Feb-94	8	8-Feb-94	4,800.00
DAO DISTRIBUTION/DRAFT	9-Feb-94	3	11-Feb-94	0.00



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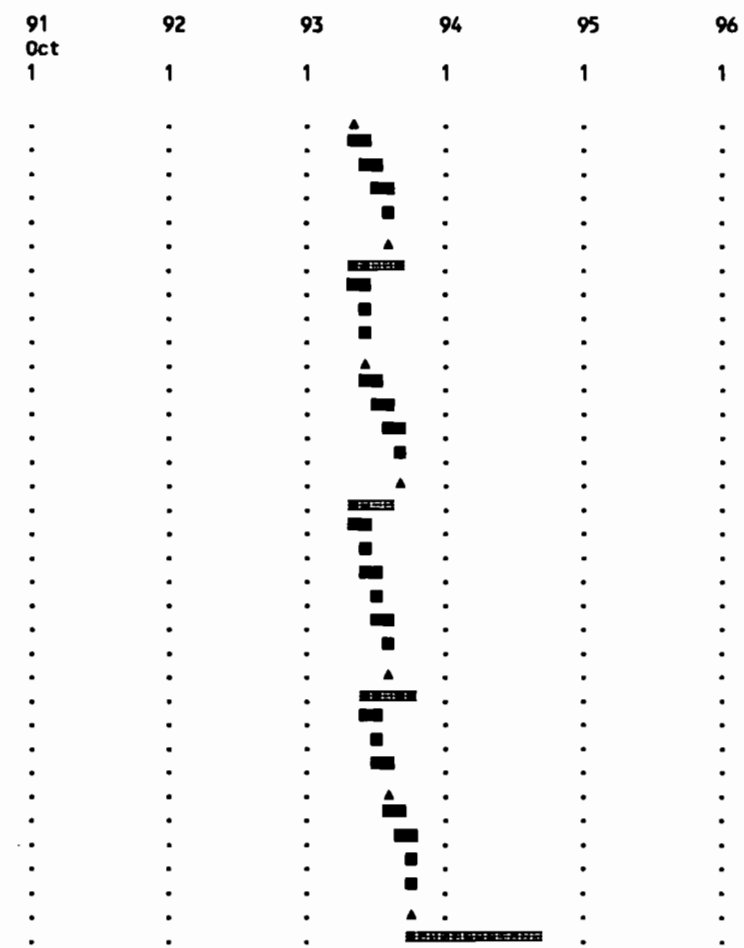
TIME LINE Gantt Chart Report, Strip 1, Page 6

Table I.2. (page 7 of 9)

Schedule Name : MOUND MISC. SITES BASELINE SCHEDULE
Responsible : ER TSO/DOE DAO
As-of Date : 1-Oct-90 Schedule File : ALMD-3DM

TASK ID # AL-MD-3

Task Name	Start Date	Duratn (Days)	End Date	Total $ (EAC)
DELIVER TM1	12-Feb-94	0	12-Feb-94	0.00
REGULATORY REVIEW	12-Feb-94	30	13-Mar-94	0.00
RESPOND TO COMMENTS	14-Mar-94	34	16-Apr-94	4,536.00
PRODUCTION/REVIEW	17-Apr-94	8	24-Apr-94	4,800.00
DAO DISTRIBUTION/COMMENT RESP	25-Apr-94	3	27-Apr-94	0.00
RESPONSE COMPLETE	28-Apr-94	0	28-Apr-94	0.00
TM2 ALT ARRAY DOC	12-Feb-94	100	22-May-94	27,290.40
PREPARE TM2	12-Feb-94	14	25-Feb-94	13,154.40
PRODUCTION/REVIEW	26-Feb-94	8	5-Mar-94	4,800.00
DAO DISTRIBUTION/DRAFT	6-Mar-94	3	8-Mar-94	0.00
DELIVER TM2	9-Mar-94	0	9-Mar-94	0.00
REGULATORY REVIEW	9-Mar-94	30	7-Apr-94	0.00
RESPOND TO COMMENTS	8-Apr-94	34	11-May-94	4,536.00
PRODUCTION/REVIEW	12-May-94	8	19-May-94	4,800.00
DAO DISTRIBUTION/COMMENT RESP	20-May-94	3	22-May-94	0.00
COMMENT RESPONSE COMPLETE	23-May-94	0	23-May-94	0.00
TM3 NEPA ENVIRON CKLIST (RA)	12-Feb-94	84	6-May-94	9,336.00
PREPARE CKLIST FOR RA	12-Feb-94	14	25-Feb-94	4,536.00
PRODUCTION/REVIEW	26-Feb-94	8	5-Mar-94	4,800.00
DAO REVIEW AND DECISION	6-Mar-94	31	5-Apr-94	0.00
DAO DISTRIBUTION/DRAFT	6-Apr-94	3	8-Apr-94	0.00
DOE AL REVIEW	9-Apr-94	14	22-Apr-94	0.00
DOE HQ REVIEW	23-Apr-94	14	6-May-94	0.00
CX APPROVED/EA DECISION	7-May-94	0	7-May-94	0.00
TM4 ENDANGERMENT ASSESSMENT	9-Mar-94	116	2-Jul-94	46,089.60
PREPARE TM4	9-Mar-94	30	7-Apr-94	31,953.60
PRODUCTION/REVIEW	8-Apr-94	8	15-Apr-94	4,800.00
DAO DISTRIBUTION/DRAFT	16-Apr-94	3	18-Apr-94	0.00
DELIVER TM4	19-Apr-94	0	19-Apr-94	0.00
REGULATORY REVIEW	19-Apr-94	30	18-May-94	0.00
RESPOND TO COMMENTS	19-May-94	34	21-Jun-94	4,536.00
PRODUCTION/REVIEW	22-Jun-94	8	29-Jun-94	4,800.00
DAO DISTRIBUTION/COMMENT RESP	30-Jun-94	3	2-Jul-94	0.00
COMMENT RESPONSE COMPLETE	3-Jul-94	0	3-Jul-94	0.00
DEVELOP FSR	19-Jun-94	347	31-May-95	263,772.10



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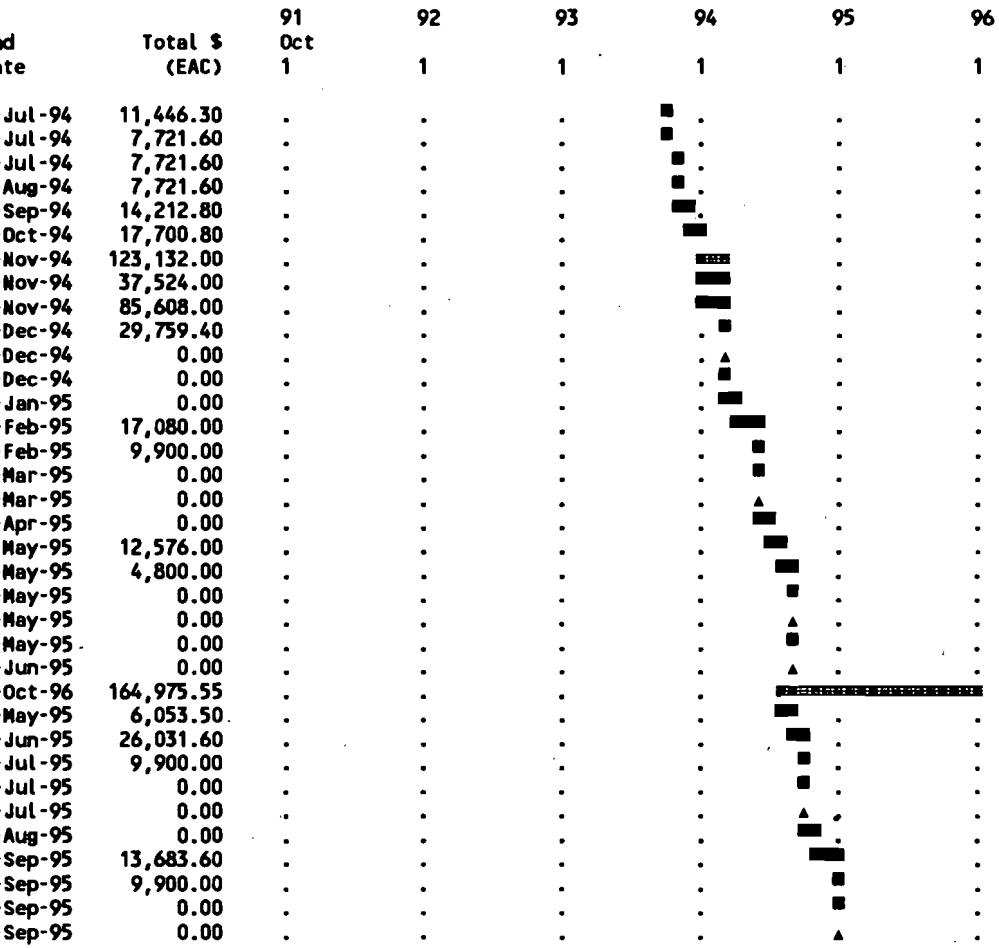
TIME LINE Gantt Chart Report, Strip 1, Page 7

Table I.2. (page 8 of 9)

Schedule Name : MOUND MISC. SITES BASELINE SCHEDULE
Responsible : ER TSO/DOE DAO
As-of Date : 1-Oct-90 Schedule File : ALMD-3DM

TASK ID # AL-MD-3

Task Name	Start Date	Duratn (Days)	End Date	Total $ (EAC)
EST SOW & COST FOR FSR	19-Jun-94	14	2-Jul-94	11,446.30
DESCRIBE POTENTIAL RESPONSES	3-Jul-94	14	16-Jul-94	7,721.60
ASSEMBLE INITIAL OBJS & ALTS	17-Jul-94	14	30-Jul-94	7,721.60
INITIAL SCREEN OF RA ALTS	31-Jul-94	14	13-Aug-94	7,721.60
EVAL ALTS & COST EFFECTIVENESS	14-Aug-94	30	12-Sep-94	14,212.80
DETAILED ANALYSIS OF ALTS	13-Sep-94	30	12-Oct-94	17,700.80
EVAL DRAFT NEPA CHAPTERS	13-Oct-94	44	25-Nov-94	123,132.00
COMPILE NEPA INFORMAION	13-Oct-94	36	17-Nov-94	37,524.00
PREPARE INTEGRATED SECTION	13-Oct-94	44	25-Nov-94	85,608.00
PRODUCTION/REVIEW	26-Nov-94	14	9-Dec-94	29,759.40
DOE WORK DRAFT COMPLETE	10-Dec-94	0	10-Dec-94	0.00
DAO DISTRIBUTION/WORK DRAFT	10-Dec-94	3	12-Dec-94	0.00
DOE REVIEW (FS & NEPA)	13-Dec-94	30	11-Jan-95	0.00
RESPOND TO COMMENTS	12-Jan-95	34	14-Feb-95	17,080.00
PRODUCTION/REVIEW	15-Feb-95	14	28-Feb-95	9,900.00
DAO DISTRIBUTION/DRAFT	1-Mar-95	3	3-Mar-95	0.00
DEL FSR/NEPA EA-REGULTRS/STATE	4-Mar-95	0	4-Mar-95	0.00
REGULATORY REVIEW	4-Mar-95	30	2-Apr-95	0.00
RESPOND TO COMMENTS	3-Apr-95	34	6-May-95	12,576.00
PRODUCTION/REVIEW	7-May-95	8	14-May-95	4,800.00
DAO DISTRIBUTION/DRAFT FINAL	15-May-95	3	17-May-95	0.00
RESPONSE COMPLETE	18-May-95	0	18-May-95	0.00
REGULATORY APPROVAL (EA FONSI)	18-May-95	14	31-May-95	0.00
REGULATORY APPROVAL RECEIVED	1-Jun-95	0	1-Jun-95	0.00
PROPOSED PLAN ROD SUPPORT	4-May-95	518	2-Oct-96	164,975.55
ESTIMATE SOW & COST	4-May-95	14	17-May-95	6,053.50
PREPARE DRAFT ROD	18-May-95	35	21-Jun-95	26,031.60
PRODUCTION/REVIEW	22-Jun-95	14	5-Jul-95	9,900.00
DAO DISTRIBUTION/WORK DRAFT	6-Jul-95	3	8-Jul-95	0.00
DOE WORK DRAFT COMPLETE	9-Jul-95	0	9-Jul-95	0.00
DOE REVIEW	9-Jul-95	30	7-Aug-95	0.00
RESPOND TO COMMENTS	8-Aug-95	34	10-Sep-95	13,683.60
PRODUCTION/REVIEW	11-Sep-95	14	24-Sep-95	9,900.00
DAO DISTRIBUTION/DRAFT	25-Sep-95	3	27-Sep-95	0.00
DELIVER DRAFT TO REGULATORS	28-Sep-95	0	28-Sep-95	0.00



Detail Task — Summary Task — Baseline
(Progress) — (Progress) — Conflict
(Slack) — (Slack) — Resource delay
Progress shows Percent Achieved on Actual — Milestone
Scale: 30 days per character

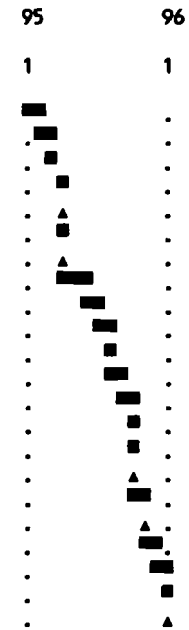
TIME LINE Gantt Chart Report, Strip 1, Page 8

Table I.2. (page 9 of 9)

Schedule Name : MOUND MISC. SITES BASELINE SCHEDULE
Responsible : ER TSO/DOE DAO
As-of Date : 1-Oct-90 Schedule File : ALMD-3DM

TASK ID # AL-MD-3

Task Name	Start Date	Duratn (Days)	End Date	Total $ (EAC)
REGULATORY REVIEW	28-Sep-95	30	27-Oct-95	0.00
RESPOND TO COMMENTS	28-Oct-95	34	30-Nov-95	8,089.20
PRODUCTION/REVIEW	1-Dec-95	8	8-Dec-95	4,800.00
DAO DISTRIBUTION/DRAFT FINAL	9-Dec-95	3	11-Dec-95	0.00
DOE DRAFT FINAL COMPLETE	12-Dec-95	0	12-Dec-95	0.00
REGULATORY REVIEW	12-Dec-95	14	25-Dec-95	0.00
RELEASE ROD TO PUBLIC	26-Dec-95	0	26-Dec-95	0.00
PUBLIC COMMENT	26-Dec-95	44	7-Feb-96	0.00
RESPOND TO PUBLIC COMMENTS	8-Feb-96	50	28-Mar-96	9,172.80
PRODUCTION/REVIEW	29-Mar-96	14	11-Apr-96	9,900.00
DAO DISTRIBUTION/COMMENT RESP	12-Apr-96	3	14-Apr-96	0.00
REGULATORY REVIEW	15-Apr-96	30	14-May-96	0.00
RESPOND TO COMMENTS	15-May-96	34	17-Jun-96	4,636.80
PRODUCTION/REVIEW	18-Jun-96	14	1-Jul-96	9,900.00
DAO DISTRIBUTION/FINAL	2-Jul-96	3	4-Jul-96	0.00
DOE FINAL ROD & COMMENT RESP	5-Jul-96	0	5-Jul-96	0.00
REGULATORY APPROVAL RECEIVED	5-Jul-96	30	3-Aug-96	0.00
ROD APPROVED	4-Aug-96	0	4-Aug-96	0.00
DOE SIGNS ROD	4-Aug-96	30	2-Sep-96	0.00
EPA SIGNS ROD	3-Sep-96	15	17-Sep-96	0.00
PUBLIC NOTICE	18-Sep-96	15	2-Oct-96	0.00
COMPLETION	3-Oct-96	0	3-Oct-96	0.00



Detail Task — Summary Task — Baseline
(Progress) — (Progress) — Conflict
(Slack) — (Slack) — Resource delay
Progress shows Percent Achieved on Actual — Milestone
Scale: 30 days per character

TIME LINE Gantt Chart Report, Strip 1, Page 9

2. PROJECT ORGANIZATION AND RESPONSIBILITY

2.1. OPERATIONAL RESPONSIBILITIES

Mound Plant was placed on the National Priority List in November, 1989. Therefore, environmental investigations are being performed under the guidelines of the EPA's Superfund Program. Because the facility is owned by DOE, it is the lead federal agency for the Mound Plant Environmental Restoration (ER) Program investigations, in conjunction with Mound EG&G Applied Technologies, which operates the facility for the DOE. Regulatory authority is provided by the EPA and the Ohio EPA. EPA Region V is responsible for the final review and approval of this QAPP. Additional review and approval must be received from the Ohio EPA remedial project manager, the DOE remedial project manager, and the Mound Plant ER Program Manager. EPA Region V Central Regional Laboratory and/or EPA Central District will also perform external system and performance audits of both the laboratories performing the analyses and field activities. Figure 2.1 summarizes the organizational structure for the Operable Unit 3 Limited Field Investigation.

The Operable Unit 3 limited field investigation will be performed by Roy F. Weston, Inc., a subcontractor designated by the ER Program Technical Support Office (TSO), which is the Los Alamos National Laboratory. The ER Program TSO is on contract to the DOE Albuquerque Operations Office and provides technical support for the ER Program at Mound Plant. Final data review/assessment will also be performed by Roy F. Weston, Inc.

Laboratory analyses will be performed by the International Technology Corporation (IT) Analytical Services Mixed Waste Laboratory in Oak Ridge, TN and the IT Analytical Services Laboratory in Knoxville, TN. Responsibilities within the IT Laboratories are described in subsection 2.2. Screening of soil samples for plutonium-238 and thorium-232 will be performed at the Mound Plant Soil Screening Facility operated by EG&G.

2.2. FIELD TEAM RESPONSIBILITIES

The subcontractor field team responsibilities for the Operable Unit 3 Limited Field Investigation will consist of performing the field activities specified in the Operable Unit 3 Work Plan. The field team will have four identified positions; the field team leader, site health and safety coordinator (SHSC), field technicians and sample document control administrator. The field team leader is responsible for directing the field activities specified in the Work Plan and communicates progress and any issues to the subcontractor site manager.

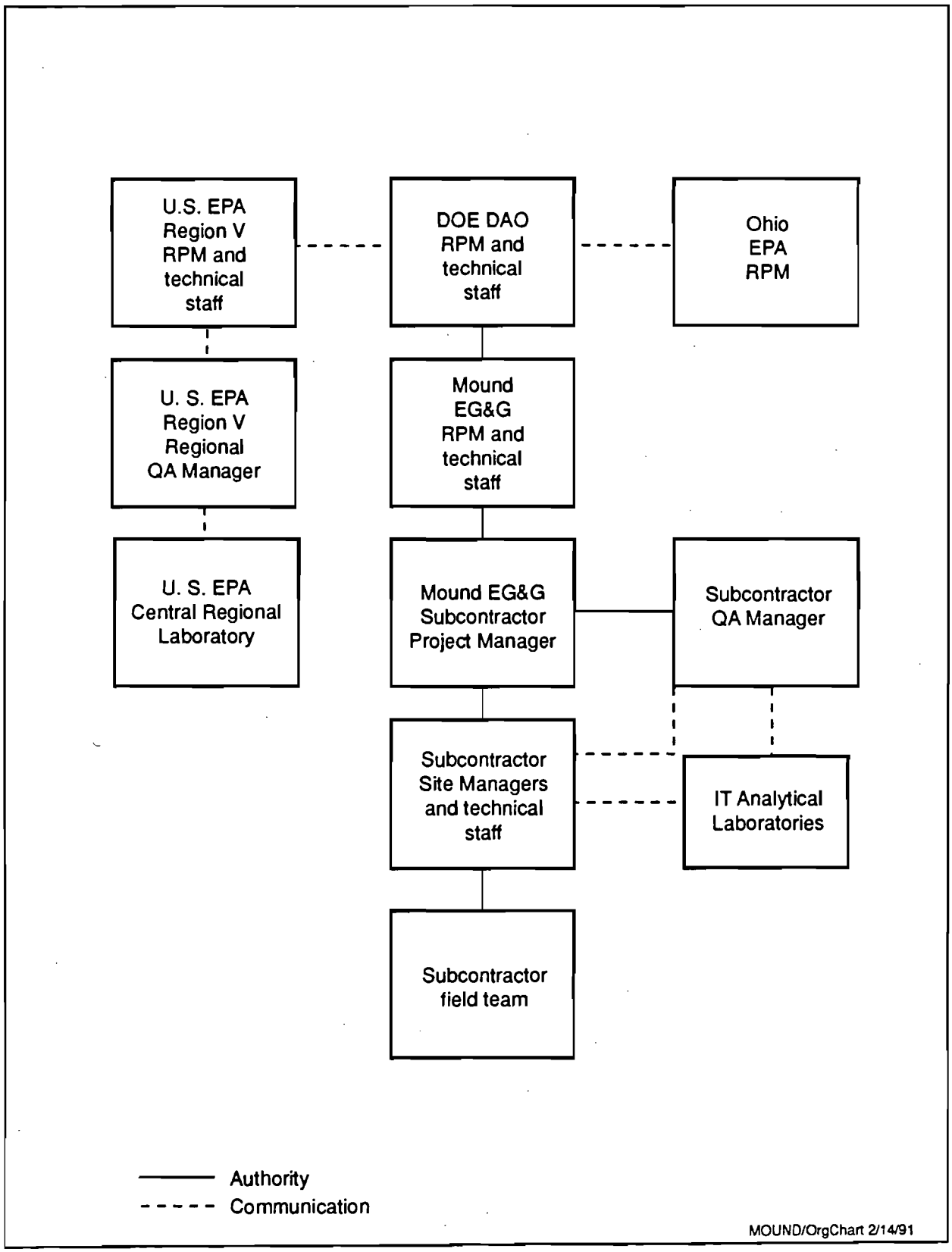


Figure 2.1. Operable Unit 3 organizational chart.

The SHSC is responsible for ensuring that the health and safety guidelines specified in the Health and Safety Plan are followed. He/she will be trained in first aid and CPR. It is the responsibility of the SHSC to make sure safe work practices are implemented and to report all incidents which occur during the field activities.

The field technicians are responsible for conducting the field activities as specified in the Work Plan under the supervision of the field team leader.

A member of the field team will be designated the Sample Document Control Administrator. He/she will be responsible for tracking the locations sampled and for ensuring that samples are properly labelled and documented prior to shipment to the laboratory. The administrator ensures that the sample control procedures specified in this QAPP are followed.

2.3. LABORATORY RESPONSIBILITIES

Laboratory responsibilities for the Operable Unit 3 Limited Field Investigation will consist of performing analytical services according to guidelines presented in this QAPP, reporting all laboratory nonconformances should they occur, and transmitting quality-assured data packages. The IT Analytical Services Laboratory in Knoxville, Tennessee, and in Earth City, Missouri will perform all nonradiological analyses for this project, while the IT Mixed Waste Laboratory in Oak Ridge, Tennessee, will perform all radiochemical and mixed waste analyses. This section identifies the various positions within the analytical laboratories and the specific responsibilities of each for the implementation of the quality assurance program and the completion of quality control activities. Specific sections of the IT laboratories' Quality Assurance Manuals are referenced throughout this QAPP. The IT Quality Assurance Manuals that apply to individual IT analytical laboratories and are designed as supplements to the IT Analytical Services Corporate Quality Assurance Manual. The February 1988 Revision 1 manual for IT Analytical Services is still current. The quality assurance program defined in this QAPP takes precedence over equivalent sections in the IT Analytical Services Quality Assurance Manuals, unless otherwise referenced. It should be noted that several sections in the IT Analytical Services Quality Assurance Manuals are not applicable to the Operable Unit 3 Limited Field Investigation. Sections of the quality assurance manuals will be referenced in this QAPP where they directly apply and when more information is available in the manuals. These sections are included as Appendix A through D of this QAPP.

2.3.1. IT Analytical Laboratory Quality-Assurance-Related Responsibilities

- The laboratory director shall
    - report directly to the Vice President of IT Analytical Services;
    - periodically determine the effectiveness of the quality assurance program in the laboratory; and
    - approve the laboratory-specific attachment to the Quality Assurance Manual, project-specific manuals, Standard Operating Procedures (SOPs), and revisions.
- The laboratory manager shall
    - approve the laboratory-specific attachment to the Quality Assurance Manual, project-specific manuals, SOPs, and all revisions;
    - implement the quality assurance program within the laboratory;
    - supervise routine quality control activities within the laboratory;
    - recommend changes in the quality assurance program to the IT Analytical Services quality assurance/quality control director;
    - approve all reports issued by the laboratory;
    - maintain current laboratory organization chart;
    - manage laboratory daily analytical operations; and
    - supervise sample storage facilities.
- The technical director shall
    - provide technical overview of laboratory activities;
    - define and oversee training program;
    - serve as an "in-house" consultant for the applicability of general quality control practices to specific needs;
    - evaluate new analytical techniques, procedures, instrumentation, and quality control procedures and provide recommendations to the laboratory manager;
    - define calibration programs for instrumentation within the laboratory;
    - define and oversee instrument preventive maintenance program and schedule;
    - supervise the verification of software for data processing; and

- recommend standards for purchasing instruments, equipment, reagents, gases, and chemicals.

- The quality assurance coordinator shall

  - prepare quality control standards, insert quality control samples into the laboratory sample stream, and review results;

  - perform statistical analyses using results of quality control sample analyses;

  - notify the laboratory manager of out-of-control or biased situations;

  - report nonconformances to the IT Analytical Services quality assurance/quality control director if the situation is not corrected within the laboratory;

  - assist in the performance of quality assurance audits and perform quality control audits;

  - establish and supervise the laboratory quality assurance training program;

  - supervise the maintenance of laboratory records;

  - monitor implementation of the quality assurance program;

  - perform an annual review of quality assurance manual and procedures; and

  - serve as focal point for the reporting and disposition of nonconformances.

- Group leaders shall

  - serve as lead analysts within the laboratory group to which they are assigned (group meaning a division of people in the lab who perform work of a similar nature or in a specific area);

  - organize and schedule the analytical testing program with consideration for sample-holding times;

  - review all analytical data and submit to the laboratory manager for approval;

  - implement data verification procedures;

  - assign analysts for data processing and validation activities;

  - evaluate instrument performance and supervise instrument calibration and preventive maintenance programs;

  - report out-of-control or nonconforming situations to the laboratory manager and quality control coordinator;

  - stop production of data in laboratory area where review of quality control data or procedures show significant problems;

- resolve ongoing and recurring nonconformances within the laboratory;
- recommend corrective actions for resolutions of nonconformances;
- review statistical data to verify the laboratory is meeting stated quality control goals;
- maintain current distribution lists for laboratory-specific attachments, project-specific manuals, and SOPs; and
- verify that corrective actions to findings and recommendations described in quality assurance audits have been performed, where appropriate.

- Technicians shall
  - perform analytical procedures and data recording in accordance with accepted methods;
  - perform and document calibration and preventive maintenance of instrumentation, as required in associated procedures;
  - perform data processing and validation; and
  - immediately report out-of-control situations, instrument malfunctions, calibration failures, or other nonconformances to the group leader and quality control coordinator, as appropriate.

2.3.2. IT Mixed Waste Laboratory Quality-Assurance-Related Responsibilities

- The regional director
  - reports directly to the Vice President of IT Analytical Services;
  - provides resources for implementation of the quality assurance program at each laboratory;
  - periodically determines the effectiveness of the quality assurance program at each laboratory; and
  - recommends changes in the quality assurance program to the IT Analytical Services director of quality assurance and compliance.

- The laboratory director
  - reports directly to the Regional Director of Analytical Operations;
  - implements the quality assurance program within the laboratory;
  - periodically determines the effectiveness of the quality assurance program in the laboratory;

- approves laboratory-specific attachments to the Quality Assurance Manual, project-specific manuals, SOPs, and revisions;

- recommends changes in the quality assurance program to the IT Analytical Services director of quality assurance and compliance;

- issues laboratory reports;

- maintains current laboratory organization chart; and

- supervises laboratory participation in inter-laboratory accreditation and proficiency programs.

- The laboratory manager

  - manages daily laboratory analytical operations and supervises quality control activities performed as part of routine analytical operations,

  - supervises the preparation and maintenance of laboratory records,

  - oversees the log-in of all samples received and supervises sample storage facilities, and

  - defines the instrument preventive maintenance schedule and oversees the preventive maintenance program.

- The technical director

  - provides technical overview of laboratory activities and serves as an "in-house" consultant for the applicability of general quality control practices to specific needs;

  - leads the training of analysts in laboratory operations and analytical procedures;

  - evaluates analytical techniques, procedures, instrumentation, and quality control procedures and provides recommendations to the laboratory manager;

  - supervises the verification of software for data processing;

  - recommends standards for purchasing instrumentation, equipment, reagents, gases, and chemicals; and

  - defines the calibration program within the laboratory.

- The quality control coordinator

  - coordinates preparation of quality control standards, arranges insertion of quality control samples into the laboratory sample stream, and reviews the results;

- performs statistical analyses, using results of quality control sample analyses to verify that the laboratory is meeting stated quality control goals;

- informs the operations manager and group leaders of data which occur outside of acceptable limits;

- serves as the "focal point" for the reporting and disposition of nonconformances within the laboratory and verifies corrective actions for all nonconformances;

- notifies the technical director and laboratory manager of out-of-control situations;

- reports unresolved nonconformances to the IT Analytical Services director of quality assurance and compliance;

- stops production of data in a laboratory area where the review of quality control data or procedures shows significant problems;

- assists in the performance of quality assurance audits and performs quality control audits and surveillances;

- closes findings and recommendations of quality assurance audits;

- establishes and supervises the laboratory quality assurance training program;

- maintains current distribution lists for laboratory-specific attachments, project-specific manuals, and SOPs; and

- reviews a minimum of 5% of all laboratory data after analyst cross-checks. If analyst cross-checks have not been performed, reviews a minimum of 20% of all laboratory data.

- Group supervisors

  - ensure calibration and preventative maintenance are performed as required,

  - perform data validation and final raw data reviews,

  - report out-of-control situations to the quality control coordinator,

  - review and create in-house data forms,

  - verify technical accuracy of internal procedures and forms, and

  - perform analytical procedures and data recording in accordance with accepted procedures.

- Analysts

  - perform analytical procedures and data recording in accordance with accepted methods;

- perform and document calibration and preventive maintenance of instrumentation, as appropriate;
- perform data processing and validation;
- immediately report out-of-control situations, instrument malfunctions, calibration failures, or other nonconformances to the group leader and quality control coordinator, as appropriate; and
- prepare data summaries for review by the laboratory manager.

2.4. QUALITY ASSURANCE RESPONSIBILITIES

Quality assurance for the Operable Unit 3 investigation is the responsibility of all ER Program personnel. Responsibilities include detailed monitoring and review of all procedures used to perform every aspect of the remedial investigation. All personnel involved with ER Program activities will strictly adhere to the implementation of this QAPP, Mound Plant ER Program SOPs, analytical laboratory procedures, data acceptance criteria, and data reporting schedules.

Primary responsibility for project quality rests with the ER Program TSO subcontractor project manager. Specific responsibilities include the management of quality assurance issues as they relate to the ER Program, insuring that nonconformances are corrected, and assuring the overall quality, timeliness, and cost effectiveness of the activities performed by ER Program TSO subcontractors. The project manager is also responsible for coordinating and maintaining consistency between this QAPP and those of the various subcontractors at the Mound Plant and across the ER Program as a whole.

The Quality Assurance Manager (QAM) has responsibility for quality assurance/quality control within the project. This person will document compliance with this QAPP and program SOPs. The project quality assurance manager's responsibilities include the following:

- conducting internal performance and system audits;
- development and implementation of the QAPP;
- ongoing review of individual quality assurance procedures;
- overall quality assurance for project activities;
- insuring that laboratory activities are consistent with the objectives and requirements of this QAPP;
- coordination of internal and external quality assurance audits;
- coordination of quality assurance training;

- quality assurance for field activities;
- serve as field/laboratory liaison to resolve custody and sample problems
- audits of analytical laboratories;
- overall coordination of the quality assurance/quality control plan;
- project quality assurance/quality control; and
- periodic reports to management, including suggestions for performing and verifying corrective actions.

Although it is the QAM's responsibility to insure compliance with this QAPP, it is the installation and site managers' responsibility to implement the quality assurance program and to maintain a strong line of communication with the QAM.

The QAM and the designated auditor will have the following qualifications at minimum:

- Bachelors degree in an appropriate scientific discipline and five years work experience in remedial investigation/feasibility studies with a focus on quality assurance.
- Two years experience in performing field activities (for field auditors).
- Two years experience in an environmental analytical laboratory (for laboratory auditors).

The QAM is also responsible for ensuring that analytical data is properly validated. Either the QAM and/or his or her designee will perform data validation. The following minimum qualifications are required to perform data validation:

- Bachelors degree in chemistry
- Two years work experience in an environmental analytical laboratory.
- Two years experience in performing data validation activities.

Subcontractors generating data for the investigation are responsible for ensuring that the precision, accuracy, completeness, and representativeness of their data are known and documented. To ensure that responsibilities are uniformly met, each subcontractor will be required to adhere to this QAPP and to Mound Plant ER Program SOP's.

An ER Program TSO subcontractor internal kickoff meeting will be held before field work begins to review the operable unit-specific project work and quality assurance plan(s) and procedures. The kickoff meeting will be attended by, at a minimum, the following ER Program TSO subcontractor personnel: the project

manager or deputy project manager, the installation manager, the site manager, the field manager, the project QAM, and all personnel assigned to the field effort. Attendance at this meeting will be documented to provide evidence of quality assurance indoctrination for the field activities to be conducted during the Operable Unit 3 investigation. Such documents are to be maintained by the QAM and filed in the Mound Plant project file.

3. QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY

The overall quality assurance objectives for field sampling, field measurements, and laboratory analysis are to produce data of known and sufficient quality to support the site evaluation and the selection of remedial alternatives. Appropriate procedures and quality control checks will be used so that known and acceptable levels of accuracy and precision are maintained for each data set. This section defines the objectives (goals) for accuracy, precision, completeness, representativeness, and comparability for measurement data. These goals are primarily expressed in terms of acceptance criteria for the quality control checks performed. The field and laboratory quality control checks planned for this investigation are presented in Tables III.1 and III.2 and are defined in section 8 of this QAPP. Quality assurance goals for field measurements and field screening are also discussed.

3.1. ACCURACY

3.1.1. Definition

Accuracy of measurement data is defined as the degree of a measurement, X, with an accepted reference or true value, T. It is usually expressed as the difference between the two values, X - T, the difference as a percentage of the reference of true value, 100(X - T)/T, and sometimes expressed as a ratio, X/T. These expressions give a measure of the bias in a system.

3.1.2. Accuracy Goals for Field Screening and Field Measurements

Field measurements will include pH, specific conductance, alkalinity, and temperature of water samples. Field screening will include organic vapor, low energy gamma radiation, and alpha surface contamination. Accuracy is measured with a calibration check (source check for radiation measurements) for all of these parameters except temperature. Table III.1 summarizes the quality control checks, acceptance criteria, and corrective actions taken for accuracy.

3.1.3 Accuracy Goals for Laboratory Measurements

Accuracy of laboratory analyses will be measured using the following quality control checks: calibration standards, surrogate spikes of all samples, laboratory control samples, and matrix spikes of selected

Table III.1. Summary of Quality Control Procedures for Field Screening and Field Measurements

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SOP 2.2	pH	Calibration with two buffer solutions (pH 4 and 7 or 7 and 10) (for accuracy)	Before and after a well purge	+/- 0.1 units of true value	Recalibrate; check pH meter; replace probe and meter if necessary.
		Calibration check with one buffer solution (for accuracy)	Once per well, after alkalinity analysis	+/- 0.1 units of true value	Recalibrate.
		Duplicate sample (for precision)	One per ten or fewer field samples collected	+/- 0.2 units difference	Evaluate data for useability.
SOP 2.2	Electrical conductivity	Calibration (3 standards) (for accuracy)	Before and after sample shift	+/- 10% of true value	Recalibrate; check meter; replace probe or meter if necessary.
		Calibration check (1 standard) (for accuracy)	One per ten or fewer field samples collected	+/- 20% of true value	Recalibrate.
		Duplicate sample (for precision)	One per ten or fewer field samples collected	≤ 20% RPD	Evaluate data for useability.
SOP 2.2	Temperature	Duplicate sample (for precision)	One per ten or fewer field samples collected	+/- 1°C	Evaluate data usability.
		Calibration (for accuracy)	NA	+/- 2°C (manufacturer's specification)	NA

NA - Not Applicable

Table III.1. (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SOP 2.2	Alkalinity	Second titration (for accuracy)	once per well sampled	+/- 10% of 1st titration	Retitrate new sample.
		Duplicate sample (for precision)	One per ten or fewer field samples collected.	≤ 20% RPD	Evaluate data for useability.
SOP 6.2	Organic vapor level (PID)	Initial calibration (for accuracy)	Once per day	+/- 10% of true value	Recalibrate.
		Duplicate standard (for precision)	Once per day	+/- 20% of initial calibration	Remeasure.
SOP 6.7	Low-energy gamma radiation	Source check (for accuracy)	Once per day	+/- 3xSD	Check instrument settings (voltage, gain, etc.); remeasure source.
		Background check (for accuracy)	Once per day	+/- 3xSD	Clean detector and nearby surfaces; ventilate room; recheck.
		Voltage plateau (for accuracy)	Once per week	Voltage should be 1100 to 1300V	Check voltage setting and re-plateau beginning at 900V. If still outside range, replace probe and check again. Do not exceed 1400V.
		Replicate measurement (for precision)	Once every 10 measurements	+/- 4xSD	Identify and correct problem; remeasure.

NA - Not Applicable

Table III.1. (concluded)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SOP 6.4	Alpha surface contamination	Source check (for accuracy)	Once per day or after instrument adjustments or repairs	+/− 10% of expected value	Check instrument settings (gain, voltage, etc.) recheck; replace if still out.
		Background count (10 minutes) (for accuracy)	Once per day	≤ 2 cpm	Clean detector and nearby surfaces; check for light leaks; replace probe if necessary.
		Replicate measurement (for precision)	once every 10 measurements	+/− 4xSD	Identify and correct problem, remeasure.
EG&G SOP (See Appendix F)	Plutonium-238 and Thorium-232 (onsite lab)	**Laboratory quality control**			
		Calibration (counting system)	Once per month	per in house statistical package	Recalibrate
		Calibration check	Once per day	per in house statistical package	Recalibrate
		Background check	Once per week	<2 x LLD	Identify and correct problem
		Sample replicate	Once every five samples analyzed	<30% RSD	Evaluate data

LLD - lower limit of detection
NA - Not Applicable
PID - photoionization detector
RPD - relative percent difference
RSD - relative standard deviation
SD - standard deviation
SOP - Standard Operating Procedure

Table III.2. Summary of Internal Quality Control Procedures For Field Activities and Laboratory Measurements: Liquid/Surface Water and Soil/Sediment Samples

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
E906.0[a]	Tritium	**Field quality control**			
		Duplicate	1 every 10 or fewer field samples (water)	+4 x SD [c,d,k]	Evaluate data for useability.
			1 every 10 or fewer field samples (soil)	Not applicable	Evaluate data for variability.
		Equipment (rinsate) blank[b]	1 every 10 or fewer field samples (water)	≤10 x level in associated samples [k]	Evaluate potential sources; Evaluate associated data for useability.
		Bottle lot blank	1 every bottle lot	≤10 x level in associated samples [k]	Evaluate potential sources; Evaluate associated data for useability.
		Laboratory quality control			
		Background	Once per day	+ 3 x SD, limit-gross contamination; background subtracts	Identify and correct problem.
		Source check	Once per day	± 3 x SD	Identify and correct problem.
		Method blank	1 every 20 or fewer samples of a similar matrix	≤ 2 x LLD	Identify and correct problem; reanalyze blank.
		Method spike	1 every 20 or fewer field samples of a similar matrix	± 3 x SD[c] normalized deviations	Identify and correct problem; associated sample results for useability.
		Matrix spike	1 every 20 or fewer field samples of a similar matrix	± 3 x SD[c] normalized deviation	Identify and correct problem; evaluate associated sample results for useability.
		Replicate sample	1 every 20 or fewer field samples of a similar matrix	+ 4 x SD[c,d] normalized range	Identify and correct problem; associated sample results for useability.

Table III.2. (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SW8080[g]	Organochlorine pesticides/PCBs (TCL)	**Field quality control**			
		Duplicate	1 every 10 or fewer field samples (water)	≤35% RPD[k]	Evaluate data for useability.
			1 every 10 or fewer field samples (soil)	Not applicable	Evaluate variability.
		Equipment (rinsate) blank[b]	1 every 10 or fewer field samples (water)	≤10 x level in associated samples[k]	Evaluate potential sources; Evaluate associated data for useability.
		Bottle lot blank	1 every bottle lot	≤10 x level in associated samples[k]	Evaluate potential sources; Evaluate associated data for useability.
		Laboratory quality control			
		Method blank	1 per 20 samples analyzed of a given matrix or fewer	≤DL	Investigate source; Reextract and reanalyze associated samples.
		Matrix spike	1 per 20 samples of a given matrix in a case or fewer	See Table III.3	Evaluate data for useability.
		Matrix spike duplicate	1 per 20 samples of a given matrix in a case or fewer	See Table III.3	Evaluate data for useability.
		Surrogate spike	All lab and field samples	See Table III.3	Evaluate data for useability.

Table III.2. (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SW8080[g] (continued)	Organochlorine pesticides/PCBs (TCL)	Calibration (initial and continuing)	5 points; when calibration check criteria exceeded.	≤ 10% RSD for calibration factors ≤ 20% breakdown for Endrin, 4,4'-DDT or combined breakdown for Endrin/ 4,4'-DDT	Recalibrate
		Calibration check	Once per day or every 10 samples, whichever is more frequent	≤15% D	Recalibrate.
		Retention times windows	Each new GC column and daily	±3 x SD of 3 standards	Identify and correct problem.
CLP SOW[e]; SW8240[g]	Volatile organic compounds (TCL), acetonitrile, and acrylonitrile	**Field quality control**			
		Trip blank	1 per shipping container to Lab	≤10 x level in associated samples[k]	Evaluate potential sources; Evaluate associated data for useability.
		Duplicate	1 every 10 or fewer field samples (water)	≤35% RPD[k]	Evaluate data for useability.
			1 every 10 or fewer field samples (soil)	Not applicable	Evaluate variability.

Table III.2. (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
CLP SOW[e]; SW8240[g]	Volatile organic compounds (TCL), acetonitrile, and acrylonitrile	Equipment (rinsate) blank[b]	1 every 10 or fewer field samples (water)	≤ 10 x level in associated samples[k]	Evaluate potential sources; Evaluate associated data for useability.
		Sample bank blank	1 every 20 or fewer field samples	≤ 10 x level in associated samples[k]	Evaluate potential sources; Evaluate associated data for useabililty.
		Field (ambient) blank	1 every 20 or fewer field samples	≤ 10 x level in associated samples[k]	Evaluate potential sources; Evaluate associated data for useability.
		Bottle lot blank	1 every bottle lot	≤ 10 x level in associated samples[k]	Evaluate potential sources; Evaluate associated data for useability.

Table III.2. (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
CLP SOW (continued)	Volatile organic compounds (TCL), acetonitrile, and acrylonitrile	**Laboratory quality control**			
		Method blank	Once per 12-hour period	≤ 5 x CRQL of [f] ≤ CRQL others	Investigate source; reextract and reanalyze associated samples.
		Matrix spike	1 per 20 samples of a given matrix in a case or fewer; see CLP SOW	See Table III.3	Evaluate data for useability.
		Matrix spike duplicate	1 per 20 samples of a given matrix in a case or fewer; see CLP SOW	See Table III.3	Evaluate data for useability.
		Surrogate spike	All lab and field samples	See Table III.3	See CLP SOW.
		Tuning criteria	Daily or each 12-hour period, whichever is more frequent	CLP SOW	Retune; reanalyze associated samples.
		Calibration	CLP SOW	CLP SOW	Recalibrate before sample analysis.
		Retention time window	CLP SOW	± 0.06 relative retention time units	See CLP SOW.

Table III.2. (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
CLP SOW (continued)	Volatile organic compounds (TCL), acetonitrile, and acrylonitrile	Calibration check compounds	With every calibration	≤ 30% RSD from RRF	Recalibrate.
		Internal standard	Every sample	CLP SOW	See CLP SOW.
		System performance check compounds	Once each 12-hour period and every calibration	RRF ≥ 0.300 system performance check compounds except, RRF ≥ 0.250 bromoform	Identify source and correct. Recalibrate if source not found and corrected.
		Continuing calibration check compounds	Once each 12-hour period	≤ 25% difference to initial RRF for selected compounds	Identify source and correct. Recalibrate if source not found and corrected.
SW3550, SW3510/mod. SW8015[i]	Extractable Petroleum Hydrocarbons	**Field quality control**			
		Duplicate	1 every 10 or fewer field samples (water)	≤ 35% RPD[k]	Evaluate data for useability.
			1 of every 10 or fewer field samples (soil)	Not applicable	Evaluate data for variability.
		Equipment (rinsate) blank[b]	1 every 10 or fewer field samples (water)	≤10 x level in associated samples[k]	Evaluate potential sources; Evaluate associated data for useability.
		Bottle lot blank	1 every bottle lot	≤ 10 x level in associated samples[k]	Evaluate potential sources; Evaluate associated data for useability.

Table III.2. (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SW3550, SW3510/mod. SW8015[i] (continued)	Extractable Petroleum Hydrocarbons	**Laboratory quality control**			
		Method blank	1 per 20 samples analyzed of a given matrix	< detection limit	Identify and correct source. Reanalyze blank and associated samples.
		Calibration	5 points; when calibration check criteria exceeded	≤ 20% RSD for calibration factors	Recalibrate.
		Calibration check	Once per 10 samples analyzed	± 15% from initial response factor	Recalibrate.
		Matrix spike	1 per 20 samples of a given matrix	75 - 125% recovery	Evaluate data for useability.
		Matrix spike duplicate	1 per 20 samples of a given matrix	30% relative percent difference	Evaluate data for useability.

Table III.2. (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
CLP SOW	Semivolatile organic compounds (TCL)	**Field quality control**			
		Duplicate	1 every 10 or fewer field samples (water)	≤ 55% RPD[k]	Evaluate data for useability.
			1 every 10 or fewer field samples (soil)	Not applicable	Evaluate variability.
		Equipment (rinsate) blank[b]	1 every 10 or fewer field samples (water)	≤ 10 x level in associated samples[k]	Evaluate potential sources; Evaluate associated data for useability.
		Bottle lot blank	1 every bottle lot	≤ 10 x level in associated samples[k]	Evaluate potential sources; Evaluate associated data for useability.
		Laboratory quality control			
		Method blank	1 per 20 samples analyzed of a given matrix or fewer; see CLP SOW	≤ 5 x CRQL for the phthalate esters and ≤ CRQL others	Investigate source; reextract and reanalyze associated samples.
		Matrix spike	1 per 20 sample of a given matrix or fewer; see CLP SOW	See Table III.3	Evaluate data for useability.

Table III.2. (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
CLP SOW (continued)	Semivolatile organic compounds (TCL)	Matrix spike duplicate	1 per 20 sample of a given matrix or fewer; see CLP SOW	See Table III.3	Evaluate data for useability.
		Surrogate spike	All lab and field samples	See Table III.3	See CLP SOW.
		Tuning criteria	Daily or each 12-hour period, whichever is more frequent	CLP SOW	Retune; reanalyze associated samples.
		Calibration	CLP SOW	CLP SOW	Recalibrate before sample analysis.
		Calibration check compounds	With every calibration	≤ 30% RSD from RRF	Recalibrate.
		Internal standard	Every sample	CLP SOW	See CLP SOW.
		System performance check compounds	Once each 12-hour period and every calibration	RRF ≥ 0.050 for selected compounds	Identify source and correct. Recalibrate if source not found and corrected.
		Continuing calibration check compounds	Once each 12-hour period	≤ 25% difference to initial RRF for selected compounds	Identify source and correct. Recalibrate if source not found and corrected.

Table III.2. (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
CLP SOW (continued)	Semivolatile organic compounds (TCL)[f]	Retention time window	CLP SOW	± 0.06 relative retention time units	See CLP SOW
CLP SOW SW3050/SW6010	Inorganics (TAL) or lithium	**Field quality control**			
		Duplicate	1 every 10 or fewer field samples (water)	≤ 25% RPD[k]	Evaluate data for useability.
			1 every 10 or fewer field samples (soil)	Not applicable	Evaluate variability.
		Equipment (rinsate) blank[b]	1 every 10 or fewer field samples (water)	≤ 10 x level in associated samples[k]	Evaluate potential sources; Evaluate associated data for useability.
		Bottle lot blank	1 every bottle lot	≤ 10 x level in associated samples[k]	Evaluate potential sources; Evaluate associated data for useability.
		Laboratory quality control			
		Initial and continuing calibration blanks	After every initial and continuing calibration verification or 10% of every 2 hours, whichever is more frequent	≤ CRDL	Correct problem; recalibrate; reanalyze preceding 10 samples or all since last good blank.
		Preparation blank	One per sample delivery group or batch, whichever is more frequent	≤ CRDL	If sample results < 10 x CRDL, but > CRDL, redigest and reanalyze.

Table III.2. (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
CLP SOW SW3050/SW6010 (continued)	Inorganics (TAL) or lithium	Laboratory control sample	1 per group of samples in a delivery group or batch, whichever is more frequent	80-120% recovery	Correct problem; redigest and reanalyze associated samples.
		Initial calibration verification standard	CLP SOW	CLP SOW	See CLP SOW.
		Continuing calibration verification standard	CLP SOW	CLP SOW	See CLP SOW.
		Linear range check standard (CRI, CRA)	CLP SOW	Not established	None.
		Interference check sample	Sample twice per 8-hour shift, or at beginning and end of analysis run, whichever is more frequent	± 20% of true value	Correct problem; recalibrate reanalyze samples since last good interference check sample.

Table III.2. (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
CLP SOW SW3050/SW6010 (continued)	Inorganics (TAL) or lithium	Inductively coupled plasma serial dilution	1 per group of samples of a given matrix, concentration, or each delivery group, whichever is more frequent	If result > 50 x instrument detection limit ± 10% difference	Evaluate data for useability.
		Spike sample	1 per group of samples of a given matrix, concentration, or sample delivery group, whichever is more frequent.	75-125% recovery	Evaluate date for useability.
		Sample duplicate (sample replicate)	1 per group of samples of a given matrix, concentration, or sample delivery group, whichever is more frequent.	If result ≥ 5 x CRDL: ± 20% RPD If result ≤ 5 x CRDL: ± CRDL	Evaluate data for useability.
		Method standard addition (graphite furnace atomic absorption only)	CLP SOW	CLP SOW	See CLP SOW.
		Linear range analysis for inductively coupled plasma	CLP SOW	CLP SOW	Reanalyze.

Table III.2. (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
CLP SOW SW3050/SW6010 (continued)	Inorganics (TAL) or lithium	Interelement corrections for inductively coupled plasma	Once per year or or when instrument adjusted	CLP SOW	CLP SOW
SW9250[g] E353.2[i]	Chloride, (Cl) Nitrate-Nitrite (NO_3-NO_2)	**Field quality control**			
		Duplicate	1 every 10 or fewer field samples (water)	≤ 25% RPD[k]	Evaluate data for useability.
			1 every 10 or fewer field samples (soil)	Not applicable	Evaluate variability.
		Equipment (rinsate) blank[b]	1 every 10 or fewer field samples (water)	≤ 10 x level in associated samples[k]	Evaluate potential sources; Evaluate associated data for useability.
		Bottle lot blank	1 every bottle lot	≤ 10 x level in associated samples[k]	Evaluate potential sources; Evaluate associated data for useability.
		Laboratory quality control			
		Method blank	1 per 20 samples analyzed	< Detection limit	Correct problem; reanalyze.
		Calibration (3 points) and Reagent Blank	When instrument conditions change or when calibration check criteria exceeded	Correlation coefficient ≥ 0.995 or plot curve for nonlinear analytes	Recalibrate

Table III.2. (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SW9250[g] E353.2[i] (continued)	Chloride, (Cl) Nitrate-Nitrite (NO_3-NO_2)	Calibration check	Prior to sample analysis and one per 10 samples analyzed	± 15% of initial calibration response	Identify and correct problem; recalibrate.
		Matrix spike	1 per 20 samples of a given matrix	75-125% Recovery	Evaluate data for useability.
		Matrix spike duplicate	1 per 20 samples of a given matrix	≤ 20% relative percent difference	Evaluate data for useability.
		Laboratory Control Sample (chloride, nitrate)	1 for each calibration	Vendor specification	Evaluate data for useability.

Table III.2. (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
USATHAMA[i] LW-06	Explosives	**Field quality control**			
		Duplicate	1 every 10 or fewer field samples (water)	Not established	Evaluate data for useability.
			1 every 10 or fewer field samples (soil)	Not applicable	Evaluate variability.
		Bottle lot blank	1 every bottle lot	≤ 10 x level in associated samples[k]	Evaluate potential sources; Evaluate associated data for useability.
		Laboratory quality control			
		Method blank	1 per 20 samples of a given matrix	< DL	Reanalyze blank.
		Calibration	(5 pt.) when calibration check limit criteria exceeded	≤ 15% RSD of average response factor	Recalibrate.

Table III.2. (continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
USATHAMA[j] LW-06 (continued)	Explosives	Matrix spike	1 per 20 samples of a given matrix	See Table III.3	Evaluate data for useability.
		Matrix spike duplicate	1 per 20 samples of a given matrix	See Table III.3	Evaluate data for useability.
		Retention time window	With every calibration check	Column and compound specific	Identify source, correct problem; reanalyze samples since last good calibration check.
		Calibration check	Prior to sample analysis and 1 per 10 samples analyzed	± 25% of peak height of initial 10 x TRL calibration standard	Recalibrate.

Table III.2. (concluded)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action

Note: An addendum to the QAPP will be submitted to USEPA for review and approval for bottle lot blanks when USEPA Region V finalizes procedures for the preparation and analysis of the blanks.

A sample bank blank monitors for VOCs potentially present in surrounding environment where samples are commonly handled before shipment. The blank is prepared with organic-free deionized water in sample vials, capped with no bubbles, and placed in the desired location during sample handling.

[a] "Prescribed Procedures for Measurement of Radioactivity in Drinking Water,"EPA, EPA-6000/4-80-032, latest version.

[b] To be prepared for sampling locations without dedicated sampling equipment.

[c] Procedures according to "Environmental Radioactivity Laboratory Intercomparison Studies Program,"EPA, EPA-600/4-81-004.

[d] "Improved Evaluation of Environmental Radiochemical Inorganic Solid Matrix Replicate Precision: Normalized Range Analysis Revisited," J.W. Dillard and R.E. Gladd, 36th Annual Conference on Bioassay, Analytical, and Environmental Radiochemistry, Oak Ridge, TN, 1990.

[e] USEPA Contract Laboratory Program Statement of Work for Organic Analysis, No. 2/88 with most current revisions or most recent version. Pesticides/PCBs will be analyzed according to CLP SOW Document No. OLM01.0, including revisions 0.1 (December 1990) and 0.2 (January 1991).

[f] For methylene chloride, acetone, toluene, or 2-butanone.

[g] "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, EPA, November 1986 or most recent version.

[h] USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis, No. 7/88 with most current revisions or most recent version.

[i] "Methods for Chemical Analysis of Water and Wastes,"EPA, EPA-600/4-79-020, revised March 1983.

[j] USATHAMA Method UW-11, "Ion Nitroexplosives in Water by HPLC," and USATHAMA Method LW-06, "Nitroexplosives in Soils by HPLC," IT Laboratories, Knoxville, TN.

[k] All field quality control samples associated with a batch of samples will be evaluated as a unit. This criterion is designed for evaluating an isolated quality control sample and does not take into account the interdependencies of quality control results. Corrective actions will be taken at all levels of detection in the blank samples associated with field sampling. The criterion applies only if there is a positive detection of the same compound in associated samples. All data will be evaluated on a case by case basis, therefore this criterion may not be applicable at times, (e.g., reported levels near detection limit).

CLP - Contract Laboratory Program
CRDL - contract-required detection limit
CRQL - contract-required quantitation limit
LLD - lower limit of detection
PCB - polychlorinated biphenyl
RRF - relative response factor
RSD - relative standard deviation
SD - standard deviation
SOW - Statement of Work
TAL - Target Analyte List
TCL - Target Compound List

samples collected in the field. Surrogate spike and matrix spike results will be expressed as a recovery of an analyte added to the sample at a known concentration:

$$\% \text{ Recovery} = \frac{(SSR-SR)}{SA} \times 100\%$$

where

SSR = spiked sample result
SR = sample results (not applicable for surrogate recovery)
SA = amount of spike added.

Calibration check standards are expressed as a percent difference from the true value, i.e., 100 (X-T)/T.

Table III.2 summarizes the frequency and acceptance criteria for the accuracy quality control checks for soil/sediment and water analyses. For those analyses performed according to the procedures in the CLP for routine analytical services, goals for accuracy are established in the CLP Statement of Work (SOW). Accuracy goals for other analyses are based on established laboratory control limits and guidance in the analytical methods. It should be noted that the matrix spike recovery criteria are based upon clean sample matrices. Due to the unique nature of some of the matrices sampled for the Operable Unit 3 investigation (e.g., sump liquid), these criteria may not be met. For those identified unique matrices in which matrix spike recoveries did not meet acceptance criteria, the results will be discussed along with the implications on associated sample results, during the data review and/or validation.

Additional quality control checks to be performed that monitor accuracy of results are trip blanks, laboratory method blanks, sample bank blanks, and equipment (rinsate) blanks. Trip blanks are analyzed for VOCs to check for cross-contamination or contamination from ambient conditions that might occur during sample shipping and storage of aqueous VOC samples. Method blanks are performed as part of the applicable analytical methods to monitor for contamination due to laboratory procedures. Sample bank blanks monitor for potential VOCs in common areas in the field where samples are handled. Equipment blanks are performed in order to monitor potential sources of contamination due to the sampling procedure. The frequency of these blanks for each analysis are presented in Table III.2.

3.2. PRECISION

3.2.1. Definition

Precision is a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is expressed as a standard deviation among a group of measurements or as a relative percent difference (RPD) between two measurements.

3.2.2. Precision Goals for Field Measurements

Where applicable, precision of field measurements will be assessed for a selected set of parameters. Precision for alkalinity, specific conductance, pH, and temperature will be measured with duplicate sample analysis for every 10 or fewer samples collected. For measurements of organic vapor, precision will be assessed by analyzing the calibration standard a second time. A replicate measurement will be made for every 10 measurements for low-energy gamma radiation and alpha surface contamination and every five measurements for the onsite screening for plutonium-238 and thorium-232. Table III.1 summarizes the acceptance criteria for duplicate measurements, usually in terms of RPD. RPD is defined as

$$RPD = \frac{C1-C2}{(C1+C2)/2} \times 100\%$$

where

C1 = Concentration of analyte in the primary sample
C2 = Concentration of analyte in the duplicate sample

3.2.3. Precision Goals for Laboratory Measurements

Precision of laboratory analyses will be assessed by analyzing duplicate samples (matrix spike duplicates [MSD] being included as duplicate samples), and/or by analyzing aliquots (sample replicates) of one sample. Analysis of duplicate samples measures the precision of both the sampling and analysis, whereas a sample replicate generally measures only the analytical precision (depending upon the nature of the sample matrix). Precision of the duplicate and replicate analyses will be expressed as an RPD. Table III.2 summarizes for each laboratory analysis the frequency and acceptance criteria for duplicate and replicate samples. Precision criteria are based on CLP guidance, where available, and best technical judgement.

3.3. COMPLETENESS

Completeness is a measure of the amount of data obtained from a measurement system that achieves the project goals, compared to the amount expected under normal conditions. Completeness is affected by unexpected conditions that may occur during the data collection process. Occurrences that reduce the amount of data collected include events such as a dry well, an instrument breakdown, or a loss of sample extract. All reasonable attempts will be made to minimize loss of data (e.g., through regular maintenance of field instruments, and replacing/repairing instruments that have broken down) and to recover lost data. Completeness for this project will be calculated for a number of the criteria established in this QAPP, including accuracy and precision acceptance criteria, detection limits, sample holding times, the number of sample points sampled, and the other analytical requirements listed on Tables III.1, III.2, and III.3 (e.g., calibration and tune criteria, and blank contamination). Completeness will be assessed for each field measurement and laboratory analysis. The following are parameters used to measure completeness of laboratory analyses:

- the number of sample holding times met,
- the number of sample analyses that meet precision criteria (on Tables III.2 and III.3),
- the number of sample analyses that meet accuracy criteria (on Tables III.2 and III.3),
- the number of sample analyses that meet calibration and tuning criteria (on Table III.2),
- the number of sample analyses with reported quantitation limits at or below the required limits (see section 6), and
- the number of sample analyses with laboratory method blanks below established criteria (on Table III.2).

It should be noted that reported quantitation limits are heavily dependent upon the nature of the sample matrix, the amount of sample used in the analysis, percent moisture (solid matrices), and the dilution factor, if any. Therefore, samples with unusual matrices (e.g., a very high petroleum hydrocarbon content) will not be included in the completeness calculation, since the acceptance criteria developed are not based upon these matrices. However, the quality control results for these samples will be noted.

Completeness goals for this program have been established for three aspects of the scope of work: field measurements, sample collection, and laboratory analyses. The completeness goal for sample collection and field measurements is 90%. If the 90% completeness goal is not met, the necessity for resampling will be determined on a case by case basis due to the many factors that could impact data completeness. The

Table III.3. Laboratory Control Limits for Matrix Spikes, Matrix Spike Duplicates, and Surrogate Spikes: Liquid/Surface Water/and Soil/Sediment Samples

ANALYTICAL METHOD	SPIKING COMPOUNDS	SPIKE CONCENTRATION		CONTROL LIMITS			
		Water	Soil	Percent Recovery (%)		Relative Percent Difference (%)	
		(µg/L)	(µg/kg)[a]	Water	Soil	Water	Soil
SW8080 Pesticides/ PCBs	Matrix Spike						
	Lindane	0.02	0.08	56-123	32-127	≤15	≤50
	Heptachlor	0.02	0.08	40-131	34-111	≤20	≤31
	Aldrin	0.02	0.08	40-120	42-122	≤22	≤43
	Dieldrin	0.05	0.2	52-126	36-146	≤18	≤38
	Endrin	0.05	0.2	56-121	30-147	≤21	≤45
	4,4'-DDT	0.05	0.2	38-127	25-160	≤27	≤50
	Surrogate						
	Dibutylchlorendate	0.1	0.1	24-154	24-150	NA	NA
CLP SOW Volatile Organic Compounds	Matrix Spike						
	1,1-DCE	50	50	61-145	59-172	≤22	≤22
	Trichloroethene	50	50	71-120	62-137	≤24	≤24
	Benzene	50	50	76-127	66-142	≤21	≤21
	Toluene	50	50	76-125	59-139	≤21	≤21
	Chlorobenzene	50	50	75-130	60-133	≤21	≤21
	Surrogates						
	Toluene-d8	50	50	88-110	81-117	NA	NA
	4-Bromo-fluorobenzene	50	50	86-115	74-121	NA	NA
	1,2,Dichloroethane-d4	50	50	76-114	70-121	NA	NA

NA - Not Applicable

Table III.3. (continued)

ANALYTICAL METHOD	SPIKING COMPOUNDS	SPIKE CONCENTRATION		CONTROL LIMITS			
		Water	Soil	Percent Recovery (%)		Relative Percent Difference (%)	
		(µg/L)	(µg/kg)[a]	Water	Soil	Water	Soil
CLP SOW Semivolatile Organic Compounds	Matrix Spike						
	Phenol	100	100	12-89	26-90	≤35	≤35
	2-chlorophenol	100	100	27-123	25-102	≤50	≤50
	1,4-dichlorobenzene	50	50	36-97	28-104	≤27	≤27
	N-nitroso-di-n-propyl-amine	50	50	41-116	41-126	≤38	≤38
	1,2,4-trichlorobenzene	50	50	39-98	38-107	≤23	≤23
	4-chloro-3-methylphenol	100	100	23-97	26-103	≤33	≤33
	Acenaphthene	50	50	46-118	31-137	≤19	≤19
	4-nitrophenol	100	100	10-80	11-114	≤50	≤50
	2,4-dinitrotoluene	50	50	24-96	28-89	≤47	≤47
	Pentachlorophenol	100	100	9-103	17-109	≤47	≤47
	Pyrene	50	50	26-127	35-142	≤36	≤36
	Surrogates						
	Nitrobenzene-d5	50	50	35-114	23-120	NA	NA
	2-Fluorobiphenyl	50	50	43-116	30-115	NA	NA
	p-Terphenyl-d14	50	50	33-141	18-137	NA	NA
	Phenol-d5	100	100	10-94	24-113	NA	NA
	2-Fluorophenol	100	100	21-100	25-121	NA	NA
	2,4,6-Tribromophenol	100	100	10-123	19-122	NA	NA

NA - Not Applicable

Table III.3. (concluded)

ANALYTICAL METHOD	SPIKING COMPOUNDS	SPIKE CONCENTRATION		CONTROL LIMITS			
		Water	Soil	Percent Recovery (%)		Relative Percent Difference (%)	
		(µg/L)	(µg/kg)[a]	Water	Soil	Water	Soil
USATHAMA Explosives	Blank Spike[b] (low concentration)						
	RDX	11.6	4.4	62-87	40-160	32[d]	40[c,d]
	1,3,5-TNB	28	2.6	85-100'	40-160	19	30
	2,4,6-TNT	5.8	2.6	78-102	40-160	29	40
	2,6-DNT	1.0	2.6	66-102	40-160	45	60
	2,4-DNT	0.8	0.6	74-99	40-160	31	40
	Blank Spike (high concentration)						
	RDX	58	22	49-71	40-160	19	30
	1,3,5-TNB	140	13	85-108	40-160	20	30
	2,4,6-TNT	29	13	83-104	40-160	19	30
	2,6-DNT	5.0	13	74-96	40-160	19	30
	2,4-DNT	4.0	3.0	77-100	40-160	20	30

[a]Spike amount is for low concentration soils.

[b]These are current ITAS laboratory-established control limits. These limits change as new data points are generated. Control limits for sample matrix spikes have not been determined.

[c]Control limits have not been established for solid matrices. These limits are project-established advisory limits for data evaluation purposes and not for validation and will be used until control charts have been developed.

[d]Precision is expressed for this analysis as the difference between the highest percent recovery and lowest percent recovery, as defined in the USATHAMA Quality Assurance Manual

RDX - hexahydro-1,3,5-trinitro-s-triazine cyclotetratmethylenetetranitramine
TNT - 2,4,6-trinitrotoluene
1,3,5-TNB - 1,3,5-trinitrobenzene
2,4-DNT - 2,4-dinitrotoluene
2,6-DNT - 2,6-dinitrotoluene

NA - Not Applicable

completeness goal for laboratory analyses is 95%. There are no critical data points for this stage of the investigation.

Table III.4 summarizes the equations used for calculating completeness of sample collection, field measurements, and laboratory analyses.

3.4. REPRESENTATIVENESS

Representativeness expresses the degree to which data accurately and precisely represent characteristics of a population, parameter, variation at a sampling point, process condition, or environmental condition. Data representativeness for this project is accomplished through implementing approved sampling procedures and analytical methods that will generate data representative of site conditions. Sampling procedures to be used (Mound Plant ER Program SOP 2.2, Field Measurements on Ground and Surface Water Samples [revision 1] [DOE 1991]) (Appendix E) are designed to minimally impact the sample generated so that conditions representative of the sampling location are obtained. An example of a sampling technique that achieves this is collection of a groundwater sample after a specified volume of groundwater is purged from the well so that a sample representative of the groundwater conditions is obtained. Analytical methods are selected that will most accurately and precisely represent the true concentration of the parameter of interest to the best of technical knowledge and to the level that suits the program's needs. The quality control procedures implemented and instrumentation used for a given analysis, for example, affect the representativeness of the data generated.

3.5. COMPARABILITY

Comparability expresses the confidence with which one data set can be compared to another. Comparability of data sets generated for this investigation will be obtained through implementation of specific protocols for sampling and analysis of samples by the use of traceable reference materials for laboratory standards, and by participation of the laboratories in external performance evaluation programs. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling and analytical methods. The procedures to be used in obtaining the planned analytical data, as documented in the following sections, are expected to provide comparable data. These new analytical data, however, may not be directly comparable to existing data because of differences in procedures and quality assurance objectives.

Table III.4. Calculations for Completeness of Measurement Data

Sample Collection:

$$\text{Completeness} = \frac{\text{Number of sample points sampled}}{\text{Number of sample points planned}} \times 100\%$$

Field Measurements (Calculated for each type of field measurement):

$$\text{Completeness} = \frac{\text{Number of sample points sampled}}{\text{Number of sample points planned}} \times 100\%$$

Laboratory Analyses (Calculated for each analysis)[a,b]:

$$\text{Total Completeness (\%) (for a given laboratory analysis)} = \frac{\text{Number of "valid" data points}}{\text{Number of data points collected}} \times 100\%$$

[a]Please note that completeness will be impacted by unusual sample matrices; therefore, these sample results will not be included in the calculations for completeness, but will be noted separately.
[b]Calculated for individual parameters. Completeness will be reported for each parameter for a given analysis.

4. SAMPLING PROCEDURES

Surface and subsurface soils, sediments, test pits, sump liquid, and liquid in the oil burn structure will be sampled as part of the Operable Unit 3 Limited Field Investigation. Table IV.4 summarizes the number of samples and their frequency for the investigation. In addition, screening for VOCs will be performed. Table IV.1 gives the Mound Plant ER Program SOPs applicable to these activities that are discussed in the work plan. These SOPs have been revised according to comments from EPA and Ohio EPA and are included in Appendix E of this QAPP. All members of the field team will be required to read the SOPs listed in Table IV.1 before the Operable Unit 3 limited field investigation begins. A volume of these SOPs will be available at the work site at all times. All samples will be shipped the same day of collection by common overnight carrier.

Tables IV.2 and IV.3 gives the container, preservation, and holding time requirements for the Operable Unit 3 limited field investigation analytes. The sample containers are received from the manufacturer (Eagle Picher or equivalent) which are certified for preparation in accordance with EPA requirements. All samples will be shipped the same day of collection by common overnight carrier.

Table IV.1. Mound Plant ER Program SOPs Applicable to Operable Unit 3

Section 1 - General	Effective Date	Revision Number	Purpose
1.1 General Instructions for Field Personnel	May 1991	1	To provide field personnel with instructions regarding activities to be performed before, during, and after field investigations.
1.3 Sample Control and Documentation	January 1991	0	To define the steps necessary for sample control and identification, data recording, and chain-of-custody documentation.
1.4 Sample Containers and Preservation	May 1991	1	To provide guidance in the selection suitable containers for samples, container cleaning, required sample volumes, sample collection, times, and the recommended holding preservation techniques for water, wastes, sediments, sludges, and soil samples.
1.5 Guide to the Handling, Packaging, and Shipping of Samples	May 1991	1	To provide a general guide for packaging and shipping samples of environmental and hazardous materials to the samples laboratory. In addition, instructions are provided to select the correct category for packaging and shipping samples of unknown contents.
1.6 General Equipment Decontamination	May 1991	1	To describe methods for the decontamination of field equipment potentially contaminated during sample collection.
1.8 Personnel Decontamination Level D Protection	January 1991	0	To describe the equipment and procedures required for the decontamination of persons who have performed field activities in Level D protective clothing.
Section 2 - Water Sampling			
2.2 Field Measurements on Ground and Surface Water Samples	May 1991	1	To obtain reliable and accurate measurements of the field chemistry of water quality samples.
2.8 Sampling for VOCs	January 1991	0	To outline procedures for collecting a representative groundwater sample and to transport it to/from its original environment to the laboratory for analysis of volatile organic compounds.
2.9 Surface Water Sampling	May 1991	1	To define guidelines followed by field personnel in sampling surface water bodies and documenting all aspects of surface water sample collection.

Table IV.1. (continued)

Section 4 - Drilling and Logging	Effective Date	Revision Number	Purpose
4.1 Soil Boring	January 1991	0	To ensure acceptable, consistent soil-boring procedures for all pertinent aspects of hazardous waste investigations.
4.2 Rock Boring	January 1991	0	To ensure acceptable, consistent rock boring procedures for all pertinent aspects of hazardous waste investigations.
4.6 Test Pit Logging and Sampling	January 1991	0	To describe the physical nature of consolidated or unconsolidated subsurface earthen materials encountered during the excavation of a pit or trench.
Section 5 - Soil Sampling			
5.1 Soil and Rock Borehole Logging and Sampling	January 1991	0	To describe the physical nature of consolidated or unconsolidated subsurface earthen materials encountered during auger, rotary, or other drilling activities and collect samples of the earthen materials for further evaluation.
5.2 Soil Sampling with a Spade and Scoop	May 1991	1	To describe a method for collecting a soil sample less than 4 feet below the land surface.
5.3 Subsurface Solid Sampling with Hand Auger and Thin-Wall Sampler	January 1991	0	To define a method of collecting subsurface solid samples with a hand auger and thin-wall tube sampler.
Section 6 - Health and Safety			
6.1 Health and Safety Monitoring of Combustible Gas Levels	January 1991	0	To describe the equipment and proper method for monitoring combustible gas levels in order to determine when an explosion hazard exists in the work environment.
6.2 Health and Safety Monitoring of Organic Vapors with a Photoionization Detector	January 1991	0	To describe the equipment and proper method for environmental monitoring of toxic gases and vapors using a portable photoionization detector.

Table IV.1. (continued)

Section 6 - (continued)		Effective Date	Revision Number	Purpose
6.4	Total Alpha Surface Contamination Measurements	January 1991	0	To provide guidance for determining levels of total surface alpha contamination on equipment, vehicles, and personnel that have been in contact with material that was potentially contaminated with alpha-emitting radionuclides.
6.7	Near Surface and Soil Sample Screening for Low-Energy Gamma Radiation Using the FIDLER	January 1991	0	To describe the procedure in which a field instrument for the detection of low-energy radiation (FIDLER) is used to monitor surfaces and soil samples for the presence of low-energy gamma radiations that accompany some alpha emissions.

Table IV.2. Sample Containers, Volumes, Preservation, and Holding Times: Liquid/Water Samples

Parameters	Analytical Method	Container[a]	Minimum Volume	Preservation	Holding Time[b]
Purgeable Halocarbons	SW5030/SW8010	Glass vial jar with Teflon-lined septum lid	3x40 mL	Cool 4°C	14 days
Purgeable Aromatic Compounds	SW5030/SW8020	Glass vial with Teflon-lined septum	3x40 mL	HCl to pH <2 Cool 4°C	14 days
Volatile Organic Compounds	CLP SOW	Glass vial with Teflon-lined septum	3x40 mL	HCl to pH <2 Cool 4°C	14 days
Semivolatile Organic Compounds	CLP SOW	Amber glass bottle with Teflon-lined lid	2000 mL	Cool 4°C	7 days extraction/40 days analysis[c]
Pesticides/PCBs	SW8080	Amber glass bottle with Teflon lined lid	2000 mL	Cool 4°C	7 days extraction/40 days analysis[c]
TAL Metals	CLP SOW	Polyethylene bottle	1000 mL	HNO_3 to pH <2, Cool 4°C	6 months, 28 days (mercury)
Cyanide	CLP SOW	Polyethylene bottle	1000 mL	NaOH to pH >12 Cool 4°C	14 days
Extractable Petroleum Hydrocarbons	SW3510/SW8015	Amber glass bottle	1000 mL	Cool 4°C	7 days extraction/ 40 days analysis

Table IV.2. (concluded)

Parameters	Analytical Method	Container[a]	Minimum Volume	Preservation	Holding Time[b]
Tritium	E906.0	Glass bottle	250 mL	None	None

NOTE: Holding times for CLP analyses are based on "Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses," EPA, February 1, 1988 and "Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses," EPA, July 1,1988.

[a]Sample containers will be certified cleaned by the manufacturer according to EPA standards.
[b]From date of collection.
[c]From date of extraction.

Table IV.3. Sample Containers, Volumes, Preservation, and Holding Times: Soil/Sediment Samples

Parameters	Analytical Method	Container[a]	Minimum Volume/Weight	Preservation	Holding Time[b]
Volatile Organic Compounds	CLP SOW	Glass jar vial with Teflon-lined lid	2x125mL (no headspace)	Cool 4°C	14 days
Semivolatile Organic Compounds	CLP SOW	Amber glass jar with Teflon-lined lid	30 grams	Cool 4°C	7 days extraction/ 40 days analysis[c]
Pesticides/PCBs	SW8080	Amber glass jar with Teflon-lined lid	30 grams	Cool 4°C	7 days extraction/ 40 days analysis[b]
TAL Metals and lithium	CLP SOW SW6010	Wide-mouth poly-ethylene bottle	50 grams	Cool 4°C	6 months 28 days (mercury)
Cyanide	CLP SOW	Wide-mouth poly-ethylene bottle	50 grams	Cool 4°C	14 days
Nitrate-Nitrite	E353.2	Wide-mouth poly-ethylene bottle	50 grams	Cool 4°C	28 days
Chloride	SW9250	Wide-mouth poly-ethylene bottle	50 grams	Cool 4°C	28 days

Table IV.3. (concluded)

Parameters	Analytical Method	Container[a]	Minimum Volume/Weight	Preservation	Holding Time[b]
Explosives	USATHAMA LW-06	125-mL wide-mouth amber glass jar with Teflon-lined lid	10 grams	Cool 4°C	7 days extraction/ 30 days analysis[c]
Extractable petroleum hydrocarbons	SW 3550/SW 8015 modified	Amber glass jar	250 mL	Cool 4°C	7 days extraction/ 40 days analysis[c]
Onsite radiation screening - plutonium-238 and thorium-232		Wide-mouth polyethylene bottle	650 grams	none	NA

NOTE: Holding times for CLP analyses are based on "Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses," EPA, February 1, 1988 and "Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses," July1, 1988.

[a]Sample containers will be certified cleaned by the manufacturer according to EPA standards.

[b]From date of collection.

[c]From date of extraction.

CLP - Contract Laboratory Program
SOW - Statement of Work
PCB - polychlorinated biphenyl

Table IV.4. Summary of Sampling and Analysis Program

Sample Matrix	Field Parameters	Laboratory Parameters	Investigative Samples No.	Investigative Samples Freq.	Investigative Samples Total	Duplicates No.	Duplicates Freq.	Duplicates Total	Equipment Blanks No.	Equipment Blanks Freq.	Equipment Blanks Total	MS/MSDs[b] No.	MS/MSDs[b] Freq.	MS/MSDs[b] Total	Matrix Total
Surface soil	Organic vapor														
	Soil classification														
		CLP TCL VOCs	55	1	55	14	1	14	–	–	–	3	1	3	69
		CLP TCL semivolatile organic compounds	71	1	71	14	1	14	–	–	–	4	1	4	85
		TCL pesticides/PCBs	71	1	71	14	1	14	–	–	–	4	1	4	85
		CLP TAL metals, cyanide	71	1	71	16	1	14	–	–	–	4	1	4	85
		Tritium	5	1	5	2	1	2	–	–	–	1	1	1	7
		EPH	14	1	14	3	1	3	–	–	–	1	1	1	17
		Plutonium-238, thorium-232 screen	71	1	71	14[c]	1	14	–	–	–	–	–	–	85
		Lithium	2	1	2	1	1	1	–	–	–	1	1	1	3
		USATHAMA explosives	26	1	26	6	1	6	–	–	–	2	1	2	32
Subsurface soil	Organic vapor														
	Soil classification														
		CLP TCL VOCs	344	1	344	40	1	40	–	–	–	18	1	18	384
		CLP TCL semivolatile organic compounds	212	1	212	25	1	25	–	–	–	11	1	11	237
		TCL pesticides/PCBs	212	1	212	25	1	25	–	–	–	11	1	11	237
		CLP TAL metals, cyanide	212	1	212	25	1	25	–	–	–	12	1	12	237
		Tritium	29	1	29	11	1	11	–	–	–	2	1	2	40
		EPH	54	1	54	7	1	7	–	–	–	3	1	3	61
		Plutonium-238, thorium-232 screen	344	1	344	40[c]	–	40	–	–	–	–	–	–	384
		USATHAMA explosives	99	1	99	12	1	12	–	–	–	5	1	5	111
		Nitrate-nitrite, chloride	21	1	21	3	1	3	–	–	–	2	1	2	24
		Lithium	16	1	16	2	1	2	–	–	–	1	1	1	18
No. 2 fuel oil		EPH	1	1	1	–	–	–	–	–	–	–	–	–	1

Field Quality Control Samples[a]

Table IV.4. (concluded)

Sample Matrix	Field Parameters	Laboratory Parameters	Investigative Samples No.	Investigative Samples Freq.	Investigative Samples Total	Duplicates No.	Duplicates Freq.	Duplicates Total	Equipment Blanks No.	Equipment Blanks Freq.	Equipment Blanks Total	MS/MSDs[b] No.	MS/MSDs[b] Freq.	MS/MSDs[b] Total	Matrix Total
Sediment	Organic Vapor														
		CLP TCL VOCs	5	1	5	2	1	2	–	–	–	1	1	1	9
		CLP TCL semivolatile organic compounds	5	1	5	2	1	2	–	–	–	1	1	1	7
		TCL pesticides/PCBs	5	1	5	2	1	2	–	–	–	1	1	1	7
		CLP TAL metals, cyanide	5	1	5	2	1	2	–	–	–	1	1	1	7
		Tritium	2	1	2	2	1	2	–	–	–	1	1	1	4
		EPH	4	1	4	1	1	1	–	–	–	1	1	1	5
		Acetonitrile, acrylonitrile	1	1	1	1	1	1	–	–	–	1	1	1	2
		USATHAMA explosives	1	1	1	1	1	1	–	–	–	1	1	1	2
		Nitrate-nitrite, chloride	1	1	1	1	1	1	–	–	–	1	1	1	2
		Plutonium-238, thorium-232 screen	5	1	5	2[c]	1	2	–	–	–	–	–	–	7
Surface water	pH, temperature, conductance, alkalinity														
		CLP TCL VOCs	3	1	3	2	1	2	2	1	2	1	1	1	7
		CLP TCL semivolatile organic compounds	3	1	3	2	1	2	2	1	2	1	1	1	7
		TCL pesticides/PCBs	3	1	3	2	1	2	2	1	2	1	1	1	7
		CLP TAL metals, cyanide	3	1	3	2	1	2	–	–	–	1	1	1	5
		Tritium	1	1	1	1	1	1	2	1	2	1	1	1	4
		EPH	3	1	3	2	1	2	2	1	2	1	1	1	7
		Acetonitrile, acrylonitrile	1	1	1	1	1	1	2	1	2	1	1	1	4
		Plutonium-238, Thorium-232 screen	3	1	3	2[c]	1	2	–	–	–	–	–	–	5
Drum sample	Organic vapor														
		CLP TCL semivolatile organic compounds	1	1	1	1	1	1	–	–	–	1	1	1	2
		TCL pesticides/PCBs	1	1	1	1	1	1	–	–	–	1	1	1	2
		CLP TAL metals, cyanide	1	1	1	1	1	1	–	–	–	1	1	1	2
		USATHAMA explosives	1	1	1	1	1	1	–	–	–	1	1	1	2

[a] In addition to the field quality control samples in this table, the following will be collected: trip blanks (1 per shipping container to lab with TCL VOCs), bottle lot blanks (1 every bottle lot), sample bank blanks (1 for every 20 field samples) and field (ambient) blanks (minimum 1 every 20 samples collected).

[b] Only a matrix spike is performed for TAL metals, cyanide, tritium, and lithium, 1 every 20 or fewer.

[c] Onsite laboratory radioactivity screening is performed on all samples taken at the site.

For water MS/MSD samples, where feasible, triple the normal sample volumes will be collected for VOCs, and double the normal sample volume will be collected for extractable organics, pesticides, and PCBs.

EPH – extractable petroleum hydrocar
MS/MSD – matrix spike/matrix spike duplicate pairs
PCB – polychlorinated biphenyl
TAL – Target Analyte List
TCL – Target Compound List
VOC – volative organic compounds

5. SAMPLE CUSTODY

Sample custody procedures for Operable Unit 3 require that the possession and handling of the sample from the moment of its collection through analysis be documented by written record. A sample is in someone's custody when one of the criteria listed below has been satisfied.

- The sample is in one's actual possession,
- The sample is in one's view after being in one's physical possession,
- The sample is in one's physical possession and is then locked up so that no one can tamper with it, or
- The sample is kept in a secured area that is restricted to authorized personnel only.

Samples will consist of material collected in the field, such as water, soil, or sediments, and any reagents added for the purposes of sample preservation. It should be noted that chain-of-custody requirements will be followed for samples that are screened onsite at the Mound Plant Soil Screening Facility.

5.1. CHAIN OF CUSTODY

5.1.1. Field Custody Procedures

5.1.1.1. Sample Labels

All samples will be identified with a label attached directly to the container. Examples of soil and water sample labels are presented in Mound Plant ER Program SOP 1.3, Sample Control and Documentation (revision 0) (DOE 1991). Sample label information will be completed using waterproof black ink. The labels will contain the following information

- sample number,
- time and date of collection,
- installation name,
- parameters to be analyzed,
- preservative (if any),

- sample source/location, and
- sampler's initials.

5.1.1.2. Chain-of-Custody Record and Seals

To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, a chain-of-custody record (Figure 5.1) will be filled out for each sample at each sampling location as it is collected by the field sampler. Each time the samples are transferred, the signatures of the persons relinquishing and receiving the samples, as well as the date and time of transfer, will be documented.

Signed and dated chain-of-custody seals will be attached to both the Operable Unit 3 sample containers and the shipping coolers at the junction between the lid and the jar or coolers by the person responsible for the packaging. These seals are used to detect any sample tampering that occurs during shipment. If all seals are not intact upon arrival at the laboratory, the laboratory project manager will notify the Operable Unit 3 field manager within 24 hours of receipt. The field manager will then complete a Non-Conformance Report (section 13).

5.1.1.3. Transfer of Custody and Shipment

Prior to shipment of samples or transfer to the onsite screening facility, the chain-of-custody record will be signed and dated by a member of the field team who has verified that those samples indicated on the record are indeed being shipped. A copy of the chain-of-custody record is shown in Figure 5.1. Mound Plant ER Program SOP 1.3, Sample Control and Documentation (revision 0) (DOE 1991) describes the completion of this form and the steps necessary for sample control, identification, and data recording (Appendix E). The completed chain-of-custody record will be sealed in a plastic bag and taped to the inside of the cooler lid so that it is immediately visible when the cooler is opened. After packaging has been completed; custody seals, signed and dated by a member of the field team, will be placed on the cooler.

All samples will be shipped by Federal Express to the IT analytical laboratory. Samples will be transported by field personnel the day of collection from Mound Plant to the courier location for subsequent shipment to the laboratory. (It should be noted that Federal Express does not claim responsibility for samples and does not sign off on the chain-of-custody. However the laboratory retains the shipping ticket, indicating acceptance and delivery of shipment.) Rental vehicles used by the field personnel may be used for transporting environmental samples from Mound Plant to Federal Express only if the samples are properly

Custody Transfer Record/Lab Work Request

WESTON

WESTON Analytics Use Only

Client _____
Work Order _____
Date Rec'd. _____ Date Due _____
RFW Contact _____
Client Contact/Phone _____

			Refrigerator#								
			#/Type Container								
			Containers/Volume								
			Preservative								
			ANALYSES REQUESTED ➡								
WA Use Only Lab ID	Client ID/Description	Matrix	Date Collected								

Matrix:
S - Soil
SE - Sediment
SO - Solid
W - Water
O - Oil
A - Air
DS - Drum Solids
DL - Drum Liquids
F - Fish
X - Other

Special Instructions:

Item/Reason	Relinquished by	Received by	Date	Time	Item/Reason	Relinquished by	Received by	Date	Time

WESTON Analytics Use Only

Samples Were:
1 Shipped or Hand-Delivered
NOTES:

2 Ambient or Chilled
NOTES:

3 Received Broken/Leaking (Improperly Sealed)
Y N
NOTES:

4 Properly Preserved
Y N
NOTES:

5 Received Within Holding Times
Y N
NOTES:

COC Tape Was:
1 Present on Outer Package Y N
2 Unbroken on Outer Package Y N
3 Present on Sample Y N
4 Unbroken on Sample Y N
NOTES:

COC Record Was:
1 Present Upon Receipt of Samples Y N

Discrepancies Between Sample Labels and COC Record? Y N
NOTES:

RFW 21-21-001/A-5/88

7-115

Figure 5.1. Chain-of-Custody record.

packaged and labeled. Upon receipt of the samples at the laboratory, the receiver will complete the transfer by dating and signing the chain-of-custody record (Figure 5.1). If additional transfers are required (i.e., a sample is sent from one IT laboratory to another), additional entries will be made on the chain-of-custody form.

5.1.2. Laboratory Custody Procedures

The following subsections describe laboratory custody procedures associated with sample receipt, storage, preparation, analysis, and general security of the Operable Unit 3 samples once they are received at the IT laboratory.

5.1.2.1. Sample Receipt

Upon receipt, the sample custodian will inspect sample containers for integrity. The presence of leaking or broken containers will be noted on the chain-of-custody record (see Figure 5.1). The IT laboratory Mound Plant project manager will notify the Operable Unit 3 field manager or sample bank manager within 24 hours of the receipt of leaking or broken containers.

The sample custodian will sign the chain-of-custody record (with date and time of receipt), thus assuming custody of the samples. The information on the chain-of-custody record will be compared with that on sample tags and labels to verify sample identity. Any inconsistencies will be resolved with the field sampling representative before sample analysis proceeds.

Samples will be moved to one of the locked sample storage refrigerators for storage prior to analysis. The storage location will be recorded on the chain-of-custody record. Samples designated for analysis of VOCs will be stored in a separate refrigerator.

The sample custodian will retain the original chain-of-custody record and will provide carbon copies to each laboratory section manager and one to the main sample log kept in the laboratory. An internal chain-of-custody is generated for tracking samples during storage and analysis and is kept with the samples/extracts at all times. The sample custodian will alert the appropriate section managers and analysts of any analyses requiring immediate attention because of short holding times.

5.1.2.2. Sample Storage

Samples will be maintained in storage in one of the locked storage refrigerators prior to sample preparation and analysis. The storage refrigerators are maintained at $4^{o} \pm 2^{o}C$. Samples designated for analysis of

volatile organic chemicals will be stored in a separate refrigerator. Analytical laboratory personnel will retrieve samples for analysis from the storage refrigerators and will sign the chain-of-custody record. The sample custodian and analyst will sign and date the chain-of-custody record to acknowledge transfer of custody to the analyst.

5.1.2.3. Sample Tracking - Organic Analysis

For samples that require extraction prior to analysis, a sample extraction form is completed during the time of extraction. When samples are extracted for analysis by gas chromatography (GC), gas chromatography/mass spectrometry (GC/MS), or liquid chromatography, all pertinent data are entered on the sample extraction form and are recorded in a bound laboratory notebook. Extraction data are entered into the laboratory information management system by the person performing the extraction. A hard copy of the form is printed out and is used as the vehicle for custody transfer to the analyst. Copies are provided to the analysts to inform them that extracts are ready for analysis. The bound laboratory notebook is kept in the extraction laboratory.

Extracts are maintained in secured, refrigerated storage by the sample preparation section until transferred to the analysts.

5.1.2.4. Sample Tracking - Metals Analysis

Samples are received by the sample preparation section for digestion prior to analysis for inorganics by atomic absorption (AA)/inductively coupled plasma (ICP) spectroscopy. Sample digests are stored at room temperature in a secured area before analysis and after analysis for a minimum of six weeks. When samples are prepared for digestion, the preparation technician fills out a sample digestion record.

All information regarding sample digestion is entered onto the sample digestion record as the sample preparation proceeds and is recorded in a bound laboratory notebook. The digestion record is maintained to acknowledge custody transfer of digestates to the inorganics analysis section. Upon completion of sample digestion, a carbon copy of the sample digestion record is provided to the inorganics analysis section to alert them that digestates are ready for analysis. The bound laboratory notebook containing the digestion record is retained by the inorganics digestion laboratory.

5.1.2.5. Recordkeeping

Information related to all sample tracking at the IT laboratories are recorded in laboratory log-in books and in the Laboratory Information Management System, which is a computerized database. The information recorded or entered includes

- project identification number;
- sample number;
- type of sample;
- date received;
- date put into storage after analysis is completed; and
- date of disposal.

Laboratory notebook pages are signed and dated daily by laboratory analysts. Corrections to notebook entries are made by drawing a single line through the erroneous entry and by writing the correct entry next to the one crossed out. All corrections are initialed and dated by the analyst. More details on laboratory records including sample tracking are presented in Section 12 of the corporate IT Analytical Services quality assurance manual and Section 12 of laboratory specific attachments.

5.2. DOCUMENTATION

5.2.1. Field Logs

All data collection activities performed during the Operable Unit 3 investigation will be documented, using waterproof, nonerasable black ink, either in a field notebook or on ER Program forms. Field notebooks will be bound books and will be assigned to individual field personnel for the duration of their stay in the field. Entries will be as detailed and descriptive as possible so that a particular situation can be recalled without reliance on the collector's memory. All field log entries will be dated and signed by the person making the entries. Information that will be entered in each logbook on a one-time basis includes the following:

- name;
- company and address;
- phone number;
- activity or location; and

- phone numbers for supervisors, emergency response, etc.

Field logbook daily entries will include the following items:

- date and time;
- name of initials of individual making the entry;
- description of test, experiment, or measurement;
- conditions that may adversely affect the work, such as weather;
- names of other people present;
- samples taken, received, or released;
- procedure (SOP) used or followed;
- any data not recorded on data sheets or by automatic methods;
- deviations from procedures, plans, or protocols;
- data and reason for any delays; and
- site visitors.

It will be the responsibility of all field personnel to photocopy all field logs (including notebook pages and ER Program forms) generated during a given field day, at the end of that day. Copies will be given to the site manager or field supervisor who will review the logs for accuracy, completeness, and consistency, and maintain field log files. At the completion of a work shift, copies of all field logs, notebook pages, and ER Program forms will be returned to the subcontractor's office. These copies will be presented to the ER Program data administrator and entered into the project file. At the completion of a field program, field logbooks will be returned to the project files.

5.2.2. Data Collection Forms

As an added means of assuring the collection of accurate field and sampling information, standardized data collection forms will be used. These forms will be used to record data in a consistent format that limits individual interpretations or preferences. By explicitly outlining reporting methods, identifying appropriate units of measure, and specifying alternative test procedures, these forms provide a measure of quality control and quality assurance in the data collection process.

The standard data collection forms associated with the Mound Plant ER Program SOPs group data and information according to problem-solving needs. They are a means of preventing the collection of invalid

or redundant data and eliminating critical data gaps. Each data collection form precisely defines what data are necessary to accurately characterize a particular property or relationship. This reduces the likelihood of initiating field sampling or laboratory analyses, only to discover that key pieces of information have not been collected and that further sampling is required.

Each Mound Plant ER Program SOP for a data collection activity provides an example of all forms required for the accurate recording of the procedure. A blank form will be used for each new location or sample, as specified by the SOP. During the field activity, each form will be completed as accurately and completely as possible, as indicated by the example contained in the SOP. All entries on the data collection form will be made using indelible black ink, with incorrect entries crossed out with a single line and initialed. Each form must be signed or initialed and dated by the person completing the form. Any additional information not recorded on the form will be recorded in the field notebook. After the field activity is completed, all data collection forms will be reviewed by a technical reviewer other than the person recording the data. The review will ensure completeness and accuracy in both form completion, as well as data integrity. The reviewer will then sign and date each form.

5.2.3. Corrections to Documentation

All measurements made, and samples collected, will be recorded as described above in subsections 5.2.2 and 5.2.3. If an incorrect entry is made, regardless of the type of data document, the incorrect data will be crossed out with a single strike mark, the correct information entered, and the correction initialed and dated by the person making the correction. There will be no erasures or deletions from any type of data document record.

5.3. SAMPLE HANDLING, PACKAGING, AND SHIPPING

All samples collected and shipped for analysis as part of the Operable Unit 3 Limited Field Investigation will be considered environmental samples unless data or information becomes available that indicates the samples are hazardous or radioactive. Mound Plant ER Program SOP 1.5, Guide to the Handling, Packaging, and Shipping of Samples (revision 1) (DOE 1991), provides information and references that must be reviewed prior to selection of appropriate packaging materials, shipping containers, and shipping labels. Mound Plant ER Program SOP 1.5 and the current U.S. Department of Transportation regulations will be the guidelines for shipment of all samples collected during the investigation.

5.4. FINAL EVIDENCE FILE DOCUMENTATION

Records will be kept by the ER Program TSO subcontractor to document the quality assurance/quality control activities and to provide support for possible evidential proceedings. An outline of project file requirements is given below.

- Communications
  - Internal
  - External
- Quality assurance/quality control
  - Procedures
  - Chain of custody
  - Audit reports
  - Laboratory quality control reports
  - Deviation notification forms
  - Nonconformance/corrective action reports
- Technical information
  - Analytical data
  - Field data
  - Field logbooks
  - Graphic resources
  - Data quality acceptance
  - Calculations/evaluations
  - Data review reports
  - Regulatory compliance
- Management
  - Schedule
  - Budget
  - Release site database
- Health and safety
  - Plans/procedures
  - Audit reports
- Documents
  - Plans
  - Reports

All evidence file documentation will be maintained by the DOE or its subcontractor under the ER Program document control system. Upon termination of the project, all records (e.g., chromatograms, spectra, and calibration records) will be archived indefinitely by the DOE. If at any time the DOE chooses to purge its

files, the EPA will be advised and offered possession. The ER Program TSO subcontractor project manager will ensure that the quality assurance/quality control records are properly stored and retrievable.

6. ANALYTICAL PROCEDURES

6.1. FIELD MEASUREMENTS AND SCREENING

As part of the remedial investigation at Mound Plant, several parameters will be measured in the field for surface water and sump liquid samples. All aqueous samples will be measured for specific conductance, temperature, pH, and alkalinity. Field screening will include combustible gas, organic vapors, low-energy gamma radiation, and alpha surface contamination. The Mound Plant ER Program SOPs for field determination of these parameters are presented in Appendix E of this QAPP. The following subsections summarized the procedures for measurement of field parameters.

6.1.1. Specific Conductance

The specific conductance of all aqueous samples from surface water and groundwater sampling locations will be taken with a YSI Model 33 meter, or equivalent. The procedures and required solutions for measuring specific conductance are described in detail in Mound Plant ER Program SOP 2.2, Field Measurements on Ground and Surface Water Samples (revision 1) (DOE 1991).

6.1.2. pH

The pH of all aqueous samples form surface water and groundwater sampling locations will be measured using a Fisher Model No. 107 portable water pH meter, or equivalent. The procedures and solutions required for measuring the pH of aqueous samples are described in detail in Mound Plant ER Program SOP 2.2, Field Measurements on Ground and Surface Water Samples (revision 1) (DOE 1991).

6.1.3. Alkalinity

A digital titration kit (Hach Kit, or equivalent) will be used to measure alkalinity of the aqueous samples collected from all surface water and groundwater sampling locations. The procedures for performing alkalinity measurements are described in detail in Mound Plant ER Program SOP 2.2, Field Measurements on Ground and Surface Water Samples (revision 1) (DOE 1991).

6.1.4. Temperature

Temperature of groundwater samples will be measured with a Celsius thermometer according to the procedures in Mound Plant ER Program SOP 2.2, Field Measurements on Ground and Surface Water Samples (revision 1) (DOE 1991).

6.1.5. Combustible Gas

Combustible gas levels will be measured using a combustible gas indicator calibrated with a National Bureau of Standards traceable combustible gas standard. The procedures for measuring combustible gas levels are described in Mound Plant ER Program SOP 6.1, Health and Safety Monitoring of Combustible Gas Levels (revision 0) (DOE 1991).

6.1.6. Organic Vapor

Organic vapor levels will be measured according to Mound Plant ER Program SOP 6.2 Health and Safety Monitoring of Organic Vapors with a Photoionization Detector (revision 0) (DOE 1991), with a portable photoionization detector (PID) calibrated with a specific gas standard such as benzene. The PID measures total vapors for a broad range of compounds that it can detect.

6.1.7. Radionuclide Screening

Soil and sediment samples will be screened for low-energy gamma radiation, primarily from the presence of thorium and plutonium isotopes, using a field instrument for the detection of low-energy radiation (FIDLER) probe. Mound Plant ER Program SOP 6.7, Near Surface and Soil Sample Screening for Low-Energy Gamma Radiation using the FIDLER (revision 0) (DOE 1991), presents the procedures for screening with a FIDLER.

The surfaces of personnel and field personnel and field equipment will be monitored for total alpha surface contamination before entering and exiting the site. Mound Plant ER Program SOP 6.4, Total Alpha Surface Contamination Measurements (revision 0) (DOE 1991), describes the procedures for this screening using an alpha scintillation probe.

Screening of the Operable Unit 3 soil samples for plutonium-238 and thorium-232 will be performed at the Mound Plant Soil Screening Facility using a FIDLER calibrated to detect these isotopes. The procedure for this screening is given in Appendix F of this QAPP.

6.2. LABORATORY ANALYTICAL METHODS

Soil/sediment, surface water, and sump liquid samples will be analyzed for Target Compound List (TCL) for VOCs compounds, semivolatile organic compounds, pesticides/PCBs, and Target Analyte List (TAL) inorganic parameters. Analysis of these parameters will be performed according to the EPA CLP Statement of Work for Organic Analysis (EPA 1988a) and for Inorganic Analysis (EPA 1988b). Pesticides/PCBs will be analyzed according to EPA Method 8080 (EPA 1986). The specific compounds contract-required quantitation/detection limits are presented on Tables VI.1, VI.2, VI.3, and VI.4.

Non-CLP laboratory analyses that will be performed for soil/sediments include lithium, chloride, nitrate, acetonitrile and acrylonitrile, extractable petroleum hydrocarbons (EPH), and U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) explosives. Sump liquid and surface water samples will be analyzed for tritium, acetonitrile and acrylonitrile, and/or extractable petroleum hydrocarbons. If insufficient water is available for all the planned analyses, the samples will be analyzed for parameters in the following priority: TCL VOCs, TCL semivolatile organics and pesticides/PCBs, acetonitrile and acrylonitrile, extractable petroleum hydrocarbons, TCL inorganics, and tritium. EPA-approved procedures will be used for all non-CLP analytes except USATHAMA explosives. ITs certification performance data package for soils by High Performance Liquid Chromatography (HPLC) is provided as Appendix G in this QAPP. The procedures that will be used by the IT Laboratory to analyze samples for these analytes are discussed below. IT Laboratory Standard Operating Procedures (SOPs) for non-CLP analyses have been submitted to U.S. EPA Region V Quality Assurance Section.

- Lithium in soil samples will be analyzed by EPA method 6010 and the sample digested by EPA method 3050, according to procedures described in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," third edition (referred to as SW846, EPA, 1986) (or most recent version). This method uses ICP spectroscopy as does the CLP SOW for several TAL analytes. The quality control procedures and data reporting requirements defined in the CLP SOW will be implemented for this analyte.

- SW 846 Method 9250 will be used to analyze soil samples for chloride (EPA 1986). Method 9250 analyzes chloride using a colorimetric automated ferricyanide procedure. Because this method is for aqueous samples, the soil/sediment samples will first be dissolved in water.

Table VI.1. Target Compound List (TCL) Volatile Organics and Contract Laboratory Program Contract-Required Quantitation Limits (CRQL) for Volatile Organic Analytes[a,b]

TCL Volatile Organic Compounds	Quantitation Limits for Water (µg/L)	Quantitation Limits for Low Soil/Sediment (µg/kg)[c,d,e]
1. Chloromethane	10	10
2. Bromomethane	10	10
3. Vinyl chloride	10	10
4. Chloroethane	10	10
5. Methylene chloride	5	5
6. Acetone	10	10
7. Carbon disulfide	5	5
8. 1,1-dichloroethene	5	5
9. 1,1-dichloroethane	5	5
10. 1,2-dichloroethene (total)	5	5
11. Chloroform	5	5
12. 1,2-dichloroethane	5	5
13. 2-butanone	10	10
14. 1,1,1-trichloroethane	5	5
15. Carbon tetrachloride	5	5
16. Vinyl acetate	10	10
17. Bromodichloromethane	5	5
18. 1,2-dichloropropane	5	5
19. *cis*-1, 3-dichloropropene	5	5
20. Trichloroethene	5	5
21. Dibromochloromethane	5	5
22. 1,1,2-trichloroethane	5	5
23. Benzene	5	5
24. *trans*-1, 3-dichloropropene	5	5
25. Tribromomethane (bromoform)	5	5
26. 4-methyl-2-pentanone	10	10
27. 2-hexanone	10	10
28. Tetrachloroethene	5	5
29. Toluene	5	5
30. 1,1,2,2-tetrachloroethane	5	5
31. Chlorobenzene	5	5
32. Ethylbenzene	5	5
33. Styrene	5	5
34. Xylenes (total)	5	5

Table VI.1. (continued)

[a]Volatile organic compounds will be analyzed according to the "EPA Contract Laboratory Program Statement for Work for Organics Analysis, Multi-Media, Multi Concentration," SOW No. 2/88, including revisions 9/88 and 4/89, or most recent version.

[b]These are the EPA quantitation limits for routine analytical services under the CLP. Specific quantitation limits are highly matrix-dependent. The quantitation limits listed here are provided for guidance and may not always be achievable (EPA 1988a).

[c]Quantitation limits achieved by GC/MS.

[d]Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher (EPA 1988a).

[e]Medium soil/sediment CLP contract-required quantitation limits for volatile TCL compounds are 125 times the individual low soil/sediment contract-required quantitation limits (EPA 1988a).

CLP - Contract Laboratory Program
GC/MS - gas chromatography/mass spectrometry
TCL - Target Compound List
VOC - volatile organic compounds

Table VI.2. Target Compound List (TCL) Semivolatiles and Contract Laboratory Program Contract-Required Quantitation Limits (CRQL) for Semivolatile Organic Analytes[a,b]

Semivolatile Organic Chemicals	Quantitation Limits for Water (µg/L)	Quantitation Limits for Low Soil/Sediment (µg/kg)[c,d,e]
1. Phenol	10	330
2. bis(2-chloroethyl) ether	10	330
3. 2-chlorophenol	10	330
4. 1,3-dichlorobenzene	10	330
5. 1,4-dichlorobenzene	10	330
6. Benzyl alcohol	10	330
7. 1,2-dichlorobenzene	10	330
8. 2-methylphenol	10	330
9. bis(2-chloroisopropyl) ether	10	330
10. 4-methylphenol	10	330
11. N-nitroso-di-n-dipropylamine	10	330
12. Hexachloroethane	10	330
13. Nitrobenzene	10	330
14. Isophorone	10	330
15. 2-nitrophenol	10	330
16. 2,4-dimethylphenol	10	330
17. Benzoic acid	50	1600
18. bis(2-chloroethoxy) methane	10	330
19. 2,4-dichlorophenol	10	330
20. 1,2,4-trichlorobenzene	10	330
21. Naphthalene	10	330
22. 4-chloroaniline	10	330
23. Hexachlorobutadiene	10	330
24. 4-chloro-3-methylphenol (para-chloro-meta-cresol)	10	330
25. 2-methylnaphthalene	10	330
26. Hexachlorocyclopentadiene	10	330
27. 2,4,6-trichlorophenol	10	330
28. 2,4,5-trichlorophenol	50	1600
29. 2-chloronaphthalene	10	330
30. 2-nitroaniline	50	1600
31. Dimethylphthalate	10	330
32. Acenaphthylene	10	330
33. 2,6-dinitrotoluene	10	330
34. 3-nitroaniline	50	1600
35. Acenaphthene	10	330

Table VI.2. (continued)

Semivolatile Organic Chemicals	Quantitation Limits for Water (µg/L)	Quantitation Limits for Low Soil/Sediment (µg/kg)[c,d,e]
36. 2,4-dinitrophenol	50	1600
37. 4-nitrophenol	50	1600
38. Dibenzofuran	10	330
39. 2,4-dinitrotoluene	10	330
40. Diethylphthalate	10	330
41. 4-chlorophenyl-phenyl ether	10	330
42. Fluorene	10	330
43. 4-nitroaniline	50	1600
44. 4,6-dinitro-2-methylphenol	50	1600
45. N-nitrosodiphenylamine	10	330
46. 4-bromophenyl-phenylether	10	330
47. Hexachlorobenzene	10	330
48. Pentachlorophenol	50	1600
49. Phenanthrene	10	330
50. Anthracene	10	330
51. Di-n-butylphthalate	10	330
52. Fluoranthene	10	330
53. Pyrene	10	330
54. Butylbenzylphthalate	10	330
55. 3,3'-dichlorobenzidine	20	660
56. Benzo(a)anthracene	10	330
57. Chrysene	10	330
58. bis(2-ethylhexyl)phthalate	10	330
59. Di-n-octylphthalate	10	330
60. Benzo(b)fluoranthene	10	330
61. Benzo(k)fluoranthene	10	330
62. Benzo(a)pyrene	10	330
63. Indeno(1,2,3-cd)pyrene	10	330
64. Dibenz(a,h)anthracene	10	330
65. Benzo(g,h,i)perylene	10	330

[a]EPA. 1988a.

Table VI.2. (continued)

[a]Semivolatile organic compounds will be analyzed according to "EPA Contract Laboratory Program Statement of Work for Organics Analysis, Multi-Media, Multi Concentration," SOW No. 2/88, including revisions 9/88 and 4/88, or most recent version.

[b]These are the EPA quantitation limits for routine analytical services under the CLP. Specific quantitation limits are highly matrix-dependent. The quantitation limits listed here are provided for guidance and may not be achievable (EPA 1988a).

[c]Quantitation limits achieved by GC/MS.

[d]Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher (EPA 1988a).

[e]Medium soil/sediment CLP contract-required quantitation limits for semivolatile Target Compound List compounds are 60 times the individual low soil/sediment contract-required quantitation limits (EPA 1988a).

CLP - Contract Laboratory Program
EPA - U.S. Environmental Protection Agency
GC/MS - gas chromatography/mass spectrometry

Table VI.3. Target Compound List (TCL) Pesticides/PCBs and Quantitation Limits (CRQL) for Pesticides/PCBs[a,b]

Pesticides/PCBs	Quantitation Limits for Water (µg/L)	Quantitation Limits for Low Soil/Sediment (µg/kg)[c,d,e]
1. Alpha-BHC	0.03	2.0
2. Beta-BHC	0.06	4.0
3. Delta-BHC	0.09	6.0
4. Gamma-BHC (Lindane)	0.04	2.7
5. Heptachlor	0.03	2.0
6. Aldrin	0.04	2.7
7. Heptachlor epoxide	0.05	8.0
8. Endosulfan I	0.14	9.4
9. Dieldrin	0.02	1.3
10. 4,4'-DDE	0.04	2.7
11. Endrin	0.06	4.0
12. Endosulfan II	0.04	2.7
13. 4,4'-DDD	0.11	7.4
14. Endosulfan sulfate	0.10	16.0
15. 4,4'-DDT	0.12	8.0
16. Methoxychlor	0.5	80.0
17. Endrin ketone	0.10	16.0
18. Endrin aldehyde	0.23	15.4
19. Alpha-chlordane	0.14	9.4
20. Gamma-chlordane	0.05	9.4
21. Toxaphene	2.4	161
22. Aroclor-1016	1.0	80.0
23. Aroclor-1221	2.0	80.0
24. Aroclor-1232	1.0	80.0
25. Aroclor-1242	1.0	80.0
26. Aroclor-1248	1.0	80.0
27. Aroclor-1254	1.0	160
28. Aroclor-1260	1.0	160

[a]Pesticide and PCB analysis will be performed according to "Test Methods for Evaluating Solid Waste Physical/Chemical Methods," SW846/EPA November 1986 or most current version.

[b]Specific quantitation limits are highly matrix-dependent. The quantitation limits listed here are provided for guidance and may not be achievable (EPA 1986).

Table VI.3. (continued)

[c]Quantitation limits achieved by GC.

[d]Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher (EPA 1988a).

Table VI.4. Target Analyte List (TAL) of Inorganics and Contract Laboratory Program Contract-Required Detection Limits (CRDL) for Inorganic Analytes[a]

Analyte	Contract-Required Detection Limit[b,c] (Water) (µg/L)	Contract-Required Detection Limit[b,c] (Soil) (mg/kg)
Aluminum	200	40
Antimony	60	12
Arsenic	10	2
Barium	200	40
Beryllium	5	1
Cadmium	5	1
Calcium	5000	1000
Chromium	10	2
Cobalt	50	10
Copper	25	5
Iron	100	20
Lead	3	0.6
Magnesium	5000	1000
Manganese	15	3
Mercury	0.2	0.1
Nickel	40	8
Potassium	5000	1000
Selenium	5	1
Silver	10	2
Sodium	5000	1000
Thallium	10	2
Vanadium	50	10
Zinc	20	4
Cyanide	10	2

[a]Inorganic analytes will be analyzed according to "EPA Contract Laboratory Program Statement of Work for Inorganics Analysis, Multi-Media, Multi Concentration," SOW No. 7/88, including revisions 2/89 and 6/89, or most recent version.

[b]Contract-required detection limits are subject to the restrictions specified in the first page of Part G, Section IV, Exhibit D (Alternate methods - Catastrophic Failure) of the EPA CLP Statement of Work (EPA 1988b). Any analytical methods specified in the EPA CLP Statement of Work, Exhibit D, may be used as long as the documented instrument or method detection limits meet the contract-required detection limits. Higher detection limits may only be used if the sample concentration exceeds five times the detection limit of the instrument or method in use, then the value may be reported even though the instrument or method detection limit may not equal the contract-required detection limit. This is illustrated in the example below (EPA 1988b):

For lead:

Method in use - ICP
Instrument Detection Limit (IDL) = 40
Sample Concentration = 220
Nominal Detection Limit (CRDL) = 3

Table VI.4. (continued)

The value of 220 may be reported even though the instrument detection limit is greater than the contract-required detection limit. The instrument or method detection limit must be documented as described in Exhibit E of the EPA CLP Statement of Work (EPA 1988b).

[c]The contract-required detection limits are the instrument detection limits obtained in pure water that must be met using the procedure in Exhibit E of the EPA CLP Statement of Work. The detection limits for samples may be considerably higher, depending on the sample matrix (EPA 1988b).

CLP - Contract Laboratory Program
ICP - inductively coupled plasma
IDL - instrument detection limit
CRDL - contract-required detection limit

- Water samples will be analyzed for tritium using the EPA-approved drinking water method, numbered 906.0, for tritium (EPA 1980a).

- EPA Method 353.2 (EPA 1983) will be used to analyze soil and sediment samples for nitrate-nitrite. Because this method is for aqueous samples, the soil/sediment samples will be dissolved in water.

- SW846 Method 8240 will be used to analyze sediment and sump liquid samples for acetonitrile and acrylonitrile from the glass melter room.

- SW 846 modified Method 8015 will be used to analyze soil/sediment samples for volatile EPH. These results will be quantified for total high boiling petroleum hydrocarbon as motor oil, #2 fuel oil, jet fuel (JP-4), or diesel fuel, as specified. Water samples will also be analyzed for EPH reported as jet fuel (JP-4) according to EPA Methods 3510 (preparation) and 8015 (analysis). (Note: Total low-boiling petroleum hydrocarbons are reported as gasoline and will not be required for this investigation.)

- IT Laboratory's USATHAMA Method LW06 (solid samples) will be used to analyze soil/sediment samples for ten explosives (Appendix G). This IT method was developed according to the USATHAMA quality assurance program (Appendix G) and uses HPLC to identify the ten explosives in soil. The certification performance data package for this method is provided in Appendix G.

Table VI.5 lists the non-CLP analytes, the analytical method reference, and the analyte quantitation limits. The limit of detection for each analyte is best expressed as a quantitation limit that is defined as the "lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions" (EPA 1986). This limit may vary significantly depending upon the sample matrix, and, as a final reported limit, will vary with the weight or volume of the sample used, percent moisture (where applicable), and the dilution factor, if any. The limits listed on Table VI.5 are the minimum achievable quantitation limits for IT Laboratories in Tennessee with analysis of clean sample matrices of a given sample volume or weight.

Table VI.5. Non-CLP Analytes, Quantitation Limits, and Analytical Methods

Analyte	Quantitation Limit[a] (Water)	Quantitation Limit[a] (Soil or Sediment)
	(µg/L)	(µg/kg)
Acetonitrile	100	NA
Acrylonitrile	100	NA
Explosives[a]		
HMX	NA	2.9
RDX	NA	2.2
1,3,5-TNB	NA	1.3
1,3-DNB	NA	1.2
NB	NA	1.3
Tetryl	NA	2.2
TNT	NA	1.3
2A,4,6-DNT	NA	1.3
2,6-DNT	NA	1.3
2,4-DNT	NA	0.3
Extractable Petroleum Hydrocarbons[b]	(mg/L)	(mg/kg)
EPH reported as jet fuel (JP-4)	50	5
EPH reported as diesel fuel	NA	5
EPH reported as motor or #2 fuel oil	NA	20
Lithium[c]	NA	10.0
Radionuclides	(pCi/L)	(pCi/g)
Plutonium-238[d]	NA	25 ± 25 (3SD)
Thorium-232[d]	NA	2 ± 1 (3SD)
Tritium[e]	500[f]	NA
		(mg/kg)
Nitrate[g]	NA	1[h]
Chloride[i]	NA	5[h]

[a]U.S. Army Toxic and Hazardous Materials Agency Method LW-06, "Nitroexplosives in Soil by HPLC," IT Laboratories, Knoxville, TN.
[b]EPHs are analyzed by SW846 modified method 8015 (EPA 1986).
[c]Analyzed by U.S. Environmental Protection Agency (EPA) Method 6010 and the sample prepared by 3050 (EPA 1986).

[d]Plutonium-238 and thorium-232 analyses will be performed at the Mound Plant Soil Screening Facility. It should be noted that values less than the detection levels given above will be reported, but should be considered estimates.
[e]Analyzed by EPA Method 906.0 in "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/4-80-032, latest version.
[f]Based on a 100mL sample size
[g]Analyzed by EPA Method 353.2 in "Methods for Chemical Analysis of Water and Wastes," U.S. EPA, EPA-600/4-79-020, revised March 1983.
[h]Based on a 10 gram soil sample and 100mL volume of extractant and a soil moisture content of 0-10% (rounded). Actual quantitation limit will vary with the sample and extractant amounts and will depend upon the nature of the soil matrix.
[i]Analyzed by EPA Method 9250 in SW846 (EPA 1986).

EPH - extractable petroleum hydrocarbons
HMX - octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine cyclotetramethylenetetranitramine
RDX - hexahydro-1,3,5-trinitro-s-triazine cyclotetratmethylenetetranitramine
NB - nitrobenzene
1,3-DNB - 1,3-dinitrobenzene
1,3,5-TNB - 1,3,5-trinitrobenzene
2,4-DNT - 2,4-dinitrotoluene
2,6-DNT - 2,6-dinitrotoluene
TNT - 2,4,6-trinitrotoluene
2A,4,6-DNT - 2-amino-4,6-dinitrotoluene
Tetryl - A-methyl-N,2,4,6-tetranitroanaline
NA - Not Applicable.

7. CALIBRATION PROCEDURES AND FREQUENCY

Before any instrument is used as a measuring device, the instrument's response to known reference materials (traceable to an appropriate agency standard such as NIST, NBS, or ASTM) must be determined. The manner in which various instruments are calibrated is dependent upon the particular type of instrument and its intended use. All sample measurements are made within the calibrated range of the instrument. For laboratory analyses, appropriate sample dilution is performed if the instrument response is greater than the upper end of the calibration range.

7.1. FIELD EQUIPMENT

Applicable field instruments to be used during the investigation will be calibrated according to the specifications set forth in the respective Mound Plant ER Program SOPs (Appendix E). Instruments will be calibrated at least once per day during field use. Table III.1 in section 3 summarizes the calibration procedures, frequency of calibration and acceptance criteria necessary for the calibration to be valid for applicable field measurements and field screening.

Records for each field instrument used as part of this program will be maintained to ensure its capability of providing accurate and precise measurements. Records will be maintained on instrument maintenance and calibration. Such records will be reviewed prior to their use in the field. Tracking of instrument records will be accomplished by assigning a unique number to each instrument that will correspond to its records file.

The field measurement and field screening instruments that may be used in the field during the environmental investigation are presented in the following subsections.

7.1.1. Photoionization Detector (PID)

The HNu™ PID is a general survey instrument capable of detecting and measuring many organic and inorganic vapors in air. The instrument can be calibrated to a particular compound; however, it cannot distinguish between detectable compounds in a mixture of gases. The HNu has a sensitivity of approximately 0.1 parts per million (ppm) of benzene. However, the instrument's sensitivity depends upon the compounds present and environmental factors such as moisture and temperature. Mound Plant ER Program SOP 6.2, Health and Safety Monitoring of Organic Vapors with a Photoionization Detector (revision 0) (DOE 1991), provides details on the calibration frequency and procedures for the HNu PID.

7.1.2. Explosimeter/Combustible Gas Indicator

Explosimeters, or combustible gas indicators (CGIs), are used to determine the potential for the combustion or explosion of unknown atmospheres. A typical CGI determines the level of organic vapors and other gases present in an atmosphere as a percentage of the lower explosive limit or lower flammability limit by measuring the change in electrical resistance in a Wheatstone bridge circuit. The CGI can reliably detect common combustible vapors such as methane, butane, and pentane. Some CGI models also contain an oxygen detector. The oxygen detector is useful for determining the existence of atmospheres deficient in oxygen. Mound Plant ER Program SOP 6.1, Health and Safety Monitoring of Combustible Gas Levels (revision 0) (DOE 1991), describes the details of the calibration frequency and procedures of CGIs.

7.1.3. Specific Conductance Meter

The YSI Model 33 specific conductance meter, or equivalent, is a portable, battery-operated, transistorized instrument used to measure salinity, specific conductance, and temperature in surface water, groundwater, and waste water systems. Instrument calibration frequency and procedures for salinity, specific conductance, and temperature measurement instruments are described in detail in Mound Plant ER Program SOP 2.2, Field Measurements on Ground and Surface Water Samples (revision 1) (DOE 1991).

7.1.4. pH Meter

The Fisher Model No. 107 pH meter, or equivalent, is a portable pH monitoring instrument for determining pH in surface water, groundwater, and waste systems, and for other water quality applications. The calibration frequency and procedures are described in detail in Mound Plant ER Program SOP 2.2, Field Measurements on Ground and Surface Water Samples (revision 1) (DOE 1991).

7.1.5. Field Alkalinity Testing

Field alkalinity testing will be performed on all groundwater samples. A digital titration kit (Hach Kit, or equivalent) will be used to measure alkalinity of the groundwater samples. The calibration frequency and procedures are described in detail in Mound Plant ER Program SOP 2.2, Field Measurements on Ground and Surface Water Samples (revision 1) (DOE 1991).

7.1.6. Zinc Sulfide Alpha Scintillometer

A portable zinc sulfide alpha scintillometer, coupled with a ratemeter/scaler, will be used to screen soil samples that are collected in areas where alpha-emitting radionuclides may exist. This screening must be performed in order to establish proper shipping/packaging procedures for the samples and to establish the average background levels for safe handling of the samples by laboratory personnel. This instrumentation can reliably measure activity levels above 350 disintegrations per minute (dpm) per 100 centimeters squared (cm^2). In addition, this detector may be used for personnel monitoring. This instrumentation can reliably measure activity levels above 350 dpm/100 cm^2, or about 70 counts per minute (cpm)/100 cm^2. Mound Plant ER Program SOP 6.4, Total Alpha Surface Contamination Measurements (revision 0) (DOE 1991), describes in detail the calibration frequency and procedures for the zinc sulfide alpha scintillometer.

7.1.7. Field Instrument for the Detection of Low-Energy Radiation

A FIDLER will be used to monitor surfaces and soil samples for the presence of low-energy gamma radiations that accompany some alpha emissions, such as the 17-kiloelectron volt (keV) gamma-ray emitted when plutonium-238 low-energy decays to uranium-234. The FIDLER, used in combination with the zinc sulfide alpha probe, can provide a determination of the presence or absence of radionuclides. The FIDLER reliably measures gamma levels above 100 picocuries per gram (pCi/g) for plutonium-238 in soil. The calibration frequency and procedures for the FIDLER are described in detail in Mound Plant ER Program SOP 6.7, Near Surface and Soil Sample Screening for Low-Energy Gamma Radiation Using the FIDLER (revision 0) (DOE 1991).

7.2. LABORATORY EQUIPMENT

Laboratory instrument calibrations typically consist of two types, initial calibration and continuing calibration. Initial calibration procedures establish the calibration range of the instrument and determine instrument response over that range. Typically, three to five analyte concentrations are used to establish instrument response over a concentration range. The instrument response over that range is commonly expressed as a correlation coefficient (e.g., UV-visible/infrared spectrophotometry) or by a response factor, amount/response (e.g., for GC, GC/MS, or HPLC).

Continuing calibration usually includes measurement of one or more calibration standards. The response is compared to the initial measured instrument response. Continuing calibration is performed at least once per operating shift for laboratory analyses.

Instrument calibration procedures for CLP analyses will be performed according to the CLP SOW for inorganic and organic analyses (most recent versions). For non-CLP analyses, calibration procedures will be performed as described in the approved analytical method. Calibration procedures for all laboratory analyses, along with frequency and acceptance criteria, are summarized in Table III.2 in section 3. The following subsections discuss the calibration procedures for each type of instrumentation.

7.2.1. Gas Chromatographic (GC) Analyses

GC will be used for analysis of EPHs (EPA 1986) and pesticides/PCBs (EPA 1988a). Initial calibration is performed when chromatographic conditions are changed (e.g., change in flow rate, detectors, new column) or when the calibration check does not meet the required acceptance criteria. A minimum of five standards of different concentrations are analyzed to determine the linearity of the GC. Response factors for each compound are calculated from the results, and a calibration curve is generated. Linearity criteria for extractable petroleum hydrocarbons are valid if there is less than or equal to 20% relative standard deviation among the calibration factors and 10% relative standard deviations for pesticides/PCBs. A quadratic curve may also be used.

For pesticide/PCB analysis, the retention-time windows are be determined for the target compounds and surrogate compound. Three injections are made over a 72-hour period and the retention-time shift for each compound is evaluated. The retention-time window is established by applying plus or minus three times the standard deviation of the three retention times for a given compound to the retention-time of the first analysis.

The initial calibration for these analysis is checked once every 10 samples analyzed, or once per day, which every is more frequent. A midrange standard is run and must be within 15% difference of the initial response factor. Recalibration is performed to further sample analysis if this criteria is not met.

7.2.2. Gas Chromatography/Mass Spectrometry (GC/MS) Analyses

GC/MS will be used for analysis of volatile and semivolatile organic compounds according to the CLP SOW (EPA 1988a). Modifications to the calibration for acetonitrile and acrylonitrile are specified in the IT Laboratory SOP. Mass spectral abundance criteria must be met prior to sample analysis. Decafluorotriphenylphosphine for semivolatile organics and bromofluorobenzene for volatile organics are used to tune the GC/MS system and must meet specific ion abundance criteria established in the CLP SOW. Meeting these criteria is demonstrated daily or every 12-hour time period, whichever is more frequent. The tuning is also verified whenever a corrective action to the GC/MS system is taken that affects the tuning (e.g., ion source cleaning or repair).

Initial calibration of the GC/MS system is accomplished with a minimum of five concentrations of target compounds (three concentrations for air analyses). Only a four-point calibration is required by the CLP SOW for nine of the target semivolatile compounds that have a lower response and for VOCs that saturate at the highest concentration (200 micrograms per liter [μg/L]). Relative response factors (RRFs) are calculated for the target compounds and relative standard deviations for CLP-identified calibration check compounds (CCCs). Relative standard deviations for CCCs must be less than or equal to 30%. Initial calibration is not valid if this criterion is not met. CLP-identified system performance check compounds (SPCCs) must have the minimum average RRFs specified in the CLP SOW for the initial calibration curve to be valid. The relative retention times of each compound in each standard run must agree within 0.06 units.

The initial calibration is verified every 12-hour period with a calibration standard containing all target semivolatile and volatile compounds and surrogate compounds. RRFs are compared to the average RRF from the initial calibration. The minimum RRF for the SPCCs must be met. The percent difference for the RRFs of the CCCs must be less than or equal to 25 % for the initial calibration to be valid. Prior to sample analysis, the GC/MS system is evaluated and corrective action taken if these criteria are not met.

7.2.3. Inductively Coupled Plasma (ICP) and Atomic Absorption(AA) for Metals and Spectrophotometry for Cyanide

TAL inorganics will be analyzed according to the procedures presented in the CLP SOW for inorganic analyses (EPA 1988b). Lithium will be calibrated for according to the IT Laboratory SOP. ICP, atomic

absorption (AA) instruments, and the spectrophotometer are calibrated daily, or once every 24 hours, and each time the instrument is set up. The AA instrument is calibrated with a blank and at least three concentrations of standards prepared each time for analysis. Minimum linearity for AA analysis is a correlation coefficient of 0.995. The ICP must be calibrated with at least two standards, with one being a blank. The minimum correlation coefficient for cyanide calibration is 0.996.

An initial calibration verification (ICV) is performed to assess the accuracy of the initial calibration using a standard of a certified concentration from an external source. When the measurement exceeds the CLP-established control limits, the problem is corrected, the instrument is recalibrated, and the ICV is run again. The initial calibration is verified by analysis of a continuing calibration verification (CCV) standard every two hours during an analysis run, or at a frequency of 10%, whichever is more frequent. This standard is also analyzed for at the beginning and end of each sample analysis run. The concentration and source of the CCV and acceptance criteria are specified in the CLP SOW. For ICP and AA analysis, linearity is required near the contract-required detection limit (CRDL). An ICP standard (CRI) at two times the CRDL or two times the instrument detection limit (IDL), whichever is greater, is analyzed for at the beginning and end of each sample analysis run, or twice per 8-hour working shift, whichever is more frequent, but not before the ICV. An AA standard (CRA) at the CRDL or IDL, whichever is greater, is analyzed for at the beginning of each sample analysis run, but not before the ICV.

7.2.4. Liquid Scintillation

Liquid scintillation is used to measure beta particle activity from tritium. A source check is performed daily to verify calibration and efficiency. The background level is also checked daily.

7.2.5. High-Performance Liquid Chromatography (HPLC)

HPLC is used for analysis of explosives (IT Analytical Services USATHAMA Method LW-06). Initial calibration is performed with a minimum of five concentrations of standards whenever there is a change in chromatographic conditions or when the check standard is outside acceptance criteria. The resulting calibration curve must have an average response factor with a relative standard deviation less than or equal to 15%.

The initial calibration is checked prior to sample analysis and once every 10 samples analyzed with a midrange standard for each analyte. The response of the check standard must be within 25% of the predicted response in order for the initial calibration to be valid. If the calibration check is outside this criteria, a new calibration curve will be performed. The retention times and peak heights of the check

standard for every 10 samples are compared to those of the check standard run at the beginning of the day. If significant deviation or visible chromatographic abnormalities are observed, then all samples analyzed after the last acceptable standard check will be reanalyzed.

7.2.6. Colorimetry

Nitrate-nitrite (method E353.2) and chloride (method SW9250) will be analyzed for by spectrophotometric methods, which use a colorimeter to identify the analyte when the analyte is complexed with, or creates the formation of, a light-absorbing compound. Calibration of the colorimeter is accomplished with a minimum of three concentrations of standards and is performed when instrument conditions are changed or when the calibration standard exceeds acceptance criteria. The calibration curve is plotted, or a minimum correlation coefficient of 0.995 is required, for acceptable linearity of the resulting calibration curve.

The initial calibration is verified with the analysis of a midrange calibration standard prior to sample analysis and for every 10 samples analyzed. The standard result must be less than or equal to a 15% difference from the response of the initial calibration. If this acceptance criterion is exceeded, then the instrument is recalibrated and the affected samples will be reanalyzed.

7.2.7. Alpha Spectrometry

Alpha spectrometry, using a 17-keV plutonium x-ray with a 1/16-inch thick, 5-inch diameter NaI detector and lead x-rays at 88 keV, will be used to measure plutonium-238 and thorium-232. The procedures for calibration are included in the Mound Plant SOP presented in Appendix E of this QAPP.

8. INTERNAL QUALITY CONTROL CHECKS

Internal quality control checks are performed as part of a field investigation in order to assess and control the quality of the data generated. Quality control checks are used to evaluate the accuracy and precision of field screening, field measurements, sampling technique, and laboratory analyses. Acceptance criteria for the quality control checks, and corrective actions to be taken if criteria are not met, have been established for this program so that data of known quality are obtained (Table III.2). The following subsections summarize those internal quality control checks.

8.1. SCREENING AND FIELD MEASUREMENTS

Quality control procedures for screening and field measurements are limited to checking the reproducibility of the measurement by obtaining multiple readings and by calibrating the instruments (when appropriate) with either internal references or external standards. The frequency of these checks and acceptance criteria are presented on Table III.1.

8.2. FIELD SAMPLING

Field conditions and sampling techniques for surface water and sump liquid samples can be assessed by the collection of trip blanks, sample bank blanks, equipment (rinsate) blanks, ambient blanks, and duplicate samples for selected laboratory analyses. Blank samples will not be labeled as such so that laboratory bias is minimized. The laboratory will be kept from using these samples for internal QC by indicating on the chain of custody which samples are to be used for internal QC. Trip, sample bank, ambient, and equipment blanks monitor environmental conditions or sampling technique during sampling in order to detect potential sample contamination. Trip blanks monitor for volatile organic contamination during sample transport and storage. Trip blanks are prepared in the laboratory by filling vials with organic-free deionized water (ASTM Type II quality), sealing the vials with no air bubbles and transporting them to the site. These blanks remain unopened while transported and stored with the collected samples (one set per sample cooler). Sample bank blanks monitor for VOCs potentially present in the surrounding environment where samples are commonly handled before shipment. Sample bank blanks are prepared, after a given batch of samples are collected, with organic-free deionized water (ASTM Type II quality) in sample vials, capped with no air bubbles, and placed in the desired location during handling. Ambient blanks will be used to monitor for VOCs potentially present in the environment surrounding a given area to be sampled. This blank is prepared prior to sampling in the area of interest with a vial filled with organic-free, deionized water (ASTM Type II quality) that is left uncapped in the area during sampling. When

sampling is completed in the area, the vial is capped, leaving no air bubbles. Ambient blanks will be used to monitor for VOCs potentially present in the environment surrounding a given area to be sampled. This blank is prepared prior to sampling in the area of interest with a vial filled with organic-free, deionized water (ASTM Type II quality) that is left uncapped in the area during sampling. When sampling is completed in the area, the vial is capped, leaving no air bubbles. Equipment blanks are used to evaluate the decontamination technique of water sampling equipment that is not dedicated to a given sampling location. Equipment (rinsate) blanks are prepared by filling sample bottles with organic-free, deionized water (ASTM Type II quality) that has been routed through a decontaminated sampling device, including the filtering apparatus (inorganics samples only).

Two water samples will be collected for this investigation; therefore, one duplicate, trip blank, ambient blank, and sample bank blank will be prepared. A trip blank is collected for each set of vials for volatile organics shipped in a given sample cooler. If dedicated samplers are not used for the water sample collection, then an equipment blank will be collected after the first sample collected. Equipment blanks are analyzed for volatile and semivolatile organic compounds, pesticides, TAL inorganics, and EPHs.

Ambient blanks and duplicates will be collected as field quality control checks for soil and sediment samples. Ambient blanks will be prepared for every 20 soil or sediment samples collected at selected locations. A duplicate sample is collected for every 10 soil samples collected in the field. Collection of duplicate samples can measure the precision of the sampling technique. A relative percent difference is calculated for the duplicate sample analysis as defined in section 3 and is used to evaluate sampling precision. As a result of the sample analysis, duplicate sample results also include the precision of the analytical technique. Monitoring analytical precision is discussed in the following section, Laboratory Analyses. Acceptance criteria for precision of duplicate field samples has not been established. A high variation in soil sample results for the primary and duplicate sample is common due to the nonhomogeneous nature of soils; therefore, duplicate soil sample results will be assessed accordingly. Sample duplicates will not be labeled as such to prevent bias in the laboratory analysis.

Bottle lot blanks will be collected for every new bottle lot number and are prepared by filling the sample bottle for a new lot of bottles with organic-free deionized water (ASTM Type II quality). Analyses to be performed on these blanks are those designed for the bottle type. When U.S. EPA Region V finalizes procedures for the preparation and analysis of bottle lot blank samples, an addendum to the QAPP will be submitted to U.S. EPA for review and approval.

8.3. LABORATORY ANALYSES

Internal quality control checks for routine analytical services from the CLP are specified in the EPA CLP Statement of Work or the most current SOW for organics (EPA 1988a), and EPA CLP Statement of Work or the most current SOW for inorganics (EPA 1988b). These quality control checks are summarized in Table III.2 with frequency and acceptance criteria. Corrective actions to be taken, if any acceptance criteria are not met, are specified in the EPA CLP SOWs for organic and inorganic analyses.

Internal quality control checks for non-CLP analyses are specified in the analytical method and in Table III.2. Additional quality control checks for some analyses will be performed and are also summarized in Table III.2. Frequency of the checks, acceptance criteria, and corrective actions (as presented in Table III.2.) are based on guidance in the analytical method and established laboratory control limits.

A definition of the internal quality checks for laboratory analysis are presented below:

- Method Blank. The method blank is an artificial sample designed to monitor the introduction of artifacts into the analysis that could result from the sample preparation or analytical method. For aqueous sample analysis, reagent water is generally used as the matrix. For solid sample analysis, a purified solid matrix is used. The method blank is carried through the entire analytical scheme (extraction, concentration, and analysis). Method blanks will be performed for all applicable analyses at the frequency stated for the analytical method in Table III.2. For CLP TAL inorganics analyses, the method blank is referred to as the preparation blank.

- Background Check. A background check is performed for liquid scintillation analysis at a given count time and frequency to determine if the instrumentation is contaminated above a maximum acceptable level. For tritium analysis, the background check is performed once per day prior to sample analysis. The maximum acceptance level for system contamination is 3 standard deviations. If this limit has been exceeded, the problem is identified and corrected prior to sample analysis.

- Source Check. A source check is a check of the counting efficiency of the detector, using a known amount of radionuclide. The check determines the detector resolution. The check is performed once per day for tritium analysis by liquid scintillation.

- Method Spike. A method spike is a method blank sample carried throughout the same process as the samples to be analyzed, with a known amount of standard added. The spike result of the sample provides information on how the method is performing. This quality control check is performed for radiochemical analyses such as tritium.

- Matrix Spike. Predetermined quantities of specific analytes are added to a sample matrix prior to sample extraction or digestion. Percent recoveries are calculated for each analyte to assess the analytical accuracy. Matrix spikes monitor the effects of the sample matrix on the analytical results. One matrix spike for every 20 samples collected will be performed for all applicable analyses (volatile organics, semivolatile organics, pesticides/PCBs, explosives, TAL inorganics, chloride, nitrate-nitrate, and radionuclides). The field sample to spike will be selected by field personnel and will not include a field blank sample (trip blank, equipment blank, ambient blank, or sample bank blank). This will ensure that a Mound Plant sample matrix with possible analyte detections will be spiked to obtain representative results of analytical accuracy.

- Matrix Spike Duplicate. A duplicate matrix spike of the same sample collected in the field will also be performed for every 20 samples collected. The matrix spike duplicate will assess the analytical and sampling precision by calculating a relative

percent difference (as defined in section 3) between the primary and duplicate spike recoveries. This sample will be a duplicate sample of the selected matrix spike.

- Surrogate Spike. Surrogate compounds are organic compounds similar to the analytes of interest in chemical composition and extraction and chromatographic properties, but are not normally found in environmental samples. These compounds are spiked into all field and laboratory samples (blanks, standards, and matrix spikes) for volatile organic, semivolatile organic, and pesticide/PCB analyses. Percent recoveries are calculated for each surrogate compound in each sample. These recoveries give an indication of the performance of the analytical method.

- Replicate Sample. A replicate sample is analyzed for given analyses by taking aliquots from a sample container to assess the precision of the analytical method. An RPD is calculated for the primary and replicate sample results. Replicate samples will be analyzed for radionuclides, TAL inorganics, and total organic carbon. Field personnel will select the CLP TAL inorganics sample to be analyzed as a replicate.

- Tuning Criteria. GC/MS analyses require that the mass spectrometer be tuned prior to calibration and sample analysis. This is accomplished with analysis of a compound that has similar properties but is not commonly found in the environment. For tunings and mass calibration, BFB and DFTPP are used for semivolatile organic and volatile organic GC/MS analyses, respectively. Specific ion abundance criteria must be met, as defined in the CLP SOW, before sample analysis begins.

- Calibration Check. The initial instrument calibration is verified at regular intervals to account for potential instrument drift or other changes in instrument conditions. A standard with a concentration within the calibration range is analyzed for usually after 10 sample analyses or with a frequency defined in the CLP SOW. The standard result is compared to the initial calibration, and a percent difference or RPD is calculated. If the result is not within the established acceptance criteria, then the analytical system is evaluated and recalibrated before continuing sample analysis. For CLP metal analyses, samples analyzed since the last acceptable standard must be reanalyzed.

- Retention-Time Window. Retention times of target analytes for GC, GC/MS, and HPLC analyses must be monitored for shifts during sample analysis. The allowed shift of retention time for a given analyte is the retention-time window. Retention-time windows are established according to the analytical method. Acceptance criteria are expressed as an established range (e.g., ±0.06 units) or for pesticide analysis as plus or minus three times the standard deviation of three retention times of the same compound. Shifts that occur outside the acceptance criteria indicate a change in the chromatographic system or an instrument problem and could lead to misidentifications unless corrective action is taken.

- Internal Standard. Internal standards are performed for volatile and semivolatile GC/MS analyses and are used to ensure that the system sensitivity and response are stable for every run. Internal standards consist of compounds that are similar in analytical behavior to the analytes, and they are added to the calibration standards. Response factors of these standards are used to quantitate sample results. Criteria for internal standard responses and retention times are defined in the CLP SOW.

- System Performance Check Compounds (SPCCs). SPCCs are specific compounds used to monitor the RRFs of the continuing calibration check compared to the initial calibration for GC/MS analysis of volatile and semivolatile organic compounds. A

minimum RRF for each of the SPCCs, as defined in the CLP SOW, must be achieved in order for the initial calibration to be valid.

- Calibration Check Compounds (CCCs). CCCs are specific compounds used to monitor the RRFs of the continuing calibration check compared to the initial calibration for GC/MS analysis of volatile and semivolatile organic compounds. The relative standard deviation of the RRF for each compound must be less than or equal to 30% in order for the initial calibration to be valid, as defined in the CLP SOW.

- Initial and Continuing Calibration Blanks (ICB, CCB). A blank consisting of reagent water is analyzed immediately after every initial and continuing calibration verification for CLP TAL inorganics analyses, at a frequency of 10% or every 2 hours, whichever is more frequent.

- Laboratory Control Sample (LCS). An LCS is a standard solution of a certified concentration prepared by a source external to the laboratory performing the analysis and is used to measure the analytical accuracy. This quality control check is performed for TAL inorganics, volatile organics, chloride, and nitrate-nitrite analyses for every batch of analytical samples. The CLP SOW requires that the recovery of the LCS analysis for TAL inorganics be within 80% to 120%. Manufacturer's specifications for acceptance criteria of the LCS are used for the other analyses. An LCS may also be prepared by the EPA Region V Central Regional Laboratory for submittal (blind) to IT Analytical Services for analysis.

- Initial Calibration Verification (ICV). After the ICP, AA, and cyanide systems are calibrated, the accuracy of the initial calibration is verified with the analysis of a calibration verification standard. The CLP SOW has established control limits for each system (ICP and AA: 90% to 110% of the true value; AA--cold vapor for mercury: 80% to 120% of true value; and cyanide: 85% to 115% of true value). If control limits are exceeded, then the problem is identified and corrected, and the instrument is recalibrated.

- Continuing Calibration Verification (CCV). The initial calibration of ICP, AA, and cyanide systems is verified during each set of analyses at a frequency of 10% or every 2 hours, whichever is more frequent. The solution, as required by the CLP SOW, is either an EPA solution, a National Bureau of Standards SRM1643a solution, or a contractor-prepared standard. The control limits for this analysis are the same as for the ICV.

- Linear Range Check Standard (CRI). For ICP analysis, a standard (or CRI) at two times the CRDL or two times the instrument detection limit is analyzed at a frequency defined in the CLP SOW to verify the linearity near the CRDL. For AA analysis, the standard (or CRA) is analyzed at a concentration at the CRDL or IDL.

- Interference Check Sample (ICS). An ICS is analyzed for the ICP analysis at a frequency defined in the CLP SOW to verify interelement and background correction factors. The ICS consists of one solution containing interferents, and a second containing analytes mixed with the interferents. The second solution must fall within ±20% of the true value. Corrective action, as defined in the CLP SOW, is taken if this criteria is not met.

- ICP Serial Dilution (L). A serial dilution is a series of different dilutions of a given sample and a comparison of sample results. An ICP serial dilution is performed on

one sample from each group of samples of a similar matrix and concentration or for each sample delivery group, whichever is more frequent, as defined in the CLP SOW. Results of the analysis must agree within ±10%. The analysis of serial dilutions gives an indication of potential chemical or physical interferences.

- Method of Standard Addition (MSA). The method of standard addition (MSA) is a method for quantitating furnace AA sample results to eliminate the effects of the sample matrix. A sample whose matrix is interfering with quantitation is spiked at three concentrations, and the results are used as a calibration curve to quantitate the sample results. The conditions under which standard addition is performed and how the sample results are qualified are defined in the CLP SOW.

- Linear Range Analysis (LRA). The linear range for ICP analyses is checked with a standard quarterly. The standard is analyzed with a routine analytical run. The results must be within ±5% of the true value. The concentration of the standard is the upper limit of the ICP linear range.

- Interelement Corrections. ICP interelement correction factors are determined prior to the start of CLP contract analyses and at least annually. The interelement corrections are for spectral interference due to aluminum, calcium, iron, and magnesium, and are checked for all wavelengths used for each analyte.

9. DATA REDUCTION, VALIDATION, AND REPORTING

9.1. FIELD AND TECHNICAL DATA

The field and technical (non-laboratory) data that will be collected during the RI effort at Mound Plant can generally be characterized as either objective or subjective data. Objective data include all direct measurements such as field screening/analytical parameters and water level measurements. Subjective data include descriptions and observations. Test boring and well logs include both types of data, in that the data recorded in the field are descriptive but can be reduced using the standardized lithologic coding system.

9.1.1. Field and Technical Data Reduction

For field measurement data which require calculations to obtain final concentrations/values (e.g., alkalinity), equations used and the calculations performed will be recorded in the appropriate field log. The equations used for measurement data are provided in the respective Mound Plant ER Program SOPs in Appendix E. All calculations will be checked at least once by the field team member performing the field measurement.

Occasionally, a field measurement will result in an outlier with a value significantly outside the expected range for most field conditions. Attempts will be made by the field team to identify outliers during the field measurement. All health and safety screening measurements will be considered. When identified, the outlier will be recorded as would any other field measurement, and at least two additional measurements will be made and recorded to verify or invalidate the suspected outlier. When appropriate, field instrumentation and calibration will be checked and the parameter remeasured when an outlier is discovered. If after this check, the value remains the same, it is considered a valid measurement.

9.1.2. Field and Technical Data Validation

Review of objective field and technical data integrity will be performed at two different levels. On the first level, field personnel at the time of collection will check to ensure that standard operating procedures are followed, all data are recorded, and quality control checks are performed. At the second level, data will be reviewed by a technical peer not involved with the data collection, who will check to ensure that all data are recorded and reported correctly, including calculations and sample collection information. Ten percent of all calculations will be checked. Any inconsistencies

or anomalies discovered will be resolved immediately, if possible, by seeking clarification from the field personnel responsible for collecting the data.

Subjective field and technical data will also be reviewed for reasonableness and completeness by the installation manager. In addition, random checks of sampling and field conditions will be made by the field supervisor, who will check recorded data at that time to confirm the recorded observations. Whenever possible, peer review also will be incorporated into the data review process, particularly for subjective data, to maximize consistency among field personnel. For example, during drilling activities, the field manager will schedule periodic reviews of archived lithologic samples to ensure that the appropriate descriptions and codes are being consistently applied by all field personnel.

After the validity of data in the field notes and on ER Program forms has been evaluated, according to the procedures described above, the data administrator will tabulate the data, wherever possible, by entering the data in computer data files. Where appropriate, the data files will be set up for direct input into the project database. Subjective data will be filed as hard copies for later review by the installation manager and for incorporation into technical reports, as appropriate.

9.1.3. Field and Technical Data Reporting

All field data will be recorded by field personnel in bound field notebooks and on ER Program forms (for specific measurements). For example, during drilling activities, the field team member supervising a rig will keep a chronological log of drilling activities, a vertical descriptive log of lithologies encountered, other pertinent drilling information (staining, odors, field screening, working conditions, water levels, geotechnical data), and a labor and materials accounting in the team member's bound notebook. It will be the responsibility of all field personnel to photocopy all field logs (including notebook pages and ER Program forms) generated during a given field day. Copies will be given to the site manager or field supervisor, who will maintain field log files. At the completion of a work shift, copies of all field logs, notebook pages, and ER Program forms will be returned to the TSO subcontractor's office. These copies will be presented to the ER Program data administrator and entered into the project file. All records of numerical analyses performed on field and technical data will be legible, reproduction-quality, and complete enough to permit logical reconstruction by a qualified individual other than the originator. All field records will be kept on file at the TSO subcontractor's office for a minimum of 10 years. EPA Region V and the TSO will be notified prior to any intent to dispose of project files.

Field notebook entries will include, on the inside cover and first pages, the following information:

- name,
- company name and address,
- phone number,
- activity or location, and
- table of contents.

Daily entries will include the following information:

- date and time;
- name of individual making the entry;
- description of test/activity;
- quantities of materials used;
- drawings and information related to the activity;
- conditions that might adversely affect the test/activity;
- names of witnesses or observers present;
- samples collected, received, or released;
- the procedures used or followed;
- data which is not recorded on the ER Program forms;
- calculations performed;
- deviations from procedures used;
- date and reason for downtime or delays; and
- visitors.

All entries will be made using a regular or ballpoint pen with indelible black ink. Corrections to entries will be made by crossing out the error with one line, dating and initialing the error, and entering the correction above or beside the error. Each page entered on in the notebook will be signed and dated by the individual. Once an entry has been signed and dated, changes, deletions, or additions are made only as a new entry and refer back to the original entry rather than crossing it out. A new page in the field notebook

is started when the previous page is full or when the previous page has been marked, dated, and signed so that no entries can be made. Pages are not removed from the bound notebook.

9.2. LABORATORY DATA

9.2.1. Laboratory Data Reduction

All raw analytical data are recorded in numbered and bound laboratory notebooks issued by the quality assurance officer. Data are recorded and are associated with the unique laboratory sample identification number and the field sample identifier. For inorganic analyses, the bound notebook pages are preformatted for each analytical method. Those pages contain the following:

- analytical method,
- analyst,
- date,
- reagent concentrations,
- instrument settings (as applicable), and
- raw data.

The laboratory analysts sign and date all notebook entries daily. The notebook pages are reviewed periodically by the section manager before final data assembly. Copies of strip-chart outputs (for example, chromatograms) are maintained on file.

At the completion of a set of analyses, all calculations are completed and are checked by the analyst. Calculations using raw data to obtain final concentrations are performed according to the procedures described in the specified analytical method. The associated quality control data (laboratory duplicates, surrogates, matrix spikes, and matrix spike duplicates [MSDs]) are entered onto quality control charts and are verified as being within control limits. If all data are acceptable, the data are entered into the laboratory computer system, and the data summaries (including raw data) are submitted to the section manager for review. After approval, data are subsequently entered into the project database format. Data outside control limits will be reported with a qualifier on the CLP reporting forms for CLP data packages. Case narratives will note any out-of-control situations for non-CLP analyses. After the laboratory section manager approves, the laboratory data manager is notified that the data are ready to be reported, and the completed analyses are removed from the laboratory backlog. A hard-copy data summary is then

generated that is reviewed and is signed by the laboratory section manager and/or the laboratory manager.

Laboratory data is stored separately for each project by IT Analytical Services. Included in the file are calibration records, raw analytical data, processing of data, data validation, quality control sample results, data reports, and project-specific requirements. These records are kept for a minimum of 10 years. The TSO subcontractor and EPA Region V will be notified of intent to dispose of Mound Plant data.

9.2.2. Laboratory Data Validation

Prior to the final production of analytical reports, laboratory data are reviewed and verified according to IT Analytical Services procedures described in the IT Analytical Services Quality Assurance Manual in Section 10.2, Data Validation (Appendix A). The IT Analytical Services data review procedures are based on two activities:

- processing quality control sample results; and
- data validation of reporting analytical results.

Precision and accuracy of the analyses are evaluated with the quality control sample results. Control charts prepared from statistical evaluation of quality control sample results (e.g., matrix spikes and MSDs) are used to compare results to established control limits.

Data validation consists of review of data processing and data reporting. Twenty percent of all data processed will be checked using one of the two procedures described in subsection 10.2.1 of the IT Analytical Services Quality Assurance Manual (Appendix A). Data reports are reviewed against the processed data by the analyst or group leader. The operations manager then reviews the data report to ensure that it meets project requirements. The laboratory manager gives final approval for issuance of the laboratory report.

In addition to the data review performed by the appropriate laboratory, an organization external to the one that generated the data will validate the analytical data. Analytical data generated from samples collected at Mound Plant will be reviewed and validated by an ER Program TSO subcontractor.

Data Generated Under CLP

Validation by the ER Program TSO subcontractor will be performed on CLP organic and inorganic analyses in a step-by-step approach. These data will first be evaluated against the requirements of this QAPP. The CLP-generated data (TCL organic compounds and TAL inorganic parameters) will then be evaluated according to EPA CLP validation procedures ("Laboratory Data Validation - Functional Guidelines for Evaluation of Inorganic Analyses" and "Laboratory Data Validation - Functional Guidelines for Evaluation of Organic Analyses," prepared by the EPA Data Review Work Group, latest revisions).

Non-CLP Data

The Mound Plant ER Program will use the criteria described in this section to evaluate the acceptability of non-CLP data. Non-CLP analyses include radiological constituents, lithium, acetonitrile and acrylonitrile, extractable petroleum hydrocarbons, organochlorine pesticides/PCBs common anions, and explosives.

The evaluation of these analytical results will consist of, at a minimum, the following components:

- Were the appropriate SOPs followed during sample collection?
- Were the samples containerized and handled as described in the FSP and SOPs?
- Were the appropriate number and type of field quality control samples collected?
- Are the data packages complete (subsection 9.2.3)?
- Did the field and laboratory quality control checks meet the established acceptance criteria (section 3)?

9.2.3. Laboratory Data Reporting

Laboratory data reporting will be provided on electronic media (as ASCII files of a specified format) and in hard-copy data reports. All laboratory data report packages for each type of analysis will contain a case narrative that summarizes the following information on the given set of samples analyzed:

- date of issue;
- the laboratory analysis performed;

- any deviations from the stated analytical method;
- the laboratory batch number;
- the number of samples and the sample matrices;
- a reference to the quality control procedures performed for the specific methods used, including the reference to the acceptance criteria used.;
- the contents of the laboratory report;
- the project name and number;
- the state of the samples received (e.g., whether preserved and packaged properly);
- whether sample holding times were met, and identification of those for which they were not;
- any observations that may have had an impact on the analyses;
- any technical problems affecting the analysis and corrective actions taken;
- laboratory quality control checks that did not meet the project criteria (as specified in the QAPP) and/or laboratory criteria (include any corrective actions taken and any known possible reasons for the results); and
- the laboratory manager's signature approving the issuance of the data package.

A copy of the chain-of-custody with all relinquished signatures will accompany each data package.

The following summarizes the contents of hard copy laboratory data report packages for each group of analyses.

- For those analyses performed under the CLP SOW for inorganic (TAL) and organic (TCL) compounds, data report packages will follow the requirements set forth in the CLP SOW. These include TCL volatile organic and semivolatile organic compounds and TAL inorganics (including lithium) for sump liquid and surface water and soil and sediment samples.
- Laboratory data packages for explosive analyses will, at a minimum, consist of the following:
    - a case narrative for each laboratory batch of samples analyzed;
    - a summary page stating the extraction and analysis dates for each field sample and reported laboratory quality control checks;
    - a cross reference of laboratory sample identification numbers to the project sample identification numbers;
    - a description of all data qualifiers used in the laboratory report;

- a record of sample extraction of all samples and laboratory quality control checks;
- sample results for the 10 explosives;
- results of matrix spikes, matrix spike duplicates, method blank, initial calibration, and continuing calibration checks; and
- labeled and dated chromatograms of sample results, matrix spikes, MSDs, the method blank, and continuing calibration checks.

- Reports covering analysis of common anions (chloride and nitrate-nitrite) will contain, at a minimum, the following where applicable:
  - a case narrative for each laboratory batch of samples analyzed;
  - a summary page stating the analysis dates for field samples and reported quality control samples;
  - a cross reference of laboratory sample identification numbers to the project sample identification numbers;
  - a description of all data qualifiers used in the laboratory report;
  - a record of sample preparation for all field samples and laboratory quality control samples;
  - sample results;
  - results of initial calibration and calibration checks (including date); and
  - results of matrix spikes, MSDs, laboratory replicates, laboratory control samples, and the method blank.

- Data packages for radiochemical analyses will consist of the following elements:
  - a case narrative for each laboratory batch of samples analyzed;
  - a cross reference of laboratory sample identification numbers to the project sample identification numbers;
  - a summary page stating the extraction and analysis dates for each field sample and reported laboratory quality control check;
  - a description of data qualifiers used in the laboratory report;
  - a record of sample preparation for all field samples and laboratory quality control;
  - instrument calibration results (date, time, technician);
  - results for standards, including instrument blanks and calibration standards;

- labeled and dated raw data for all sample results and laboratory quality control checks, including counting time and number of disintegrations per sample; and

- calculated activity, per unit mass or liquid volume, with the following associated statistics:

    - relative counting error at the 95% confidence level,

    - lower detection limit, and

    - normalized deviation for method spikes and matrix spikes.

- Data report packages for EPH and pesticides/PCBs will consist, at a minimum, of the following items where applicable:

    - a case narrative for each laboratory batch of samples analyzed;

    - a cross reference of laboratory sample identification numbers to the project sample identification numbers;

    - a description of data qualifiers used in the laboratory report;

    - a record of sample extraction/preparation for all field samples and laboratory quality control checks;

    - a summary page stating the extraction and analysis dates for each field sample and reported laboratory quality control check;

    - sample results;

    - results of matrix spikes, MSDs, method blank, initial calibration, continuing calibration checks, and replicate samples;

    - labeled and dated chromatograms/spectra of sample results and the laboratory quality control checks listed above; and

10. PERFORMANCE AND SYSTEM AUDITS

Audits may consist of two types, system audits and performance audits. The purpose of a system audit is to determine whether appropriate project systems such as equipment, instruments, procedures, qualified personnel, etc., are in place. Performance audits are used to indicate whether those systems are functioning properly and are capable of meeting project requirements. Internal audits will be conducted by the TSO subcontractor's project QAM or a designated auditor to verify the existence of a quality control system and evaluate the level of compliance with that system for quality control measures, standards, records, and project documentation and control. At least one internal audit will be conducted during the Operable Unit 3 Limited Investigation. External audits will be performed by EPA Region V, the Region V Central Regional Laboratory, and/or the Region V Central District Office.

The auditor must have a working knowledge of Mound Plant and of the associated operable unit, which includes a detailed understanding of the QAPP, the associated SOPs, and the Work Plan. The auditor is also expected to have an understanding of technical issues related to the field and laboratory sampling program. Finally, the QAM or the designated auditor must exhibit experience with quality assurance audits.

10.1. TECHNICAL PERFORMANCE AUDITS

Technical performance audits will be conducted by the QAM or assigned qualified personnel of the TSO subcontractor on an ongoing basis during the project, as field data are generated, reduced, and analyzed. These audits will be scheduled and executed as part of work on each operable unit. All numerical analyses, including manual calculations, mapping, and computer modeling, will be documented and will be the subject of performance audits in the form of quality control review, numerical analysis, and peer review. Technical peer review is the responsibility of the Mound Plant ER Program TSO subcontractor installation manager and will be performed for project data reductions, analyses, and reports.

10.2. FIELD PERFORMANCE AUDITS

The TSO subcontractor's QAM or a designated auditor will perform at least one internal field performance audit during each phase (i.e., drilling, soil sampling, water sampling, etc.) of the investigation. Field sampling and associated activities will be audited at least once. The purpose of field performance audits is to ensure that the methods and protocols detailed in this QAPP are being consistently adhered to in the field.

Field performance audits will be performed using an audit checklist of the format presented in Figure 10.1. The field performance audit will include a review of the following activities:

SAMPLE AUDIT CHECKLIST

FACILITY	AUDITOR(S)	DAT E

ACTIVITY/REQUIREMENT	AUDIT QUESTIONS	ANSWER			AUDITOR COMMENTS
		YES	NO	N/A	

MOUND/SamAudCheck 1/15/91

Figure 10.1. Sample audit checklist form.

- air, soil/sediment, groundwater/surface water, and biological tissue sampling procedures;
- field measurements and field screening procedures;
- field logbook documentation;
- sample labeling, handling, and custody procedures;
- completion of ER Program field forms;
- the number and type of quality control samples collected; and
- drilling procedures.

These activities will be reviewed for their adherence to the procedures established in the FSP, Mound Plant ER Program SOPs, and the QAPP. Audit questions will be developed by the QAM. These checklists are used to ensure completeness of the review and to document the results of the audit.

As part of the field audit, field operation records will be reviewed to verify that field-related activities were performed in accordance with appropriate project procedures. Items reviewed will include, but are not limited to, field equipment calibration records, daily field logs, and chain-of-custody documentation. Upon audit completion, an audit report containing observations and any findings and associated corrective actions will be submitted to the ER Program TSO installation coordinator, the ER Program subcontractor project manager, and the installation manager. These reports will be delivered to U.S. EPA upon their request.

EPA Region V, the Region V Central Regional Laboratory, and/or the Region V Central District Office are responsible for performing external audits of field activities.

10.3. PROJECT SYSTEM AUDITS

The DOE or its subcontractor director of quality assurance may periodically, on an unannounced basis, call for a corporate project audit (system audit). The installation manager must respond by submitting the Operable Unit 3 QAPP, and the auditor will then determine whether the QAPP is in place. The auditor will also determine whether the audits called for in the QAPP have been, and are being, conducted. Certain tasks are identified by the director of quality assurance for a more formal audit on a scheduled basis. Those audits evaluate, in depth, the implementation of the QAPP in the project as it applies to field and laboratory data analysis and reduction procedures. The division operations manager, in the role of division quality assurance officer, may ask the director of quality assurance to perform an audit or may personally conduct the audit.

10.4. LABORATORY AUDITS

10.4.1. Laboratory System Audits

The EPA Region V Central Regional Laboratory will perform external system audits of IT laboratories as necessary for this program. This audit may include an onsite visit of the laboratories. An audit of the IT Analytical and Mixed Waste Laboratories may also be performed by the ER Program TSO subcontractor QAM prior to the implementation of the remedial investigation. The system audit will consist of review of the laboratory quality assurance manual; the instrumentation and/or analytical system developed for the analyses of interest; sample preparation methodologies; laboratory sample handling, log-in, and custody procedures; data reduction and reporting procedures; data validation procedures; instrument calibration procedures; the quality control program developed for the methods; and other laboratory procedures that may impact the laboratory analyses to be performed for this investigation.

10.4.2. Laboratory Performance Audits

A performance audit of IT Analytical and Mixed Waste Laboratories will be performed at least once and as determined necessary during this investigation by the ER Program TSO subcontractor QAM. The performance audit will evaluate at a minimum the laboratories' performance on the following activities specific to the investigation:

- implementation and follow-through of the laboratory quality control program established for this investigation as defined in the QAPP,
- sample custody and handling procedures,
- analytical methods followed as defined in the QAPP,
- sample tracking,
- data reduction,
- data validation,
- instrument calibration,
- sample preparation, and
- documentation of data analysis/data reduction.

The IT Analytical Laboratory currently participates in the CLP round-robin program. The IT Mixed Waste Laboratory participates in the EPA cross-check program for radiation analysis and the DOE Environmental Measurement Laboratory cross-check program. The results of the EPA cross-check program are

evaluated by the EPA, and the results are published as part of the public record. The DOE Environmental Measurement Laboratory performance samples will be reviewed by the DOE or its subcontractor to ensure acceptable laboratory performance.

The laboratory quality assurance coordinator has responsibility for monitoring the internal quality assurance program. The quality assurance coordinator is responsible for scheduling, coordinating, and conducting internal performance audits and for reviewing data for performance samples received. The quality assurance officer supplies blind performance samples to the laboratory at least semiannually.

External performance audits of IT laboratories will also be conducted by the EPA Region V Central Regional Laboratory as necessary. This audit may include an onsite visit of the laboratories.

11. PREVENTIVE MAINTENANCE

Proper preventive maintenance of field and laboratory equipment is a necessary element in achieving equipment reliability and minimizing equipment downtime.

11.1. FIELD EQUIPMENT

Field equipment will be properly calibrated, properly charged, and in good general working condition before the beginning of each working day. Mound Plant, ER Program SOPs (DOE 1991), Appendix E, define the required equipment checks and their frequency for each type of field equipment to be used, and Table III.1 in section 3 summarizes the checks to be performed during the Operable Unit 3 investigation. Any nonoperational field equipment will be removed from service and returned to the supplier, and a replacement will be obtained. Field equipment will not be repaired in the field. The following spare parts will be kept in the field: extra titration kit, pH electrodes, pH meter, electrical conductivity meter, water level indicator, and pump motor. Maintenance records will be maintained for each field instrument according to a unique number affixed to the instrument. These records will be reviewed prior to the instruments' use in the field to ensure that instrument maintenance and calibration are up-to-date.

All field instruments will be properly protected against inclement weather conditions during the field investigation. Each instrument is specially designed to maintain its operating integrity during variable temperature ranges that may be encountered during cold-weather working conditions. At the end of each working day, all field equipment will be taken out of the field and placed in a cool, dry room for overnight storage.

All subcontractor equipment (e.g., drill rigs and water trucks) will arrive at the site in proper working condition each day. All lubricating hydraulic motor oils will be checked by the subcontractors before the start of each work day to make certain all fluid reservoirs are full and that there are no leaks. Before the start of work each day, the field supervisor will also inspect all equipment for fluid leaks. If a leak is detected, the equipment will be removed from service for repair or replacement.

11.2. LABORATORY EQUIPMENT

The ability to generate valid analytical data requires that all analytical instrumentation be properly maintained. The IT Laboratories maintain full service contracts on all major instruments. These service contracts not only provide routine preventive maintenance, but also emergency repair service. Each analytical instrument is assigned an instrument logbook. All maintenance activities are recorded in the

instrument log. The information entered in the instrument log includes

- date of service,
- person performing service,
- type of service performed and reason for service,
- replacement parts installed (if appropriate), and
- miscellaneous information.

Table XI.1 summarizes the type and frequency of maintenance of major instruments that will be used for the analytical program. Those parts commonly replaced, as specified, in Table XI.1 are the spare parts kept in the laboratory. A listing of spare parts is also routinely maintained in files for each instrument.

Table XI.1.

Preventive Maintenance Requirements for Major Instruments
IT Laboratories

APPLICABLE ANALYTICAL METHOD	INSTRUMENT	ITEMS CHECKED/SERVICED	MINIMUM FREQUENCY
CLP SOW-7/88 for TAL inorganics	Inductively Coupled Argon Plasma	Replace pump tubing	As needed
		Inspect and clean sample introduction system	As needed
		Drain air filters	Semi-weekly
		Check drain receptacle level	Weekly
		Clean torch	Monthly
CLP SOW-7/88 for TAL inorganics	Atomic Absorption Spectrometer for Mercury Analysis	Replace check valves	As needed
		Check pump delivery	As needed
		Check tubing integrity	As needed
		Clean burner/adsorption cell mount	Monthly
		Clean adsorption cell	Monthly
		Replace system tubing	Semi-annually
CLP SOW-7/88 for TAL inorganics	Atomic Absorption Spectro-photometer--Flame Units	Inspect backflash diaphram	As needed
		Check drain receptacle level	As needed
		Replace nebulizer chamber O-rings	As needed
		Replace beam splitter	As needed
		Clean nebulizer	Weekly
		Clean burner head	Weekly
		Clean spray chamber	Weekly
		Remove and clean burner mount	Monthly

Table XI.1. (continued)

APPLICABLE ANALYTICAL METHOD	INSTRUMENT	ITEMS CHECKED/SERVICED	MINIMUM FREQUENCY
CLP SOW-7/88 for TAL inorganics	Graphite Furnace Atomic Absorption Spectrometer (Model 951/655)	Replace graphite cuvette	As needed
		Replace dry-rite and glass wool	As needed
		Check drain receptacle	As needed
		Replace temperature sensor	As needed
		Clean electrodes	As needed
		Replace graphite inserts	As needed
		Replace electrodes and sensor posts	As needed
		Check graphite insert	Weekly
		Check recirculator water level	Weekly
		Replace set screw	Weekly
		Check electrode appearance	Weekly
		Clean nebuulizer and spray chamber	Monthly
		Clean furnace chamber and windows	Monthly
		Flush furnace head with acetic acid	Semi-annually
		Replace recirculator water	Semi-annually
CLP SOW-7/88 for TAL inorganics	Graphite Furnace Atomic Absorption (Model 12E/188)	Check drain receptacle	As needed
		Clean sapphire window	As needed
		Replace graphite cuvette	As needed
		Clean cell body and windows	Weekly
		Replace marble chips	Weekly
		Check recirculator water level	Weekly
		Clean temperature sensor filter	Semi-monthly
		Clean FASTAC pump	Monthly
		Clean nebulizer and spray chamber	Monthly
		Clean air filter	Monthly
		Flush furnace head with acetic acid	Semi-annually
		Replace recirculator water	Semi-annually

Table XI.1. (continued)

APPLICABLE ANALYTICAL METHOD	INSTRUMENT	ITEMS CHECKED/SERVICED	MINIMUM FREQUENCY
USATHAMA Explosives	High Performance Liquid Chromatography	Degas solvents	Daily when used
		Check for leaks	Daily when instrument used
		Flush column	Daily when instrument used
		Check injector for blockage/pressure drop	Daily when instrument used
		Check column pressure	Daily when instrument used
		Replace lamp	As needed
		Replace guard column	As needed
	Organic Prep: Sonicator	Inspect prope tips for etching/pitting	Daily when used
		Replace probe tip	As needed
		Disassemble and clean sonicator probe tips	Semi-annually
		Tune sonicator	Semi-annually
	pH Meters	Gel Filled	Maintenance free
		Electronics checked	As needed
Chloride, nitrate-nitrite	VIS Spectrophotometer	Replace lamp	As needed
		Check tubing	Daily
	Millipore Water System	Conductivity checked	Daily
		Pressure check	Daily
		Change ion exchange bed and filters	As needed
	Analytical Balance	Internal weight train, gears, electronics	Annually by service contract
NA	Refrigerators	Temperature checked and logged	Daily
NA	Walk-In Coolers	Temperature checked and logged	Daily
NA	Ovens	Temperature checked and logged	Daily

Table XI.1. (continued)

APPLICABLE ANALYTICAL METHOD	INSTRUMENT	ITEMS CHECKED/SERVICED	MINIMUM FREQUENCY
Extractable Petroleum Hydrocarbons CLP SOW-2/88 for Pesticides/PCBs	Gas Chromatograph	Inlet, detector and column temperatures	Daily when instrument in use
		Gas pressures	Daily
		Column and dectector flow rates	Daily when instrument in use
		Check zero and noise level	Daily when instrument in use
		Check responses, retention times using a check standard	Daily when instrument in use
		Gas purity check	As needed
		Check autosampler for bubbles, flush volume	Daily when instrument in use
		Check electrolyte level and flow rate (HALL only)	Daily when instrument in use
		Reactor tube temperature (HALL only)	Daily when instrument in use
		Check pressure in gas tanks, replace in <500 PSI	Weekly
		Clean outside surfaces of instruments	Weekly
		Replace septa	As needed
		Replace column or part of column packing	As needed
		Clean/replace detector	As needed
		Replace glass wool/glass insert	As needed
		Clean injection port	As needed
		Replace reaction tube (HALL only)	As needed

Table XI.1. (continued)

APPLICABLE ANALYTICAL METHOD	INSTRUMENT	ITEMS CHECKED/SERVICED	MINIMUM FREQUENCY
CLP SOW-2/88 for Volatile and Semivolatile Organic Compounds	Gas Chromatograph Mass Spectrometry	Diffusion pump oil	Bi-weekly[a]
		Mechanical pump oil	Quarterly[b]
		Power con. air filter	Bi-weekly[c]
		QEM filter	Bi-weekly[c]
		Water bay filter	Bi-weekly[c]
		Interface box	Bi-weekly[c]
		Vacuum chaff filter	Monthly[c]
		Turbo pump oil	Quarterly[d]
		Water filter (if applicable)	Observe and change as needed
		Computer air filter	Monthly[c]
		Card cage air filter	Monthly[b]
		Source-clean ceramics, polish lenses	As needed
		Clean poles and ceramics on the poles	As needed
		Clean contacts on the component boards	As needed
		Vacuum the component boards	As needed
		Clean all fan screens	Weekly
		Vacuum outside of instrument	Weekly
		Clean grob and replace quartz insert	As needed
		Replace septum	Daily (each shift)
		Injection port liner checked	Daily
		Column maintenance	As needed

Table XI.1. (concluded)

APPLICABLE ANALYTICAL METHOD	INSTRUMENT	ITEMS CHECKED/SERVICED	MINIMUM FREQUENCY
CLP SOW-2/88 for Volatile and Semivolatile Organic Compounds (continued)	Gas Chromatograph/ Mass Spectrometry (continued)	Disk drive (CMD only)	Semi-annual (service engineer) or as needed
		Printer	Quarterly
	Manual Alpha/Beta Counter	Clean fan filter	Monthly
	Gamma Spec System		
	Equipment Rack	Clean fan filter	Monthly
		Check for operation	Monthly
	Vacuum pump	Clean fan filter	Monthly
		Change pump oil	Annually
	Germanium detectors	Fill LN_2 dewars	Weekly

Note: "As needed" under minimum frequency refers to the fact that the item is routinely checked during analysis, and the item is replaced/repaired when it fails or is found to be worn.

[a] This is an ITAS Knoxville frequency. ITAS Mixed Waste Laboratory performs this every 2 to 5 years.
[b] This is an ITAS Knoxville frequency. ITAS Mixed Waste Laboratory performs this every 6 months.
[c] This is not applicable to the ITAS Mixed Waste Laboratory.
[d] This is an ITAS Knoxville frequency. ITAS Mixed Waste Laboratory performs this annually.

CLP - Contract Laboratory Program
TAL - Target Analyte List
NA - Not Applicable

12. SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

The procedures to assess precision, accuracy, and completeness are presented in section 3. Equations are used to calculate, and acceptance criteria are used to assess precision, accuracy, and completeness (subsections 3.1.1, 3.1.3, 3.2.2, and Tables III.1 through III.4). Accuracy, precision, and completeness are defined in subsections 3.1.1, 3.2.1, and 3.3, respectively.

The precision and accuracy requirements for TCL organic analyses, TAL inorganic analyses, and non-CLP analyses are given in Tables III.2 and III.3. All analytical data are reviewed relative to those criteria. For any quality control checks outside the acceptance criteria, the results will be assessed by the TSO subcontractor for data usability. The acceptance criteria for field quality control checks does not take into account the interdependencies of the checks. Since many of the quality control checks are interrelated (e.g., both a method blank and a trip blank could have contaminants), each batch of analytical data will be assessed on a case by case basis. Field blank results associated with soil/sediment data will be used only to provide information on the potential for contamination to exist in the soil/sediment samples, and will not be used for quantitative purposes.

Precision and accuracy of laboratory analyses are assessed by IT Analytical Services prior to reporting analytical results according to the procedures described in sections 10.1.2.2, 10.1.2.3, and 10.1.3 of the IT Analytical Services Corporate Quality Assurance Manual (Appendix A) and section RS10.2 of the Radiological Sciences Laboratory QA Manual for radionuclide analysis (Appendix C). These procedures include evaluating all quality control samples and using control charts for major parameters. Control charts with warning limits and control limits are prepared for replicate analyses (using RPD), matrix spikes, and blank spikes (using percent recovery). Upper and lower warning limits and control limits are defined by the following equations:

$$\text{Upper Control Limit} = m + 3s_m$$

$$\text{Lower Control Limit} = m - 3s_m$$

$$\text{Upper Warning Limit} = m + 2s_m$$

$$\text{Lower Warning Limit} = m - 2s_m$$

Where:

m = average of the RPD determinations or percent recoveries of at least 20 measurements

s_m = standard deviation of the RPD determinations or percent recoveries

These upper and lower control limits are those used internally by the laboratory and are not necessarily those presented in Tables III.2 and III.3. Control limits for CLP analyses are established as guidance or contract-required limits in the CLP SOW for organic and inorganic analysis. The laboratory calculates control limits for these analyses to determine if they fall within these limits.

The initial responsibility for monitoring the quality of an analytical system lies with the analyst. The analyst will verify that all quality control procedures are followed and that results of analysis of quality control samples are within acceptance criteria. If acceptance criteria limits are exceeded, appropriate corrective actions will be taken and out-of-control situations will be described in the analytical report case narrative.

13. CORRECTIVE ACTION PROTOCOLS

13.1. FIELD CORRECTIVE ACTION

During the Operable Unit 3 Limited Field Investigation, failure to comply with the requirements of this QAPP, the requirements of the work plan, or any referenced procedures will be considered nonconformances. The initial responsibility for monitoring the quality of field measurements and observations and compliance with plans and procedures lies with the field personnel. The field manager is responsible for verifying that all quality control procedures are followed. This requires that the field manager assess the correctness of field methods and the ability to meet quality assurance objectives. If a problem occurs that might jeopardize the integrity of the project or cause some specific quality assurance objective not to be met, it is the responsibility of all field project staff to report it. Field project staff must report all such suspected nonconformances by initiating a nonconformance report and submitting it to the field manager (Figure 13.1). The field manager will submit the nonconformance report to the site manager, the installation manager, and the project quality assurance manager for formal investigation. An appropriate corrective action will then be decided upon and implemented. The field manager will document the problem, the corrective action, and the results, using the form shown in Figure 13.2. Copies of the documentation form will be provided to the site manager, the installation manager, and the project quality assurance manager. All nonconformance reports and corrective action forms will be included in the quality assurance section of the Operable Unit 3 Limited Field Investigation Report.

13.2. LABORATORY CORRECTIVE ACTION

The initial responsibility for monitoring the quality of an analytical system lies with the analyst. In this pursuit, the analyst will verify that all quality control procedures are followed and that the results of analysis of quality control samples are within acceptance criteria.

If this assessment by the analyst reveals that any of the quality control acceptance criteria are not met, the analyst must immediately assess the analytical system to correct the problem. The deficiency is reported to the appropriate supervisor, who will complete and sign a nonconformance memorandum and notify the Quality Control Coordinator. Written laboratory nonconformances are reported monthly to the Quality Assurance Director.

The nature of the corrective action obviously depends on the nature of the problem. For example, if a continuing calibration verification is determined to be out of control, the corrective action may require recalibration of the analytical system and reanalysis of all samples since the last acceptable continuing

NONCONFORMANCE REPORT (NCR)

ACTIVITY **NCR NO.:**

NCR DATE: **APPLICABLE TEST/PLAN/SOW/DRAWING/SPECIFICATIONS:**

ORGANIZATION/CONTRACTOR: **CONTRACT NO.:**

ORIGINATOR: **ORGANIZATION:** **PHONE NO.:**

APPROVAL: ( ) SIGNIFICANT NCR (Requires client concurrence)
( ) INTERNAL

DESCRIPTION OF NONCONFORMANCE (Including cause, location, and date):

DISPOSITION (Actions taken to solve the problem):

CORRECTIVE ACTION (Actions taken to prevent recurrence):

Date Corrective Action is to be completed:
Person responsible for corrective action:

QA VERIFICATION OF CORRECTIVE ACTION:
( ) VERIFIED
( ) CANNOT VERIFY (Note reasons)

_____
Signature of QA Officer

APPROVAL/DISTRIBUTION:

APP	DIST	TITLE	NAME	ORG	APPROVAL INITIALS	DATE
( )	( )	PROJECT DIRECTOR				
( )	( )	DEPUTY PROJECT DIRECTOR				
( )	( )	PROJECT MGR.				
( )	( )	QA OFFICER				
( )	( )	CLIENT PI				
( )	( )	CLIENT PL				
( )	( )	CORP QA MGR				
( )	( )	INSTALLATION MANAGER				
( )	( )	_____				
	( )	FILE COPY -				

SHEET _____ **OF** _____

form 4427

Figure 13.1. Nonconformance report (NCR).

Audit Report #_____
Page _____ of _____

CORRECTIVE ACTION DOCUMENTATION

DATE/ORIGINATOR: _____

PERSON RESPONSIBLE FOR RESPONSE: _____

DESCRIPTION OF PROBLEM and when identified: _____

State cause of problem if known or suspected: _____

SEQUENCE OF CORRECTIVE ACTION: (If no responsible person is identified, bring this form directly to the QA Coordinator.) State date, person, and action planned: _____

CA Initially Approved By: _____ Date: _____

Description of Follow-Up: _____

Follow-Up Dates: _____

Final CA Approved By: _____ Date: _____

MOUND/CorActDoc 1/15/91

Figure 13.2. Corrective Action Documentation form.

calibration standard. Specific corrective actions to exceeded acceptance criteria for laboratory quality control checks are summarized in Table III.2. Corrective actions to be taken for CLP analyses (TCL VOCs, semivolatile organic compounds, pesticides/PCBs and TAL inorganics) are presented in the CLP SOW, or the most current, for organic analyses, and CLP SOW, or the most current, for inorganic analyses. If samples analyzed under the CLP procedures exceed holding times, the laboratory will analyze new samples at no cost to the client. Section 13 of each IT Analytical Services Quality Assurance Manual in Appendixes A through D provide additional corrective action protocols for the analyses of interest.

13.3. CORRECTIVE ACTIONS RESULTING FROM AUDITS

The project quality assurance manager and audit team will prepare a formal report of all audit proceedings. The programmatic impact of a negative finding, such as the lack of or failure to use an appropriate procedure, will be determined by the project quality assurance manager or lead auditor and reported to the site or installation manager. A corrective action plan and schedule will be requested, and the installation manager will be responsible for ensuring that action to correct the nonconformance has been developed and initiated, and that any special expertise has been made available. The installation manager will also be responsible for implementing the corrective action and ensuring that no additional work, dependent on the nonconforming activity, is performed until the nonconformance is corrected. Corrective action may include reanalyzing the samples (if holding time permits), resampling, and evaluating and amending the sampling and analytical procedures.

The project manager will approve the corrective action and will be responsible for ensuring that the corrective action adequately addresses any nonconformances noted during internal or external audits. The project quality assurance manager will ensure that corrective actions for nonconformances are implemented by

- evaluating all reported nonconformances,
- controlling additional work on nonconforming items,
- maintaining the log of nonconformances, and
- ensuring that nonconformance and correction reports are included in the site documentation files.

Following implementation of satisfactory corrective action, the project quality assurance manager will conduct sufficient follow-up activities to verify this implementation. Such confirmation will be documented,

along with any further recommendations, in a formal closeout report for the audit. The closeout report will be included in the Operable Unit 3 Limited Field Investigation Report.

14. QUALITY ASSURANCE REPORTS TO MANAGEMENT

Quality assurance reports to management will consist of prior notification of activities, and reports on activities. Reports will encompass both routine reports and special reports, including written reports and memoranda documenting data assessment activities, results of data validations, audits, nonconformances, corrective actions, and quality notices. The management hierarchy receiving some or all of the reports will include DOE Dayton Area Office (DAO) (the Remedial Project Manager), the EG&G Mound Manager of Environmental Restoration, the DOE Albuquerque Operations Program Manager, and the DOE RI/FS Contractor Project Manager. Reports may be distributed to RI/FS subcontractors at the discretion of the DOE Remedial Project Manager and the RI/FS Contractor Project Manager.

Prior notification of all quality assurance activities will be provided to all managers through the vehicle of RI/FS monthly progress reports that also contain information about all RI/FS activities, and through special trip notices that notify managers of contractor and subcontractor travel. Monthly RI/FS progress reports will routinely discuss upcoming activities including field work and scheduled audits. Any of the managers listed above may use the notification to identify the need for, and schedule quality assurance audits in connection with, scheduled field work.

Monthly reports will also describe the progress, the completion, and sometimes the results, of quality assurance activities. Descriptions of the completion of activities will serve as notice to all managers of the availability of quality assurance reports. Results of quality assurance activities may be summarized for inclusion in monthly reports. Each quality assurance activity shall be documented in a report separate from any summary or description included in the monthly RI/FS progress report.

Within IT Analytical Services, the Quality Control Coordinator and the Quality Assurance/Quality Control Director will prepare routine reports to management indicating effectiveness of the laboratory quality assurance program. The Quality Control Coordinator will prepare a monthly summary report of the internal review activities of laboratory performance and submit the report to the Quality Assurance/Quality Control Director. Reports of the semiannual system audits will be distributed to the Vice President of Analytical Services, laboratory manager, regional Director of analytical operations, and the IT Analytical Services Quality Assurance/Quality Control Director.

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APPENDIX A

IT ANALYTICAL SERVICES CORPORATE QUALITY ASSURANCE MANUAL

INTERNATIONAL TECHNOLOGY CORPORATION

QUALITY ASSURANCE MANUAL
IT ANALYTICAL SERVICES

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STATEMENT OF MANAGEMENT POSITION

IT Corporation is committed to providing quality services for environmental management; services which meet the needs of our clients, satisfy regulatory requirements, and are commensurate with the current state of the art.

To satisfy our clients' quality objectives, to meet regulatory requirements, and to comply with IT Corporate-wide requirements, IT Analytical Services (ITAS) has adopted a comprehensive Quality Assurance Program. The principles and practices of the Program apply to every employee at every level within ITAS; they are fundamental to the way we do business and to the services we provide.

The Quality Assurance Manual is an overall statement of Program policy. This Manual provides guidance to ITAS personnel in fulfilling their responsibilities, and serves as a statement to external parties of ITAS' commitment to quality.

Implementation of the Program is the responsibility of all ITAS personnel. Management at every level has the commitment, duty, and authority to insist that these responsibilities are met, and that the principles and practices of the Program are followed and implemented.

Quality Assurance Coordinators are assigned in all ITAS laboratories to see that the Program is implemented on a day-to-day basis as intended. Each Coordinator has the authority and duty to stop work if and when necessary to satisfy Program requirements.

To verify that the Quality Assurance Program is successfully implemented, the laboratories are independently audited by the ITAS Director of Quality Assurance/Control, who is directly responsible to the Vice President, Analytical Services. In addition, the laboratories are subject to audits by the Environmental Projects Group Director of Quality Assurance and Discipline Management, and by various regulatory authorities and other outside agencies.

Brad S. Figley
Vice President
Analytical Services
February 1, 1988

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1.0 INTRODUCTION

Analytical data are used for many purposes, including: compliance with regulatory requirements; determination of the presence, concentration, and movement of hazardous materials in the environment; potential effects upon or protection required for persons; and the actions necessary for disposal or treatment of hazardous materials.

Analytical data may be used within IT Corporation to support a broader-based project involved with: site characterization and/or remediation; on-site treatment; treatment and/or disposal at an IT facility; or health and safety protection of IT personnel and the public. Data may also be produced for outside commercial testing and submitted directly to clients for their decision making.

It is the purpose of the ITAS Quality Assurance Program, as expressed in this Quality Assurance Manual, to provide data which are of acceptable quality. To achieve this, a system is described which controls:

- Preservation of samples
- Receipt and handling of samples
- Processing and analyses of samples
- Analytical equipment
- Data verification
- Data reporting
- Records management
- Management review.

ITAS recognizes that all laboratory personnel affect data quality. This Manual has been prepared so that staff members will be cognizant of the procedures adopted by ITAS for the production of analytical data, and so they will be aware of their responsibilities. This Manual applies to chemical analysis only; microbiology, bioassay, geotechnical and radiological analyses are described in Laboratory-Specific Attachments to this Quality Assurance Manual.

The remainder of this introductory section discusses:

- Quality Assurance objectives of ITAS, which are subsequently discussed in detail in this Manual
- The organization of ITAS
- Quality Assurance documents within ITAS
- Control, distribution, and revision of Quality Assurance documents.

Within this Manual, the terms Quality Assurance and Quality Control are defined and used as follows:

- Quality Assurance - The overall controls imposed upon laboratory operations. Actions taken by personnel and the documentation of laboratory performance are included as specified in the Quality Assurance Program.
- Quality Control - The daily, specific actions taken within the laboratory to verify sample integrity, performance of analyses, data processing, and record maintenance.

1.1 OBJECTIVES OF THE QUALITY ASSURANCE PROGRAM

As stated, the overall objective of the Quality Assurance Program for the ITAS laboratories is to provide data of known quality. To accomplish this, each laboratory must:

- Maintain an effective, ongoing Quality Control Program to measure and verify laboratory performance.
- Meet data requirements for accuracy, precision, and completeness through the use of proven methodologies.
- Provide sufficient flexibility to allow controlled changes in routine methodology to meet specific data requirements.

- Monitor operational performance of the laboratory on a routine basis and provide corrective action as needed.

- Recognize and promptly correct for any factors which adversely affect quality.

- Maintain complete records of sample submittal, laboratory performance, and completed analyses to verify resulting data.

Specifically, the following actions are taken:

- Procurement and control of instrumentation and supplies required for laboratory operation

- Sample receipt, chain-of-custody completion, and sample storage

- Calibration and preventive maintenance of instrumentation

- Establish a Quality Control sample program within the laboratory so that statistical evaluation of analyses can be done

- Analyses in accordance with recognized analytical procedures

- Data processing, validation, and reporting

- Control and maintenance of laboratory records

- Identification and resolution of nonconformances requiring corrective action

- Audits to verify laboratory performance and the reporting of audit results to management

- Training of analysts in technical and quality control procedures, including an orienetation to the ITAS Quality Assurance Program.

1.2 ITAS QUALITY ASSURANCE DOCUMENTS

Complete documentation of ITAS operations requires several types of ITAS Quality Assurance documents. This Quality Assurance Manual provides

overall policy for ITAS. Other documents provide supplementary information or project-specific requirements. Each of the document types is discussed below. Figure 1-1 illustrates the basic purpose of each document.

- Quality Assurance Manual - The Quality Assurance Manual provides ITAS Quality Assurance policy. The Manual discusses all aspects for Quality Assurance and Quality Control, both administrative and technical. However, it is not intended that the Manual provide in-depth technical discussion. The Quality Assurance Manual has precedence in policy matters over all other ITAS quality-related documents. The Manual is applicable to and provides direction for all ITAS analytical laboratories.

- Laboratory-Specific Attachments - Laboratory-Specific Attachments to the Quality Assurance Manual are prepared for two purposes:

  - If an individual ITAS laboratory provides services that are unique to that laboratory, the service and necessary Quality Assurance requirements will be described in an attachment.

  - Certain aspects of laboratory operations discussed in the Quality Assurance Manual are dependent upon laboratory facilities. Quality Assurance activities such as preventive maintenance, calibration, and data verification are functions of the equipment in a specific laboratory. Attachments will be prepared by each laboratory to document their specific operations. Laboratory-Specific Attachments are adopted as part of the Quality Assurance Manual for the preparing laboratory.

- Manuals of Practice - Manuals of Practice are developed to provide in-depth technical discussions of specific topics. For example, a Manual of Practice for the field collection, preservation, and shipment of samples to ITAS laboratories provides specific uniform direction to IT personnel. Manuals are usable across ITAS laboratories. A Manual may be a collection of SOPs.

- Standard Operating Procedures (SOPs) - These are written, detailed instructions describing the specific laboratory operations and performance of routine laboratory tasks. They specify what is done, whose responsibility it is to perform tasks and whose to verify their correctness; they are sufficiently detailed to provide data of acceptable quality and integrity, with a minimum loss of data due to out-of-control situations. They also provide for documentation to record the performance of all tasks and their results, and demonstrate the validation of the data each time the data are recorded, calculated, or transcribed. SOPs address the major elements upon which analytical quality depends.

  - Organization and personnel
  - Facilities and equipment
  - Analytical methodology
  - Sample custody procedures
  - Quality control
  - Data handling.

- Project-Specific Manuals - Frequently, contractual and regulatory demands, or uniqueness of a project's scope of work, require the preparation and implementation of a Project-Specific Quality Assurance Program and Manual, generally referred to as a Quality Assurance Project Plan (QAPP). Project demands can include:

  - The development and/or use of new testing methods

  - Different requirements for equipment calibration and maintenance

  - Different detection limits for testing

  - Different accuracy and precision limits or the statistical treatment of data

  - Additional or different document and record formats and maintenance.

  If a specific project requires a unique Quality Assurance Program, that program will be implemented. Full documentation will be provided in a Project-Specific Manual. The requirements of the project will take precedence over conventional ITAS Quality Assurance practices for that work.

1.3 DOCUMENT CONTROL, DISTRIBUTION, AND REVISION

The Quality Assurance Manual, Attachments, Manuals of Practice, SOPs, and Project-Specific Manuals are approved and controlled documents.

Approvals required before issue for the various documents follow:

- ITAS Quality Assurance Manual
  - Senior Vice President, Environmental Projects Group
  - Vice President, IT Analytical Services
  - Director, Quality Assurance/Control, ITAS
  - Director, Quality Assurance and Discipline Management, Environmental Projects Group
- Laboratory-Specific Attachment
  - Laboratory Manager
  - Regional Director, Analytical Operations
  - Director, Quality Assurance/Control, ITAS
  - Director, Quality Assurance and Discipline Management, Environmental Projects Group
- Manuals of Practice
  - Vice President, IT Analytical Services
  - Director, Quality Assurance/Control, ITAS
- Standard Operating Procedures
  - Laboratory Manager
  - Director, Quality Assurance/Control, ITAS
  - Laboratory Quality Control Coordinator
- Project-Specific Manuals
  - Laboratory Manager
  - Laboratory Quality Control Coordinator
  - Project Manager (if analytical services are part of a broader-based project, and if required by contract).

Approval of these documents shall be denoted by a signature-and-date page in each document, which includes the required signatures above and the manual author. In addition, IT prepared documents shall be reviewed and approved by regulatory agencies, as appropriate.

In general, documents will be distributed as needed to all ITAS

personnel. When the document is no longer needed or the copyholder leaves IT Corporation, it shall be returned. Copies distributed within IT and ITAS will be numbered for document control, or as uncontrolled documents. Distribution of the Quality Assurance Manual and Manuals of Practice will be by the Director, Quality Assurance/Control, ITAS. Laboratory-Specific Attachments and Project-Specific Manuals (QAPP), and SOPs will be distributed by the appropriate Laboratory Managers and QC Coordinators.

So that all Quality Assurance documents can be revised as necessary, distribution lists will be maintained, as applicable, by the ITAS Quality Assurance Staff and Laboratory Managers. Before issue, revisions will require the same approvals as the original document. Also, revisions will be consecutively numbered and each page revised shall denote the numbered revision. To facilitate revisions, each page of all manuals shall contain the following:

Title or Document Identification
Section No. _____
Revision No. _____
Date: (of issue)
Page ___ of ___

Issuing a revision requires a revised signature page.

Revisions will be issued to all holders of controlled copies. Each copyholder will sign a revision receipt verifying that the revision has been received and properly placed in the document. The receipt will be returned to the issuing manager.

Copies of Quality Assurance documents will be issued external to IT Corporation as required. These documents may be controlled (numbered) or uncontrolled (unnumbered) copies depending upon the reason for issue. Controlled copies will be revised in accordance with the above. In all cases, external parties will be asked to return Quality Assurance

documents when the need for the document has ended. Exceptions to this will be if the external party requires a copy for record purposes.

DOCUMENT	PURPOSE
QUALITY ASSURANCE MANUAL	• Provides ITAS Quality Assurance policy • States responsibilities of laboratory staff • Describes basic Quality Control practices
LABORATORY-SPECIFIC ATTACHMENT	• States supplementary implementing practices and services of a specific laboratory • For specific laboratory; becomes incorporated with Quality Assurance Manual
MANUALS OF PRACTICE	• Detailed discussion of specific technical subjects • Provides detailed information related to technical topics discussed in the Quality Assurance Manual
STANDARD OPERATING PROCEDURES	• Detailed description of specific tasks relating to laboratory operation and procedures
PROJECT-SPECIFIC MANUAL (QAPP)	• Prepared as required for specific contract • May supplement or change ITAS Quality Assurance practices for specific project • Takes precedence over other ITAS documents for a specific project

FIGURE 1-1

ITAS QUALITY ASSURANCE DOCUMENTS

2.0 LABORATORY ORGANIZATION

This section discusses general positions and quality-related responsibilities which each laboratory must provide for the implementation of the Quality Assurance Program and completion of Quality Control activities. Also discussed is the role of the IT Analytical Services Quality Assurance/Control Director.

2.1 ITAS QUALITY-RELATED RESPONSIBILITIES WITHIN A LABORATORY

The positions discussed below are part of ITAS laboratories. The quality-related responsibilities may be modified in the Laboratory-Specific QA Attachments' organization section:

- Laboratory Manager
  - Report directly to Vice President, IT Analytical Services, or Regional Director Analytical Operations
  - Implement the Quality Assurance Program within the laboratory
  - Periodically determine the effectiveness of the Quality Assurance Program in the laboratory
  - Approve Laboratory-Specific Attachments to the Quality Assurance Manual and Project-Specific Manuals (QAPPs), SOPs, and revisions
  - Recommends to the Quality Assurance/Control Director, ITAS, changes in the Quality Assurance Program
  - Issues laboratory reports
  - Maintain current laboratory organization chart
  - Supervise laboratory participation in inter-laboratory accreditation and proficiency programs.

- Technical Director

  - Provides technical overview of laboratory activities

  - Serves as an "in-house" consultant for the applicability of general Quality Control practices to specific needs

  - Leads the training of analysts in laboratory operations and analytical procedures

  - Evaluates analytical techniques, procedures, instrumentation and Quality Control procedures, and provide recommendations to the Laboratory Manager

  - Supervises the verification of software for data processing

  - Recommends standards for purchasing instrumentation, equipment, reagents, gases, and chemicals

  - Defines the instrument preventive maintenance schedule

  - Defines the calibration program within the laboratory.

- Quality Control Coordinator:

  - Prepares Quality Control standards, arranges insertion of Quality Control samples into the laboratory sample stream, and reviews the results

  - Performs statistical analyses utilizing results of QC samples analyses

  - Informs the Operations Manager and Group Leaders of data which lies outside of acceptable limits

  - Serves as the "focal point" for the reporting and disposition of nonconformances

  - Resolves ongoing and recurring nonconformances within the Laboratory

- Recommends corrective actions for resolution of nonconformances

- Reviews statistical data to verify the laboratory is meeting stated Quality Control goals

- Maintains current distribution lists for Laboratory-Specific Attachments and Project-Specific Manuals and Standard Operating Procedures

- Closes findings and recommendations of Quality Assurance Audits

- Notifies the Technical Director, Operations Manager, and Laboratory Manager of out-of-control situations

- Stops production of data in a laboratory area where the review of quality control data or procedures shows significant problems

- Reports nonconformances to the ITAS Quality Assurance/Control Director or the Director, Quality Assurance, Environmental Project Group if the situation is not corrected within the laboratory

- Assists in the performance of Quality Assurance audits and performs Quality Control audits

- Establishs and supervises the laboratory Quality Assurance training program.

• Operations Manager/Assistant Laboratory Manager:

- Oversees the log-in of all samples received, completion of chain-of-custody records, and maintenance of sample log books

- Manages laboratory daily analytical operations

- Supervises Quality Control activities performed as part of routine analytical operations

- Supervises sample storage facilities

- Supervises the preparation and maintenance of laboratory records

- Oversees preventive maintenance program.

- Group Leaders:

  - Serve as the Lead Analyst within the group (group meaning organic analysis, inorganic analysis, special projects, etc.)

  - Organize and schedule the analytical testing program with consideration for sample-holding times

  - Implement data verification procedures

  - Assign analysts for data processing and validation activities

  - Review and approve all analytical data and submit to Operations Manager and Laboratory Manager for issue

  - Evaluate instrument performance and supervise instrument calibration and preventive maintenance programs

  - Report out-of-control or nonconforming situations to Operations Manager, Laboratory Manager, and Quality Control Coordinator, as appropriate.

- Analysts

  - Perform analytical procedures and data recording in accordance with accepted methods

  - Perform and document calibration and preventive maintenance of instrumentation, as appropriate

  - Perform data processing and validation

  - Immediately report out-of-control situations, instrument malfunction, calibration failure, or other nonconformances to the Group Leader and Quality Control Coordinator, as appropriate.

2.2 ITAS QUALITY ASSURANCE RESPONSIBILITIES OUTSIDE OF THE LABORATORY

Ultimate responsibility for Quality Assurance within the laboratories

lies with the Vice President, IT Analytical Services. To fulfill this responsibility, the Vice President has assigned responsibility within the laboratory to the respective Regional Directors Analytical Operations and Laboratory Managers. External review of laboratory quality performance is assigned to the IT Analytical Services Quality Assurance/Control Director.

The Director, Quality Assurance/Control, reports directly to the Vice President and is independent of laboratory management. This independence allows the Director to assess laboratory quality performance without being influenced by schedule and cost considerations. Responsibilities of the Quality Assurance/Control Director are:

- Approves ITAS Quality Assurance documents
- Performs audits of ITAS laboratories (audits are discussed in Section 14.0) to verify compliance with the Quality Assurance Program
- Verifies completion of corrective action cited in audits
- Acts as the collection point for proposed changes in the Quality Assurance Program and initiate changes in the program
- As necessary, discusses with Laboratory Managers and the Vice President unresolved nonconformances brought to the Director's attention by Quality Control Coordinators.

IT Analytical Services is a Division of the Enviromental Project Group. The Director, Quality Assurance and Discipline Management for the Group provides a further independent quality assurance review by:

- Participating in laboratory audits with the ITAS Quality Assurance/Control Director

- Approving the ITAS QA Manual and its Laboratory-Specific Attachments

- Maintaining communication so that QA problems which are not readily resolved within ITAS or are detrimental to the performance of a project within the Group are brought to the Group Director's attention for resolution.

IT Analytical Services has several Regional Directors of Analytical Operations, each responsible for two or more laboratories within a particular region. Quality Assurance duties of these Directors include:

- Providing resources for each laboratory's QA Program's effective implementation

- Reporting directly to the Vice President, IT Analtyical Services

- Periodically determining the effectiveness of the QA Programs at each laboratory

- Recommending changes in the QA Program to the Director, Quality Assurance/Control, ITAS.

3.0 STANDARD LABORATORY PRACTICE

Daily activities within an analytical laboratory are directed toward the analysis of samples. However, there are many laboratory functions which precede and follow analysis that are necessary to control and verify the analysis. The functions, as a total, represent the daily implementation of the Quality Assurance Program by the laboratory staff.

This section serves as an introduction to these functions and provides an overview of them as the total system providing Quality Assurance within the laboratory. Figure 3-1 presents a flow chart of functions for laboratory analyses. The functions shown can be divided into those which directly involve a sample, laboratory functions, and those which are independent (in general) of specific sample analysis, auxiliary functions.

Each function is affected by those which precede it. Therefore, it is imperative that each function be controlled in a specified manner. It is the purpose of the Quality Assurance Program to provide control by specifying how functions are to be performed, and by providing "checks and balances" to verify performance.

Each function is discussed in detail in the sections which follow (Sections 4.0 through 13.0 as denoted in Figure 3-1). Following is a brief discussion of the activities performed for each function and their role in the Quality Assurance Program.

3.1 RECEIPT OF SAMPLES AND INITIATION OF TESTING PROGRAM

Upon receipt of samples in the laboratory, the following is done:

- Samples are examined for damage, checked for proper preservatives and temperature
- The Chain of Custody Form is signed
- Samples are placed in the proper storage environment

- The testing program is defined by the Request for Analysis form or Purchase Order, or other appropriate means
- Acceptable holding times until samples must be analyzed are defined
- Samples are logged into the laboratory sample stream
- Appropriate laboratory personnel are notified of sample receipt.

3.2 MATERIAL AND INSTRUMENT PREPARATION

Concurrent with the performance of analysis, auxiliary functions are performed to provide appropriate materials to the Analysts, and verify and maintain instrument performance.

Material Procurement and Control

- Specifying grades of reagents, solvents, gases, and water used within the laboratory for specific analyses, and verifying adequacy of these materials before use
- Specifying material composition and volumetric tolerance of laboratory vessels
- Controlling reagents, solvents, etc., during storage
- Cleaning protocol for laboratory vessels.

Calibration

- Scheduled comparison of instrument performance against national standards for instruments which measure physical parameters, such as mass, time, and temperature. This type of calibration is independent of use in specific analyses and projects.

- Determination of instrument response to known chemical composition and concentration. Calibration may be part of daily instrument usage to define response for post analysis data processing of analytical raw data, or as response checks during or at the completion of a instrument run. This type of calibration may be independent of or part of the analysis for a specific project.

Preventive Maintenance

- Servicing instruments on a scheduled basis to maintain performance
- Maintaining a stock of instrument parts which are known to regularly degrade because of usage
- Preparing log books so that the historical performance of an instrument can be assessed.

3.3 ANALYTICAL PROCEDURES

The actual analysis of samples occurs during this function; however, for the analysis to be complete, several activities must be coincident:

- Sample holding times and storage environment are reviewed by the Analyst to verify that sample integrity has been maintained
- Analysis is performed in accordance with standard methods adopted by ITAS or as specified by the client. Analysis results include:
  - Prescribed daily instrument calibration or instrument tuning and documentation
  - Preparation and analysis of Quality Control samples and/or standards as part of the sample stream at the level of samples required.

3.4 PROCESS QUALITY CONTROL DATA

There are four steps involved in the total verification of analytical data:

- Computation of Quality Control sample data

- Comparison of Quality Control data with analytical acceptance limits
- Computation of analytical results from the data
- Independent validation of analytical results.

In general, Quality Control data and analytical data are interconnected. However, for the purposes of this discussion, the preparation of Quality Control data and the processing and evaluation of analytical data will be discussed separately because of the difference in quality assurance requirements for each.

Section 3.4 discusses Quality Control data, and Section 3.6 discusses the treatment of analytical data.

Quality Control samples results are analyzed and the QC data are reviewed and compared against stated acceptance levels for accuracy and precision. The data are used, as appropriate, to update control charts. If the QC data meets acceptance levels, processing of the analytical data begins. If the QC data are unacceptable, corrective action must be taken.

3.5 CORRECTIVE ACTION

If Quality Control data are unacceptable, the cause must be determined. If the cause can be resolved so that the integrity of the analytical data are not affected or can be corrected, the processing of the analytical data can proceed. Nonconformances which affect the integrity of analytical data will require resolution which may include reanalysis of the affected samples. Results shall be qualified in the report.

3.6 DATA PROCESSING AND VALIDATION

- Analytical test results are calculated by the

Analyst using computational methods prescribed for the various analytical methods

- The resulting data are validated, as required, by an individual independent of the analysis. Validation demonstrates and documents that the analyses have been properly performed, that proper input parameters (such as response factors or dilutions) have been used, and that mathematical manipulation is correct.

3.7 REPORTING

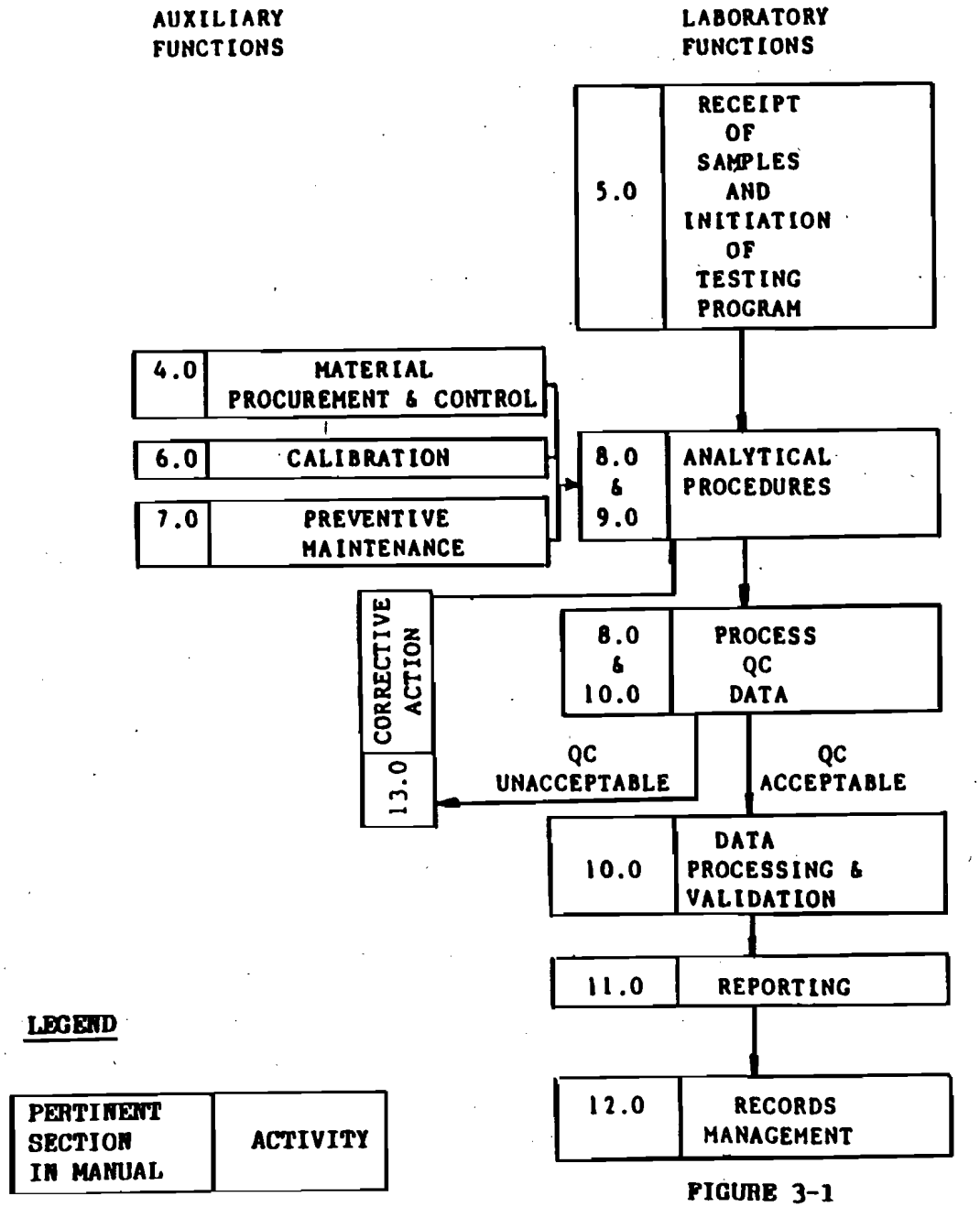
- Analytical data, and Quality Control data if appropriate, are summarized in presentation format
- The data are reviewed by Group Leaders to verify that the objectives of the analysis have been met
- After approval the data are issued by the Operations Manager and/or the Laboratory Manager.

3.8 RECORDS MANAGEMENT

There are two categories of records prepared within an ITAS laboratory:

- Project-specific records which are related solely to the analysis performed for a group of samples such as chain-of-custody and raw analytical data
- Records which demonstrate overall laboratory operation, and are, in general, independent of specific projects, such as the master laboratory sample log-in book, instrument performance, and maintenance log books and control charts.

Separate files are maintained for each category of records. However, the document control system enables cross-referencing of records (such as for instrument performance and maintenance log books) so that implementation of the Quality Assurance Program can be demonstrated.



ACTIVITIES

- Complete chain of custody
- Sample log in
- Define tests to be performed on specific samples
- Define QC sample requirements

- Analysis of samples
- Analysis of QA samples
- Summarize QC data
- Update control charts
- Statistical treatment of data

- Calculate analysis results
- Independent data review

- Summarize data
- Issue within IT or to client

- Prepare project file
- Maintain records

FIGURE 3-1

LABORATORY ANALYSIS FLOW CHART

4.0 MATERIAL PROCUREMENT AND CONTROL

The quality of reagents, solvents, gases, water, and laboratory vessels used in analyses must be known so that their effect upon analytical results can be defined. Materials purchased by an ITAS Laboratory shall meet the requirements stated below or as denoted in specific analytical procedures, and be controlled as stated. Requirements are also stated for internally prepared materials such as water and compressed air.

Each laboratory shall assign an individual responsible for purchasing materials and controlling them in the laboratory. This person can be the Quality Control Coordinator or other as assigned by the Laboratory Manager. Responsibility for this work shall be defined in the respective Laboratory-Specific Attachment to this Manual and the laboratory organization charts. Duties include:

- Specifying in purchase orders or requisitions, suitable grades of materials (grade should be defined by the QC Coordinator or Group Leaders)
- Verifying upon receipt that materials meet requirements and that, as applicable, material certificates are provided and maintained in the laboratory record system
- Identifying and storing materials
- Verifying that material storage is properly maintained, and removing materials from use when shelf life has expired.

4.1 REQUIREMENTS FOR REAGENTS, SOLVENTS. AND GASES

Chemical reagents, solvents, and gases are available in a variety of grades of purity, ranging from technical grade to ultrapure grades. The purity required varies with the type of analysis. The parameter measured and the sensitivity and specificity of the detection system are important factors in determining the purity required. For many analyses, including most inorganic analyses, analytical reagent (AR) grade is satisfactory. Other analyses, such as trace organic and radiological,

frequently require special ultrapure reagents, solvents, and gases. If the analytical method does not specify the purity of materials, it is generally intended that analytical grade be used. Materials of lesser purity than specified shall not be used. Prior to performing an analysis, the label of the material container should be checked by the Analyst to verify that the material purity meets analytical needs.

Materials are prepared and standardized against reliable primary standards whenever possible. They are restandardized, or prepared fresh, as often as indicated by their stability.

4.1.1 General Inorganic Analyses

In general, AR grade reagents and solvents are adequate for inorganic analyses. Primary standard reagents shall be used for standardizing all volumetric solutions. All prepared reagents shall be checked for accuracy.

Individual analytical methods specify the reagents that require frequent standardization, or special treatment. The Analyst must comply with these special operations. To minimize potential deterioration, the Analyst should prepare a limited volume of such reagents, depending on the quantity required over a given period of time.

4.1.2 Trace Metals Analyses

All standards used for atomic absorption and emission spectroscopy shall be spectro-quality. It is recommended that other reagents and solvents also be spectro-quality. Standards are prepared by the Analyst, or purchased provided purchased materials meet the requirements of the analytical method.

In general, fuel and oxidant gases used for atomic absorption can be commercial grade. Compressed air can be commercially supplied, dry grade, or supplied by laboratory air compressors if adequate pressure is

maintained and the air is filtered to remove oil, water, and possible trace metals.

4.1.3 Organic Chemical Analyses

In general, pesticide grade (nanograde) is the minimum acceptable grade for materials used for organic analyses. Reference grade standards should be used as necessary. Special note should be made of the assay of standard materials.

Some gas chromatography (GC) detectors require that solvents, standards, and samples be free of certain classes of compounds. For example, use of the flame photometric detector requires that reagents and solvents be free of sulfur and phosphorus interference.

For sample cleanup procedures, the adsorbents most commonly used for column chromotographic cleanup of sample extracts are Florisil, carbon, silica gel, and alumina. These are preactivated according to the analytical method requirements and checked for interfering constituents. The contents of each solvent lot must be checked to determine suitability for the analyses. Similarly, all analytical reagents and other chemicals must also be routinely checked.

4.1.4 Water

Deionized water is used as appropriate for dilution, preparation of reagent solutions, and final rinsing of glassware as described in Laboratory-Specific Attachments to this Quality Assurance Manual. Distilled water is usually not of sufficient purity because distillation does not remove certain contaminants. The specifications for ASTM Type II quality water shall be met: Maximum Electrical Conductivity at 25°C of 1.0 µmho/cm, or Minimum Electrical Resistivity at 25°C of 1.0 M ohm/cm. Water quality shall be determined daily by measuring either conductivity or resistivity and recording measurements in a log book.

Organic-free water is required for microbiological and volatile organic analyses. Organic-free water may be verified by the purge-and-trap technique on the GC. However, when determining trace organics by solvent extraction and gas chromatography, specialty water such as high performance liquid chromatography (HPLC) grade water with sufficiently low background must be used. Preextraction of the water with the solvent used in the analysis, or nitrogen purging, may be helpful in eliminating organic compounds from the water.

4.1.5 Compressed Air

Compressed air must be free of oil, water, and dirt. If produced in the laboratory, appropriate filters shall be used at the compressor to prevent moisture from entering the piping system. Filters to remove oil shall be installed at the point of use for atomic absorption and gas chromatographic applications.

If purchased, or as a backup to an in-house compressor system, compressed air shall be high quality, dry grade.

4.2 REQUIREMENTS FOR LABORATORY CONTAINERS

Containers used in the laboratories can affect the quality of results. Material composition and volumetric tolerances are discussed below.

4.2.1 Material Composition of Laboratory Vessels

Soft glass containers are not recommended for general use, especially for the storage of reagents. The glass recommended for general use is chemically resistant borosilicate glass, such as is manufactured under the trade names of Pyrex or Kimax. This glassware is satisfactory for analyses performed by an ITAS laboratory unless otherwise noted in the sampling or testing procedure.

The use of plastic vessels; containers; and other apparatus made of Teflon, polyethylene, polystyrene, and polypropylene is desirable for

certain specified applications.

The following guidelines should be considered when selecting the material composition of laboratory vessels:

- Borosilicate or polyethylene bottles are to be used for the storage of reagents and standard solutions, unless otherwise specified.
- Plastic containers should not be used for reagents and solvents used in organic analyses.
- Dilute metal solutions have a tendency to plate out on container walls over long periods of time; therefore, dilute metal standard solutions should be prepared at the time of analysis.
- The use of disposable glassware is satisfactory for some analyses, such as the use of disposable test tubes as sample containers for use with some automatic samplers.
- Borosilicate glassware is not completely inert, particularly to alkalis. Standard solutions of silica, boron, and the alkali metals should be stored in polyethylene bottles.

4.2.2 Volumetric Container Specifications

ITAS laboratories shall use glassware of sufficient accuracy as required for the analytical procedure for the measurement of sample or reagent volumes. This includes volumetric flasks, volumetric pipets, and accurately calibrated burets. Less accurate types of glassware, including graduated cylinders and serological and measuring pipets, have specific uses when less exact volumes are permitted by the analytical procedure.

In general, volumetric containers will not be calibrated by ITAS unless required by a specific analytical method. However, containers, primarily glassware, shall be purchased with the objective of meeting the correct end use of the container in an analytical procedure. Thus, for

example, if an analytical method requires that Class A glassware be used, the Analyst shall fully comply with the method.

4.3 STORING AND MAINTAINING REAGENTS AND SOLVENTS

The following shall apply for storing and maintaining reagents and solvents:

- Standard reagents and solvents are stored in accordance with manufacturer's recommendations
- Standard solutions shall be stored separately from samples to avoid possible cross-contamination
- Light-sensitive standard reagents or solvents are stored in a cool, dark place
- Materials used for radiological determinations are stored in dark bottles
- Organic reagent standards are stored at or below 4°C
- Organic reference materials are stored in a freezer (at or below 0°C)
- Adsorbents for thin-layer and column chromatography are stored in the containers in which they are supplied, or according to the requirements of individual analytical methods
- When fresh stock solutions are required, dilutions of the new standard are compared to the current standard or an independent Standard Reference Material to determine consistency or document traceability
- Standards are not maintained longer than recommended by the manufacturer or as specified in the analytical method.

4.4 GLASSWARE CLEANING REQUIREMENTS

Methods of cleaning glassware are selected according to the substances that are to be removed and the analytical analysis required. Water-soluble substances can be washed out with hot or cold water and the

vessel finally rinsed with successive small amounts of deionized water. Other substances more difficult to remove may require the use of a detergent, organic solvent, dichromate cleaning solution, nitric acid, or aqua regia. In all cases, it is good practice to rinse a vessel with tap water followed by deionized water as soon as possible after use. Material allowed to dry on glassware is much more difficult to remove.

Chromic acid is an effective but dangerous cleaning agent. It may be prepared by adding 1 liter of concentrated sulfuric acid slowly, with stirring, to a 35-ml saturated sodium dichromate solution. This mixture must be allowed to stand for approximately 15 minutes in the vessel to be cleaned and may then be returned to a storage bottle. Following the chromic acid wash, the vessels are rinsed thoroughly with tap water, then with small successive portions of deionized water.

For certain determinations, especially trace metals, glassware shall also be rinsed with a 1:1 nitric acid-water mixture. This is followed by thoroughly rinsing with tap water and successive portions of deionized water. This may require as many as 12 to 15 rinses, especially if chromium is determined. A nitric acid rinse is also especially important if lead is to be determined.

Glassware used for phosphate determinations should not be washed with detergents containing phosphates. This glassware must be thoroughly rinsed with tap water and deionized water. For ammonia and Kjeldahl nitrogen determination, the glassware must be rinsed with ammonia-free water.

Glassware used in the determination of trace organic constituents in water, such as chlorinated pesticides, should be as free as possible of organic contaminants. A chromic acid wash of at least 15 minutes is necessary to destroy these organic residues. Rinse thoroughly with tap water and, finally, with deionized water. Glassware may be dried for

immediate use by rinsing with redistilled acetone. Otherwise, non-volumetric glassware may be oven dried or placed in the muffle furnace at 400°C for two hours. Glassware should be stored immediately after drying to prevent any accumulation of dust and stored inverted or with the mouth of the glassware covered with foil.

Bottles used for the collection of samples for organic analyses should be rinsed successively with chromic acid cleaning solution; tap water; deionized water; and, finally, several times with a redistilled solvent such as acetone, hexane, petroleum ether, or chloroform. Caps should be washed with detergent; rinsed with tap water, deionized water, and solvent. Liners are treated in the same way as the bottles and are stored in a sealed container.

Alternate methods for cleaning may be used if it is demonstrated (such as by blank analysis) that the result is satisfactory. Also, disposable or precleaned glassware may be purchased if applicable to the analytical procedure.

5.0 SAMPLE RECEIPT AND INITIATION OF TESTING PROGRAM

Laboratory analyses are performed to produce data representative of conditions when the sample was obtained. To provide representative samples for analysis, both field and laboratory personnel must satisfactorily perform their activities. Although the purpose of this manual is to define the laboratory Quality Assurance Program, the interrelationship of field and laboratory operations in maintaining sample integrity is briefly discussed because the effect of field operations upon resulting data quality cannot be totally separated from laboratory operations. Figure 5-1 shows the sample collection, transport, and holding process. The steps presented are the basis for the following discussion.

5.1 FIELD COLLECTION AND SHIPMENT

Prior to collecting samples, the collection team must consider the analyses to be performed and the "hazard level" of the media being sampled so that proper sample containers and shipping containers can be assembled and the proper preservatives added to samples. In addition, field logs and record sheets, Chain of Custody forms, and Request for Analysis records must be assembled.

All records required for documentation of field collection must be completed by the field team. Several of the documents that affect laboratory operations are discussed herein. The primary documenting record is the Chain of Custody form.

In addition to initiating the Chain of Custody form, field personnel are responsible for uniquely identifying (required on the Chain of Custody form) and labeling samples, providing proper preservation, and packaging samples to preclude breakage during shipment.

Referring to Figure 5-2, every sample shall be labeled to identify:

- Project number
- Unique sample number

- Sample location (such as borehole and depth, or grid coordinates)
- Sampling date and time
- Person obtaining the sample
- Method of sample preservation/conditioning.

Samples must be placed in containers compatible with the intended analysis and properly preserved to maintain sample integrity. Also, the collection of samples must consider the time interval between acquiring the sample and analysis (holding time) so that the sample is representative. Table 5-1 provides requirements for various analytical parameters with respect to the type of container, preservation method, and maximum holding time between collection and analysis.

As can be seen, polyethylene or glass containers are required; and, in most cases, samples must be cooled to four degrees Centigrade. The table also provides the recommended sample volume for a specific analysis.

High hazard (medium/high concentration) samples must be packaged according to DOT regulations. The ITAS Manual of Practice: "Sample Packaging and Shipment" should be used, in conjunction with 49 CFR, Parts 100-177, when packaging hazardous samples. The Manual also presents detailed instructions on packaging non-hazardous or environmental samples.

Shipping containers are to be sealed with custody tape prior to shipment (see Figure 5-5), whether shipped by direct transport by field personnel or commercial carrier. The only exception to this is if sufficient holding time exists so that the samples can be held in the field; then it is necessary to re-ice the containers and seal them prior to transport.

As soon as field personnel are ready to transport samples from the field to the laboratory, they shall notify the laboratory by telephone of the shipment. If the samples are transported by field personnel, the estimated time of arrival at the laboratory should be given. If the samples are shipped by commercial carrier, the laboratory should be telephoned as soon as the shipping containers are consigned to the shipper.

The final step in providing information to the laboratory is shown on Figure 5-3. The Request for Analysis form shall be completed by the field personnel and included with the Chain of Custody form. The QC level required (see Section 8.3) should be stated on this form. It is imperative that the Request for Analysis form be provided so that analytical requirements are defined and sample holding times are not exceeded. The analyses requested should be consistent with the project work plan, or Quality Assurance Project plan; the Request for Analysis form takes precedence if these documents conflict.

5.2 CHAIN OF CUSTODY

An overriding consideration for resulting data is the ability to demonstrate that the samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Evidence of collection, shipment, laboratory receipt, laboratory custody and disposal must be documented to accomplish this. Documentation is accomplished through a Chain of Custody record that lists each sample and the individuals responsible for sample collection, shipment, and receipt. A sample is considered in custody if it is:

- In a person's actual possession
- In view after being in physical possession
- Locked or sealed so that no one can tamper with it after having been in physical custody
- In a secured area, restricted to authorized personnel.

Figure 5-4 is the Chain of Custody form to be used by IT personnel in collecting and shipping samples. An ITAS laboratory shall not accept samples collected by IT personnel for analysis without a correctly prepared Chain of Custody form.

The Chain of Custody form shall be signed by each individual who has the samples in their possession. Preparation of the Chain of Custody shall be as follows:

- The Chain of Custody record shall be initiated in the field by the person collecting the sample, for every sample. Every sample shall be assigned a unique identification number that is entered on the Chain of Custody form. Samples can be grouped for shipment and use a common form. The form on Figure 5-4 allows for ten samples. If more than ten samples are shipped in the same container, more than one Chain of Custody form is required.

- The record shall be completed in the field to indicate project, sampling team, etc.

- If the person collecting the sample does not transport the samples to the laboratory or deliver the sample containers for shipment, the first block for Relinquished By _____, Received By _____ shall be completed in the field.

- The person transporting the samples to the laboratory or delivering them for shipment shall sign the record form as Relinquished By _____.

- If the samples are shipped to the laboratory by commercial carrier, the Chain of Custody form shall be sealed in a watertight container, placed in the shipping container, and the shipping container sealed prior to giving it to the carrier.

- If the samples are directly transported to the laboratory, the Chain of Custody form shall be kept in possession of the person delivering the

samples.

- For samples shipped by commercial carrier, the waybill shall serve as an extension of the Chain of Custody record between the final field custodian and receipt in the laboratory.

- Upon receipt in the laboratory, the Sample Custodian, or representative, shall open the shipping containers, compare the contents with the Chain of Custody form, and sign and date the record. Any discrepancies shall be noted on the Chain of Custody form.

- If discrepancies occur, the samples in question shall be segregated from normal sample storage and the field personnel immediately notified.

- The Chain of Custody form is completed after sample disposal. Note the blocks for this purpose on Figure 5-4.

- Chain of Custody records shall be maintained with the records for a specific project, becoming part of the data package.

Multipart Chain of Custody forms may be used so that a copy (yellow) remains with the person shipping the samples.

5.3 LABORATORY SAMPLE RECEIPT

The first step in the laboratory receipt of samples is notification of the Operations Manager or Group Leaders of the incoming samples.

Upon sample receipt, the Sample Custodian, or representative shall:

- Note whether the samples were shipped as hazardous materials; if so, the sample container should be opened in a hood, the Custodian should wear gloves and a respirator in case of improper packing or breakage.

- Examine the shipping containers to verify that the custody tape is intact. If not, the sample shipper is notified. This nonconformance is

documented, together with corrective actions and notification of field personnel, and Lab and Project Management. Figure 5-5 shows examples of custody seals.

- Examine all samples and determine if proper temperature has been maintained during shipment. Document temperatures on the Chain of Custody forms. If samples have been damaged during shipment, the remaining samples shall be carefully examined to determine whether they were affected. Any samples affected shall also be considered damaged. It will be noted on the Chain of Custody form that specific samples were damaged and that the samples were removed from the sampling program. Field personnel will be notified as soon as possible that samples were damaged and that they must be resampled, or the testing program changed, and an estimate of the cause of damage.

- Compare samples received against those listed on the Chain of Custody form.

- Verify that sample holding times have not been exceeded.

- Sign and date the Chain of Custody form and attach the waybill.

- Attach appropriate laboratory sample container labels with test and preservative information (Figure 5-6).

- Place the samples in adequate laboratory storage.

- Enter the samples in the laboratory sample log-in book, and/or the sample management computer system which contains the following information:

  - Project identification number
  - Sample numbers
  - Type of samples
  - Date received in laboratory
  - Date put into storage after analysis is completed
  - Date of disposal.

The last two items will be added to the log when the action is taken.

- Notify the Operations Manager or Group Leaders of sample arrival.

- Place the completed Chain of Custody records in the project file.

If samples collected by IT personnel arrive without Chain of Custody forms or with incorrect Chain of Custody records or broken custody seals, the following shall be done by the Sample Receiving Clerk:

- If the Chain of Custody form is incorrect or a custody seal is broken, a telephone call is made to the Project Manager and field personnel, stating the nonconformance reporting and correction. A record of the call memorandum must be signed and dated by the Sample Custodian and the QC Coordinator. This will serve as an amendment to the Chain of Custody for the project file. If the information on the Chain of Custody form cannot be corrected by the Sample Receiving Clerk, the samples affected shall be removed from the sampling program.

- If the Chain of Custody form is not shipped with the samples, the field personnel shall be called by the Sample Receiving Clerk and a memorandum prepared by field personnel which lists the persons involved in collecting, shipping, and receiving the samples and the times, dates, and events. Each person involved must sign and date this memorandum. The completed memorandum will be maintained in lieu of the Chain of Custody.

5.4 LABORATORY STORAGE OF SAMPLES

The primary considerations for sample storage are:

- Maintenance at prescribed temperature, if required, which is typically four degrees Centigrade.

- Extracting and/or analyzing samples within the prescribed holding time for the parameters of interest.

The requirements of Table 5-1 for temperatures and holding times shall be used. Placing of samples in the proper storage environment is the responsibility of Sample Receiving personnel who should notify the Operations Manager, or Group Leaders, if there are any samples which must be analyzed immediately because of holding-time requirements.

5.5 INITIATION OF TESTING PROGRAM

As stated in Section 5.1, a Request for Analysis form shall be submitted with the samples to the laboratory. If the analytical program is not defined with the sample shipment, the Sample Receiving Clerk shall immediately notify the Operations Manager or designee for definition of the analysis program; if the samples were collected by IT personnel, the Project Manager and field personnel are notified. If the samples are external to IT Corporation, the Operations Manager or designee shall contact the client to determine the testing program.

The analytical program shall be entered in the laboratory Master Analytical Logbook and/or computerized information manangement system which includes the following information; project number and name; project contact name; date received; received by; submitted by; report to; requested report date; sample identifications; sample parameters; comments; special instructions.

The Operations Manager and Group Leaders are responsible for prioritizing samples on the basis of holding time and required reporting time into the laboratory sample stream.

5.6 SAMPLE DISPOSAL

The chain of custody for the sample is completed as part of sample disposal; these may take one form of a list of project samples signed upon disposition. There are several possibilities for sample disposition:

- The sample may be completely consumed during analysis.
- Samples may be returned to the client or location of sampling for disposal.
- The samples may be stored after analysis. Proper environmental control and holding time must be observed if reanalysis is anticipated. If reanalysis is not anticipated, environmental conditions for storage will not be observed.In general, ITAS will not maintain samples and extracts longer than sixty days beyond completion of analysis, unless otherwise specified.
- Disposal by the laboratory.

The Operations Manager shall determine disposition of samples if not specified in the Request for Analysis, Figure 5-3. Disposition must meet IT Corporation policies which are outlined below.

When samples are removed from storage for disposal, they become classified as hazardous wastes. Laboratory wastes include several types of material: precisely known substances from quality control analyses; unused portions of samples that may or not have been completely analyzed; and solvents, filter media, contaminated protective clothing, etc. used in the analytical process. Certain quantitites of some laboratory wastes must be identified as hazardous because of their general characteristics (ignitability, corrosivity, reactivity, or EP toxicity) or because they are specifically listed in 40 CFR Part 261 due to acute or toxic hazards. Some laboratories composite compatible waste and conduct more thorough analyses of the larger composited quantity to more completely identify their wastes. Certain laboratory waste may be disposed of in a sanitary sewer pursuant to 40 CFR 261.3(a)(2)(iv), for laboratories that connect to a facility wastewater treatment or pretreatment system. Careful review of local, state, and federal wastewater treatment regulations, and a knowledge of the material being disposed of, is required before this disposal option is taken.
A container should be chosen for the wastes in which the wastes can be

ultimately shipped, so it will not be necessary to transfer hazardous materials from one container to another. The laboratory must provide at least enough information about container contents (an inventory listing is appropriate) to properly manifest hazardous waste shipments. Federal Regulations of the Department of Transportation (49 CFR Transportation) apply to the packaging and labeling and shipment of these materials. All labels and markers must meet DOT specifications in accordance with 49 CFR 172.300 and 172.400. Shipping papers (which may be a shipping order, a bill of lading, a manifest, or other document) must contain the information required by Section 172.202, 173.203, 172.204 of the regulation. A copy of the hazardous waste manifest, identifying all wastes, must accompany each shipment of waste from pick-up to destination. Accompanying the manifest should be a contents listing describing each container's contents.

All operations should be performed in a safe manner in accordance with applicable federal, state, local, and IT requirements. Personnel who handle and pack hazardous waste should be trained regarding chemical incompatibility, spills, first aid procedures and use of personal protective equipment. Training records on current personnel must be kept until closure of the facility and training records on former employees must be kept for at least 3 years from the date the employee last worked there.

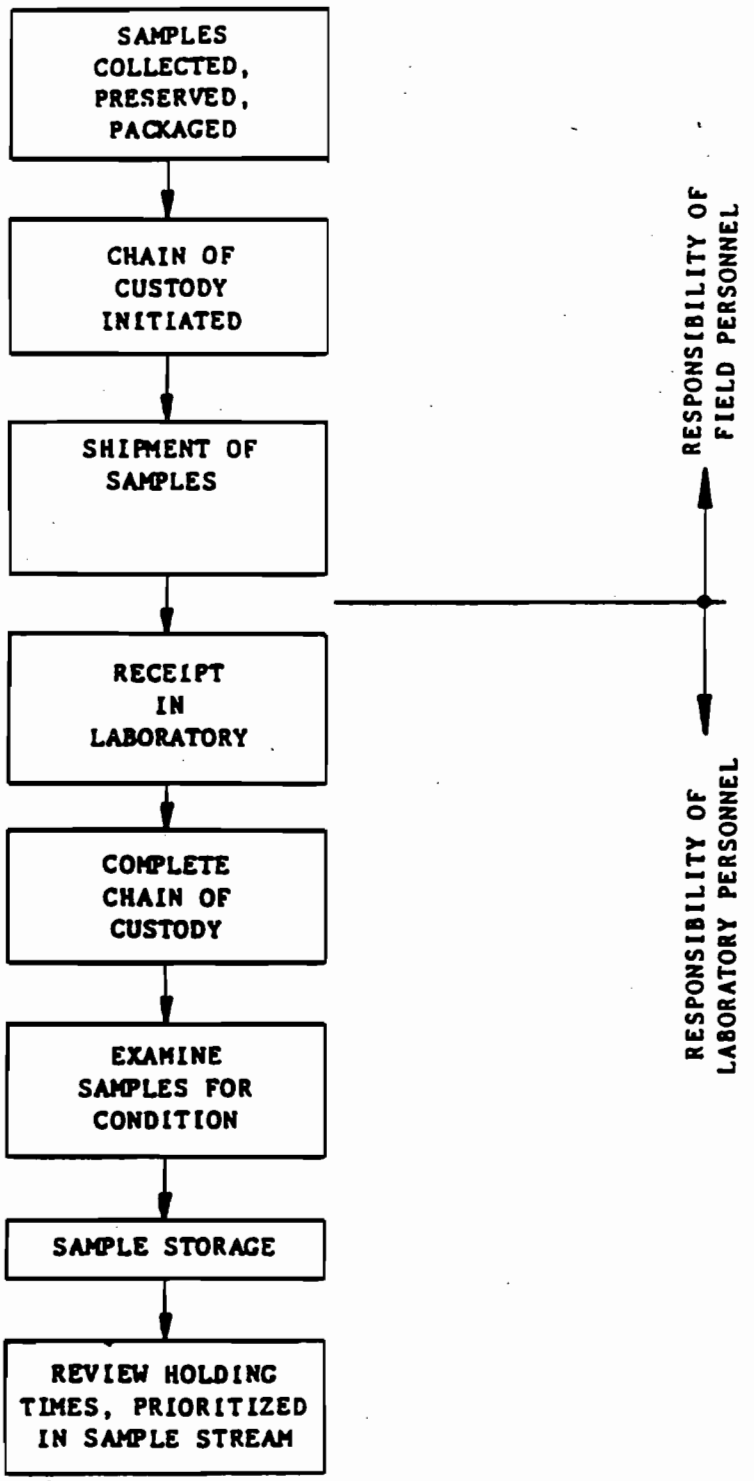


FIGURE 5-1
SAMPLE COLLECTION, TRANSPORT, AND HOLDING FLOW CHART

IT CORPORATION

Project Name _____

Project No. _____

Sample No. _____

Collection Date/Time _____

Collector's Name _____

Sample Location _____

Sample Type/Depth/Description _____

_____ Preservative _____

Analyze For _____

Bottle _____ of _____ _____ Filtered _____ Nonfiltered

FIGURE 5-2

EXAMPLE - SAMPLE FIELD LABEL

INTERNATIONAL TECHNOLOGY CORPORATION

REQUEST FOR ANALYSIS

R/A Control No.
C/C Control No. _____

PROJECT NAME _____
PROJECT NUMBER _____
PROJECT MANAGER _____
BILL TO _____

PURCHASE ORDER NO. _____

DATE SAMPLES SHIPPED _____
LAB DESTINATION _____
LABORATORY CONTACT _____
SEND LAB REPORT TO _____

DATE REPORT REQUIRED _____
PROJECT CONTACT _____
PROJECT CONTACT PHONE NO. _____

Sample No.	Sample Type	Sample Volume	Preservative	Requested Testing Program	Special Instructions

TURNAROUND TIME REQUIRED: (Rush must be approved by the Project Manager.)

Normal _____ Rush _____ (Subject to rush surcharge)

POSSIBLE HAZARD IDENTIFICATION: (Please indicate if sample(s) are hazardous materials and/or suspected to contain high levels of hazardous substances)

Nonhazard _____ Flammable _____ Skin Irritant _____ Highly Toxic _____ Other _____ (Please Specify)

SAMPLE DISPOSAL (Please indicate disposition of sample following analysis. Lab will charge for packing, shipping, and disposal.)

Return to Client _____ Disposal by Lab _____

FOR LAB USE ONLY

Received By _____ Date/Time _____

INTERNATIONAL TECHNOLOGY CORPORATION

CHAIN-OF-CUSTODY RECORD

R/A Control No. _____

C/C Control No. [illegible]

PROJECT NAME/NUMBER _____ LAB DESTINATION _____

SAMPLE TEAM MEMBERS _____ CARRIER/WAYBILL NO. _____

Sample Number	Sample Location and Description	Date and Time Collected	Sample Type	Container Type	Condition on Receipt (Name and Date)	Disposal Record No.

Special Instructions _____

Possible Sample Hazards _____

SIGNATURES (Name, Company, Date and Time)

1 Relinquished By: _____ 3 Relinquished By: _____

Received By: _____ Received by: _____

2 Relinquished By: _____ 4 Relinquished By: _____

Received By: _____ Received By: _____

FIGURE 5-4

CHAIN OF CUSTODY RECORD FORM

WHITE - To accompany samples
YELLOW - Field copy

SAMPLES
INTERNATIONAL TECHNOLOGY ANALYTICAL SERVICES

INTERNATIONAL TECHNOLOGY CORPORATION	LABORATORY SAMPLES Do Not Tamper Date:
	Signature:
	Name & Title (PRINT):

FIGURE 5-5

EXAMPLE - CUSTODY SEALS

International Technology Corp.
165 Fieldcrest Ave.
Edison, New Jersey 08818-7809

CLIENT	WORK-ORDER	FRAC	DATE-RECEIVED
	[illegible]0144	02AS	02/27/87

CLIENT NAME:
SAMPLE ID: A1-0-[illegible]
DATE/TIME COLLECTED: 02/26/87
Old Lot # [illegible]194

TESTS:

[illegible]	609AB	[illegible]	625	625A	AS
AS	BE	CD	CNT_R	CNT_S	CR
CU	DGST_S	DGS_AS	DGS_CU	HG	NI
PB	PL_P	PL_S	SB	SE	TL
TS	[illegible]				

No Preservative [] White
Sulfuric Acid [] Yellow
Nitric Acid [] Red
Phosphoric Acid [] Dark Blue
Sodium Hydroxide [] Green
Zinc Acetate [] Black
Sodium Thiosulfate [] Light Blue
Acetone Rinsed [] Brown
Other [] Orange

COLOR KEY

JOB# LOT# SAMPLE DATE SAMPLE# BOTTLE#

PROJECT NAME:

SAMPLE ID:

No Preservative ☐ White
Sulfuric Acid ☐ Yellow
Nitric Acid ☐ Red
Phosphoric Acid ☐ Dark Blue
Sodium Hydroxide ☐ Green
Zinc Acetate ☐ Black
Sodium Thiosulfate ☐ Light Blue
Acetone Rinsed ☐ Brown
Other ☐ Orange

COLOR KEY	SAMPLE NUMBER

FIGURE 5-6

EXAMPLE - LABORATORY SAMPLE CONTAINER LABELS

TABLE 5.1
SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	CONTAINER[a]	VOLUME REQUIRED (mL)	PRESERVATION[b]	MAXIMUM HOLDING TIMES[c]
Bacterial Tests				
o Coliform, fecal and total	P,G	200	Cool 4°C, 0.008% $Na_2S_2O_3$[d]	6 hours
o Fecal streptococci	P,G	200	Cool 4°C, 0.008% $Na_2S_2O_3$[d]	6 hours
Classical Wet Chemistry Tests				
o Acidity	P,G	50	Cool 4°C	14 days
o Alkalinity	P,G	50	Cool 4°C	14 days
o Ammonia	P,G	100	Cool 4°C, H_2SO_4 to pH <2	28 days
o Biochemical Oxygen Demand	P,G	1,000	Cool 4°C	48 hours
o Biochemical Oxygen Demand (carbonaceous)	P,G	1,000	Cool 4°C	48 hours
o Bromide	P,G	200	None required	28 days
o Chemical Oxygen Demand	P,G	75	Cool 4°C, H_2SO_4 to pH <2	28 days
o Chloride	P,G	50	None required	28 days
o Chlorine, Total Residual	P,G	200	None required	Analyze immediately
o Color	P,G	50	Cool 4°C	48 hours
o Cyanide, Total and Amenable to Chlorination	P,G	1,500	Cool 4°C, NaOH to pH >12, 0.6g ascorbic acid[d]	14 days[e]
o Fluoride	P	300	None required	28 days
o Hardness	P,G	100	HNO_3 to pH <2, H_2SO_4 to pH <2	6 months

MAN:sampling

See footnotes at end of table.

TABLE 5.1

(Continued)

PARAMETER	CONTAINER[a]	VOLUME REQUIRED (mL)	PRESERVATION[b]	MAXIMUM HOLDING TIMES[c]
o Hydrogen Ion (pH)	P,G	25	None required	Analyze immediately
o Kjeldahl and Organic Nitrogen	P,G	500	Cool 4°C, H_2SO_4 to pH <2	28 days
Metals[f]				
o Chromium VI	P,G	50	Cool 4°C	24 hours
o Mercury	P,G	100	HNO_3 to pH <2	28 days
o Metals, Except Chromium VI and Mercury	P,G	200	HNO_3 to pH <2	6 months
o Nitrate	P,G	100	Cool 4°C	48 hours
o Nitrate-Nitrite	P,G	100	Cool 4°C, H_2SO_4 to pH <2	28 days
o Nitrite	P,G	50	Cool 4°C	48 hours
o Oil and Grease	G	1,000	Cool 4°C, H_2SO_4 to pH <2	28 days
o Orthophosphate	P,G	50	filter immediately, cool 4°C	48 hours
o Oxygen, Dissolved Probe	G bottle and top	300	None required	Analyze immediately
o Phenols	G	500	Cool 4°C, H_2SO_4 to pH <2	28 days
o Phosphorus (Elemental)	G	50	Cool 4°C	48 hours
o Phosphorus, Total	P,G	50	Cool 4°C, H_2SO_4 to pH <2	28 days
o Residue, Total	P,G	100	Cool 4°C	7 days
o Residue, Filterable	P,G	100	Cool 4°C	7 days
o Residue, Nonfilterable	P,G	250	Cool 4°C	7 days

MAN:sampling See footnotes at end of table.

TABLE 5.1

(Continued)

PARAMETER	CONTAINER[a]	VOLUME REQUIRED (mL)	PRESERVATION[b]	MAXIMUM HOLDING TIMES[c]
o Residue, Settleable	P,G	1,000	Cool 4°C	48 hours
o Residue, Volatile	P,G	100	Cool 4°C	7 days
o Silica	P	50	Cool 4°C	28 days
o Specific Conductance	P,G	100	Cool 4°C	28 days
o Sulfate	P,G	100	Cool 4°C	28 days
o Sulfide	P,G	500	Cool 4°C, add zinc acetate plus sodium hydroxide to pH >9	7 days
o Sulfite	P,G	50	None required	Analyze immediately
o Surfactants	P,G	250	Cool 4°C	48 hours
o Temperature	P,G	1,000	None required	Analyze immediately
o Turbidity	P,G	100	Cool 4°C	48 hours
<u>Organic Tests</u>[g]				
o Organic Carbon	P,G	25	Cool 4°C, HCl or H_2SO_4 to pH <2	28 days
o Organic Carbon (TOC)	G, Amber bottle Teflon-Lined Cap	4 X 15	Cool 4°C, 1 ml of 1.1 M $Na_2S_2O_3$	28 days
o Purgeable Halocarbons	G,Teflon-lined septum	2 X 40	Cool 4°C, 0.008% $Na_2S_2O_3$[d] HCl pH 2[h]	14 days

MAN:sampling See footnotes at end of table.

TABLE 5.1

(Continued)

PARAMETER	CONTAINER[a]	VOLUME REQUIRED (mL)	PRESERVATION[b]	MAXIMUM HOLDING TIMES[c]
o Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	2 X 40	Cool 4°C, 0.008% $Na_2S_2O_3$[d], HCl to pH 2[h]	14 days
o Acrolein and Acrylonitrile	G, Teflon-lined septum	2 X 40	Cool 4°C, 0.008% $Na_2S_2O_3$[d], adjust pH to 4-5[i]	14 days
o Phenols[j]	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$[d]	7 days until extraction, 40 days after extraction
o Benzidines[j]	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$[d]	7 days until extraction[l] 40 days after extraction
o Phthalate Esters[j]	G, Teflon-lined cap	1,000	Cool 4°C	7 days until extraction, 40 days after extraction
o Nitrosamines[j,m]	G, Teflon-lined cap	1,000	Cool 4°C, store in dark, 0.008% $Na_2S_2O_3$[d]	7 days until extraction, 40 days after extraction
o PCBs[j]	G, Teflon-lined cap	1,000	Cool 4°C	7 days until extraction, 40 days after extraction
o Nitroaromatics and isophorone[j]	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$[d], store in dark	7 days until extraction, 40 days after extraction
o Polynuclear Aromatic Hydrocarbons[j]	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$[d], store in dark	7 days until extraction, 40 days after extraction
o Haloethers[j]	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$[d]	7 days until extraction, 40 days after extraction

MAN:sampling

See footnotes at end of table.

TABLE 5.1
(Continued)

PARAMETER	CONTAINER[a]	VOLUME REQUIRED (mL)	PRESERVATION[b]	MAXIMUM HOLDING TIMES[c]
o Chlorinated Hydrocarbons[j]	G, Teflon-lined cap	1,000	Cool 4°C	7 days until extraction, 40 days after extraction
o TCDD[j]---------- Tetrachlorodibenzo-p-dioxin	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$[d]	7 days until extraction, 40 days after extraction
o Total Organic Halogens	G, Teflon-lined Cap, Amber Bottle	4 X 15	Cool 4°C, H_2SO_4 to pH ≤ 2	14 days
Pesticides				
o Pesticides	G, Teflon-lined cap	1,000	Cool 4°C, pH 5-9[n]	7 days until extraction, 40 days after extraction
Radiological Tests				
o Alpha, Beta, and Radium	P,G	1,000	HNO_3 to pH <2	6 months

MAN:sampling

See footnotes at end of table.

TABLE 5.1

(Continued)

Reference: This table includes the requirements of the U.S. Environmental Protection Agency, as published in the Code of Federal Regulations, Vol. 49, No. 209, 40 CFR 136, October 26, 1984, pg, 43260.

(a) Polyethylene (P) or glass (G).

(b) Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automatic sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

(c) Samples should be analyzed as soon as possible after collection. The times listed are maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter period if knowledge exists to show this is necessary to maintain sample stability.

(d) Should only be used in the presence of residual chlorine.

(e)

Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustment to determine if sulfide is present.

TABLE 5.1

(Continued)

(f) Filtration, acid preservation, etc., are to be performed at the time the sample is collected or as soon as possible thereafter. For the determination of dissolved elements the sample must be filtered through a 0.45-μm membrane filter as soon as practical after collection. Acidify the filtrate with (1+1)HNO_3 to a pH of 2 or less. For the determination of suspended elements a measured volume of unpreserved sample must be filtered through a 0.45-μm membrane filter as soon as practical after collection. The filter plus suspended material should be transferred to a suitable container for storage and/or shipment. No preservative is required. For the determination of total or total recoverable elements, the sample is acidified with (1+1)HNO_3 to pH 2 or less as soon as possible, preferably at the time of collection. The sample is not filtered before processing.

(g) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

(h) Aqueous sample receiving no pH adjustment must be analyzed within seven days of sampling. Soils, sediments and sludge samples do not require acidification.

(i) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within three days of sampling.

(j) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to six to nine; samples preserved in this manner may be held for seven days before extraction and 40 days after extraction. Exceptions to the optional preservation and holding time procedure are noted in footnote (d) (re the requirement for thiosulfate reduction of residual chlorine) and footnotes (k) and (l) (re the analysis of benzidine).

TABLE 5.1

(Continued)

(k) If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.

(l) Extracts may be stored up to seven days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

(m) For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to seven to ten with NaOH within 24 hours of sampling.

(n) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extacted within 72 hours of collection. For the analysis of aldrin add 0.008% $Na_2S_2O_3$.

6.0 CALIBRATION PRACTICES

Instruments and equipment used in an ITAS laboratory are controlled by a formal calibration program. The program verifies that equipment is of the proper type, range, accuracy, and precision to provide data compatible with specified requirements. All instruments and equipment which measure a quantity, or whose performance is expected at a stated level, are subject to calibration. Calibration may be performed by ITAS personnel using reference standards, or externally by calibration agencies or equipment manufacturers.

This section of the QA Manual prescribes the practices used by an ITAS laboratory to implement a calibration program. Specifics are not provided herein because the requirements for the calibration of instruments and equipment are dependent upon the type and expected performance of individual instruments and equipment. Therefore, an ITAS laboratory shall use the guidelines provided herein to develop a calibration program as part of its Laboratory-Specific Attachment to the Quality Assurance Manual. Development and documentation of the laboratory calibration program is the responsibility of the Technical Director. Implementation is the responsibility of the Group Leaders and Analysts. The Quality Control Coordinator shall review the implementation of the program, as discussed in Section 14.0.

Two types of calibration are discussed in this section:

- Operational calibration which is routinely performed as part of instrument usage, such as the development of a standard curve for use with an atomic absorption spectrophotometer. Operational calibration is generally performed for instrument systems.

- Periodic calibration which is performed at prescribed intervals for equipment, such as balances and ovens. In general, equipment which can be calibrated periodically is a distinct, singular purpose unit and is relatively stable in performance.

6.1 CALIBRATION SYSTEM

Following is a discussion of the elements comprising the calibration system.

6.1.1 Calibration Procedures

Written procedures shall be developed by an ITAS laboratory within the requirements of this manual for all instruments and equipment subject to calibration. Whenever possible, recognized procedures, such as those published by ASTM or the USEPA, or procedures provided by manufacturers shall be adopted. If established procedures are not available, a procedure shall be developed considering the type of equipment, stability characteristics of the equipment, required accuracy, and the effect of operational error on the quantities measured. As a minimum, the procedures shall include:

- Equipment to be calibrated
- Reference standards used for calibration
- Calibration technique and sequential actions
- Acceptable performance tolerances
- Frequency of calibration
- Calibration documentation format.

6.1.2 Equipment Identification

Equipment that is subject to calibration shall be uniquely identified so that calibration records can be designated with a specific instrument. Equipment identification shall be by manufacturer's serial number, IT inventory control number, or a unique number assigned by an ITAS laboratory.

6.1.3 Calibration Frequency

Instruments and equipment shall be calibrated at prescribed intervals and/or as part of the operational use of the equipment. Frequency shall be based on the type of equipment, inherent stability, manufacturer's recommendations, values provided in recognized standards, intended data use, specified analytical methods, effect of error upon the measurement process, and prior experience.

6.1.4 Calibration Reference Standards

Two types of reference standards are used within ITAS laboratories for calibration:

- Physical standards, such as weights for calibrating balances and certified thermometers for calibrating working thermometers and ovens, which are generally used for periodic calibration

- Chemical standards such as Standard Reference Materials (SRMs) provided by the National Bureau of Standards (NBS) or the United States Environmental Protection Agency (USEPA). These may include vendor certified materials traceable to NBS or EPA SRMs. These are primarily used for operational calibration.

Whenever possible, physical reference standards shall have known relationships to nationally recognized standards (e.g., NBS) or accepted values of natural physical constants. If national standards do not exist, the basis for the reference standard shall be documented.

Physical reference standards shall be used only for calibration and shall be stored separately from equipment used in analyses. In general, physical reference standards shall be at least four to ten times as accurate as the requirements for the equipment which they are used to calibrate. In general, physical standards should be recalibrated every three years by a certified external agency.

Whenever possible, chemical reference standards shall be directly traceable to NBS SRMs. If SRMs are not available, compounds of vender-certified high purity will be used to prepare calibration standards.

6.1.5 Calibration Failure

Equipment that fails calibration or becomes inoperable during use shall be removed from service and segregated to prevent inadvertent use, or shall be tagged to indicate it is out of calibration. Such equipment shall be repaired and satisfactorily recalibrated before reuse. For equipment that fails

calibration, a record such as shown on Figure 6-1 shall be prepared and maintained with the equipment calibration file.

Scheduled calibration of equipment does not relieve the laboratory staff of the responsibility for using properly functioning equipment. If an equipment malfunction is suspected, the equipment shall be tagged and removed from service and recalibrated. If it fails recalibration, the above process shall apply.

6.1.6 Calibration Records

Records shall be prepared and maintained for each piece of equipment subject to calibration. Records demonstrating accuracy of preparation, stability, and proof of continuity of reference standards shall also be maintained.

Records for periodically calibrated equipment shall include, as appropriate:

- Identification number of equipment and type of equipment, or assigned unique equipment number
- Calibration frequency and acceptable tolerances
- Identification of calibration procedure used
- Date calibration was performed
- Identity of ITAS personnel and/or external agencies performing calibration
- Reference standards used for calibration
- Calibration data
- Certificates or statements of calibration provided by manufacturers and external agencies, and traceability to national standards
- Information regarding calibration acceptance or failure and any repair of failed equipment.

Records for periodically calibrated equipment shall be maintained in the Laboratory Operations Records as discussed in Section 12.0. Records for each instrument/equipment and physical reference standard shall be kept in a separate folder. The title sheet for each file shall be a summary of calibrations performed, such as shown in Figure 6-2. It is recommended that an index precede the equipment files which lists in matrix form all equipment and physical standards, calibration frequency, and dates for upcoming calibration. The use of a calibration due date matrix provides ready reference so that calibration can be maintained by the Group Leaders.

For instruments and equipment that are calibrated on an operational basis, calibration generally consists of determining instrumental response against compounds of known composition and concentration or the preparation of a standard response curve of the same compound at different concentrations. Records of these calibrations can be maintained in several ways:

- The calibration data can be kept with analytical sample data
- A log book can be prepared for each instrument which contains all calibration data.

The former method provides response factor information, etc., directly with analytical data so that the analytical data can be readily processed and verified. Also, the raw data package is complete as a unit. However, if samples from several projects are processed together, the calibration data must be copied and included with each group of data.

The latter method provides an ongoing record of the calibration undertaken for a specific instrument; however, to process and verify the analytical data the log must be used in conjunction with the raw data.

For operational calibration, the following is recommended:

- Calibration data be included with the raw analytical data. If samples from different projects are processed together, calibration data should be copied and included with each group of samples.

- A file is maintained for each instrument in the Laboratory Operating Records which includes:

  - Calibration instructions (curve preparation, linear range, etc.)

  - Procedures for chemical standards preparation

  - A bound calibration log book prepared by the Analyst which contains:

    a. Notice of calibration failure and repairs

    b. Midpoint standard response (as discussed in Section 8.0)

    c. Brief notice of calibrations performed.

The log book should be indexed in the Laboratory Operations Records as discussed in Section 12.0, but should be maintained at the instrument by the Analyst. All entries should be signed and dated by the Analyst.

6.2 OPERATIONAL CALIBRATION

Operational calibration is generally performed as part of the analytical procedure. Included may be the analysis of a method blank and the preparation of a standard response (standard calibration) curve. Operational calibration is dependent upon the instrumentation within an ITAS laboratory, and as previously discussed, each ITAS laboratory shall prepare a Laboratory-Specific Attachment to this manual which includes the Laboratory program for calibration.

Following is a brief discussion of the analysis of method blanks and preparation of standard curves. Guidelines for the major instrument systems within ITAS laboratories follow. (See also Table 6-1).

6.2.1 General Calibration Procedures

The initial phase of a laboratory testing program requires the selection and certification of the method best suited for an individual parameter. Certification, or verification, is the elimination, or minimizing, of determinate errors which may be due to Analyst error, the use of less-than-optimum equipment, reagents, solvents, or gases. The quality of materials, even though they are AR grade or better, may vary from one source to another. The Analyst must determine, through the use of reagent and/or solvent blanks, if materials are free from interfering substances which could affect the analysis. Other steps in certifying the method include the determination of a method blank and the preparation of a standard calibration curve.

6.2.1.1 Method Blank

After determining the individual reagent or solvent blanks, the Analyst defines the method blank to determine if the cumulative blank interferes with the analysis. The method blank is prepared by following the procedure step by step, including the addition of all of the reagents and solvents, in the quantity required by the method. If this cumulative blank interferes with the determination, steps must be taken to eliminate or reduce the interference to a level that will permit the combination of solvents and reagents to be used. If the blank interference cannot be eliminated, the magnitude of the interference must be considered when calculating the concentration of specific constituents in the samples analyzed.

A method blank should be determined whenever an analysis is made. The number of blanks is determined by the method of analysis and the number of samples analyzed at a given time. (See also Section 8.1.4.)

6.2.1.2 Preparation of Standard Calibration Curve

Concurrent with the preparation of reagent and method blanks, a standard calibration curve is prepared for the instrumentation. Preparation of a

standard calibration curve is accomplished by using calibration standards. The process is summarized as:

- Preparation of a standard calibration curve is accomplished by using calibration standards prepared by mixing the species to be analyzed into the solvent that is to be introduced into the instrument.
- The concentrations of the calibration standards are chosen to cover the working range of the instrument.
- All sample measurements are made within this working range.
- The calibration curve is prepared by plotting instrument response versus concentration of the species analyzed.
- Concentrations of the sample prepared with the same procedure are read directly from the calibration curve or determined by interpolation.

6.2.2 Instrument Calibration Procedures

This section outlines the minimum operations necessary to satisfy analytical requirements associated with the determination of specific parameters in water, soil/sediment, and hazardous waste samples. Table 6-1 presents summary calibration requirements for analytical instrumentation. Specific methods may have slightly different requirements. Details of calibration are described in Laboratory-Specific Manuals or in Standard Operating Procedures.

6.3 PERIODIC CALIBRATION

Periodic calibration shall be performed for equipment such as balances, thermometers, ovens, and furnaces that are required in analytical methods, but which are not routinely calibrated as part of the analytical procedure. Documentation of calibration shall be kept for each equipment item as discussed in Section 6.1.6.

Calibration requirements are determined within each ITAS laboratory

depending upon the equipment used and its operating function. Following are brief example discussions for the calibration of balances and thermometers with examples of calibration data sheets to serve as a guideline for the preparation of laboratory-specific procedures. (See also Table 6-2).

6.3.1 Balances (Example Procedure)

All balances shall be calibrated at least every three months using weights traceable to the National Bureau of Standards (NBS). Calibration weights shall be Class S or better and shall be recertified every three years. If balances are calibrated by an external agency, verification of their weights shall be provided.

Calibration of balances shall be to approximately 95 percent of balance capacity. Testing intervals over the operational range of the balance shall be approximately 5, 10, 15, 20, 25, 30, 40, 50, 60, 70, 80, 90, and 95 percent of capacity. Acceptance for balances which are direct reading to 0.01 gram shall be ±0.01 g for 0 to 100 g and ±0.1 percent of the applied weight over 100 g.

Figure 6-3 provides an example data sheet that can be used for balance calibration. Figure 6-4 provides a data form which can be used for calibration weight certification.

6.3.2 Thermometers (Example Procedure)

Certified, or reference, thermometers shall be maintained for use in calibrating working thermometers. Reference thermometers shall be provided with NBS traceability for initial calibration and shall be recertified every three years with equipment directly traceable to the NBS.

Working thermometers shall be compared with the reference thermometers every 12 months. In addition, working thermometers shall be visually

inspected by laboratory personnel prior to use.

Calibration temperatures and acceptance criteria shall be based upon the working range of the thermometer and the accuracy required for its use.

Figure 6-5 shows an example of a calibration record sheet for thermometer calibration.

IT ANALYTICAL SERVICES

NOTICE OF EQUIPMENT
CALIBRATION FAILURE

EQUIPMENT NUMBER _____

EQUIPMENT NAME _____

REASON FOR FAILURE TO PASS CALIBRATION _____

_____

_____

WAS EQUIPMENT REPAIRED OR REPLACED? _____

_____

IF REPAIRED, WAS THE CORRECTIVE ACTION ADEQUATE? _____

_____

_____

_____

_____

SIGNED _____

DATE _____

FIGURE 6-1
EXAMPLE CALIBRATION FAILURE NOTICE

IT ANALYTICAL SERVICES

LABORATORY EQUIPMENT CALIBRATION RECORD

EQUIPMENT NUMBER _____

EQUIPMENT NAME _____

_____

REQUIRED CALIBRATION PERIOD _____

DATE CALIBRATION PERFORMED	CALIBRATION PERFORMED BY	DATE CALIBRATION PERFORMED	CALIBRATION PERFORMED BY

FIGURE 6-2
EXAMPLE EQUIPMENT CALIBRATION RECORD

BALANCE CALIBRATION

EQUIPMENT NUMBER _____

EQUIPMENT NAME _____

DATE _____ DATE LAST CALIBRATED _____

CALIBRATION PERIOD _____

WEIGHTS APPLIED TO BALANCE	BALANCE READING	DOES BALANCE MEET STANDARD	WEIGHTS APPLIED TO BALANCE	BALANCE READING	DOES BALANCE MEET STANDARD

Signed _____

FIGURE 6-3

EXAMPLE - BALANCE CALIBRATION RECORD

WEIGHT CALIBRATION BY EXTERNAL AGENCY*

EQUIPMENT NUMBER _____

EQUIPMENT NAME _____

WEIGHT CLASS _____

NAME OF EXTERNAL AGENCY PERFORMING CALIBRATION _____

_____

DATE _____

PROVIDE IN THE TABLE BELOW THE LABELED WEIGHTS VERSUS THE CERTIFIED WEIGHT DETERMINATION. ALL CERTIFICATION RECORDS ARE TO BE ATTACHED TO THIS FORM.

WEIGHT AS LABELED	WEIGHT AS CERTIFIED	DOES WEIGHT MEET CLASS STANDARD?	WEIGHT AS LABELED	WEIGHT AS CERTIFIED	DOES WEIGHT MEET CLASS STANDARD?

* WEIGHT CALIBRATION IS TO BE PERFORMED EVERY THREE YEARS

FIGURE 6-4

EXAMPLE - WEIGHT CERTIFICATION RECORD

THERMOMETER CALIBRATION

EQUIPMENT NUMBER _____

EQUIPMENT NAME _____

DATE _____ DATE LAST CALIBRATED _____

CALIBRATION PERIOD _____

TEMPERATURE ACCORDING TO REFERENCE	THERMOMETER BEING CALIBRATED

WORKING RANGE _____

ACCEPTANCE CRITERIA _____ ± _____ °C

Signed _____

FIGURE 6-5

EXAMPLE - THERMOMETER CALIBRATION RECORD

Table 6.1. Summary of Operational Calibration Requirements

Instrument	Calibration Standards Used, Initial & Daily Minimum	Acceptance Limits	Corrective Actions	Reference[1]
Atomic Absorption Spectrophotometer	Initial: 3 levels + blank Daily: 1 check standard (midrange) per 10 samples;	Linear regression correlation coefficient >0.995; ±5% of true value	Make new standards or establish new calibration curve	1,2
Gas Chromatograph	Initial: 5 levels + blank Daily: 1 level of check standards standards	Calibration factor % RSD < 10%; ±10% of original curve	Make new standards, or establish new calibration curve	1
Inductively Coupled Plasma Emission Spectrophotometer	Initial: Manufacturer's instructions; Daily: Instrument check standard and calibration blank every 10 samples Weekly: QC sample, EPA/NBS source	Maximum response <0.006 nm of reference line, ±5% of original curve; blank within 2 standard deviations of limits. ±5% of true value	Establish new curve " Repeat twice; if average is outside control limit, then: Make new standards, and recalibrate	3 3 3
Ion Chromatograph	Initial: 3 levels ± blank Daily: 1 level of check standards every 20 samples (midrange)	±10% of original curve	Make new standards recalibrate	4
pH Meter	Daily: 3 levels	±0.05 pH unit	Clean or replace electrode; recalibrate	1
UV-Visible Spectrophotometer	Initial: 3 levels + blank Daily: Check standard Quarterly: Wavelength accuracy and photometric linearity	+10% of original curve Manufacturer specifications ±10% previous observations	Recalibrate Service Service	

MAM:PM-t-6-1

Table 6.1. (Continued)

Instrument	Calibration Standards Used, Initial & Daily Minimum	Acceptance Limits	Corrective Actions	Reference[1]
Infrared Spectrophotometer	Daily: Wavelength calibration and calibration curve, 6 levels + blank	±5 cm^{-1} true (Sadtler Index)	Service Make new standards, and recalibrate	5
Total Organic Carbon	Daily: 3 levels + blank	±10% of original curve	Make new standards	1
GC/MS	Initial: 5 levels + blank Daily: 1 level	%RSD <30% ±5% of initial curve	Make new standard recalibrations	1
Total Organic Halogen	Daily: 3 levels + blank	±10% of original curve	Make new standards	1

References:
1) EPA SW-846, "Test Methods for Evaluating Solid Waste"
2) USEPA Methods for Chemical Analysis of Water and Wastes, 1979
3) USEPA 200.7,1982
4) USEPA 300.0, 1984
5) USEPA "Procedure for the Evaluation of Environmental Monitoring Laboratories", 1978

Table 6.2. Summary of Periodic Calibration Requirements

Instrument	Calibration Frequency	Acceptance Limit	Corrective Actions
Analytical Balance	Daily: Sensitivity (with a Class "S" weight)	0.001 gm	Adjust, sensitivity
	Monthly: Reproducibility	Mean, Std. Dev. less than 0.1 mg	Service balance
	Monthly: Consistency	Difference less than 0.1 mg	Replace weights
	Quarterly: Class "S" weights Check	Difference less than 0.1 mg	Service balance
Autoclaves and Sterilizers	(a) Sterilization effectiveness check daily		Service
	(b) Temperature-recording device calibrated semi-annually		Service
	(c) Automatic timing mechanism checked monthly	±0.5 minutes	Service
Thermometers	Calibrate in constant temperature baths at two temperatures against precision thermometers certified by NBS annually	±0.5°C	Tag and remove from service, replace
Pipettors	Gravimetric check quarterly	High volume (>100 µL): ≤ 1.0% relative error and RSD Low volume (<100 µL): ≤ 2.0% relative error and RSD	Service or replacement

7.0 PREVENTIVE MAINTENANCE

Within ITAS laboratories, preventive maintenance is an organized program of actions (such as equipment cleaning, lubricating, reconditioning, adjustment and/or testing) taken to maintain proper instrument and equipment performance and to prevent instruments and equipment from failing during use. An adequate preventive maintenance program increases reliability of a measurement system. A preventive maintenance program considers the following:

- Instruments, equipment, and parts thereof that are subject to wear, deterioration, or other change in operational characteristics without periodic maintenance.
- Spare parts that should be available within the laboratory to minimize downtime.
- Frequency that maintenance is required.

The implementation of a preventive maintenance program is dependent upon the specific instruments and equipment used within an ITAS laboratory; therefore, this manual does not designate specific practices for instruments and equipment. Each ITAS laboratory shall prepare a preventive maintenance program which meets the guidelines presented in this section. The program shall be included in the Laboratory Specific Attachment to this Quality Assurance Manual.

Within a laboratory, the Operations Manager is responsible for preparation and documentation of the program. Group Leaders shall implement the program, and the QC Coordinator shall review implementation to verify compliance.

Each ITAS laboratory preventive maintenance program shall include the following:

- A listing of the instruments and equipment that are included in the program

- The frequency of maintenance considering manufacturer's recommendations and/or previous experience with equipment. The listing and maintenance frequency should be provided on a schedule such as Figure 7-1. Frequency should be stated in terms of monthly, quarterly, etc. following guidance in Table 7-1

- For each instrument in the program provide:

  - A list of spare parts maintained by the laboratory

  - External service contracts

  - Items to be checked and/or serviced during maintenance and directions for performing maintenance (if external service is not provided, or if not stated in manufacturer's instrument manuals).

Preventive maintenance should be documented as discussed below and the records stored in accordance with Section 12.2 of this manual. (See examples Figure 7-3 through 7-9). The master schedule (Figure 7-1) should be kept at the beginning of the maintenance records. Behind the schedule, a separate file should be maintained for each instrument. The instrument file should include:

- Spare parts list

- External service contracts

- Checklist of items to be serviced and directions for maintenance or manufacturer's instrument manuals

- Record/results of periodic maintenance.

The record of maintenance can be documented using Figure 7-2 or equivalent. Comments should note any parts which were replaced, observed deterioration, etc.

PREVENTIVE MAINTENANCE SCHEDULE

INSTRUMENT/EQUIPMENT	SERIAL NUMBER	MAINTENANCE FREQUENCY	MAINTENANCE REQUIRED	TO BE PERFORMED BY

FIGURE 7-1

EXAMPLE - PREVENTIVE MAINTENANCE SCHEDULE

INSTRUMENT _____

SERIAL NUMBER _____

MAINTENANCE FREQUENCY _____

MAINTENANCE PERFORMED BY	DATE	COMMENT

FIGURE 7-2

EXAMPLE - PREVENTIVE MAINTENANCE RECORD

pH METER/SIE

Maintenance Performed	Date/Initials				
Clean Electrode					
Calibrate / Check Slope					
Calibrate Meter. Electronics					

Clean Electrode Each Use

Check Slope Each Use

Calibrate Meter Electronics Monthly

FIGURE 7-3

EXAMPLE - pH METER/SIE
PREVENTIVE MAINTENANCE RECORD

CONDUCTIVITY METER

Maintenance Performed	Date/Initials				
Clean Cell					
Meter Span Check					

Clean Cell Each Use

Meter Span Check Monthly

FIGURE 7-4

EXAMPLE - CONDUCTIVITY METER PREVENTIVE MAINTENANCE RECORD

ATOMIC ABSORPTION SPECTROPHOTOMETER

Maintenance Performed	Date / Initials				
Clean Burner Head					
Empty Waste Receptacle					
Clean Nebulizer/ Burner Chamber					
Optics/Gen. Cleaning					
Gases Checked					
Air Purifier Drained					

Burner Head Cleaned Each Use

Waste Reservoir Emptied as Needed

Nebulizer/Burner Chamber Cleaned Weekly

Optics Cleaned by Contract Personnel Quarterly

Gases Checked Each Change of Tank

Air Purifier Drained Quarterly

FIGURE 7-5

EXAMPLE - ATOMIC ABSORPTION SPECTROPHOTOMETER PREVENTIVE MAINTENANCE RECORD

GC PREVENTIVE MAINTENANCE

Maintenance Performed	Date / Initials				
Detector Cleaned					
Hall					
FID					
Septa Changed					
Gases Changed					
Other (Specify) _____ _____ _____ _____					

Detector Cleaned as Needed

Septa Changed as Needed

Gases Changed as Needed

Others as Needed

FIGURE 7-6

EXAMPLE - GC PREVENTIVE MAINTENANCE RECORD

GC/MS PREVENTIVE MAINTENANCE

Maintenance Performed	Date / Initials				
Source Cleaned					
Septa Changed					
Turbo Oil Changed					
Pump Oil Changed					
Gases Changed					
Other (Specify) _____ _____ _____ _____					

Source Cleaned as Needed

Septa Changed as Needed

Turbo Oil Changed Quarterly

Pump Oil Changed Quarterly

Gases Changed as Needed

Others as Needed

FIGURE 7-7

EXAMPLE - GC/MS PREVENTIVE MAINTENANCE RECORD

SPECTROPHOTOMETER MAINTENANCE FORM

Maintenance Performed	Date / Initials				
Lamp Changed					
Wavelength Checked					
Cell Changed / Cleaned					
Service					

Lamp Changed as Needed

Wavelength Checked Quarterly

Cell Changed/Cleaned as Needed

Service as Needed

FIGURE 7-8

EXAMPLE - SPECTROPHOTOMETER PREVENTIVE MAINTENANCE RECORD

TOTAL ORGANIC CARBON ANALYZER

Maintenance Performed	Date / Initials				
Perchlorate Tubes Changed					
Ascarite Changed					
Clean Cell / Detachable Parts					
Insure Gas Quality					

Perchlorate and Copper Tubes - as Needed

Ascarite - as Needed

Cleaning Detachable Parts - Each Use

Gas Quality - Each Tank

FIGURE 7-9
EXAMPLE - TOTAL ORGANIC CARBON ANALYZER
PREVENTIVE MAINTENANCE RECORD

TABLE 7-1
PREVENTIVE MAINTENANCE GUIDANCE

INSTRUMENT	ITEM CHECKED/SERVICED	FREQUENCY
Atomic Absorption Spectrophotometer	3 point calibration performed	When serviced by repairman
	Burner head	Each shift
	Electrical	Each shift
	Lamps	Each shift
	Nebulizer	Each shift
	Optics	Quarterly
	Pump	Each shift
	Tygon tubing	Each shift
	Graphite tube	As necessary
	Replace graphite tube	As needed, or 80-100 Samples
	Replace contact rings	Quarterly
	Replace quartz windows	Semi annual
	Clean optics	Quarterly
	Align background lamp	Quarterly
	Check wave length	Quarterly
Gas Chromatograph	EC (Ni-63) wipe test	Semi annual
	Replace column packing	< 2 months
	Clean detector	As needed or EC Semi annually; FID monthly
	Change column	< 6 weeks
	Change glass wool plug	Weekly
	Clean insert	Weekly
	Replace septum	Daily
	Gases purity checks	Upon receipt of new cylinders
	Flow controller	Semi annually
	Purge and trap	As needed
	Change fuses	As needed
	Reactivate external carrier gas filler dryers	Weekly
	Reactivate flow controller filter dryers	Semi annually
	Clean and silanize or replace glass liners on injectors	As needed or quarterly
	Clean Detectors a) ECD	As needed
	b) FID/NPD	As needed or annually
	Clean Purge Vessel	As needed or monthly
	Replace Purge Vessel	As needed
	Bake Trap	As needed
	Replace Trap	Semi annually
	Replace carbon filter	Annually

Table 7-1 continued

Inductively Coupled Plasma Spectrophotometer	Sample introduction system	Daily
	Clean optics	Daily
	Clean burner heads	Daily
	Check pumps	Daily
	Check electronics	Daily
	Clean, realign torch	Monthly
	Clean nebulizer	Monthly
	Clean mixing chamber	Monthly
	Check nebulizer press	Monthly
	Replace pump tubing	Monthly
	Clean air filters	Monthly
Ion Chromatograph	Change plunger seals	Six months
	Check plumbing	Daily
	Oil pumps	Weekly (leak check monthly)
	Check filter (inlet)	Weekly
	Change column	Replaced < 2 weeks
	Check bed support	Change when pressure drops
	Change fuses	As needed
	Change pump motor	As needed
	Replace plunger seals	Semi annually
	Clean check valve	Semi annually
	Clean, replace solvent reservoir filter	Semi annually
	Degas pump head	As needed
	Clean, replace cell packing, degas floor cell	As needed or quarterly
Refrigerators	Temperature checked and logged	Daily
Walk-in Coolers	Temperature checked and logged	Daily
Balances	Service representative calibration	Annually
Deionized/Organopure Water	Conductivity check	Daily
	Ion exchange bed changed	Weekly
	Replace filters	As needed
Vacuum Pumps and Air Compressor	Check performance	Weekly
	Lubrication, belts, etc.	As needed
Analytical Balance	Internal Weight Train, Gears, Electronics	Annual Service

Table 7-1 continued

GC/MS	GC/MS maintenance is the same as GC with the following additions:	
	Diffusion pump oil	Bi-Weekly
	Mechanical pump oil	Quarterly
	Power Con. air filter	Bi-Weekly
	QEM filter	Bi-Weekly
	Water bay filter	Bi-Weekly
	Interface Box	Bi-Weekly
	Vacuum chaff filter	Monthly
	Turbo pump oil	Quarterly
	Water filter (if applicable)	Observe and change as needed
	Computer air filter	Monthly
	Card cage air filter	Monthly
	Source-clean ceramics, polish lenses	As needed
	Clean poles and ceramics on the poles	As needed
	Clean contacts on the component boards	As needed
	Vacuum the component boards	As needed
	Clean all fan screens	Weekly
	Vacuum outside of instrument	Weekly
	Clean grob and replace quartz insert	As needed
	Replace septum	Daily (each shift)
	Injection port liner checked	Daily
	Column maintenance	As needed
	Disk Drive	Semi-Annual (Service the Engineer) or as needed
	Printer	Quarterly

Table 7-1 continued

Infrared Spectrophotometer	Clean cells	Daily
pH Meter	Electronics checked	Daily
	Electrolyte changed	Checked weekly, changed when low
Threshold Limit Valve Meter (HCVP)	Check valves	Weekly
	Check pump	Weekly
	Check electronics	Weekly
	Check detector	Weekly
Total Organic Carbon Instrument	Check oxygen purity	Each new cylinder
	Check heater	
	Add phosphoric acid	Monthly
UV/VIS Spectrophotometer	Lamp	As needed
	Wavelength checked	Quarterly
	Serviced	As needed
HPLC	Carbon check	Daily
	Oil levels	Weekly
	Filters	Monthly
	Pump Drives	Quarterly

8.0 ANALYSIS OF QUALITY CONTROL SAMPLES

This section discusses samples which are routinely added to the normal laboratory sample stream to demonstrate that the laboratory is operating within prescribed requirements for accuracy and precision. Quality control samples are of known content and concentration (with the exception of field blanks) so that accuracy and precision can be determined and control charts can be prepared. Evaluation of these data are discussed in Section 10.1.

Table 8.1 summarizes the quality control samples which are analyzed in ITAS laboratories. Included in the table are:

- Type of sample.
- Purpose of the sample.
- Frequency with which the sample is to be analyzed within the normal sample stream.
- Applicability of the sample to organic or inorganic analyses, with a citation if the sample is for GC/MS analysis only.
- Whether the sample is used for the statistical evaluation of accuracy and/or precision.
- Person responsible for introducing the quality control sample into the sample stream. If the sample is introduced by the Quality Control Coordinator, the content and/or concentration of the sample and its occurrence is unknown to the Analyst. Samples unknown to the analyst provide independent verification of laboratory operation.

Following is a discussion of the major types of quality control samples. QC samples will be analyzed as recommended herein, unless analytical procedures prescribe specific sample analysis. If the procedure is specific, the procedural requirements will be met. A discussion of the ITAS interlaboratory testing program follows. Figures 8.1 and 8.2 show how different QC samples may be used to provide bias and precision estimates.

As stated, Section 10.1 presents the statistical analyses of these samples.

8.1 TYPES OF QUALITY CONTROL SAMPLES

Type of Quality Control Sample	Section No.
Trip Blank Analyses	8.1.1
Field Blank Analyses	8.1.2
Rinsate Blank Analyses	8.1.3
Method Blank Analyses	8.1.4
Reagent Blank Analyses	8.1.5
Bottle Blank Analyses	8.1.6
Duplicate Sample Analyses	8.1.7
Blind Duplicate Analyses	8.1.8
Check Standard Analyses	8.1.9
Surrogate Standard Analyses	8.1.10
Laboratory Matrix Spike Analyses	8.1.11
Laboratory Matrix Spike Duplicate Analyses	8.1.12
Verification/Reference Standard Analyses	8.1.13
Blank Spike Analyses	8.1.14
Field Matrix Spike Analyses	8.1.15
Analysis Matrix Spike Analyses	8.1.16
Internal Standard Spike Analyses	8.1.17
Collocated Samples Analyses	8.1.18
Replicated Sample Analyses	8.1.19
Split Sample Analyses	8.1.20

8.1.1 Trip (Travel) Blank Analyses

Volatile organics samples are susceptible to contamination by diffusion of organic contaminants through the teflon-faced silicone rubber septum of the sample vial; therefore, trip blanks (also referred to as travel blanks) shall be analyzed to monitor for possible sample contamination during shipment. Trip blanks will be prepared by filling two VOA vials (40 ml) with organic-free water and shipping the blanks with the field kit. Trip blanks accompany the sample bottles through collection and shipment to the laboratory and are stored with the samples. Following the analyses, if the trip blanks indicate possible contamination of the samples, depending upon the nature and extent of th contamination, the samples may be corrected for the trip blank concentration or t sources resampled. Results of trip blank analyses should be maintained with the corresponding sample analytical data in the project file.

8.1.2 Field Blank Analyses

A field blank is a volume of water (or soil) that is provided by the sample collectors to demonstrate the absence of contamination during sampling. Deionized, distilled laboratory water, or previously-prepared solid materials (lab sand, etc.) is placed into sample containers by the sample collection crew, packaged, and shipped with the other field samples. If analysis of the field blanks indicates possible contamination of the samples, depending upon the nature and extent of the contamination, the samples may be corrected for the field blank concentration or the sources resampled. Contamination sources to be checked include: Containers; sample storage facilities; field handling procedures; sampling tools (also see rinsate blanks). Results should be maintained with the corresponding sample analytical data in the project file.

8.1.3 Rinsate Blank Analyses

A rinsate blank is a volume of rinse solution (deionized, distilled lab water or organic solvent) used to rinse a sampling tool which contacts multiple samples. The rinse solution is collected after the tool has collected a sample and has been cleaned, to demonstrate that there is no residual contamination remaining on the tool to carry over into the next sample. If the rinsate blank indicates possible contamination of the succeeding samples, the samples may be corrected for the rinsate blank concentration or the sources resampled. Results of rinsate blank analyses should be maintained with the corresponding sample analytical data in the project file.

8.1.4 Method Blank Analyses

A method blank is a volume of deionized, distilled laboratory water for water samples, or a purified solid matrix for soil/sediment samples carried through the entire analytical procedure. The volume or weight of the blank must be approximately equal to the sample volume or sample weight processed. A method blank should be performed with each group of samples. Analysis of the blank verifies that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware are known and

minimized. Optimally, a method blank should contain no greater than two times (2X) the method detection limit for the parameter. Results of method blank analyses should be maintained with the corresponding analytical data in the project file. (See also Section 6.2.1.1).

8.1.5 Reagent Blank Analyses

A reagent blank is composed of the materials which will be added to client samples during preparation, and then will be analyzed for specific parameters. It is run prior to the use of the materials on "real" samples, to verify that no contaminants are present at levels which would affect sample results. If such contaminants are found, corrective actions must be taken. Records of reagent blanks, including solvent lots and column adsorbent test results, are stored in Quality Operation files. The reagent blank may be run as a method blank with client samples, but if contamination is present, samples, will require reanalysis with a clean blank demonstrating reagent purity.

8.1.6 Bottle Blank Analyses

At a frequency of 1 percent or greater, laboratory-prepared sample containers are tested to verify that the glassware cleaning procedure is performed acceptably. Parameters of concern for the particular container are tested (metals for plastic containers, organics for glove, etc.). Results of the tests from each batch are filed with the shipping/receiving clerk or sample custodian. Records of traceability of these containers through each project must be maintained. Vendors must supply certificate of cleanliness for purchased, pre-cleaned, containers, and these records must also be maintained.

8.1.7 Duplicate Sample Analyses

Duplicate analyses are performed to evaluate the precision of an analysis. Results of the duplicate analyses are used to determine the relative percent difference between replicate samples. Criteria for evaluating duplicate sample results are provided in Section 10.1. A duplicate analysis should be performed whenever a group of five or more samples are analyzed at one time.

Duplicate analysis results should be summarized on the quality control data summary form.

8.1.8 Blind Duplicate Analyses

A blind duplicate sample is a duplicate sample which has been introduced as a separate sample by the Quality Control Coordinator during the log-in process or prior to analysis. Evaluation of the replicate is discussed in Section 10.1. This data is reported to and summarized by the Quality Control Coordinator.

8.1.9 Check Standard Analyses

Because standards and calibration curves are subject to change and can vary from day to day, a midpoint standard or check standard should be analyzed with each group of samples. Analysis of this standard is necessary to verify the standard curve and may serve in some cases as sufficient for calibration. This value should be entered in the instrument calibration log whenever performed. Check standard analyses results should be summarized on the quality control data summary form.

8.1.10 Surrogate Standard Analyses

Surrogate standard determinations should be performed on all samples and blanks for GC/MS analyses. All samples and blanks are fortified with surrogate spiking compounds before purging or extraction to monitor preparation and analysis of samples. Recoveries should meet EPA acceptance criteria which are established as laboratory results become available. Surrogate standard data should be summarized on the surrogate standard recovery form.

8.1.11 Laboratory Matrix Spike Analyses

To evaluate the effect of the sample matrix upon analytical methodology, a separate aliquot sample should be spiked with the analyte of interest and analyzed with the sample. The percent recovery for the respective compound will then be calculated. If the percent recovery falls outside established

quality control limits the data should be evaluated and the sample reanalyzed if criteria are not met. Matrix spike results should be summarized on the quality control data summary sheets. This type of matrix spike does not necessarily reflect the behavior of the field-collected target analyte, especially if the target analyte is not stable during shipping or storage.

8.1.12 Laboratory Matrix Spike Duplicate Analyses

Similar in concept to the matrix spike sample above, it is a separate aliquot sample that is spiked with the analyte(s) of interest and analyzed with the associated sample and sample matrix spike. If the percent recovery falls outside established quality control limits, that data should be evaluated and the sample reanalyzed if criteria are not met. The comparison of the recoveries of the spiked compounds in the matrix spike and the matrix spike duplicate samples is made to determine the relative percent difference between the MS/MSD samples. RPD criteria and results should be summarized on the quality control data summary form.

8.1.13 Verification/Reference Standard Analyses

On a quarterly basis, the Quality Control Coordinator should introduce a group of prepared verification samples, or standard reference materials, into the analytical testing regime. Results of these data will be summarized, evaluated, and presented to laboratory management for review and corrective actions, if appropriate. The data are reported to and summarized by the Quality Control Coordinator.

8.1.14 Blank Spike Analyses

A blank spike is a volume of deionized, distilled laboratory water for water samples, or a purified solid matrix for soil/sediment samples which is spiked with parameters of interest and carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of spiked materials demonstrates that the laboratory techniques for this method are in control. This sample is recommended in conjunction with matrix spike/matrix spike duplicate (MS/MSD) samples on those sample matrices which are

anticipated to cause analytical difficulties due to matrix interferences. If the MS/MSD pair shows poor recoveries due to interferences, yet the blank spike sample is acceptable, this is strong evidence that the method has been performed correctly by the laboratory for these samples, but matrix interferences have affected the results. Results of blank spike analyses should be maintained with the corresponding MS/MSD and sample analytical data in the project file.

8.1.15 Field Matrix Spike Analyses

This is a sample created by spiking target analytes into a portion of a sample in the field at the point of sample acquisition. This sample provides information on the target analyte stability after collection and during transport and storage.

8.1.16 Analysis Matrix Spike Analyses

This is a sample created by spiking target analytes into a prepared portion of a sample just prior to analysis. It only provides information on matrix effects encountered during analysis, i.e., suppression or enhancement of instrument signal levels. It is most often encountered with elemental analyses involving the various forms of atomic spectroscopy and is often referred to as "standard additions."

8.1.17 Internal Standard Spike (or Recovery Standard) Analyses

This is an analyte which has the same characteristics as the surrogate, but is added to a sample just prior to analysis. It provides a short-term indication of instrument performance, but it may also be an integral part of the analytical method in a non-quality control sense, e.q., to normalize data for quantitation purposes.

8.1.18 Collocated Samples Analyses

Collocated samples are independent samples collected in such a manner that they are equally representative of the variable(s) of interest at a given point in space and time. Examples of collocated samples include: samples

from two air quality analyzers sampling from a common sample manifold or two water samples collected at essentially the same time and from the same point in a lake.

Collocated samples when collected, processed, and analyzed by the same organization provide intralaboratory precision information for the entire measurement system including sample acquisition, handling, shipping, storage, preparation, and analysis. Both samples can be carried through the steps in the measurement process together to provide an estimate of short-term precision for the entire measurement system. Likewise, the two samples, if separated and processed at different times or by different people, and/or analyzed using different instruments, provide an estimate of long-term precision of the entire measurement system.

Collocated samples when collected, processed and analyzed by different organizations provide interlaboratory precision information for the entire measurement system.

8.1.19 Replicated Sample Analyses

A replicated sample is a sample that has been divided into two or more portions, at some step in the measurement process. Each portion is then carried through the remaining steps in the measurement process (see also duplicate sample 8.1.5).

8.1.20 Split Sample

A split sample is a sample divided into two portions, one of which is sent to a different organization or laboratory and subjected to the same environmental conditions and steps in the measurement process as the one retained.

A replicated or split sample can be divided into portions (or split) at different points in the sampling and analysis process to obtain precision information on the various components of the measurement system. For example, a field replicated, or field split sample, provides precision information

about all steps after sample acquisition including effects of storage shipment, analysis, and data processing; whereas, information on the intra- and interlaboratory precision of sample preparation and analysis steps of the measurement system is provided by samples subdivided once they are received in the laboratory, i.e., laboratory replicated or laboratory split samples, respectively. A sample divided into two portions just prior to analysis, i.e., an analysis replicate, provides information on the precision of the analytical instrumentation.

The replicated sample can provide short-term or long-term precision estimates by processing the two portions together or separating them for processing at different times and under different conditions as discussed above for collocated samples.

8.2 INTERLABORATORY (ROUND ROBIN) VERIFICATION SAMPLES

On an annual basis, ITAS will conduct an interlaboratory round robin testing program for organic and inorganic parameters which are common to all laboratories. Samples will be prepared on an annual rotating basis by the Quality Control Coordinator of an ITAS laboratory. Results of the round-robin program will be determined by the Quality Control Coordinator preparing and distributing the samples. A report of the "true" values, acceptable statistical ranges and values determined by a specific laboratory will be issued to the respective Laboratory Manager. Results of all ITAS laboratories will be presented to the Vice President, Analytical Services for use in the annual management laboratory review.

8.3 QC LEVELS

There are several types of QC samples which may be applied to different projects at varying frequencies; these may also be reported in summary fashion, or in detail with all raw and associated data provided. The costs to the laboratory and the client will vary depending upon the types of samples, analyses, and data reported. This section describes the levels available from ITAS laboratories and their applications. The QC level should be specified

where requesting analyses (use the Request for Analysis form).

Level I -- ITAS standard practice. Use available analytical procedures. Fifteen percent QC samples (blank/spike/duplicate) for every 20 samples. QC samples may not be performed for a specific project, but as part of compiled sets of samples. QC data are not reported as part of analytical results. ITAS published rates apply to client samples tested.

Level II - Use available analytical (reference) methods. Fifteen percent QC samples minimum (blank, duplicate, spike, or duplicate spike). QC samples are project or client-specific. QC summary report is included as part of the analytical results reported. No raw data are included. Each QC sample is billed as a "real" analytical sample.

Level III - The highest level requires use of referenced regulatory procedures, and/or established/verified procedures using confirmatory type techniques (i.e., GC/MS, dual column GC). Method blank plus 20 percent or two QC samples minimum per each matrix. A QC summary report is supplied with supporting data. Where applicable, this level is the USEPA Contract Laboratory Program (CLP) package. A surcharge is added and/or QC samples are billed at sample rates. Costs will be based on the analytical program required.

Project-specific requirements must be defined in a specific QA Project Plan or Work Plan. Project specific documentation must be submitted to the laboratory before beginning work. Project requirements for QC samples cannot be less than Level I.

Table 8.2 provides a quick reference for the QC levels and their types of QC sample information.

TABLE 8.1. QUALITY CONTROL SAMPLES

TYPE	PURPOSE OF SAMPLE	FREQUENCY	APPLICABILITY		ACCURACY AND PRECISION APPLICATION	INTRODUCED BY FIELD SAMPLER/ ANALYST/QC COORDINATOR
			INORGANIC	ORGANIC		
Trip Blank	40-ml VOA vial filled with organic free water and taken with field sample collection kit. Used to verify that contamination of soil/ water VOA samples has not occurred due to shipment and sample containers being in field.	Two trip blank vials with every set of collection vials sent to field; each VOA cooler, returned to the lab should contain two trip blanks.		X	Accuracy	Supplier of Containers
Field Blank	A volume of "clean" collection media is added to the container to verify absence of field contamination. May be water, soil, or other material.	Specified in Project Work Plan	X	X	Accuracy	Field Sampler
Rinsate Blank	Rinse of field sample collection equipment to verify cleanliness, eliminate carryover of contamination to later samples.	Specified in Project Work Plan	X	X	Accuracy	Field Sampler
Method Blank (Process Blank)	The analysis is performed using only the reagents and solvents used in the method. Determines cumulative interference. If interference cannot be eliminated, it must be	With each group of samples, or beginning and end of each run. For GC/MS, method is spiked with surrogates 1 out of 10.	X	X	Accuracy	Analyst

TABLE 8.1. (Continued)

TYPE	PURPOSE OF SAMPLE	FREQUENCY	APPLICABILITY		ACCURACY AND PRECISION APPLICATION	INTRODUCED BY FIELD SAMPLER/ ANALYST/QC COORDINATOR
			INORGANIC	ORGANIC		
	considered when computations are performed.					
Reagent Blank	Determine the background of each reagent/solvent to be used in an analyses. Must use identical conditions to actual analyses including detection system. Background must not interfere with intended analyses.	Can be done as part of method blank, determine separately with each new batch of reagent/solvents.	X	X	Accuracy	Analyst
Bottle Blank	Rinse of cleaned sample container, analyzed prior to field shipment to demonstrate lack of contaminants.	At least 1% of each container batch; does not apply to vendor-certified containers	X	X	Accuracy	QC Coordinator or Shipping/Receiving Clerk
Duplicate	An aliquot of a sample known to Analyst. Calculate Relative Percent Difference.	1 out of 20 or at least 1/month/run	X	X	Precision	Analyst
Blind Duplicate	An aliquot of a sample introduced unknown to analyst at time of sample log in by QC Coordinator (known to QC Coordinator).	1 out of 100	X	X	Precision	QC Coordinator

TABLE 8.1. (Continued)

TYPE	PURPOSE OF SAMPLE	FREQUENCY	APPLICABILITY		ACCURACY AND PRECISION APPLICATION	INTRODUCED BY FIELD SAMPLER/ ANALYST/QC COORDINATOR
			INORGANIC	ORGANIC		
Check Standard	Analysis of standard with concentrations at mid-point or low end of standard curve to verify standard calibration curve.	With each group of samples.	X		Accuracy	Analyst
Surrogate Standards	For GC/MS and GC analy-, sis the addition of non-priority pollutants as spikes in standards, method blanks and samples. 3-point calibration curve, When used with daily determines Mean and Warning and Control Limits.	All standards, method blanks, and samples.		X GC/MS only	Accuracy	Analyst
Spiked Samples (Laboratory Matrix Spikes)	A known concentration of a specific parameter is added to an aliquot of a sample with the matrix of interest. Percent recovery is determined and spike is compared against an unspiked aliquot.	1 out of 20 or at least 1/month/ run	X	X	Accuracy	Analyst
Matrix Spike Duplicate	Percent recovery is determined and compared against matrix spiked sample.	1 out of 20 or at least 1/batch		X	Both	Analyst

TABLE 8.1. (Continued)

TYPE	PURPOSE OF SAMPLE	FREQUENCY	APPLICABILITY		ACCURACY AND PRECISION APPLICATION	INTRODUCED BY FIELD SAMPLER/ ANALYST/QC COORDINATOR
			INORGANIC	ORGANIC		
Standard Reference Materials	Standards prepared by recognized external agency and used to determine performance of entire system, instrumentation and Analysts.	Scheduled periodic basis such as quarter or semi-annually.	X	X	Accuracy	Analyst or QC Coordinator
Blank Spike	Blank material spiked with parameters of interest. Recovery evaluated for proper execution of technique or analytical method.	1 out of 20 or at least 1/batch with MS/MSD pair		X	Accuracy	Analyst
Field Matrix Spike	Sample spiked in field to show target analyte stability after collection, shipment, storage, preparation, and analysis	5 percent recommended	X	X	Accuracy	Field sampler
Analysis Matrix Spike	Sample or extract spiked just prior to analysis. Shows matrix effects during analysis. Also known as "Method of Standard Additions" spike	As specified in methods, or as needed	X		Accuracy	Analyst
Internal Standards	Nonpriority pollutant spiked into sample and method blank after extraction to monitor instrument performance and sensitivity should surrogate standards indicate a problem.	Each sample and standard		GC/MS VOA and Base-Neutral/ Acid Analysis Base-Neutral	Accuracy	Analyst

TABLE 8.1. (Continued)

TYPE	PURPOSE OF SAMPLE	FREQUENCY	APPLICABILITY		ACCURACY AND PRECISION APPLICATION	INTRODUCED BY FIELD SAMPLER/ ANALYST/QC COORDINATOR
			INORGANIC	ORGANIC		
Collocated samples	Independent samples collected to be equally representative of the variables of interest	5 percent recommended	X	X	Precision	Field sampler
Replicated sample	Collected sample that has been split into two or more parts in the field, sent to the same laboratory for analysis	5 percent recommended	X	X	Precision	Field sampler
Split sample	Sample split into two or more portions, and sent to several laboratories for analysis	1 percent recommended	X	X	Precision	Field sampler or QC coordinator

MAN:8-1 (5)

Table 8.2 QC Samples and QC Levels

QC level	QC Samples*			Are QC Results Reported?	Are QC Samples Project-specific?	QC Sample Costs
	Method Blanks (%)	Matrix Spikes (%)	Duplicates and/or Duplicate Spikes (%)			
I (15%)	5	5	5	No	No	Unbilled
II (15%)	5	5	5	Yes, summary	Yes	Per sample
III (blank +20%)	5	10	10	Yes, summary plus raw data	Yes	Surcharge and/or per sample
Project-specific	To be established individually on project-specific basis			Project-specific	Project-specific	Project-specific

*Frequencies listed are minimum goals but may vary depending upon rate of sample receipt and number of samples per analytical batch. Other types of samples, such as "blank spikes," which may be analyzed to demonstrate laboratory technique is in control despite matrix interferences in samples received, may also be billed.

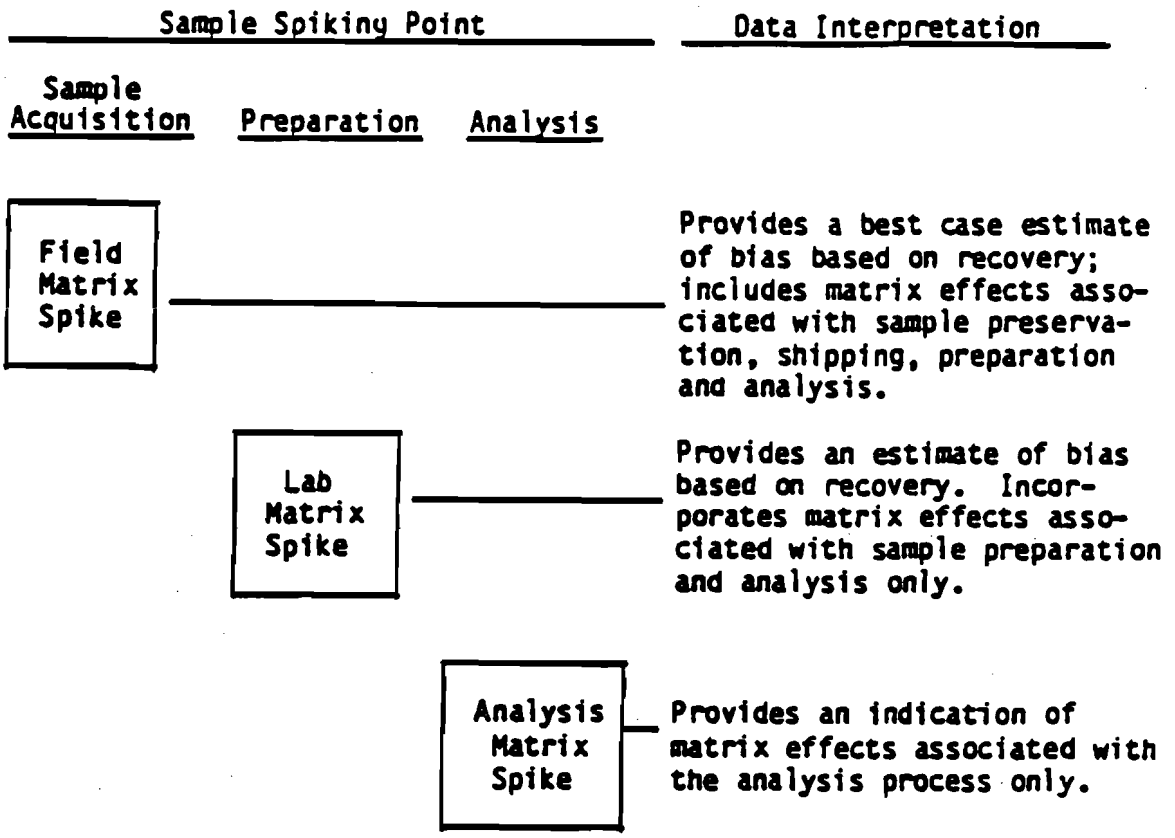


Figure 8-1 The Use of Target Analyte Spikes for Bias Estimation

from "Chapter 5, Calculation of Precision, Bias, and Method Detection Limit for Chemical and Physical Measurements"; issued by Quality Assurance Management and Special Studies Staff, Office of Monitoring Systems and Quality Assurance, Office of Research and Development, USEPA, Washington, D.C. 20460

Data Quality Assessment Sample Point of Origination			Data Interpretation
Sample Acquisition	Preparation	Analysis	
Collocated Samples			Best estimate of intra- or interlaboratory precision of the entire measurement system
Field Replicate			Second best estimate of intralaboratory precision of measurement system from sample acquisition through analysis.
Field Split			Second best estimate of interlaboratory precision of the measurement system from sample acquisition through analysis.
	Lab Replicate		Best estimate of intralaboratory precision of sample preparation and analysis.
	Lab Split		Best estimate of interlaboratory precision of sample preparation and analysis.
		Analysis Replicate	Estimate of intralaboratory precision of analysis.
		Analysis Split	Estimate of interlaboratory precision of analysis.

Figure 8-2 Precision Evaluation Samples.

from "Chapter 5, Calculation of Precision, Bias, and Method Detection Limit for Chemical and Physical Measurements"; issued by Quality Assurance Management and Special Studies Staff, Office of Monitoring Systems and Quality Assurance, Office of Research and Development, USEPA, Washington, D.C. 20460

9.0 ANALYTICAL PROCEDURES

Analytical procedures for the analysis of samples include the following:

- Prescribed method for sample preparation, including observance of stated sample holding times (such as shown on Table 5.1) and necessary extractions, dilutions, etc.
- Instrument standardization, including calibration and preventive maintenance
- Analytical techniques to be used in processing the sample
- Prescribed format on prepared data sheets for recording raw data which include:
  - Identification of project(s)
  - Identification of sample number(s)
  - Identification of Analyst
  - Identification of Analyst performing data validation
  - Dates for sample analysis and data validation
  - Raw data resulting from the analysis with appropriate calibration standards and blanks.
- Method for computation of analytical results, which can be included on the data sheet.

Documentation of the above as an analytical procedure is dependent upon the specific instrumentation and data collection and reduction methods used within a laboratory. Therefore, ITAS laboratories shall prepare and maintain as a Laboratory Specific Attachment to this manual an Analytical Procedures Manual which includes the above for all analyses performed. The requirements of this Quality Assurance Manual for activities such as calibration, preventive maintenance, and quality control sample analysis shall be incorporated into the laboratory specific manuals as appropriate.

9.1 ANALYTICAL METHODS

Whenever possible, ITAS laboratories utilize industry recognized analytical methods from source documents published by agencies such as the U.S. Environmental Protection Agency (USEPA), American Public Health Association (APHA), American Society for Testing and Materials (ASTM), and the National Institute for Occupational Safety and Health (NIOSH).

Tables 9.1 through 9.8 provide a compendium of the analytical methods typically used throughout ITAS. The format presented in Tables 9.1A through 9.1E and Table 9.2 has been adopted from the referenced USEPA document. Tables 9.3 through 9.8 provide analytical procedures for analyses not cited in Tables 9.1A through 9.1E and procedures for different matrices. Table 9.9 lists detection limits for the USEPA's Toxicity Characteristic Leaching Procedure (TCLP).

9.2 DETECTION LIMITS

All analytical methodologies have an associated method detection limit below which an analyte present in the sample cannot be accurately measured.

The detection limit of a method is generally defined as the quantity of analyte which results from the lowest differential between a signal caused by the analyte and that of random noise. A factor of 2 is generally used. Analytes present in concentrations below the detection limit WILL NOT appear as present in the sample when using this analytical technique. A detection limit quantity is reported as a less than value(<). This less than value does not indicate that an analyte is not present in a sample but only that it is present at levels below the detection limit. For results produced by USEPA CLP methods, values which are below required detection limits, but can still be quantified, are reported inside brackets [ ] as "estimated concentrations."

The detection limits attained by the EPA methods used in the laboratory are equal to or better than required by EPA regulations. The accompanying pages

indicate these detection limits. It is important to remember that specific detection limits are highly matrix dependent and the detection limits listed herein are provided for guidance and may not always be achievable.

9.2.1 Inorganics

Wet chemical techniques for nutrient, demand, and mineral constituents are relatively free from interferences in the analysis of aqueous samples. Analyses of these constituents in solid phase samples are not routinely performed. High concentrations of any of these analytes require dilution of the sample, with corresponding changes to detection limits.

9.2.2 Metals

The detection limit for metals in aqueous samples is affected by the salt content total dissolved solids (TDS). If the TDS is above 5,000 mg/liter, the sample may require dilution to prevent background light-scattering effects and false positives. Solids and sludges for analysis require a digestion prior to analysis. The detection limit for the analyte of interest is equal to the detection limit in water times a digestion factor equal to the final volume of digestate divided by the weight of solids digested.

9.2.3 Organics

Organic compounds analyzed by GC or GC/MS are analyzed as either volatile or extractable constituents. Factors which affect the detection limit of these constituents fall into the following categories.

- Contaminated samples (samples containing the analytes of interest). Samples containing as little as 0.5 ppm of chromatographable organics may require dilution. The analyst must make a dilution decision based on the knowledge of the sample and the observation of the sample.

- Background contamination, that is, a sample may have a high organic loading but not of the compounds of interest. This background may mask the compounds of interest and must be diluted from the matrix.

- Sample preparation techniques--Water samples are extracted at a 1000:1 concentration factor. One liter of sample is extracted in triplicate and concentrated to 1 ml. Soil samples are prepared by extracting 20 g of soil and concentrating to 1 ml. The resultant detection limits are 50 times greater than for water.

- Clean-up preparations--All analytical techniques for organics allow for the use of column chromatographic clean-up techniques to eliminate background and non-chromatographic organic loadings. This preparation step also dilutes the sample with the resultant rise in detection limit.

In short, many factors affect the specific detection limit of sample and no absolute may be made except for the use of average conditions. The preceding factors all affect the detection limit from sample to sample. The following tables provide the detection limits for various parameters analyzed within the analytical laboratory.

9.3 VARIANCE FROM STATED ANALYTICAL METHODS

Analyses will be performed in accordance with the methods cited herein unless specific project requirements or needs dictate adoption of an alternate method or modification of the cited methods. For example, GC/MS procedures may be "CLP-modified", as specified by the USEPA's Contract Laboratory Program (CLP).

Based on laboratory experience with a procedure and matrix type, ITAS has developed for some analytes in-house Standard Operating Proceudres (SOPs) based on regulatory methods but which may include modifications to improve reproducibility and/or accuracy.

If analysis is performed in an alternate manner, the method shall be documented in the project records as discussed in Section 12.1 of this manual.

Table 9.1
Method Summary for Organic Compounds in Water and Wastewater

Parameter	Method No.	Preferred Equipment	Detection Limit	Reference
Purgeable Hydrocarbons	601	Purge and Trap Gas Chromatography	0.005 to 0.04)g/l, 0.02 to 1.81)g/l	Federal Register[1] Federal Register[4]
Purgeable Aromatics	602	Purge and Trap Gas Chromatography	0.01 to 0.04)g/l 0.02 to 0.04)g/l	Federal Register[1] Federal Register[4]
Acrolein/Acrylonitrile	603	Purge and Trap Gas Chromatography	10 to 100)g/l 0.5 to 0.7)g/l	Federal Register[1] Federal Register[4]
Phenols	604	Gas Chromatography	1 to 10)g/l 0.14 to 16.0)g/l	Federal Register[1] Federal Register[4]
Benzidines	605	High Performance Liquid Chromatography	0.5 to 0.1)g/l 0.08 to 0.13)g/l	Federal Register[1] Federal Register[4]
Phthalate Esters	606	Gas Chromatography	2.0 to 3.0)g/l FID, 0.02 to 0.20)g/l ECD 0.29 to 3.0)g/l	Federal Register[1] Federal Register[4]
Nitrosamines	607	Gas Chromatography	0.3 to 1.0)g/l 0.15 to 0.81)g/l	Federal Register[1] Federal Register[4]
Organochlorine Pesticides and PCBs	608	Gas Chromatography	0.1 to 0.10)g/l 0.002 to 0.24)g/l	Federal Register[1] Federal Register[4]
Nitroaromatics and Isophorone	609	Gas Chromatography	5.0)g/l FID, 0.05)g/l ECD 3.6 to 5.7)g/l	Federal Register[1] Federal Register[4]
Polynuclear Aromatic Hydrocarbons	610	High Performance Liquid Chromatography	0.1 to 5.0)g/l 0.013 to 2.3)g/l	Federal Register[1] Federal Register[4]

Table 9.1 (Continued)
Method Summary for Organic Compounds in Water and Wastewater

Parameter	Method No.	Preferred Equipment	Detection Limit	Reference
Haloethers	611	Gas Chromatography	0.4 to 1.1)g/l 0.3 to 3.9)g/l	Federal Register[1] Federal Register[4]
Chlorinated Hydrocarbons	612	Gas Chromatography	0.001 to 0.015)g/l 0.03 to 1.34)g/l	Federal Register[1] Federal Register[4]
2,3,7,8-Tetrachloro-dibenzo-p-dioxin	613	Gas Chromatograph, Gas Chromatography Mass Spectrometry	0.003)g/l GC, 0.003)g/l GC/MS 0.002)g/l	Federal Register[1] Federal Register[4]
Organics Screen	503.1	Gas Chromatography	0.1 to 100)g/l	EPA, May 1980[3]
Purgeable Organics	624	Gas Chromatography/ Mass Spectrometry	10 to 100)g/l 1.6 to 7.2)g/l	Federal Register[1] Federal Register[4]
Base-Neutral Extractables and Acid Extractables	625	Gas Chromatography/ Mass Spectrometry	10 to 25)g/l 1.6 to 36)g/l	Federal Register[1] Federal Register[4]
Chlorophenoxy Acid Herbicides	615	Gas Chromatography	0.05 to 0.50)g/l	EPA, 1978[2]
Safe Drinking Water Act Pesticides	-	Gas Chromatography	0.02 to 1.0)g/l	EPA, 1978[2]
Organophosphorous Pesticides	-	Gas Chromatography	0.1 to 1.0)g/l	EPA, 1978[2]
Polychlorinated Biphenyls (PCBs)	-	Gas Chromatography	10)g/l	EPA, 1978[2]
Triazine Pesticides	-	Gas Chromatography	10 to 20)g/l	EPA, 1978[2]

Table 9.1 (Continued)

(1) Federal Register, Volume 44, No. 233, Monday, December 3, 1979, 40 CFR Part 136, pp. 69464 to 69575.

(2) "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol, and Pesticides in Water and Wastewater," Environmental Monitoring and Support Laboratory, U. S. Environmental Protection Agency, Cincinnati, Ohio, September, 1978.

(3) "The Analysis of Aromatic Chemicals in Water by the Purge and Trap Method," Method 503.1, U.S. Environmental Protection Agency, Physical and Chemical Methods Branch, Cincinnati, Ohio, May, 1980.

(4) "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," 40 CFR Part 136, Federal Register, Friday, October 26, 1984.

Table 9.1A
List of Approved Biological Test Procedures

Parameter and Units	Method[1]	Reference (Method Number or Page) EPA[2]	Standard Methods 16th Ed.	ASTM	USGS[3]
Bacteria:					
1. Coliform (fecal), number per 100 ml	MPN, 5 tube, 3 dilution;	p. 132	908C	-	-
	or membrane filter (MF),[4] single step	p. 124	909C	-	B-0050-7
2. Coliform (fecal) in presence of chlorine, number per 100 ml	MPN, 5 tube, 3 dilution	p. 132	908C	-	-
	or membrane filter (MF), single step [4A]	p. 124	909C	-	B-0050-7
3. Coliform (total), number per 100 ml	MPN, 5 tube, 3 dilution; or MF[4] single step or two step	p. 114	908A	-	-
		p. 108	909A	-	B-0025-7
4. Coliform (total) in presence of chlorine, number per 100 ml	MPN, 5 tube, dilution; or MF[4] with enrichment	p. 114	908A	-	-
		p. 111	909 (A+A.5c)	-	-
5. Fecal streptococci, number per 100 ml	MPN, 5 tube, 3 dilution; MF;[4] or plate count	p. 139	910A	-	-
		p. 136	910B	-	B-0055-7[5]
		p. 143	910C	-	-

Table 9.1A Notes

[1] The method must be specified when results are reported.

[2] "Microbiological Methods for Monitoring the Environment, Water and Wastes, 1978," EPA-600/8-78-017, U.S. Environmental Protection Agency."

[3] Greeson, P.E. et al., "Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A4, Laboratory Analysis, 1977.

[4] 0.45 m membrane filter or other pore size certified by the manufacturer to fully retain organisms to be cultivated, and free of extractables which could interfere with their growth and development.

[4A] Since the membrane filter technique usually yields low and variable recovery from chlorinated wastewaters, the MPN method will be required to resolve any controversies.

[5] Approved only if dissolution of the KF Streptococcus Agar (Section 5.1, USGS Method B-0055-77) is made in a boiling water bath to avoid scorching of the medium.

Table 9.1B
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page)				
	EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
1. Acidity, as $CaCO_3$, mg/l: Electrometric end point or phenolphthalein end point.	305.1	402(4.a)	D1067-82(E)	-	-
2. Alkalinity, as $CaCO_3$, mg/L: Electrometric or colorimetric:					
Titration to pH 4.5, manual	310.1	403	D1067-82(B)	I-1030-84	33.014[2]
Or automated	310.2	-	-	I-2030-84	-
3. Aluminum--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	202.1	303C	-	I-3051-84	-
AA furnace	202.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or colorimetric (Eriochromu cyanine R)	-	306B	-	-	-
4. Ammonia (as N), mg/L: Manual distillation[5] (at pH 9.5) followed by:	350.2	417A	-	-	33.057[2]
Nesslerization	350.2	417B	D1426-79(A)	I-3520-84	33.057[2]
Titration	350.2	417D	-	-	-
Electrode	350.3	417E or F	D1426-79(D)	-	-
Automated phenate, or	350.1	417G	D1426-79(C)	I-4523-84	-
Automated electrode	-	-	-	-	(6)
5. Antimony--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	204.1	303A	-	-	-
AA furnace, or	204.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]

Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page) EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
6. Arsenic--Total,[3] mg/l:					
Digestion[3] followed by	206.5	-	-	-	-
Hydride	206.3	303E	D2972-84(B)	I-3062-84	-
AA furnace	206.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or, colorimetric (SDDC)	206.4	307B	D2972-84(A)	I-3060-84	-
7. Barium--Total,[3] mg/l: Digestion[3] followed by:					
AA direct aspiration	208.1	303C	-	I-3084-84	-
AA furnace, or	208.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
8. Beryllium--Total,[3] mg/l: Digestion[3] followed by:					
AA direct aspiration	210.1	303C	D3654-84[A]	I-3095-84	-
AA furnace	210.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or colorimetric (aluminon)	-	309B	-	-	-
9. Biochemical oxygen demand (BOD_5), mg/l:					
Dissolved oxygen depletion	405.1	507	-	I-1578-78[7]	33.019[2], p. 17[8]
10. Boron--Total, mg/l:					
Colorimetric (curcumin) or	212.3	404B	-	I-3112-84	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
11. Bromide, mg/l: Titrimetric	320.1	-	D1246-82(C)	I-1125-84	P.544[9]

Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page)				
	EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
12. Cadmium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	213.1	303A or 303B	D3557-84(A or B)	I-3135-84 or I-3136-84	33.089[2]
AA furnace	213.2	304	-	-	Pg. 37[8]
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Polarometry[10] or	-	-	D3557-78(C)	-	-
Colorimetric (Dithizone)	-	310B	-	-	-
13. Calcium--Total,[3] mg/L: Digestion[3] followed by:					
Atomic absorption	215.1	303A	D511-84(B)	I-3152-84	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or EDTA titration	215.2	311C	D511-84(A)	-	-
14. Carbonaceous Biochemical oxygen demand[11] ($CBOD_5$), mg/L: Winkler (Azide modification) or electrode method with nitrification inhibitor	-	507(5.a.6)	-	-	-
15. Chemical oxygen demand (COD), mg/L:					
Titrimetric colorimetric	410.1	508A	D1252-83	I-3560-84 or	33.034[2]
Manual or	410.2	-	-	I-3562-84	Pg. 17[8]
	410.3	-	-	-	
Automated	410.4	-	-	-	-
Spectrophotometric	-	-	-	I-3561-84	(12,13)
16. Chloride, mg/L:					
Titrimetric (silver nitrate) or	-	407A	D512-81(B)	I-1183-84	-
Mercuric nitrate	325.3	407B	D512-81(A)	I-1184-84	33.067[2]
Colorimetric (ferricyanide) manual or	-	-	D512-81(C)	I-1187-84	-
Automated	325.1 or 325.2	407D	-	I-2187-84	-

Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page) EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
17. Chlorine--Total residual, mg/L:					
Titrimetric-amperometric,	330.1	408C	D1253-76(A)	-	-
Starch end point direct,	330.3	408A	D1253-76(B)	-	-
Back titration either end point[14] or	330.2	408B	-	-	-
DPD-FAS	330.4	408D	-	-	-
Spectrophotometric, DPD, or	330.5	408E	-	-	-
Electrode	-	-	-	-	(15)
18. Chromium VI dissolved, mg/L: 0.45 micron filtration with:					
Extraction and atomic absorption, or	218.4	312B	-	I-1232-84	-
Colorimetric (Diphenylcarbazide)	-	-	-	I-1230-84	3070[16]
19. Chromium--Total,[3] mg/L:					
Digestion[3] (optional extraction) followed by:	218.3	303B	-	-	-
AA direct aspiration	218.1	303A	D1687-84(D)	I-3236-84	33.089[2]
AA furnace	218.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or colorimetric (Diphenylcarbazide)	-	312B	D1687-77(A)	-	-
20. Cobalt--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	219.1	303A or 303B	D3558-84(A or B)	I-3239-84 or I-3240-84	P. 37[8]
AA furnace, or	219.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
21. Color, platinum Cobalt units or dominant wavelength hue, luminance, purity:					
Colorimetric, ADMI	110.1	204D	-	-	(17)
Platinum cobalt; or	110.2	204A	-	I-1250-84	-
Spectrophotometric	110.3	204B	-	-	-

Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page) EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
22. Copper--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	220.1	303A or 303B	D1688-84(D or E)	I-3271-84 or I-3270-84	33.089[2], P.37[8]
AA furnace	220.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Colorimetric (Neocuproine)	-	313B	D1688-84(A)	-	-
Bicinchoninate	-	-	-	-	(18)
23. Cyanide--Total, mg/L:					
Manual distillation with $MgCl_2$, followed by:		412D	-	-	-
Titrimetric,		412C	-	-	P.22[8]
Manual or	335.2	412D	D2036-82(A)	-	-
Automated[19] spectrophotometric	335.3	-	D2036-82(A)	I-3300-84	-
24. Cyanide amenable to chlorination, mg/L: Manual distillation with $MgCl_2$; followed by titrimetric, manual or automated[15] spectrophotometric	335.1	412F	D2036-82(B)	-	-
25. Fluoride--Total, mg/L:					
Manual distillation[3]	-	413A	-	-	-
Followed by manual or	340.2	413B	D1179-80(B)	-	-
Automated electrode	-	-	-	I-4327-84	-
SPADNS	340.1	413C	D1179-80(A)	-	-
Or automated complexone	340.3	413E	-	-	-
26. Gold--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	231.1	303A	-	-	-
Or AA furnace	231.2	304	-	-	-
27. Hardness--Total as $CaCO_3$, mg/L:					
Automated colorimetric	130.1	-	-	-	-
EDTA titration, or Ca plus Mg as their carbonates, by inductively coupled plasma	130.2	314B	D1126-80	I-1338-84	33.082[2]
Or atomic absorption (see parameters 13 and 33)	-	-	-	-	-

Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page) EPA 1979	Standard Methods 18th Ed.	ASTM	USGS[1]	Other
28. Hydrogen ion (pH), pH units:					
Electrometric measurements	150.1	423	D1293-84(A or B)	I-1586-84	33.062[2]
Or automated electrode	-	-	-	-	(20)
29. Iridium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	235.1	303A	-	-	-
Or AA furnace	235.2	304	-	-	-
30. Iron--Total,[3] mg/L:					
Digestion[3] followed by	-			-	-
AA direct aspiration	236.1	303A or 303B	D1068-84 (C or D)	I-3381-84	33.089[2]
AA furnace	236.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4] (21)
Or colorimetric (Phenanthroline)	-	315B	D1068-84(A)	-	
31. Kjeldahl nitrogen--Total (as N), mg/L:					
Digestion and distillation	351.3	420A or B	D3590-84(A)	-	33.051[2]
Followed by titration	351.3	417D	D3590-84(A)	-	-
Nesslerization or	351.3	417B	D3590-84(A)	-	-
Electrode	351.3	417E or F	-	I-4551-78	-
Automated phenate	351.1	-	-	I-4552-78	-
Semi-automated block digestor	351.2	-	D3590-84(A)	-	-
Or potentiometric	351.4	-	D3590-84(A)	-	-
32. Lead--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	239.1	303A or 303B	D3559-85(A or B)	I-3399-84	33.089[2]
AA furnace	239.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Voltametry[9] or	-	-	D3559-85(C)	-	-
Colorimetric (Dithizone)	-	316B	-	-	-

Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page) EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
33. Magnesium--Total,[3] mg/L: Digestion[3] followed by:					
Atomic absorption	242.1	303A	D511-84(B)	I-3447-84	33.089[2]
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or gravimetric	-	318B	D511-77(A)	-	-
34. Manganese--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	243.1	303A or 303B	D858-84(B or C)	I-3454-84	33.089[2]
AA furnace	243.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or colorimetric (persulfate), or	-	319B	D858-84(A)	-	33.126[2]
Periodate	-	-	-	-	(22)
35. Mercury--Total,[3] mg/L:					
Cold vapor, manual or	245.1	303F	D3223-80	I-3462-84	33.095[2]
Automated	245.2	-	-	-	-
36. Molybdenum--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	246.1	303C	-	I-3490-84	-
AA furnace, or	246.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
37. Nickel--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	249.1	303A or 303B	D1886-84(C or D)	I-3499-84	-
AA furnace	249.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or colorimetric (Heptoxime)	-	321B	-	-	-
38. Nitrate (as N), mg/L:					
Brucine sulfate, or	352.1	-	D992-71	-	33.063[2] 4190
Nitrate-nitrite N minus Nitrite N	See parameters 39 and 40	See parameters 39 and 40	See parameters 39 and 40	See parameters 39 and 40	P. 28[8]

Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page)				
	EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
39. Nitrate-nitrite (as N), mg/L: Cadmium reduction,					
Manual or	353.3	418C	D3867-85(B)	-	-
Automated; or	353.2	418F	D3867-85(A)	I-4545-84	-
Automated hydrazine	353.1	-	-	-	-
40. Nitrite (as N), mg/L: Spectrophotometric,					
Manual or	354.1	419	D1254-67	-	(24)
Automated (Diazotization)	-	-	-	I-4540-84	-
41. Oil and grease--total recoverable, mg/L: Gravimetric (extraction)	413.1	503A	-	-	-
42. Organic carbon--total (TOC), mg/L: Combustion or oxidation	415.1	505	D2579-85(A or B)	-	33.044[2] Note 23
43. Organic nitrogen (as N), mg/L: Total Kjeldahl N (parameter 31) minus ammonia N (parameter 4)					
44. Orthophosphate (as P), mg/L: Ascorbic acid method,					
Automated or	365.1	424G	-	I-4601-84	33.116[2]
Manual single reagent or	365.2	424F	D515-82(A)	-	33.111[2]
Manual two reagents	365.3	-	-	-	-
45. Osmium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration, or	252.1	303C	-	-	-
AA furnace	252.2	304	-	-	-
46. Oxygen, dissolved, mg/L:					
Winkler (Azide modification)	360.2	421B	D888-81(C)	I-1575-78	33.028[2]
Or electrode	360.1	421F	-	I-1576-78	-

Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page)				
	EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
47. Palladium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	253.1	-	-	-	P.527[9]
Or AA furnace	253.2	-	-	-	P.528[9]
48. Phenols, mg/L:					
Manual distillation[25]	420.1	-	D1783-80 (A or B)	-	(26)
Followed by manual	420.1	-	-	-	(26)
Or automated[15] colorimetric (4AAP)	420.2	-	-	-	-
49. Phosphorus (elemental), mg/L: Gas-liquid chromatography	-	-	-	-	(27)
50. Phosphorus--Total, mg/L:					
Persulfate digestion	365.2	424C(111)	-	-	33.111[2]
Followed by manual or	365.2 or 365.3	424F	D515-82(A)	-	-
Automated ascorbic acid reduction;	365.1	424G	-	I-4600-78	-
or semi-automated block digestor	365.4	-	-		33.116[2]
51. Platinum--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	255.1	303A	-	-	-
Or AA furnace	255.2	304	-	-	-
52. Potassium-Total,[5] mg/L: Digestion[3] followed by:					
Atomic absorption,	258.1	303A	-	I-3630-84	33.103[2]
Inductively coupled plasma,	-	-	-	-	Method 200.7[4]
Flame photometric, or	-	322B	D1428-82(A)	-	-
Colorimetric (Cobaltinitrate)	-	-	-	-	317B
53. Residue--total, mg/L: Gravimetric, 103-105°C	160.3	209A	-	I-3750-84	-
54. Residue--filterable, mg/L: Gravimetric, 180°C	160.1	209B	-	I-1750-84	-

Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page) EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
55. Residue--nonfiltrable, (TSS), mg/L: Gravimetric, 103-105°C post washing of residue	160.2	209C	-	I-3765-84	-
56. Residue--settleable, mg/L: Volumetric (Imhoff cone) or gravimetric	160.5	209E	-	-	-
57. Residue--volatile, mg/L: Gravimetric, 550°C	160.4	209D	-	I-3753-84	-
58. Rhodium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	265.1	303A	-	-	-
Or AA furnace	265.2	304	-	-	-
59. Ruthenium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	267.1	303A	-	-	-
Or AA furnace	267.2	304	-	-	-
60. Selenium--Total,[3] mg/L: Digestion[3] followed by:					
AA furnace	270.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or AA gaseous hydride	270.3	303E	D3859-84(A)	I-3667-84	-
61. Silica--Dissolved, mg/L: 0.45 micron filtration followed by:					
Colorimetric, manual,	370.1	425C	D859-80(B)	I-1700-84	-
Automated (Molybdosilicate), or	-	-	-	I-2700-84	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
62. Silver--Total,[28] mg/L: Digestion[3] followed by:					
AA direct aspiration	272.1	303A or 303B	-	I-3720-84	33.089[2] and 37[8]
AA furnace, or	272.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]

Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page)				
	EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
63. Sodium--Total,[3] mg/L: Digestion[3] followed by:					
Atomic absorption	273.1	303A	-	I-3735-84	33.107[2]
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or flame photometric	-	325B	D1428-82(A)	-	-
64. Specific conductance, mhos/cm: Wheatstone bridge	120.1	205	D1125-82(A)	I-1780-84	33.002[2]
65. Sulfate (as SO_4), mg/L:					
Automated colorimetric (barium chloranilate),	375.1	-	-	I-2022-78	-
Gravimetric, or	375.3	426A or 426B	D516-82(A)	-	33.124[2]
Turbidimetric	375.4		D516-82(B) (15th Ed.)	-	426C[29]
66. Sulfide (as S), mg/L:					
Titrimetric (iodine) or	376.1	427D	-	I-3840-84	228A[30]
Colorimetric (methylene blue)	376.2	427C	-	-	-
67. Sulfite (as SO_3), mg/L: Titrimetric (iodine iodate)	377.1	428A	D1339-84(C)	-	-
68. Surfactants, mg/L: Colorimetric (methylene blue)	425.1	512B	D2330-82(A)	-	-
69. Temperature, °C: Thermometric	170.1	212	-	-	(31)
70. Thallium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	279.1	303A	-	-	-
AA furnace, or	279.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
71. Tin--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration or	282.1	303A	-	I-3850-78	-
AA furnace	282.2	304	-	-	-

Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page)				
	EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
72. Titanium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration or	283.1	303C	-	-	-
AA furnace	283.2	304	-	-	-
73. Turbidity, NTU: Nephelometric	180.1	214A	D1889-81	I-3860-84	-
74. Vanadium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	286.1	303C	-	-	-
AA furnace	286.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or colorimetric (Gallic acid)	-	327B	D3373-84(A)	-	-
75. Zinc--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	289.1	303A or 303B	D1691-84(C or D)	I-3900-84	33.089[2]
AA furnace	289.2	304		-	
Inductively coupled plasma, or	-	-	-	-	Method 200.7[4]
Colorimetric (Zincon or Dithizone)	-	328C	-	-	(32)

TABLE 9.1B NOTES

[1] "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments," U.S. Department of the Interior, U.S. Geological Survey, Open-File Report 85-495, 1986.

[2] "Official Methods of Analysis of the Association of Official Analytical Chemists" methods manual, 14th ed. (1985).

[3] For the determination of total metals the sample is not filtered before processing. A digestion procedure is required to solubilize suspended material and to destroy possible organic-metal complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes," 1979. One (§4.1.3), is a vigorous digestion using nitric acid. A less vigorous digestion using nitric and hydrochloric acids (§4.1.4) is preferred; however, the analyst should be cautioned that this mild digestion may not suffice for all samples types. Particularly, if a colorimetric procedure is to be employed, it is necessary to ensure that all organo-metallic bonds be broken so that the metal is in a reactive state. In those situations, the vigorous digestion is to be preferred making certain that at no time does the sample go to dryness. Samples containing large amounts of organic materials would also benefit by this vigorous digestion. Use of the graphite furnace technique, inductively coupled plasma, as well as determinations for certain elements such as arsenic, the noble metals, mercury, selenium, and titanium require a modified digestion and in all cases the method write-up should be consulted for specific instructions and/or cautions.

Table 9.1B (Continued)

List of Approved Inorganic Test Procedures

NOTE: If the digestion procedure for direct aspiration or graphite furnace atomic absorption analysis included in one of the other approved references is different than the above, the EPA procedure must be used.

Dissolved metals are defined as those constituents which will pass through a 0.45 micron membrane filter. Following filtration of the sample, the referenced procedure for total metals must be followed. Sample digestion for dissolved metals may be omitted for AA (direct aspiration or graphite furnace) and ICP analyses provided the sample solution to be analyzed meets the following criteria:

(a) Has a low COD (<20)
(b) Is visibility transparent with a turbidity measurement of 1 NTU or less
(c) Is colorless with no perceptible odor, and
(d) Is of one liquid phase and free of particulate or suspended matter following acidification.

(4) The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes," is given at Appendix C of 40 CFR Part 136 as published in the October 26, 1984, Federal Register.

(5) Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.

(6) Ammonia, Automated Electrode Method, Industrial Method Number 379-75WE, dated February 19, 1976, Technicon AutoAnalyzer II, Technicon Industrial Systems, Tarrytown, New York 10591.

(7) The approved method is that cited in "Methods for Determination of Inorganic Substances in Water and Fluid Sediments," USGS TWRI, Book 5, chapter A1 (1979).

(8) American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, NY 10018.

(9) "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).

(10) The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.

(11) Carbonaceous biochemical oxygen demand ($CBOD_5$) must not be confused with the traditional BOD_5 test which measures "total BOD". The addition of the nitrification inhibitor is not a procedural option, but must be included to report the $CBOD_5$ parameter. A discharger whose permit requires reporting the traditional BOD_5 may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states $CBOD_5$ is required can the permittee report data obtained using the nitrification inhibitor.

(12) DIC COD Method, Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, Texas 77840.

(13) Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

(14) The back titration method will be used to resolve controversy.

Table 9.1B (Continued)

List of Approved Inorganic Test Procedures

(15) ORION Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977, Orion Research Incorporated, 840 Memorial Drive, Cambridge, Massachusetts 02138.

(16) The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition, 1976.

(17) National Council of the Paper Industry for Air and Stream Improvement, Inc., Technical Bulletin 253, December 1971.

(18) Copper, Bicinchoninate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

(19) After the manual distillation is completed, the auto-analyzer manifolds in EPA Methods 335.3 (Cyanide) or 420.2 (phenols) are simplified by connecting the re-sample line directly to the sampler. When using the manifold setup shown in Method 335, the buffer 6.2 should be replaced with the buffer 7.6 found in Method 335.2.

(20) Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Technicon Auto-Analyzer II, Technicon Industrial Systems, Tarrytown, New York 10591.

(21) Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

(22) Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2-113 and 2-117, Hach Chemical Company, Loveland, Colorado 80537.

(23) Goerlitz, D., Brown, E., "Methods for Analysis of Organic Substances in Water," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A3, p.4 (1972).

(24) Nitrogen, Nitrite, Method 8507, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

(25) Just prior to distillation, adjust the sulfuric acid preserved sample to pH 4 with 1 + 9 NaOH.

(26) The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition. The colorimetric reaction is conducted at a pH of 10.0 ± 0.2. The approved methods are given on page 576-81 of the 14th edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrophotometric procedures.

Table 9.18 (Continued)

List of Approved Inorganic Test Procedures

(27) R. F. Addison and R. G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," Journal of Chromatography, Vol. 47, No. 3, pp. 421-426, 1970.

(28) Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to a pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2M $Na_2S_2O_3$ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.

(29) The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 15th. Edition.

(30) The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 13th Edition.

(31) Stevens, H. H., Ficke, J. F., and Scott, G. F., "Water Temperature-Influential Factors, Field Measurement and Data Presentation," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 1, Chapter D1, 1975.

(32) Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2-231 and 2-333, Hach Chemical Company, Loveland, Colorado 80537.

Table 9.1C
List of Approved Test Procedures for Non-Pesticide Organic Compounds

Parameter[1]	EPA Method Number[2,7] GC	GC/MS	HPLC	Other
1. Acenaphthene	610	625, 1625	610	
2. Acenaphthylene	610	625, 1625	610	
3. Acrolein	603	[4]624, 1624	-	
4. Acrylonitrile	603	[4]624, 1624	-	
5. Anthracene	610	625, 1625	610	
6. Benzene	602	624, 1624	-	
7. Benzidine	-	[5]625, 1625	605	Note 3, p. 1
8. Benzo(a)anthracene	610	625, 1625	610	
9. Benzo(a)pyrene	610	625, 1625	610	
10. Benzo(b)fluoranthene	610	625, 1625	610	
11. Benzo(ghi)perylene	610	625, 1625	610	
12. Benzo(k)fluoranthene	610	625, 1625	610	
13. Benzyl Chloride	-	- -	-	Note 3, p. 130; Note 6, p. S102
14. Benzyl Butyl Phthalate	606	625, 1625	-	
15. Bis(2-chloroethoxy) methane	611	625, 1625	-	
16. Bis(2-chloroethyl) ether	611	625, 1625	-	
17. Bis(2-ethylhexyl) phthalate	606	625, 1625	-	
18. Bromodichloromethane	601	624, 1624	-	
19. Bromoform	601	624, 1624	-	
20. Bromomethane	601	624, 1624	-	
21. 4-Bromophenylphenyl ether	611	625, 1625	-	

Table 9.1C (Continued)

List of Approved Test Procedures for Non-Pesticide Organic Compounds

Parameter[1]	EPA Method Number[2,7]			
	GC	GC/MS	HPLC	Other
22. Carbon tetrachloride	601	624, 1624	-	Note 3, p. 130
23. 4-Chloro-3-methylphenol	604	625, 1625	-	
24. Chlorobenzene	601, 602	624, 1624	-	Note 3, p. 130
25. Chloroethane	601	624, 1624	-	
26. 2-Chloroethylvinyl ether	601	624, 1624	-	
27. Chloroform	601	624, 1624	-	Note 3, p. 130
28. Chloromethane	601	624, 1624	-	
29. 2-Chloronaphthalene	612	625, 1625	-	
30. 2-Chlorophenol	604	625, 1625	-	
31. 4-Chlorophenylphenyl ether	611	625, 1625	-	
32. Chrysene	610	625, 1625	610	
33. Dibenzo(a,h)anthracene	610	625, 1625	610	
34. Dibromochloromethane	601	624, 1624	-	
35. 1,2-Dichlorobenzene	601, 602, 612	624, 625, 1625	-	
36. 1,3-Dichlorobenzene	601, 602, 612	624, 625, 1625	-	
37. 1,4-Dichlorobenzene	601, 602, 612	625, 1624, 1625	-	
38. 3,3'-Dichlorobenzidine	-	625, 1625	605	
39. Dichlorodifluoromethane	601	- -	-	
40. 1,1-Dichloroethane	601	624, 1624	-	

Table 9.1C (Continued)

List of Approved Test Procedures for Non-Pesticide Organic Compounds

Parameter[1]	EPA Method Number[2,7] GC	GC/MS	HPLC	Other
41. 1,2-Dichloroethane	601	624, 1624	-	
42. 1,1-Dichloroethene	601	624, 1624	-	
43. trans-1,2-Dichloroethene	601	624, 1624	-	
44. 2,4-Dichlorophenol	604	625, 1625	-	
45. 1,2-Dichloropropane	601	624, 1624	-	
46. cis-1,3-Dichloropropene	601	624, 1624	-	
47. trans-1,3-Dichloropropene	601	624, 1624	-	
48. Diethyl phthalate	606	625, 1625	-	
49. 2,4-Dimethylphenol	604	625, 1625	-	
50. Dimethyl phthalate	606	625, 1625	-	
51. Di-n-butyl phthalate	606	625, 1625	-	
52. Di-n-octyl phthalate	606	625, 1625	-	
53. 2,4-Dinitrophenol	604	625, 1625	-	
54. 2,4-Dinitrotoluene	609	625, 1625	-	
55. 2,6-Dinitrotoluene	609	625, 1625	-	
56. Epichlorohydrin	-	- -	-	Note 3, p. 130; Note 6, p. S102
57. Ethylbenzene	602	624, 1624	-	
58. Fluoranthene	610	625, 1625	610	

Table 9.1C (Continued)

List of Approved Test Procedures for Non-Pesticide Organic Compounds

Parameter[1]	EPA Method Number[2,7]			
	GC	GC/MS	HPLC	Other
59. Fluorene	610	625, 1625	610	
60. Hexachlorobenzene	612	625, 1625	-	
61. Hexachlorobutadiene	612	625, 1625	-	
62. Hexachlorocyclopentadiene	612	[5]625, 1625	-	
63. Hexachloroethane	612	625, 1625	-	
64. Ideno(1,2,3-cd)pyrene	610	625, 1625	610	
65. Isophorone	609	625, 1625	-	
66. Methylene Chloride	601	624, 1624	-	Note 3, p. 130
67. 2-Methyl-4,6-Dinitrophenol	604	625, 1625	-	
68. Naphthalene	610	625, 1625	-	
69. Nitrobenzene	609	625, 1625	-	
70. 2-Nitrophenol	604	625, 1625	-	
71. 4-Nitrophenol	604	625, 1625	-	
72. N-Nitrosodimethylamine	607	625, 1625	-	
73. N-Nitrosodi-n-propylamine	607	[5]625, 1625	-	
74. N-Nitrosodiphenylamine	607	[5]625, 1625	-	
75. 2,2-oxybis(1-chloropropane)	611	625, 1625	-	
76. PCB-1016	608	625	-	Note 3, p. 43
77. PCB-1221	608	625	-	Note 3, p. 43

Table 9.1C (Continued)

List of Approved Test Procedures for Non-Pesticide Organic Compounds

Parameter[1]	EPA Method Number[2,7] GC	GC/MS	HPLC	Other
78. PCB-1232	608	625	-	Note 3, p. 43
79. PCB-1242	608	625	-	Note 3, p. 43
80. PCB-1248	608	625	-	Note 3, p. 43
81. PCB-1254	608	625	-	Note 3, p. 43
82. PCB-1260	608	625	-	Note 3, p. 43
83. Pentachlorophenol	604	625, 1625	-	Note 3, p. 140
84. Phenanthrene	610	625, 1625	610	
85. Phenol	604	625, 1625	-	
86. Pyrene	610	625, 1625	610	
87. 2,3,7,8-Tetrachlorodibenzo-p-dioxin	-	[5a]613	-	
88. 1,1,2,2-Tetrachloroethane	601	624, 1624	-	Note 3, p. 130
89. Tetrachloroethene	601	624, 1624	-	Note 3, p. 130
90. Toluene	602	624, 1624	-	
91. 1,2,4-Trichlorobenzene	612	625, 1625	-	Note 3, p. 130
92. 1,1,1-Trichloroethane	601	624, 1624	-	
93. 1,1,2-Trichloroethane	601	624, 1624	-	Note 3, p. 130
94. Trichloroethene	601	624, 1624	-	
95. Trichlorofluoromethane	601	624	-	
96. 2,4,6 Trichlorophenol	604	625, 1625	-	
97. Vinyl Chloride	601	624, 1624	-	

Table 9.1C Notes

(1) All parameters are expressed in micrograms per liter (µg/L).

(2) The full text of Methods 601-613, 624, 625, 1624, and 1625, are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of 40 CFR 136 as published in the October 26, 1984 Federal Register. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of 40 CFR 136 as published in the October 26, 1984 Federal Register.

(3) "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978.

(4) Method 624 may be extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, Method 603 or Method 1624 are preferred methods for these compounds.

(5) Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625,are preferred methods for these compounds.

(5a) 625, Screening only.

(6) "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).

(7) Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 601-613, 624, 625, 1624, and 1625 (See Appendix A of 40 CFR 136 as published in the October 26, 1984 Federal Register) in accordance with procedures in Section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for Methods 1624, and 1625) of all samples to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these Methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported.

Table 9.1D
List of Approved Test Procedures for Pesticides[1]

Parameter (µg/L)	Method	EPA[2,7]	Standard Methods 15th Ed.	ASTM	OTHER
1. Aldrin	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30
	GC/MS	625	-	-	
2. Ametryn	GC	-	-	-	Note 3, p.83; Note 6, p.S68
3. Aminocarb	TLC	-	-	-	Note 3, p.94; Note 6, p.S16
4. Atraton	GC	-	-	-	Note 3, P.83; Note 6, P. S68
5. Atrazine	GC	-	-	-	Note 3, p.83; Note 6, p. S68
6. Azinphos methyl	GC	-	-	-	Note 3, p.25; Note 6, p.S51
7. Barban	TLC	-	-	-	Note 3, p.104; Note 6, p. S64
8. a-BHC	GC	608	509A	D3086	Note 3, p.7
	GC/MS	[5]625	-	-	
9. b-BHC	GC	608	-	D3086	
	GC/MS	625	-	-	
10. w-BHC	GC	608	-	D3086	
	GC/MS	[5]625	-	-	
11. g-BHC (Lindane)	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30
	GC/MS	625	-	-	
12. Captan	GC	-	509A	-	Note 3, p.7
13. Carbaryl	TLC	-	-	-	Note 3, p.94; Note 6, p.S60
14. Carbophenothion	GC	-	-	-	Note 4, p.30; Note 6, p.S73
15. Chlordane	GC	608	509A	D3086	Note 3, p.7
	GC/MS	625	-	-	
16. Chloropraphan	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
17. 2,4-D	GC	-	509B	-	Note 3, p.115; Note 4, p.35
18. 4,4'-DDD	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30
	GC/MS	625	-	-	
19. 4,4'-DDE	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30
	GC/MS	625	-	-	
20. 4,4'-DDT	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30
	GC/MS	625	-	-	
21. Demeton-O	GC	-	-	-	Note 3, p.25; Note 6, p.S51
22. Dementon-S	GC	-	-	-	Note 3, p.25; Note 6, p. S51
23. Diazinon	GC	-	-	-	Note 3, p.25; Note 4, p.30; Note 6, p. S51

Table 9.10 (Continued)
List of Approved Test Procedures for Pesticides[1]

Parameter (µg/L)	Method	EPA[2,7]	Standard Methods 15th Ed.	ASTM	OTHER
24. Dicamba	GC	-	-	-	Note 3, p.115
25. Dichlofenthion	GC	-	-	-	Note 4, p.30; Note 6, p.S73
26. Dichloran	GC	-	509A	-	Note 3, p.7
27. Dicofol	GC	-	-	D3086	
28. Dieldrin	GC	608	509A	-	Note 3, p.7; Note 4, p.30
	GC/MS	625	-	-	
29. Dioxathion	GC	-	-	-	Note 4, p.30; Note 6, p.S73
30. Disulfoton	GC	-	-	-	Note 3, p. ; Note 6, p. S51
31. Diuron	TLC	-	-	-	Note 3, p. 104; Note 6, p. S64
32. Endosulfan I	GC	608	509A	D3086	Note 3, p.7
	GC/MS	[5]625	-	-	
33. Endosulfan II	GC	608	509A	D3086	Note 3, p.7
	GC/MS	[5]625	-	-	
34. Endosulfan sulfate	GC	608	-	-	
	GC/MS	625	-	-	
35. Endrin	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30
	GC/MS	[5]625	-	-	
36. Endrin aldehyde	GC	608	-	-	
	GC/MS	625	-	-	
37. Ethion	GC	-	-	-	Note 4, p.30; Note 6, p.S73
38. Fenuron	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
39. Fenuron-TCA	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
40. Heptachlor	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30
	GC/MS	625	-	-	
41. Heptachlor epoxide	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30; Note 6, p. S73
42. Isodrin	GC/MS	625	-	-	
43. Linuron	GC	-	-	-	Note 4, p.30; Note 6, p.S73
44. Malathion	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
45. Methiocarb	GC	-	509A	-	Note 3, p.25; Note 4, p.30; Note 6, p.S51
46. Methoxychlor	TLC	-	-	-	Note 3, p.94; Note 6, p.S60
	GC	-	509A	D3086	Note 3, p.7; Note 4, p.30

Table 9.1D (Continued)
List of Approved Test Procedures for Pesticides[1]

Parameter (µg/L)	Method	EPA[2,7]	Standard Methods 15th Ed.	ASTM	OTHER
47. Mexacarbate	TLC	-	-	-	Note 3, p.94; Note 6, p.S60
48. Mirex	GC	-	509A	-	Note 3, p.7
49. Monuron	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
50. Monuron-TCA	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
51. Neburon	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
52. Parathion methyl	GC	-	509A	-	Note 3, p.25; Note 4, p.30
53. Parathion ethyl	GC	-	509A	-	Note 3, p.25
54. PCNB	GC	-	509A	-	Note 3, p.7
55. Perthane	GC	-	-	D3086	
56. Prometon	GC	-	-	-	Note 3, p.83; Note 6, p.S68
57. Prometryn	GC	-	-	-	Note 3, p.83; Note 6, p.S68
58. Propazine	GC	-	-	-	Note 3, p.83; Note 6, p.S68
59. Propham	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
60. Propoxur	TLC	-	-	-	Note 3, p.94; Note 6, p.S60
61. Secbumeton	TLC	-	-	-	Note 3, p.83; Note 6, p.S68
62. Siduron	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
63. Simazine	GC	-	-	-	Note 3, p.83; Note 6, p.S68
64. Strobane	GC	-	509A	-	Note 3, p.7
65. Swep	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
66. 2,4,5-T	GC	-	509B	-	Note 3, p.115; Note 4, p.35
67. 2,4,5-TP (Silvex)	GC	-	509B	-	Note 3, p.115
68. Terbuthylazine	GC	-	-	-	Note 3, p.83; Note 6, p.S68
69. Toxaphene	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30
	GC/MS	625	-	-	
70. Trifluralin	GC	-	509A	-	Note 3, p.7

Table 9.1D Notes

(1) Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table 9.1C, where entries are listed by chemical name.

(2) The full text of methods 608 and 625 are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of 40 CFR 136 as published in the October 26, 1984 Federal Register. The standardized test procedure to be used to determine the method detection limit (MDL) for these procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of 40 CFR 136 as published in the October 26, 1984 Federal Register.

(3) "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978. This EPA publication includes thin-layer chromatography (TLC) methods.

(4) "Methods for Analysis of Organic Substances in Water," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A3 (1972).

(5) The method may be extended to include a-BHC, w-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

(6) "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).

(7) Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A of 40 CFR 136 as published in the October 26, 1984 Federal Register) in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported.

Table 9.1E
List of Approved Radiological Test Procedures

Parameter and Units	Methods	Reference (Method No. or Page) EPA[1]	Standard Methods 16th Ed.	ASTM	USGS[2]
1. Alpha-Total, pCi per liter	Proportional or scintillation counter	900.0	703	D1943-81	pp. 75 and 78[3]
2. Alpha-Counting error, pCi per liter	Proportional or scintillation counter	Appendix B	703	D1943-81	p. 79
3. Beta-Total, pCi per liter	Proportional counter	900.0	703	D1890-81	pp. 75 and 78[3]
4. Beta-Counting error, pCi per liter	Proportional counter	Appendix B	703	D1890-81	p. 79
5. (a) Radium-Total, pCi per liter	Proportional counter	903.0	705	D2460-70	
(b) ^{226}Ra, pCi per liter	Scintillation counter	903.1	706	D3454-79	p. 81
6. Radium -228, pCi per liter	Beta counter	9320[4]	-	-	-
7. Gross Alpha and Gross Beta	Proportional counter	9310[4]	-	-	-
8. Alpha-Emitting Radium Isotopes	Proportional counter	9315[4]	-	-	-

Table 9.1E Notes

(1) "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/4-80-032 (1980) update), U.S. Environmental Protection Agency, August 1980.

(2) Fishman, M. J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survay, Open-File Report 76-177 (1976).

(3) The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the "total."

(4) "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, USEPA, September 1986.

Table 9.1F References and Sources

Table	Parameters	References and Sources
9.1A - EPA	1-5	"Microbiological Methods for Monitoring the Environment, Water and Wastes," United States Environmental Protection Agency, EPA-600/8-78-017, 1978. ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268
9.1A - Standard Methods	1-5	Standard Methods for the Examination of Water and Wastewater, Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 16th Edition, 1986. Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, DC 20036.
9.1B - Standard Methods	1-46, 48-75	
9.1D - Standard Methods	1, 8, 11, 12, 15, 17-20, 26, 28, 32, 33, 35, 40, 41, 44, 46, 48, 52-54, 64, 66, 67, 69, 70	
9.1E - Standard Methods	1-5	
9.1C - Other (Standard Methods Supplement)	13, 56	"Selected Analytical Methods Approved and Cited by the the United States Environmental Protection Agency," Supplement to the 15th Edition of Standard Methods for the Examination of Water and Wastewater (1981). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20036.
9.1D - Other (Standard Methods Supplement)	2-7, 13, 14, 16, 21-23, 25, 29-31, 37, 38, 39, 41-45, 47, 49, 50, 51, 56-63, 65, 68	
9.1A - U.S. Geological Survey (USGS)	1, 2, 3, 5	"Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples," edited by P. E. Greeson, T. A. Ehlke, G. A. Irwin, B. W. Lium, and K. V. Slack: U.S. Geological Survey, Techniques of Water-Resources Investigation (USGS TWRI), Book 5, Chapter A4 (1977). Revised edition, 332 pages. Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. Government Printing Office.

Table 9.1F (Continued)

Table	Parameters	References and Sources
9.1B - EPA	1-13, 15-48, 50-75	"Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, United States Environmental Protection Agency, March, 1979. Available from: ORD Publications, CERI, U.S. Enviornmental Protection Agency, Cincinnati, Ohio 45268.
9.1B - ASTM	1, 2, 4, 6, 8, 11-13, 15-17, 19, 20, 22-25, 27, 28, 30-35, 37-40, 42-44, 46, 48, 50, 52, 60, 61, 63-65, 67, 68, 73-75	"Annual Book of Standards, Part 31, Water," American Society for Testing and Materials, (1979-1984 as noted in Table 9.1B). Available from: American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.
9.1D - ASTM	1, 8-11, 15, 18-20, 27, 32, 33, 35, 40, 41, 46, 55, 69	
9.1E - ASTM	1-5	
9.1B - USGS	2, 3, 4, 6-13, 15, 16, 18-23, 25, 27, 28, 30-40, 44, 46, 50, 52-55, 57, 60-66, 71, 73, 75	"Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments," U.S. Department of the Interior, Open File Report, 85-495 (1986). Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. Government Printing Office.
9.1B - Other (AOAC)	2, 4, 9, 12, 15, 16, 19, 22, 30-35, 38, 42-44, 46, 50, 52, 62-65, 75	Official Methods of Analysis of the Association of Official Analytical Chemists, methods manual, 14th Edition (1985). The Association of Official Analytical Chemists, 1111 N. 19th St., Suite 210, Arlington, VA 22209.
9.1B - Other (ANSI)	9, 12, 15, 20, 22, 23, 38, 62	"American National Standard on Photographic Processing Effluents," April 2, 1975. American National Standards Institute, 1430 Broadway, New York, New York 10018.

Table 9-1F. (Continued)

Table	Parameters	References and Sources
9.1B - Other (EPA)	3, 5-8, 10, 12, 13, 19, 20, 22, 27, 30, 32-34, 36, 37, 52, 60-63, 70, 74, 75	The full text of the inductively coupled plasma optical emission spectroscopic test procedure, Method 200.7, is found in Appendix C of 40 CFR Part 136 as published in the October 26, 1984 Federal Register.
9.1B - Other	21	"An Investigation of Improved Procedures for Measurement of Mill Effluent and Receiving Water Color," NCASI Technical Bulletin No. 253, December, 1971. National Council of the Paper Industry for Air and Stream Improvements, Inc., 260 Madison Avenue, New York, New York.
9.1B - Other	4	Ammonia, Automated Electrode Method, Industrial Method Number 379-75WE, dated February 19, 1976, Technicon AutoAnayzer II. Technicon Industrial Systems, Tarrytown, New York 10591.
9.1B - Other	15	Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979. Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.
9.1B - Other	15	OIC Chemical Oxygen Demand Method. Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, Texas 77840.
9.1B - Other	17	ORION Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977. Orion Research Incorporated, 840 Memorial Drive, Cambridge, Massachusetts 02138.

Table 9-1F (Continued)

Table	Parameters	References and Sources
9.1B - Other	22	Bicinchoninate Method for Copper, Method 8506, Hach Handbook of Water Analysis, 1979. Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.
9.1B - Other	28	Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Technicon AutoAnalyzer II. Technicon Industrial Systems, Tarrytown, New York 10591.
9.1B - Other	30	1,10-Phenanthroline Method for Iron, Hach Method 8008. Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.
9.1B - Other	34	Periodate Oxidation Method for Manganese, Method 8034, Hach Handbook for Water Analysis, 1979. Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.
9.1B - Other	40	Nitrite Nitrogen, Hach Method 8507. Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.
9.1B - Other	75	Zincon Method for Zinc, Method 8009, Hach Handbook for Water Analysis, 1979. Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.
9.1B - Other	49	"Direct Determination of Elemental Phosphorous by Gas-Liquid Chromatography," by R. F. Addison and R. G. Ackman, Journal of Chromatography, Volume 47, No. 3, pp. 421-426, 1970. Elsevier/North-Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164.

Table 9-1F (Continued)

Table	Parameters	References and Sources
9.1B - Other (USGS)	69	"Water Temperature-Influential Factors, Field Measurement, and Data Presentation," by H. H. Stevens, Jr., J. Ficke, and G. F. Smoot: USGS-TWRI Book 1, Chapter D1, 1975, 65 pages. U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202.
9.1D - Other (USGS)	1, 11, 14, 17-20, 23, 25, 28, 29, 35, 37, 40-42, 44, 46, 52, 68, 69	"Methods for Analysis of Organic Substances in Water," by D. F. Goerlitz and Eugene Brown: USGS-TWRI Book 5, Chapter A3, 1972, 40 pages. Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202.
9.1C - EPA	1-12, 14-55, 57-97	The full texts of Methods 601-613, 624, 625, 1624 and 1625 are found in Appendix A of 40 CFR Part 136 as published in the October 26, 1984 Federal Register.
9.1D - EPA	1, 8-11, 15, 16, 19, 20, 28, 32-36, 40, 41, 69	
9.1C - Other (EPA)	7, 13, 22, 24, 27, 56, 66, 76-83, 88, 89, 91, 93	"Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater."
9.1D - Other (EPA)	1-8,11-13, 15-24, 26, 28, 30-33, 35, 38-41, 43-54, 56-70	Environmental Monitoring and Support Laboratory, United States Environmental Protection Agency, Cincinnati, Ohio 1978. ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.
9.1E - EPA	1-5	"Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/4-80-032 (1980 Update), United States Environmental Protection Agency, 1980. ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.
9.1E - USGS	1-5	"Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," by M. J. Fishman and Eugene Brown; U.S. Geological Survey Open File Report 76-77 (1976). U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202.

Table 9.2
Method Summary for Organic Compounds in
Transformer Fluid, Waste Oils, Soil, Sediment, Solid and Liquid Wastes

Parameter	Method No.	Preferred Equipment	Detection Limit	Reference	Sample Matrix
Polychlorinated Biphenyls (PCBs)	-	Gas Chromatography	1.0 mg/kg	EPA, 1981[1]	Transformer Fluid and Waste Oil
Polychlorinated Biphenyls (PCBs)	-	Gas Chromatography	0.1 mg/kg	EPA, 1981[2]	Solid, Sediment
Organochlorine Pesticides	-	Gas Chromatography	0.002 mg/kg	EPA, 1981[2]	Solid, Sediment
Purgeable Organics	-	Gas Chromatography/ Mass Spectrometry	0.1 mg/kg	EPA, 1981[2]	Solid, Sediment, Tissue
Base-Neutrals and Acid Extractables	-	Gas Chromatography/ Mass Spectrometry	1.0 mg/kg	EPA, 1981[2]	Solid, Sediment, Tissue
Halogenated Volatile Organics	8010	Gas Chromatography	0.03 to 0.52)g/l[a]	EPA, 1986[3]	Solid Waste
Nonhalogenated Volatile Organics	8015	Gas Chromatography	Not cited	EPA, 1986[3]	Solid Waste
Aromatic Volatile Organics	8020	Gas Chromatography	0.2 to 0.4)g/l[a]	EPA, 1986[3]	Solid Waste
Acrolein, Acrylonitrile, and Acetonitrile	8030	Gas Chromatography	0.5 to 0.7)g/l[a]	EPA, 1986[3]	Solid Waste
Phenols	8040	Gas Chromatography	0.14 to 16)g/l[b]	EPA, 1986[3]	Solid Waste
Phthalate Esters	8060	Gas Chromatography	0.29 to 31)g/l[b]	EPA, 1986[3]	Solid Waste
Organochlorine Pesticides and PCBs	8080	Gas Chromatography	0.002 to 0.24[b]	EPA, 1986[3]	Solid Waste

Table 9.2 (Continued)
Method Summary for Organic Compounds in
Transformer Fluid, Waste Oils, Soil, Sediment, Solid and Liquid Wastes

Parameter	Method No.	Preferred Equipment	Detection Limit	Reference	Sample Matrix
Nitroaromatics and Cyclic Ketones	8090	Gas Chromatography	0.01 to 15.7)g/l[b]	EPA, 1986[3]	Solid Waste
Polynuclear Aromatic Hydrocarbons	8100	High Performance Liquid Chromatography	Not cited	EPA, 1986[3]	Solid Waste
Chlorinated Hydrocarbons	8120	Gas Chromatography	0.03 to 1.34)g/l[b]	EPA, 1986[3]	Solid Waste
Organophosphorous Pesticides	8140	Gas Chromatography	0.03 to 5.0)g/l[b]	EPA, 1986[3]	Solid Waste
Chlorinated Herbicides	8150	Gas Chromatography	0.07 to 249)g/l[b]	EPA, 1986[3]	Solid Waste
Volatile Organics	8240	Gas Chromatography/ Mass Spectrometry	(c)	EPA, 1986[3]	Solid Waste
Semivolatile Organics; Packed Column Technique	8250	Gas Chromatography/ Mass Spectrometry	(d)	EPA, 1986[3]	Solid Waste
Semivolatile Organics Capillary Column Technique	8270	Gas Chromatography/ Mass Spectrometry	(d)(e)	EPA, 1986[3]	Solid Waste
Polychlorinated Dibenzo-P-Dioxins and Polychlorinated Dibenzofurans	8280	Gas Chromatography/ Mass Spectrometry	Not cited	EPA, 1986[3]	Solid Waste
Polynuclear Aromatic Hydrocarbons	8310	HPLC	0.013 to 2.3)g/l[b]		Solid Waste

Table 9.2 (Continued)

(1) "The Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils," Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, U. S. Environmental Protection Agency, Cincinnati, Ohio, April, 1981.

(2) "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue," Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, U. S. Environmental Protection Agency, Cincinnati, Ohio, April, 1981.

(3) "Test Methods for Evaluating solid Waste, Physical/Chemical Methods," Waste Characterization Branch, Office of Solid Waste, U. S. Environmental Protection Agency, Washington, D.C., July 1982.

(a) Practical quantitation limits, for various matrixes are provided for guidance and may not always be achievable. Methods detection unit listed in column of table is multiplied by the following factors to determine PQL(s):

Ground water	10
Low-level soil	10
Water miscible liquid waste	500
High level soil and sludge	1250
Non-water miscible waste	1250

(b) Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable. Multiply the listed method detection limit times the following factors for these matrixes:

Ground water	10
Low level soil by sonication with GPC cleanup	670
High level soil and sludges by sonication	10,000
Non-water miscible waste	100,000

(c) PQL of Method 8240 for an individual compound is approximately 5)g/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 5)g/l for ground water. PQLs will be proportionately higher for sample extracts and samples that require dilution or reduced sample size to avoid saturation of the detector. Sample PQLs are highly matrix dependent. The PQLs listed are provided for guidance and may not always be achievable. Multiply the listed method detection limit times the following factors for these matrixes:

Water miscible liquid waste	50
High-level soil and sludges	125
Non-water miscible waste	500

Table 9.2 (Continued)

(d) The PQL of Method 8250 for determining an individual compound is approximately 1 mg/kg (net weight) for soil/sediment samples, 1-200 mg/kg for wastes (dependent on matrix and method of preparation), and 10)g/l for ground water samples. PQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector. Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable. Multiply the listed method detection limit times the following factors for these matrixes:

Ground water	10
Low-level soil by sonication with GPC cleanup	670
High-level soil and sludges by sonication	10,000
Non-water miscible waste	100,000

(e) The PQLs listed are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable. Multiply the listed method detection limit times the following factors for these matrixes:

Medium level soil and sludges by sonicator	7.5
Non-water miscible waste	75

Table 9.3
Method Summary for Trace Metals by Atomic Absorption Spectrophotometry
and Inductively Coupled Argon Plasma Emission Spectrometry

Parameter	Direct Aspiration Detection Limit (mg/l)	Direct Aspiration Reference	Graphite Furnace Detection Limit (mg/l)	Graphite Furnace Reference	ICAP[1],[8] Detection Limit (mg/l)
Trace Metals Analysis					
Aluminum	0.1	EPA 202.1[2];7020[7]	0.005	EPA 202.2	0.05, 0.045[8]
Antimony	0.2	EPA 204.1; 7040	0.005 0.003	EPA 204.2 7041	0.03, 0.032
Arsenic	1.0	EPA 206.2	0.001	EPA 206.2; 7060	0.05, 0.053
Barium	0.1	EPA 208.1; 7080	0.002	EPA 208.2	0.002, 0.002
Beryllium	0.01 0.005	EPA 210.1 7090	0.001 0.0002	EPA 210.2 7091	0.0003, 0.0003
Boron	0.1	SM 404 A[4]; EPA 212.3			0.005, 0.005
Cadmium	0.005	EPA 213.1; 7130	0.0001	EPA 213.2; 7131	0.004, 0.004
Calcium	0.01	EPA 215.1; 7140	-	-	0.01, 0.01
Chromium	0.05	EPA 218.1; 7190	0.005 0.001	EPA 218.2 7191	0.007, 0.007
Cobalt	0.1 0.05	EPA 219.1 7200	0.001	EPA 219.2; 7201	0.007, 0.007
Copper	0.02	EPA 220.1; 7210	0.001	EPA 220.2	0.006, 0.006
Iron	0.03	EPA 236.1; 7380	0.005	EPA 236.2	0.007, 0.007
Lead	0.1	EPA 239.1; 7420	0.005 0.001	EPA 239.2 7421	0.040, 0.042
Lithium	0.01	Perkin-Elmer[3]	-	-	-
Magnesium	0.001	EPA 242.1; 7450	-	-	0.03, 0.30
Manganese	0.01	EPA 243.1; 7460	0.0005	EPA 243.2	0.002, 0.002
Mercury	0.5	Perkin-Elmer[3]	0.0002[6]	EPA 245.1[6];7471[7]	-

Table 9.3 (Continued)

Method Summary for Trace Metals by Atomic Absorption Spectrophotometry and Inductively Coupled Argon Plasma Emission Spectrometry

	Direct Aspiration		Graphite Furnace		ICAP[1],[8]
Parameter	Detection Limit (mg/l)	Reference	Detection Limit (mg/l)	Reference	Detection Limit (mg/l)
Molybdenum	0.1	EPA 246.1; 7480	0.005 0.001	EPA 246.2 7481	0.008, 0.008
Nickel	0.04	EPA 249.1; 7520	0.001	EPA 249.2	0.015, 0.015
Osmium	1.0	7550			
Potassium	0.01	EPA 258.1; 7610	-	-	-
Selenium	0.1	Perkin-Elmer[3]	0.002	EPA 270.2; 7240	0.08, 0.075
Silicon	0.1	SM 303C[4]	-	-	0.005, 0.058
Silver	0.01	EPA 272.1; 7760	0.001	EPA 272.2	0.007, 0.007
Sodium	0.005 0.002	EPA 273.1 7770	-	-	0.03, 0.029
Strontium	0.1	SM 303A[4]	-	-	-
Thallium	0.1	EPA 279.1; 7840	0.005 0.001	EPA 279.2 7841	0.04, 0.040
Tin	1.0 0.8	EPA 282.1 7870	0.005	EPA 282.2	-
Titanium	0.5	EPA 283.1	0.005	EPA 283.2	-
Tungsten	10	Perkin-Elmer[3]	-	-	-
Uranium	-	-	0.1	Perkin-Elmer[5]	-
Vanadium	0.2	EPA 286.1; 7910	0.005 0.004	EPA 286.2 7911	0.008, 0.008
Zirconium	10	Perkin Elmer[3]	-	-	-
Zinc	0.01 0.005	EPA 289.1 7950	0.001	EPA 289.2	0.002, 0.002

Table 9.3 (Continued)

(1) Samples to be analyzed according to EPA Method 200.7 as per manufacturer's specifications.

(2) "Methods for Chemical Analysis of Water and Wastes," Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio, March, 1979. The numerical designation following the EPA prefix indicates the pertinent method number in the above reference.

(3) "Analytical Methods for Atomic Absorption Spectrophotometry," Perkin-Elmer Corporation, Norwalk, Connecticut, September 1976, or as prescribed by other instrument manufacturers.

(4) Standard Methods for the Examination of Water and Wastewater, 16th edition, American Public Health Association, American Waterworks Association, and the Water Pollution Control Federation, 1986. The numerical designation following the SM prefix indicates the pertinent method number in the above reference. The Standard Methods reference for boron is curcumin colorimetric procedure. All other references to Standard Methods are atomic absorption spectrophotometry procedures.

(5) "Analytical Methods for Atomic Absorption Spectrophotometry Using the HGA Graphite Furnace," Perkin-Elmer Corporation, Norwalk, Connecticut, 1978.

(6) The indicated analytical procedure for mercury is the cold vapor technique described in the referenced EPA procedure.

(7),(8) "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, USEPA, September 1986.

Table 9.4
Method Summary
General Chemical Analysis of Water and Wastewater

Parameter	Method	Instrumentation or Equipment	Detection Limit	Reference Method
• Acidity	Titrimetric	Buret, pH meter	1 mg/l	
- Available				SM 402[1]
- Total				EPA 305.1[3]; SM 402[1]
• Alkalinity	Titrimetric	Buret, pH meter	1 mg/l	SM 403[1], EPA 310.1[3]
- Phenolphthalein				
- Total				
- Bicarbonate				
- Carbonate				
- Hydroxide				
• Ammonia	Colorimetric	Spectrometer	0.1 mg/l	SM 417B[1]
	Titrimetric	Buret	1.0 mg/l	SM 417D[1]; 350.2[3]
	Specific Ion Electrode	Specific Ion Meter	0.1 mg/l	EPA 350.3[3]; SM 417E or F[1]
• BOD	Electrometric	Dissolved Oxygen Meter/ Electrode	1.0 mg/l	SM 507[1], 405.1[3]
• Bromide	Colorimetric	UV/VIS Spectrometer	1.0 mg/l	320.1[3]
• Carbon				
- Total	Combustion, Infrared	TOC Analyzer	1 mg/l	EPA 415.1[3]
- Inorganic	Combustion, Infrared	TOC Analyzer	1 mg/l	EPA 415.1[3]
- TOC	Combustion, Infrared	TOC Analyzer	1 mg/l	EPA 415.1[3]
• Carbon Dioxide (CO_2)				
- Free	Titrimetric	Buret/pH Meter	1 mg/l	SM 406B[1]
- Total	Calculation			SM 406C[1]
• Chloride	Titrimetric	Buret/Indicator	0.5 mg/l	EPA 325.3[3]; SM 407[1]

Table 9.4 (Continued)
Method Summary
General Chemical Analysis of Water and Wastewater

Parameter	Method	Instrumentation or Equipment	Detection Limit	Reference Method
(Mercuric Nitrate)				
• Chlorine Demand	Colorimetric	UV/VIS Spectrometer	1 mg/l	SM 409A[1]
• Chlorine Residual	Colorimetric	UV/VIS Spectrometer	1 mg/l	SM 408E[1]; EPA 330.5[3]
• Chromium (Hexavalent)	Colorimetric	UV/VIS Spectrometer	0.1 mg/l	SM 312B
• COD	Dichromate Reflux/ Titration	Buret/Indicator	1 mg/l	SM 508A[1];410.1[3]
• Color				
- Apparent	Dominant Wavelength	UV/VIS Spectrometer	-	SM 204B[1]; EPA 110.3[3]
- True				SM 204B[1]
• Cyanate (CNO)[a]	Specific Ion Electrode	Specific Ion Analyzer	0.1 mg/l	SM 412J[1]
• Cyanide				
- Free	Distillation/Colorimetric	UV/VIS Spectrometer	0.02 mg/l	EPA 335.2[3]; SM 412D[1]
- Total	Distillation/Colorimetric	UV/VIS Spectrometer		EPA 335.2[3]; SM 412D[1]
- Amenable and Total	Distillation/Colorimetric	UV/VIS Spectrometer		EPA 335.1[3]; SM 412F[1]
• Eh (Redox potential)	Electrometric	Specific Ion Meter	1 mV	ASTM 31-D1498
• Fluoride				
- Electrode (CDTA)	Specific Ion Electrode	Specific Ion Meter	0.1 mg/l	EPA 340.2[3]; SM 413B[1]
- Distillation				EPA 340.1[3]; SM 413C[1]
• Halogen (Total Organic)	Combustion/Electrolytic	TOX Analyzer	0.01 mg/l	EPA 450.1 Interim[3]
(TOX)	Conductivity Detector			
• Hardness				
- Total	Titrimetric	Buret/Indicator	1.0 mg/l	EPA 130.2[3]; SM 314B[1]
- Calcium			1.0 mg/l	ASTM 31-D511
- Magnesium			1.0 mg/l	ASTM 31-D511

Table 9.4 (Continued)
Method Summary
General Chemical Analysis of Water and Wastewater

Parameter	Method	Instrumentation or Equipment	Detection Limit	Reference Method
• Iodide	Colorimetric	UV/VIS Spectrometer	0.1 mg/l	EPA 345.1[3]
• Iron	Specific Ion	Specific Ion Meter	1.0 mg/l	-
- Ferrous	Colorimetric	UV/VIS Spectrometer	0.1 mg/l	SM 315.B[1]
- Ferric	Spectrometric	AAS	0.1 mg/l	SM 315A[1]
• Nitrate				
- Electrode	Specific Ion Electrode	Specific Ion Meter	1.0 mg/l	SM 418B, 419[1]
- Colorimetric	Brucine Sulfate	UV/VIS Spectrometer	0.1 mg/l	EPA 352.1[3]
• Nitrite	Colorimetric	UV/VIS Spectrometer	0.05 mg/l	EPA 354.1[3]
• Nitrogen				
- Organic (Kjeldahl-Ammonia)	Digestion/Distillation/ Specific Ion	Digestion Rack Specific Ion Meter	1.0 mg/l	SM 420[1]; EPA 351.3[3] EPA 351.3[3], SM 417E or F[1]
- Kjeldahl	Digestion/Distillation/ Specific Ion	Digestion Rack Specific Ion Meter	0.1 mg/l	EPA 351.3[3], SM 420[1]
	Titrimetric	Buret	0.1 mg/l	EPA 351.3[3]; SM 417D[1]
• Oil and Grease				
- Gravimetric	Extraction-Gravimetric	Separatory Funnel/Balance	0.5 mg/l	EPA 413.1[3]; SM 503A[1]
• Oxygen, Dissolved	Elecrometric	Dissolved Oxygen Meter	0.1 mg/l	EPA 360.1[3]; SM 421F[1]
Phenolics				
- Distillation/Extraction	Extraction/Colorimetric	UV/VIS Spectrometer	0.01 mg/l	EPA 420.1; EPA 420.2[3]
• Phosphorus				
- Total (Persulfate)	Persulfate Digestion/ Colorimetric	UV/VIS Spectrometer	0.01 mg/l	SM 424C (III); F or G[1]

Table 9.4 (Continued)
Method Summary
General Chemical Analysis of Water and Wastewater

Parameter	Method	Instrumentation or Equipment	Detection Limit	Reference Method
• Residue				
- Filterable, Dissolved (TDS)	Filtration/Gravimetric	Analytical Balance	1.0 mg/l	EPA 160.1[3]; SM 209B[1]
- Nonfilterable, Suspended (TSS)	Filtration/Gravimetric	Analytical Balance	1.0 mg/l	EPA 160.2[3]; SM 209C[1]
- Total (TS)	Gravimetric	Analytical Balance	1.0 mg/l	EPA 160.3[3]; SM 209A[1]
- Settleable (volumetric)	Settling Procedure	Imhoff Cone	1.0 mg/l	EPA 160.5[3]; SM 209E[1]
- Volatile (TVS,VSS)	Filtration/Gravimetric	Analytical Balance	1.0 mg/l	EPA 160.4[3]; SM 209D[1]
• Specific Conductance	Conductimetric	Conductivity Meter	1 0 mho/cm	EPA 120.1[3]; SM 205[1]
• Specific Gravity	Gravimetric	Analytical Balance	0.1 mg/l	SM 213E[1]
• Sulfate	Turbidimetric	UV/VIS Spectrometer	1.0 mg/l	EPA 375.4
• Sulfide	Titrimetric	Buret/Indicator	0.5 mg/l	EPA 376.1[3]; SM 427D[1]
• Sulfite	Titrimetric	Buret/Indicator	0.5 mg/l	EPA 377.1[3]; SM 427C[1]
• Surfactants (MBAS)	Extraction/Colorimetric	UV/VIS Spectrometer	0.05 mg/l	SM 512B[1]; EPA 425.1[3]
• Thiocyanate	Colorimetric	UV/VIS Spectrometer	1.0 mg/l	SM 412L[1]
• Thiosulfate	Titrimetric	Buret Indicator	0.1 mg/l	Guo, Phm & Jank, B. E. Wastewater Technology Center Environmental Protection Service, Environmental Canada, May, 1980.

Table 9.4 (Continued)
Method Summary
General Chemical Analysis of Water and Wastewater

Parameter	Method	Instrumentation or Equipment	Detection Limit	Reference Method
• Turbidity	Nephelometric	Turbidimeter	1 NTU	EPA 180.1[3]; SM 214A[1]
• Viscosity	Saybolt Method	Viscometer/Water Bath	-	USGS Methods for Collection and Analysis of Water Samples, 1968.

References

1 American Public Health Association. Standard Methods for the Examination of Water and Wastewater (16th ed.). APHA: Washington D.C., 1986.
2 Environmental Protection Agency. Methods for the Chemical Analysis of Water and Wastes. EPA: Cincinnati, OH, 1974.
3 Environmental Protection Agency. Methods for the Chemical Analysis of Water and Wastes. EPA: Cincinnati, OH, 1979.
4 Federal Register. Volume 44 (75050). December 18, 1979.
5 Federal Register. Volume 44 (69464). December 3, 1979.
6 Federal Register. Volume 41 (57280-86). December 1, 1976.
7 Perkin Elmer Methods Manual, 1975.

Table 9.5
Analytical Procedures Summary
Soil, Sediment, Sludge Analysis

Parameter	Method	Instrumentation or Equipment	Detection Limit	Reference Method
• Acid/Base Potentials	Calculation from Neutralization Potential and % Sulfur Determination	Sulfur Analyzer	0.1%	(1)
• Neutralization Potential	Titration to pH 7.0	Burat, pH Meter	0.1%	(1)
• Maximum Potential Acidity	Calculation from % Total Sulfur Determination	Sulfur Analyzer	0.1%	(1)
• Leaching Testing				
- TEP	Aqueous Extraction at controlled pH	Rotary Extractor, pH Meter	-	(2)
- ASTM	Aqueous Extraction	Agitator/Extractor	-	(3)
- CAM-WET Procedure	30 day Cumulative Extraction Two Extracting Solutions; Water and Citrate-HaCl Buffer	Agitator/Extractor	-	(4)
• pH (1:1)	1:1 Soil to Water Paste	pH Meter	0.05	(1)
• Electrical Conductivity	Extract from Saturated Soil Paste	Conductivity Meter	1 mhos/cm	(1)
• Saturation Paste Determinations (Sodium Absorption Ratio)	Atomic Absorption Spectrophotometry of Extract from Saturated Soil Paste of Calcium, Magnesium, Sodium	Atomic Absorption Spectrophotometer	1 ppm	(1)

Table 9.5
Analytical Procedures Summary
Soil, Sediment, Sludge Analysis

Parameter	Method	Instrumentation or Equipment	Detection Limit	Reference Method
• Particle Size Determinations	Hydrometer or Pipette, Drying Oven	Hydrometer, Oven	1% of size	(1)
• Exchangeable Cations (Cation Exchange Capacity)	Atomic Absorption Spectrophotometry of Calcium Magnesium, Potassium, or Sodium in Soil Extract	Atomic Absorption Spectrophotometer	1 ppm	(1)
• Organic Matter (Carbon)	Walkley-Black Dichromate Oxidation, Titration	Digester, Buret	0.1%	(1)
• Heavy Metals	Digestion followed by Atomic Absorption Spectrophotometry	Atomic Absorption Spectrophotometer	Variable	(1)
• Proximate Analysis				
- Percent Ash	Gravimetric	Analytical Balance, Muffle Furnace	1%	(5)
- Percent Moisture	Gravimetric	Analytical Balance, Drying Oven	1%	(5)
- Percent Solids	Gravimetric	Analytical Balance, Drying Oven	1%	(5)
• Sulfur Determinations				
- Total	Sulfur Analyzer	Sulfur Analyzer	0.05%	(6)
- Sulfate	Extraction, Sulfur Analyzer	Sulfur Analyzer	-	(6)
- Pyritic	Extraction, Sulfur Analyzer	Sulfur Analyzer	0.05%	(6)
- Pyritic + Organic	Extraction, Sulfur Analyzer	Sulfur Analyzer	0.05%	(6)
• Specific Gravity	Pycnometer	Pycnometer	-	(7)

Table 9.5
Analytical Procedures Summary
Soil, Sediment, Sludge Analysis

Parameter	Method	Instrumentation or Equipment	Detection Limit	Reference Method
• Oil and Grease	Drying with $MgSO_4 \cdot H_2O$ followed by Freon extraction	Soxhlet Extactor, Vacuum Pump	0.5 ppm	(8)
• Total Organic Carbon	Oxidation to CO_2 and IR Detection	Total Carbon Analyzer	1 ppm	(9)
• Bomb Preparation	Elevated Temperature and Pressure Acid Digestion	Digestion Bomb	-	(10)
• NH_3-N	KCl Extraction Ion Meter	Specific Ion Meter	0.1 ppm	(11)
• NO_3-N	Colorimetric	Spectrometer	0.1 mg/l	(8)
• TKN-N	Kjeldahl Digestion, Specific Ion Electrode	Kjeldahl Digestion Apparatus, Specific Ion Meter	1 ppm	(12)
• Phosphorus	Persulfate Digestion, Colorimetric	Spectrophotometer	0.01 ppm	(12)
• Phosphate-Phosphorus	NH_4F or $NaHCO_3$ Extraction (Dependent on pH <7.00 or >7.00	Spectrophotometer	0.05 ppm	(11)
• Potassium	SAR	AAS	0.1 ppm	(11)
• Potassium	Double-Acid Extraction	AAS	0.1 ppm	(11)
• Lithium Metaborate Fusion	Muffle Furnace Digestion	Muffle Furnace	0.1 ppm	(1)

Table 9.5 (Continued)

Method References

(1) Field and Laboratory Methods Applicable to Overburdens and Minesoil, by Andrew A. Sobek, William A. Schuller, John R. Freeman, and Richard M. Smith, College of Agriculture and Forestry, West Virginia University, Morgantown, West Virginia, March, 1978.

(2) Federal Register, Volume 45, No. 98, Book 2, May 19, 1980.

(3) 1982 Annual Book of ASTM Standards, Part 31, "Standard Test Method for Shake Extraction of Solid Waste with Water," American Society for Testing and Materials, Philadelphia, PA.

(4) California Assessment Manual for Hazardous Wastes, Hazardous Materials Management Section, California Department of Health Services, Sacramento, CA, June 1981.

(5) Methods of Soil Analysis, Part I, Physical and Mineralogical Properties, Including Statistics of Measurement and Sampling, C.A. Black, Monograph 9, American Society of Agronomists, Madison, WI, 1965.

(6) Methods of Analyzing and Testing Coal and Coke, Bulletin 638, U. S. Bureau of Mines, United States Department of the Interior, Washington, D.C. 1967.

(7) 1978 Annual Book of ASTM Standards, Part 19," Natural Building Stones; Soil and Rock," American Society for Testing and Materials, Philadelphia, PA.

(8) Standard Methods for the Examination of Water and Wastewater, 16th Edition, Method 503A, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1986, pp. 464 to 465.

(9) Operating Procedures Manual for 0524B Total Carbon System, Oceanography International Corporation, College Station, Texas, no date, pp. 10-2 to 10-4.

(10) "Perkin Elmer Autoclave-3 Instructions," Instrument Division, Perkin-Elmer Corporation, Norwalk, CT, no date.

(11) Methods of Soil Analysis, Part II, Chemical and Microbiological Properties, C. A. Black, Monograph 9, American Society of Agronomists, Madison, WI, 1965, pp. 1191 to 1195.

(12) Soil Testing and Plant Analysis, L. M. Walsh and J. D. Beaton, Soil Science Society of America, Madison WI, 1973.

Table 9.6
Method Summary
Waste Disposal Analyses
of Water, Wastewater, Soil and Waste Samples

Parameter	Method	Reference	Detection Limit	Equipment Description
BTU (Heat Content)	D240(L)(1) D271(S)(2)	5 5	0.05 Wt.%	Bomb Calorimeter
% Moisture (Water Content)	E203(L) D2216(S)	5 5	1 mg/ℓ 0.05 wt.%	Karl Fischer Titrator
% Solids (Solid Content)	160.3	6	1% (wt.)	Filtration/Oven
Organic Halogen Content	D808(L) D1305(S)	5	0.05 wt.%	Bomb Calorimeter
Organic Sulfur Content	D129(L) D3177(S)	5	0.05 wt.%	Bomb Calorimeter
% Ash Content	160.4	2	0.05 wt.%	Muffle Furnace, Balance
Ignitability	1010 1020	2 2	±2°F ±1°F	Pensky Martens Flash Point Tester Setaflash Method Closed Cup
Reactivity	8.3	2	1 ppm	Reaction Vessel
EP Toxicity	1310	2	0.1 mg/ℓ	Extractors/AAS Instrumentation
Corrosivity	1110	2	0.1 pH unit	pH Meter
Organochlorine Pesticides/PCBs	608(L) 8080(S)	1,3 2	0.1 µg/ℓ See Table 9.2	Gas chromatograph
% Acidity	402	4	1 mg/ℓ	Titration
% Alkalinity	403	4	1 mg/ℓ	Titration
Conductivity (Specific Conductance)	120.1	6	1 µmho/cm	Conductivity Meter
Ammonia Nitrogen	350.3	6	1 mg/ℓ	Distillation/Specific Ion Meter

Table 9.6 (Continued)
Method Summary
Waste Disposal Analyses
of Water, Wastewater, Soil and Waste Samples

Parameter	Method	Reference	Detection Limit	Equipment Description
TOC (Total Organic Carbon)	415.1	6	1 mg/ℓ	TOC Analyzer
TOX (Total Organic Halogen)	450.1 (9020)	6 2	0.05 mg/ℓ 5 µg/ℓ	TOX Analyzer
Chloride	325.3	6	1 mg/ℓ	Titration
Density (Specific Gravity)	213E	4	-	Pycnometer
Oil and Grease	413.1	6	0.5 mg/ℓ	Extraction/ Gravimetric
Viscosity	D88	5	1 CPS	Viscometer (Water Bath)
pH	9040	2	0.1 pH unit	pH Meter
Sulfate	375.4	6	1 mg/ℓ	Titration
Cyanide	9010	2	0.05 mg/ℓ 1 µg/g	Colorimetric

Table 9.6 (Continued)

REFERENCES:

1. Federal Register, Volume 44, December 3, 1979, 40 CFR Part 136, pp. 69464 to 69575.
2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Waste Characterization Branch, Office of Solid Waste, 3rd Edition U.S. Environmental Protection Agency, Washington, D.C., September 1986.
3. Manual of Analytical Methods for Analysis of Pesticides in Human and Environmental Samples, U.S. Environmental Protection Agency, EPA 600/8-80-038, June 1980.
4. Standard Methods for the Examination of Water and Wastewater, 16th Edition, 1986, Washington, D.C.
5. American Society for Testing and Materials, Philadelphia, PA.
6. Methods for Chemical Analysis of Water and Wastes, U.S. Environmental Protection Agency, EPA-600/4-79-020, March 1979.

FOOTNOTES:

(1) L indicates the method for liquid samples.

(2) S indicates the method is for semisolid and solid samples.

Table 9.7
Method Summary
Compatibility/Characterization Analyses
of Hazardous Wastes

Parameter	Method No.	Reference	Limit	Equipment Description
PCBs	608 8080 Section 8	1 2 3	0.1)g/P (water) See Table 9.2 1.0 ppm (air)	Gas Chromatograph
Water Reactivity	15.5.11	6	-	Reaction Vessel
pH	9040 -	2 4	0.01 pH unit 1 pH Unit	pH meter Test Paper
Flammability	7.1	2	±2°F	Flash Point Tester
Radioactivity	-	4 6	- Sensitivity 800 Counts/Minute	Survey Meter Geiger-Mueller Counter
Water Solubility	15.5.1	6	-	Beaker
Cyanide	412E Hach (spot test)	6 4	0.1 ppm 2.0 ppm	Specific Ion Electrode Colorimetric (semiquantitative)
Sulfide	-	4	N/P(1)	Test Paper
Oxidizers	-	4	N/P	Test Paper
Peroxides	-	4	N/P	Test Paper

Table 9.7 (Continued)
Method Summary
Compatibility/Characterization Analyses
of Hazardous Wastes

Parameter	Method No.	Reference	Limit	Equipment Description
Oxidation/Reduction Potential (Eh)	D1498	7	1mV	Ion Analyzer
Specific Gravity	213E	5	-	Pycnometer
Hexane Solubility	-	6	-	Reaction Vessel

References:

1. Federal Register, Volume 44, December 3, 1979, 40 CFR Part 136, pp. 69464 to 69575.
2. Test Method for Evaluating Solid Waste, Physical/Chemical Methods, Waste Characterization Branch, Office of Solid Waste, U.S. Environmental Protection Agency, Washington, D.C., 3rd Edition, September 1986.
3. Manual of Analytical Methods for Analysis of Pesticides in Human and Environmental Samples, U.S. Environmental Protection Agency, EPA 600/8-80-038, June 1980.
4. Analysis performed as per manufacturer's recommended procedure.
5. Standard Methods for the Examination of Water and Wastewater, 16th Edition, 1986, Washington, D.C.
6. Drum Consolidation Protocol (Draft), U.S. Environmental Protection Agency, August 1981.
7. American Society for Testing and Materials, Philadelphia, PA.

Footnotes:

(1) N P indicates results are qualitative, and the quantitative results for precision are not applicable.

TABLE 9.8
LIST OF APPROVED TEST PROCEDURES
FOR SOIL AND SOLID WASTES(1)

Parameter	Section No.	Method No.
CHARACTERISTICS OF SOLID WASTE	8.0	
• Ignitability	8.1	
- Pensky-Martens Closed Cup Method	8.1	1010
- Setaflash Closed Cup Method	8.1	1020
• Corrosivity	8.2	
- Corrosion Towards Steel	8.2	1110
• Reactivity	8.3	
• Extraction Procedure Toxicity	8.4	
- Method and Structural Integrity Test	8.4	1310
- Multiple Extraction Procedure	6.0	1320
- Extraction Procedure for Oily Wastes	6.0	1330
SAMPLE PREPARATION TECHNIQUES	4.0	
• Inorganic Techniques	3.0	
- Acid Digestion of Waters for Flame AAS or ICP	3.0	3005
- Acid Digestion of Aqueous Samples and Extracts Flame AAS or ICP	3.0	3010
- Acid Digestion of Aqueous Samples and Extracts for Total Metals for Furnace AAS	3.0	3020
- Dissolution Procedure for Oil, Grease, or Wax	3.0	3040
- Acid Digestion of Sediments, Sludges, and Soils	3.0	3050
• Organic Techniques, Extractions and Preparations	4.2.1	
- Organic Extraction and Sample Preparation	4.2.1	3500
- Separatory Funnel Liquid-Liquid Extraction	4.2.1	3510
- Continuous Liquid-Liquid Extraction	4.2.1	3520
- Soxhlet Extraction	4.2.1	3540
- Sonication Extraction	4.2.1	3550
- Waste Dilution	4.2.1	3580
- Purge and Trap	4.2.1	5030
- Protocol for Analysis of Sorbent Cartridges from Volatile Organic Sampling Train	4.2.1	5040
• Cleanup		
- Cleanup	4.2.2	3600
- Alumina Column Cleanup	4.2.2	3610
- Alumina Column Cleanup and Separation of Petroleum Wastes	4.2.2	3611
- Florisil Column Cleanup	4.2.2	3620
- Silica Gel Cleanup	4.2.2	3630

Table 9.8
(Continued)

Parameter	Section No.	Method No.
- Gel-Permeation Cleanup	4.2.2	3640
- Acid-Base Partition Cleanup	4.2.2	3650
- Sulfur Cleanup	4.2.2	3660
• Screening	4.4	
- Headspace	4.4	3810
- Hexadecane Extraction and Screening if Purgeable Organics	4.4	3820
METHODS FOR DETERMINATION OF METALS	3.0	
• Inductively Coupled Plasma Atomic Emission Spectroscopy	3.3	6010
• Atomic Absorption Methods	3.0	7000
• Aluminum (AA, Direct Aspiration)	3.0	7020
• Antimony (AA, Direct Aspiration)	3.0	7040
• Antimony, (AA, Furnace Technique)	3.0	7041
• Arsenic, (AA, Furnace Technique)	3.0	7060
• Arsenic, (AA, Gaseous Hydride)	3.0	7061
• Barium, (AA, Direct Aspiration)	3.0	7080
• Beryllium, (AA, Direct Aspiration)	3.0	7090
• Beryllium, (AA, Furnace Technique)	3.0	7091
• Cadmium, (AA, Direct Aspiration)	3.0	7130
• Cadmium, (AA, Furnace Technique)	3.0	7131
• Chromium, (AA, Direct Aspiration)	3.0	7190
• Chromium, (AA, Furnace Technique)	3.0	7191
• Chromium, Hexavalent, (Chelation/Extraction)	3.0	7197
• Chromium, Hexavalent, (Colorimetric)	3.0	7196
• Chromium, Hexavalent, (Coprecipitation)	3.0	7195
• Chromium, Hexavalent, (Differential Pulse Polarography)	3.0	7198
• Cobalt (AA, Direct Aspiration)	3.0	7200
• Cobalt (AA, Furnace Technique)	3.0	7201
• Copper, (AA, Direct Aspiration)	3.0	7210
• Iron, (AA, Direct Aspiration)	3.0	7380
• Lead, (AA, Direct Aspiration)	3.0	7420
• Lead, (AA, Furnace Technique)	3.0	7421
• Magnesium (AA, Direct Aspiration)	3.0	7450
• Manganese, (AA, Direct Aspiration)	3.0	7460
• Mercury, in Liquid Waste (Manual Cold Vapor Technique)	3.0	7470

Table 9.8
(Continued)

Parameter	Section No.	Method No.
• Mercury, in Solid or Semisolid Waste (Manual Cold Vapor Technique)	3.0	7471
• Molybdenum (AA, Direct Aspiration)	3.0	7480
• Molybdenum (AA, Furnace Technique)	3.0	7481
• Nickel, (AA, Direct Aspiration)	3.0	7520
• Osmium, (AA, Direct Aspiration)	3.0	7550
• Potassium, (AA, Direct Aspiration)	3.0	7610
• Selenium, (AA, Furnace Technique)	3.0	7740
• Selenium, (AA, Gaseous Hydride)	3.0	7741
• Silver, (AA, Direct Aspiration)	3.0	7760
• Sodium, (AA, Direct Aspiration)	3.0	7770
• Thallium, (AA, Direct Aspiration)	3.0	7840
• Thallium, (AA, Furnace Technique)	3.0	7841
• Tin (AA, Direct Aspiration)	3.0	7870
• Vanadium, (AA, Direct Aspiration)	3.0	7910
• Vanadium, (AA, Furnace Technique)	3.0	7911
• Zinc, (AA, Direct Aspiration)	3.0	7950
ORGANIC ANALYTICAL METHODS	4.3	
• Gas Chromatographic Methods	4.3.1	8000
- Acrolein, Acrylonitrile, Acetonitrile	4.3.1	8030
- Aromatic Volatile Organics	4.3.1	8020
- Chlorinated Herbicides	4.3.1	8150
- Chlorinated Hydrocarbons	4.3.1	8120
- Halogenated Volatile Organics	4.3.1	8010
- Nitroaromatics and Cyclic Ketones	4.3.1	8090
- NonHalogenated Volatile Organics	4.3.1	8015
- Organochlorine Pesticides and PCBs	4.3.1	8080
- Organophosphorus Pesticides	4.3.1	8140
- Phenols	4.3.1	8040
- Phthalate Esters	4.3.1	8060
- Polynuclear Aromatic Hydrocarbons	4.3.1	8100
• Gas Chromatographic/Mass Spectroscopy Methods (GC/MS)	4.3.2	
- GC/MS for Volatile Organics	4.3.2	8240
- GC/MS for Semivolatile Organics, Packed Column Technqiue	4.3.2	8250
- GC/MS for Semivolatile Organics, Capillary Column Technqiue	4.3.2	8270
- Analysis of Polychlorinated Dibenzo-P-Dioxins and Polychlorinated Dibenzofurans	4.3.2	8280
Appendix A: Signal-to-Noise Determination Methods		
Appendix B: Recommended Safety and Handling Procedures for PCDD's/PCOFI		

Table 9.8
(Continued)

Parameter	Section No.	Method No.
• High Performance Liquid Chromatographic Methods (HPLC)	4.3.3	
- Polynuclear Aromatic Hydrocarbons	4.3.3	8310
MISCELLANEOUS TEST METHODS	5.0	
• Cation Exchange Capacity of Soils (Ammonium Acetate)	6.0	9080
• Cation Exchange Capacity of Soils (Sodium Acetate)	6.0	9081
• Chloride (Colorimetric, Automated Ferricyanide AAI)	5.0	9250
• Chloride (Colorimetric, Automated Ferricyanide AAII)	5.0	9251
• Chloride (Titrimetric, Mercuric Nitrate)	5.0	9252
• Total Coliform: Multiple Tube Fermentation Technique	5.0	9131
• Total Coliform: Membrane Filter Technique	5.0	9132
• Total and Amendable Cyanide (Colorimetric, Manual)	5.0	9010
• Total and Amendable Cyanide (Colorimetric, Automated UV)	5.0	9012
• Paint Filter Liquids Test	6.0	9095
• Gross Alpha and Gross Beta	6.0	9310
• Nitrate	5.0	9200
• pH Electrometric Measurement	6.0	9040
• pH Paper Method	6.0	9041
• Phenolics (Spectrophotometric, Manual 4-AAP with Distillation)	5.0	9065
• Phenolics (Colorimetric, Automated 4-AAP with Distillation)	5.0	9066
• Phenolics (Spectrophotometric, MBTH with Distillation)	5.0	9067
• Alpha-Emitting Radium Isotopes	9.0	9315
• Radium, 228	5.0	9320
• Soil pH	6.0	9045
• Specific Conductance	6.0	9050
• Sulfate (Colorimetric, Automated, Chloranilate)	9.0	9035
• Sulfate (Colorimetric, Automated, Methylthymol, Blue,AA II)	9.0	9036
• Sulfate (Turbidimetric)	9.0	9038
• Sulfides	5.0	9030
• Total Recoverable Oil and Grease (Gravimetric, Separatory Funnel Extraction)	5.0	9070
• Oil and Grease Extraction Method for Sludge Samples	5.0	9071

Table 9.8
(Continued)

Parameter	Section No.	Method No.
• Total Organic Carbon	5.0	9060
• Total Organic Halides (TOX)	5.0	9020
• Total Organic Halides (TOX) by Neutron Activation	5.0	9022
• Saturated Hydraulic Conductivity, Saturated Leachate Conductivity and Intrinsic Permeability	6.0	9100

FOOTNOTES:

(1) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, SW-846, U.S. Environmental Protection Agency, 1986.

Table 9.9 Toxicity Characteristic Leaching Procedure (TCLP) Detection Limits

Compound	Regulatory Level* (mg/liter)	Detection Limit Low Samples (mg/liter)	Detection Limit High Samples (mg/liter)
	Volatile Organics		
Acrylonitrile	5.0	0.2	1.0
Benzene	0.07	0.01	0.05
Carbon disulfide	14.4	0.01	0.05
Carbon tetrachloride	0.07	0.01	0.05
Chlorobenzene	1.4	0.01	0.05
1,2-Dichloroethane	0.4	0.01	0.05
1,1-Dichloroethylene	0.1	0.01	0.05
Isobutanol	25	0.5	2.5
Methylene chloride	8.6	0.05	0.25
Methyl ehtyl ketone	7.2	0.01	0.05
1,1,1,2-Tetrachloro-ethane	10	0.01	0.05
1,1,2,2-Tetrachloro-ethane	1.3	0.01	0.05
Tetrachloroethylene	0.1	0.01	0.05
Toluene	14.4	0.01	0.05
1,1,1-Trichloroethane	25	0.01	0.05
1,1,2-Trichloroethane	1.2	0.01	0.05
Trichloroethylene	0.7	0.01	0.05
Vinyl chloride	0.05	0.01	0.05

*Test Methods for Evaluating Solid Waste, USEPA SW-846, Third Edition, September 1986.

Table 9.9 (Continued)

Compound	Regulatory Level (mg/liter)	Detection Limits (mg/liter)
	Extractable Organics	
Bis(2-chloroethyl)ether	0.05	0.02
Cresols (total)	10	0.10
1,2-Dichlorobenzene	4.3	0.02
1,4-Dichlorobenzene	10.8	0.02
2,4-Dinitrotoluene	0.13	0.02
Hexachlorobenzene	0.13	0.02
Hexachlorobutadiene	0.72	0.02
Hexachloroethane	4.3	0.02
Nitrobenzene	0.13	0.02
Pentachlorophenol	3.6	0.10
Phenol	14.4	0.02
Pyridine	5.0	0.10
2,3,4,6-Tetrachlorophenol	1.5	0.10
2,4,5-Trichlorophenol	5.8	0.05
2,4,6-Trichlorophenol	0.3	0.05
	Extractable Pesticides	
Chlordane	0.03	0.005
2,4-D	1.4	0.05
Endrin	0.003	0.003
Heptachlor	0.001	0.001
Lindane	0.06	0.005
Methoxychlor	1.4	0.01
Toxaphene	0.07	0.05
2,4,5-TP (Silvex)	0.14	0.05

Table 9.9 (Continued)

Metal	Regulatory Level (mg/liter)	Detection Limit (mg/liter)
Arsenic	5.0	0.005
Barium	100.0	1.0
Cadmium	1.0	0.01
Chromium	5.0	0.05
Lead	5.0	0.05
Mercury	0.2	0.0002
Selenium	1.0	0.005
Silver	5.0	0.01

10.0 DATA VERIFICATION

Data verification is the steps taken within an analytical laboratory so that reported results correctly represent the analyses performed. There are two basic verification activities:

- The processing of quality control sample results to demonstrate that analyses are within laboratory-prescribed bounds for accuracy, precision, and completeness.
- Data validation to demonstrate that numerical computation of data is correct and that it is correctly reported.

This section discusses how data verification is performed. Section 12.0 discusses maintenance of resulting records.

10.1 PROCESSING OF QUALITY CONTROL DATA

This section discusses the analytical treatment of the data resulting from the quality control samples discussed in Section 8.0.

10.1.1 Specific Routine Procedures Used to Assess Data Precision and Accuracy

Following are the procedures recommended for evaluating the precision and accuracy of all data generated within ITAS Laboratories. Quality control sample analyses are performed as appropriate for organic or inorganic sample analyses as discussed in Section 8.0. The protocol used will be in accordance with specific analytical procedures if QC requirements are stated in the procedure.

- A reagent and/or method blank is prepared and analyzed with each set of samples.
- Field and trip blanks are analyzed to determine possible sample contamination during collection and shipment to the laboratory. Trip blanks are applicable to volatile organics analysis (VOA) where volatile contaminants can be introduced from ambient air during shipment.

- Bottle blanks are prepared and analyzed to demonstrate that clean containers were shipped to the field for sample collection. If purchased, vendor certificates should be retained to doucment cleanliness and lot numbers.

- Rinsate blanks from the field are analyzed to show sampling equipment was properly cleaned between samples.

- A daily calibration curve consisting of at least three standards and a reagent blank is prepared for each parameter. If the standard curve is known to be stable, the standard curve can be verified daily by the analysis of a midpoint standard.

- One sample in every sample set of 20 samples is analyzed in duplicate, or as a matrix sample duplicate.

- One sample in every sample set of 20 samples is spiked at a level to provide a final concentration equivalent to the original concentration of the sample before dilution.

- A blind duplicate, unknown to the Analyst, is introduced by the Quality Control Coordinator at least once monthly. Blind duplicates are routinely used for the analysis of metals, water quality parameters, and organics analyses.

- Standard Reference Materials (SRMs) are introduced periodically into the testing scheme as verification or reference standards, by the Quality Control Coordinator to evaluate the accuracy of standards, the testing procedure, and the analyst's performance.

- A blank spike consisting of deionized water or a purified solid matrix spiked with the parameter of interest is analyzed. Blank spikes are used to show the analytical technique was in control, although matrix effects may have been present in associated matrix spike cycles.

- Every sample is spiked with the appropriate surrogate standards prior to extraction and analysis

for volatile organic compounds, base-neutrals, acids, and pesticides/PCBs.

- Internal standards are added to all samples or extracts prior to GC/MS analysis.

- A check standard or midpoint standard is run to verify the continued acceptability of a calibration curve.

- Analysis matrix spikes (or Method of Standard Additions) of target analytes added to samples or extracts just prior to analysis to give information on matrix effects encountered during analysis.

- Field matrix spikes are sample portions spiked with target analytes spiked into samples in the field to provide data on stability.

- Collocated samples provide information on precision of intralaboratory or interlaboratory analytical systems when sent to the same or different laboratories, respectively.

- A replicated or split sample provides information about precision depending on the location in the sampling and analysis process where the split occurs. An analysis replicate gives information about the analytical precision; a field replicated or field split sample provides precision information for all steps after sample collection.

When the analyses of a sample set are completed, the results will be reviewed as described in Laboratory-Specific Attachments and/or SOPs, and evaluated to assess the validity of the data set. Review is based on the following criteria:

- Reagent Blank Evaluation - The reagent and/or method blank results are evaluated for high readings characteristic of background contamination. If high blank values are observed, laboratory glassware and reagents should be checked for contamination and the analysis halted until the system can be brought under control before

further sample analysis proceeds. A high background is defined as a background value sufficient to result in a difference in the sample value, if not corrected, greater than or equal to smallest significant digit known to be true. A reagent blank should contain no greater than two times (2X) the parameter detection limit for most parameters (1,2,3,4).*

- Field Trip, Bottle, and Rinsate Blank Evaluation
  Field blank results are evaluated for high readings similar to the reagent and/or method blanks described above. If high field blank readings are encountered, the procedure for sample collection, shipment, and laboratory analysis should be reviewed. If both the reagent and/or method blanks and the trip blanks exhibit significant background contamination, the source of contamination is probably within the laboratory. In the case of VOAs, ambient air in the laboratory and reagents should be checked as possible sources of contamination. High field blank readings for other parameters may be due to contaminated sample bottles (unless bottle blanks are clean) or cross-contamination due to sample leakage and poorly sealed sample containers, or cross-contamination between samples collected if the rinsate blank is contaminated (1, 2, 3, 4).

- Calibration Standard Evaluation - The daily calibration curve is evaluated to determine linearity through its full range, and that sample values are within the range defined by the low and high standards. If the curve is not linear ($r \geq 0.995$), sample values must be corrected for nonlinearity by deriving sample concentrations from a graph or by using an appropriate algorithm

*The criteria are denoted as applicable to:

1 - Volatile organic compounds

2 - Base-neutral, acid, and pesticide organic compounds

3 - Metals

4 - Water quality.

to fit a nonlinear curve to the standards (1,2,3,4).

- Duplicate Sample Evaluation - Duplicate sample analysis for the sample set is used to determine the precision of the analytical method for the sample matrix. The duplicate results are used to calculate the precision as defined by the relative percent difference (RPD). The precision value, RPD, should be plotted on control charts for the parameter determined. If the precision value exceeds the warning limit (Section 10.1.2.2) for the given parameter, the appropriate Group Leader, Operations Manager, or the Quality Control Coordinator is notified. If the precision value exceeds the control limit, the reason for the nonconformance must be determined; corrective action may include reanalysis for the parameter in question. Attainable precision limits will be specified by the Quality Control Coordinators and updated periodically following review of data (1,2,3,4).

- Matrix and Blank Spike Evaluations - The observed recovery of the matrix spike versus the theoretical spike recovery is used to calculate accuracy as defined by the percent recovery. The accuracy value, the percent recovery, may be plotted on a control chart for the parameter determined. If the accuracy value exceeds the warning limit for the given parameter, the appropriate Group Leader, Operations Manager, or the Quality Control Coordinator is notified. If the accuracy value exceeds the control limit, the reason for nonconformance must be determined; corrective actions may include reanalysis for the parameter in question. If interferences are present in the samples spiked, a blank spike is used to demonstrate that the laboratory technique is in control. The results of matrix spike and matrix spike duplicate parts of samples are compared as duplicate samples described above (RPD) (1,2,3,4).

- QC Check, or Laboratory Control, Standard - Prepared as blank spikes, these samples are used for initial proficiency demonstration of analysts, methods, and equipment. Material is obtained with known values (such as river sediment); it may be analyzed without spiking as

a Laboratory Control Standard (LCS), similiar to a Reference Standard (1,2,3,4).

- Blind Duplicate Evaluation - The blind duplicate analysis is evaluated in the same manner as described above for the duplicate sample analysis and is treated as a duplicate result for purposes of evaluating the precision of the analytical method. This evaluation is performed independently by the Quality Control Coordinator (1,2,3,4).

- Reference Standard Evaluation - Standard Reference Materials analyses are compared with true values and acceptable ranges. Values outside the acceptable ranges require corrective action to determine the source of error and provide correction. Analyses data reporting should be halted pending this evaluation. Following correction of the problem, the Standard Reference Material should be reanalyzed (1,2,3,4).

- Check Standard Evaluation - The results of check standard analysis are compared with the original calibration curve, and the relative percent difference of the check standard is calculated to determine if the calibration system is in control. If correction is required, the check standard should be reanalyzed to demonstrate that the corrective action has been successful (1,2,3,4).

- Surrogate Standard Evaluation - The results of surrogate standard determinations are compared with the true values spiked into the sample matrix prior to extraction and analysis and the percent recoveries of the surrogate standards are determined. Percent recoveries attained shall be in accordance with current EPA recommendations/requirements, or in their absence, laboratory-generated control limits (1,2).

- Analysis Matrix Spikes Evaluation - Suppression or enhancement of instrument signal levels is demonstrated when recoveries are lowered or raised, respectively. Matrix-suppressing agents may be added to the sample or the extract to reduce such effects in routine sample analysis when the evaluation indicates samples are affected in this manner. One to three levels of

analysis matrix spike concentrations are used to determine the unaffected concentration level native to the sample. The observed recovery versus the theoretical recovery is used to calculate the accuracy as defined by the percent recovery. Percent recovery may be plotted on control charts. If the accuracy value exceeds the warning limits for a given parameter, the appropriate Group Leader, Operation Manager, or QC Coordinator is notified. The reason for nonconformance must be determined, and appropriate corrective actions taken. These may include reanalysis of the sample(s) for the parameter in question(3).

- Field Matrix Spike Evaluation - Target analytes spiked into a portion of a field sample are measured to determine the amounts recovered after collection, shipment, storage, preparation and analytical processes have acted upon the analytes. Decreased recoveries may be due to bacteriological action on the poorly preserved sample, to photodegradation, to leaks from improperly capped/sealed containers, or degradation during physical treatment for analysis such as evaporation of organic solvents to reduce the sample extract volumes. The observed recovery versus the theoretical recovery is used to calculate the accuracy as defined by the percent recovery. Percent recovery may be plotted on control charts. If the accuracy value exceeds the warning limits for a given parameter, the appropriate Group Leader, Operation Manager, or QC Coordinator is notified. The reason for nonconformance must be determined, and appropriate corrective actions taken. These may include reanalysis of the sample(s) for the parameter in question (1,2,3,4).

- Collocated Sample Evaluation - Two or more samples processed independently by different people at different times using different techniques, or combinations of these variables, will yield results which provide an estimate of long-term precision of measurement systems. Or, if processed identically together, short-term precision is estimated (1,2,3,4).

- Replicate Sample Evaluation - Replicate sample analysis for the sample set is used to determine

the precision of the sampling and analytical method for the sample matrix. Field splits provide precision information about all steps after collection; analytical splits, or laboratory duplicates, provide information about instrument precision. The replicate results are used to calculate precision as RPD. Precision values exceeding warning limits require the appropriate Group Leader, Operations Manager, or QC Coordinator to be notified. The reason for the nonconformance must be determined and corrective actions taken. Laboratory duplicate precision limits are specified by the QC Coordinator and updated periodically (1,2,3,4).

10.1.2 Statistical Evaluation of Quality Control (QC) Data

As part of the analytical quality control program, ITAS laboratories will determine precision and accuracy for each parameter or class of parameter analyzed. Initially, when these data are compiled, the evaluation is applied over a broad concentration range. As more data are accumulated, precision and accuracy determinations will be updated and criteria developed to define precision and accuracy over specific concentration ranges.

10.1.2.1 Evaluation of Data Using Control Charts

ITAS laboratories will apply precision and accuracy criteria to each parameter that is analyzed. When analysis of a sample set is completed, the quality control data are reviewed and evaluated through the use of control charts to validate the data set.

Control charts are established for all major analytical parameters. This discussion of control charts is a guideline which shall be used unless other provisions are stated. A minimum of 20 measurements of precision and accuracy are required before control limits can be established. In general, warning limits of two standard deviations shall be used; three standard deviations should be used as control limits. Once established, control limits are updated as additional

10.1.2.2 Evaluation of Analytical Precision

General Considerations

To determine the precision of the method and/or laboratory analyst, a routine program of duplicate analyses is performed. These may also be matrix spike/matrix spike duplicate pairs. The results of the duplicate analyses are used to calculate the relative percent difference (RPD), which is the governing quality control parameter for precision.

The RPD for replicate analyses is defined as the difference (range) of each replicate set, divided by the average value (mean) of the replicate set, times 100 percent. For replicate results D_1 and D_2, the RPD is calculated from Equation 10-1:

$$RPD\% = \frac{D_1 - D_2}{\frac{D_1 + D_2}{2}} \times 100\% \qquad (10\text{-}1)$$

When the RPD is obtained for at least 20 replicate pairs, the average RPD and the standard deviation are calculated using:

$$\bar{m} = \frac{\sum_{i=1}^{n} m_i}{n} \qquad (10\text{-}2)$$

and

$$Sm = \sqrt{\frac{\sum_{i=1}^{n} (m-\bar{m})^2}{n-1}} \qquad (10\text{-}3)$$

where

- m = the RPD of a replicate pair,
- $\bar{m}$ = the average of the Relative Percent Difference determinations,
- Sm = the standard deviation of the data set of RPD determinations, and
- n = the number of RPD determinations.

When constructing a control chart for a specific parameter, the Warning and Control Limits are then calculated from the following:

Upper Control Limit = $\bar{m} + 3\ Sm$
Lower Control Limit = $\bar{m} - 3\ Sm$
Upper Warning Limit = $\bar{m} + 2\ Sm$
Lower Warning Limit = $\bar{m} - 2\ Sm$

A control chart is established by plotting the RPD of each replicate pair on a graph generated as follows:

- The average of the RPD determinations for the original data set is established as the midpoint on the Y axis of the graph.
- The Upper Warning and Control Limits calculated above are plotted as solid horizontal lines across the graph at their respective points on the Y axis above the mean of the RPD determinations.
- The Lower Warning and Control Limits calculated above are plotted as solid horizontal lines across the graph at their respective points on the Y axis below the mean of the RPD determinations.
- The calculated RPD of each replicate pair is plotted on the graph to determine whether the RPD is within the Warning and Control Limits of the Control Chart.
- If the RPD plots between the Warning and Control

Limits, the Group Leader, Operations Manager, or Quality Control Coordinator is notified for a decision as to how to proceed.

- If the RPD plots outside the Control Limits, the data set must be reviewed and the results of the analysis considered preliminary until the source of error has been determined and corrective action taken.

- Factors that influence precision include: Analyst skill; instrument stability; sample homogeneity; reagents quality; environmental fluctuations.

10.1.2.3 Evaluation of Analytical Accuracy

In addition to the evaluation of analytical precision, ITAS laboratories evaluate accuracy.

When a program for evaluation of analytical accuracy is established, the evaluation is applied over the entire range of spiking concentrations. As more data are accumulated, the evaluation procedure is refined to define the analytical accuracy of the method over specific concentration ranges.

To determine the accuracy of an analytical method and/or the laboratory analyst, a periodic program of sample and blank spiking is conducted. The results of matrix, matrix spike duplicate, and blank spikes are used to calculate the quality control parameter for accuracy evaluation, the Percent Recovery (%R).

The %R is defined as the observed concentration, minus the sample concentration, divided by the true concentration of the spike, times 100 percent.

$$\%R = \frac{O_i - O_s}{T_i} \times 100\% \qquad (10\text{-}4)$$

where

$\%R$ = the Percent Recovery,

O_i = the Observed Spiked Sample Concentration,

O_s = the Sample Concentration, and

T_i = the True Concentration of the Spike.

The True Concentration is calculated from Equation 10-5:

$$T_i = \frac{\text{Spike Concentration [c] (mg/L) x Volume of Spike (in mL)}}{\text{Volume of Sample [in mL] + Volume of Spike [in mL]}} \qquad (10\text{-}5)$$

When the Percent Recovery is obtained for at least twenty spiked samples, the mean percent recovery and the standard deviation are calculated using the formula:

$$\overline{\%R} = \frac{\sum_{i+1}^{n} \%R_i}{n} \qquad (10\text{-}6)$$

and

$$S_R = \sqrt{\frac{\sum_{i=1}^{n} (\%R_i - \overline{\%R})}{n-1}} \qquad (10\text{-}7)$$

where

$\overline{\%R}$ = the Mean Percent Recovery,

$\%R_i$ = the Percent Recovery of a Single Spiked Sample,

n = the number of results, and

S_R = the Standard Deviation of the data set of Percent Recovery determinations.

The Warning and Control Limits are then calculated from the following equations:

$$\text{Upper Control Limit} = \overline{\%R} + 3\ S_R$$
$$\text{Lower Control Limit} = \overline{\%R} - 3\ S_R$$
$$\text{Upper Warning Limit} = \overline{\%R} + 2\ S_R$$
$$\text{Lower Warning Limit} = \%R - 2\ S_R$$

A control chart (as shown in Figure 10-1) is generated by plotting the Percent Recovery data on a graph as follows:

- The average of the Percent Recovery determinations for the original data set is established as the midpoint on the Y axis on the graph.
- The Upper Warning and Control Limits calculated above are plotted as solid horizontal lines across the graph at their respective points on the Y axis above the mean of the Percent Recovery determinations.
- The Lower Warning and Control Limits calculated above are plotted as solid horizontal lines across the graph at their respective points on the Y axis below the mean of the Percent Recovery determinations.
- The calculated Percent Recovery of each spiked sample is plotted on the graph to determine whether the Percent Recovery is within the Warning and Control Limits of the Control Chart.
- If the Percent Recovery plots between the Warning and Control Limits, the Group Leader, Operations Manager, or Quality Control Coordinator is notified for a decision as to how to proceed.
- If the Percent Recovery plots outside the Control Limits, the data set must be reviewed and the results of the analysis considered preliminary until the source of error has been determined and corrective action taken.
- The Warning and Control Limits are recalculated for the data set on a periodic basis and the Control Chart for the corresponding parameter is updated.

- Factors that influence bias (accuracy) include: interference, calibration, contamination, losses during preparation, instrument drift, sensitivity resolution, analyst techniques.

Completed control charts are filed by the Quality Control Coordinator; current charts are kept at the work station for use in decision-making and updating.

10.1.3 Example Precision and Accuracy Data

The USEPA has compiled historical precision and accuracy data which are presented in Tables 10.1A through 10.2C. Footnotes to each table cite the sources of the data and concentration or concentration range at which the precision and accuracy were determined. When a concentration is not cited, concentration information was not available in the source material. These tables are provided as guidance for precision and accuracy that has been achieved on environmental samples.

The different levels of analytical techniques are described below. (Note: These levels are not the same as the source QC levels described in Section 8.3):

- Level I - Field screening or analysis using portable instruments. Results are often not compound specific and not quantitative but results are available in real-time.

- Level II - Field analysis using more sophisticated portable analytical instruments; in some cases, the instruments may be set up in a mobile or on-site laboratory. There is a wide range in the quality of data that can be generated. Quality depends on the use of suitable calibration standards, reference materials, sample preparation equipment, and the training of the operator. Results are available in real-time or several hours.

- Level III - All analyses performed in an off-site analytical laboratory using standard, documented procedures. The laboratory may or may not be a CLP laboratory.

- Level IV - CLP routine analytical services (RAS). All analyses are performed in an off-site CLP analytical laboratory following CLP protocols.

10.2 DATA VALIDATION

Data validation (review) is the process whereby data are screened and accepted or rejected, based on a set of criteria. Data will have been provided by trained analysts using approved methods and instrument systems in control.

Data validation is a systematic procedure of reviewing a body of data against a set of criteria to verify its validity prior to its intended use. It is applied to a body of data after the fact, systematically and uniformly. It must be applied close to the origin of the data by an independent and objective reviewer.

Data validation begins with the processing of data and continues through review of the data and the reporting of analytical results. Each laboratory will document its data validation procedures in its laboratory-specific attachment to this manual. Data processing can be performed by the analyst who obtained the data or another analyst. Data review starts with an analyst independent of the data acquisition and processing, or the Group Leader, reviewing (validating) that data processing has been correctly performed and continues through verifying that the reported analytical results correspond to the data acquired and processed. Checks are made for internal consistency, proper identification, transmittal errors, calculation errors and transcription errors. Final review of the data to be reported is by the Operations Manager. The procedure is outlined in Figure 10-2.

As stated, the first step in validation is data processing. In general, data

will be processed by an analyst in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets
- Input of raw data for computer processing
- Direct acquisition and processing of raw data by a computer.

If data are manually processed by an analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors, dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, calculations should be done on standard IT calculation paper and attached to the data sheets. The analyst shall sign or initial and date in ink each page of calculations.

For data that are input by an analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the project number(s) and other information as needed. The samples analyzed shall be evident and the input signed and dated by the analyst.

If data are directly acquired from instrumentation and processed, the analyst shall verify that the following are correct: project and sample numbers, calibration constants and response factors, output parameters such as units, and numerical values used for detection limits (if a value is reported as less than). The analyst shall sign and date the resulting output.

10.2.1 Review of Data Processing

Following is a discussion of two methods to be used for reviewing (checking) data processing. At least 20 percent of all data shall be checked using one of these procedures. If, during the checking process, errors are determined in a data set, checking shall be completely (100

percent) performed for the data set. The first procedure is called the "checkprint" procedure:

- The analyst performing the data processing shall give an analyst independent of the work the data package. The package shall include, as appropriate, raw data, data sheets, strip charts, computer input/output, calculations, sources for input parameters such as response factors, etc.

- The independent analyst (checker) will make and review copies of the data for:

  - Appropriateness of equations used

  - Correctness of numerical input

  - Numerical correctness of all calculations; this should be done by reperforming numerical computations

  - Correct interpretation of strip charts, etc.

- All entries and calculations that the checker reviews shall be marked in ink with a check mark or yellow highlighting on the copies. The checking process must be thorough enough to validate that the results are correct. If the checker disagrees with any part of the computations, the checker shall mark through the number with a single line and place the revised number above it.

- Any changes made by the checker shall be back-checked by the originator. If the originator agrees with the change, no action is necessary. If the originator disagrees, the originator and checker must resolve the difference so they agree with the result presented.

- The checker shall sign originals and date in ink all pages of the data package (except for groups of printout such as chromatograms). Signing and dating indicates that the reviewer agrees with the calculations and that any changes made have been agreed to by the originator and entered on the originals.

- If the data have been processed by computer, the

reviewer shall check at least 20 percent of input entries. Agreement should be indicated by a check mark for every line. If the checker disagrees with the input, the number should be marked through with a line and the corrected number indicated above it. Corrections must be backchecked by the originator as discussed above.

If an input error is identified and the data have been processed, it will be necessary to reprocess the data. In this event, the checker shall mark the second set of input to indicate agreement with the input changes. The checker shall sign and date in ink the computer input to indicate agreement.

- Raw data that are automatically acquired and processed do not require any validation at this point beyond that previously discussed.
- The reviewed data are maintained as discussed in Section 12.0.
- Use of the copies of data for checking produces a "checkprint" which should be maintained as a record to document the review performed.

The "original check" procedure is an alternative to the checkprint process; instead of a copy (checkprint) being used, analysts

- Mark on originals for checking in black ink, initialling and dating these entries as above.
- Backcheck changes in black ink using lineouts, initials, and dates as above.
- Summary checklists may be used and attached to the checked data which clearly describes the data checked, what the check was for, who did it and when, and if the data were acceptable.
- Nonconformances and corrective actions such as reprocessing must be documented.

10.2.2 Review of Data Reporting

Review of data reports is required to verify that information reported by an ITAS laboratory corresponds with processed analytical results. Review is onl required of the data as it is presented for issuance. Intermediate steps per formed after the processed data are checked to prepare the data report (such as data summaries) do not require validation. Referring to Figure 10-1, preparation of the report is the responsibility of the Group Leader.

After the draft data report is prepared (generally in tabular form), the reported results should be checked against the reviewed processed data (not subsequent summaries) so that transcription errors do not occur. The checkin; process follows:

- Using the draft report, all data entries are checked. The checker can be an Analyst or Group Leader. The checker is not required to be independent of the work because only the transcription from the reviewed data to the data report is being checked.

- The draft data report should be checked so that the items cited for data presentation in Section 11.0 are complete and correct. As the reviewer checks the entries on the draft report, an ink check mark is placed beside each correct entry. Corrected entries are marked through with a single line and the correct entry provided; changes are documented with initials and date. The reviewer will indicate that corrections have been made in the report by placing a second check mark by the correction after comparing the change with the revised copy. The checker shall sign and date every page of the data report in black ink.

- Use of the draft data report results in a checkprint which should be maintained as a record to demonstrate the review.

- If data printouts, such as chromatograms or GC/MS data processing, are included in the data report, review is not required for the data printout.

- If computer output is used directly as the data

report without further transcription, only the input requires review as discussed in Section 10.2.1.

After checking of the data report is complete, it is given to the Operations Manager for final review. This step is not intended to verify the reported data. This review is intended to determine that the report meets project requirements. From the Operations Manager, the data report is given to the Laboratory Manager for approval to issue.

usually generated by performing hand calculations using the same equations and procedures as the software. Verification of the software must test all options of the program. Problems must test both the theory, or basis for computation, and the ability of the software to store and manage files.

- Software that performs as part of instrument operation should be verified by processing reference materials through the instrument system. Processed instrument response should be compared against the standards used.

Whenever a program is modified, reverification is necessary. If the software has had features added, previous test problems should be rerun to demonstrate their function has not been affected. New test problems should be processed as discussed above to verify added performance. If software revision changes the basic operation of the program, complete reverification of the program is required.

Software should be reverified on a semiannual basis. The test problems used to provide initial verification shall be reprocessed and the results compared to demonstrate that performance of the software is unchanged. If software performance has changed, the effect of the change upon intended function and usage since last verification shall be assessed. "Effect" must be determined on a case-by-case basis for the scope and impact of incorrectly reported results. If necessary, the data shall be reprocessed and recipients of affected data reports notified.

Software verification shall be documented by the individual performing the work, signing and dating in ink the computer output, and supporting calculations. If test problems are used, the input shall be marked with check marks to indicate correct usage and the output checked to indicate acceptable comparison. If reference materials are used as the basis for

verifying instrumental related software, the "true" values or certificates for the materials shall be included with the output to demonstrate performance.

All software verifications, whether for initial or subsequent revalidation, shall be maintained in the laboratory operations records management system as discussed in Section 12.0. A separate file shall be maintained for each program. The file shall include the basis for the verification, such as the test problems or hand calculations, results of the software performance, and the results of subsequent reverifications.

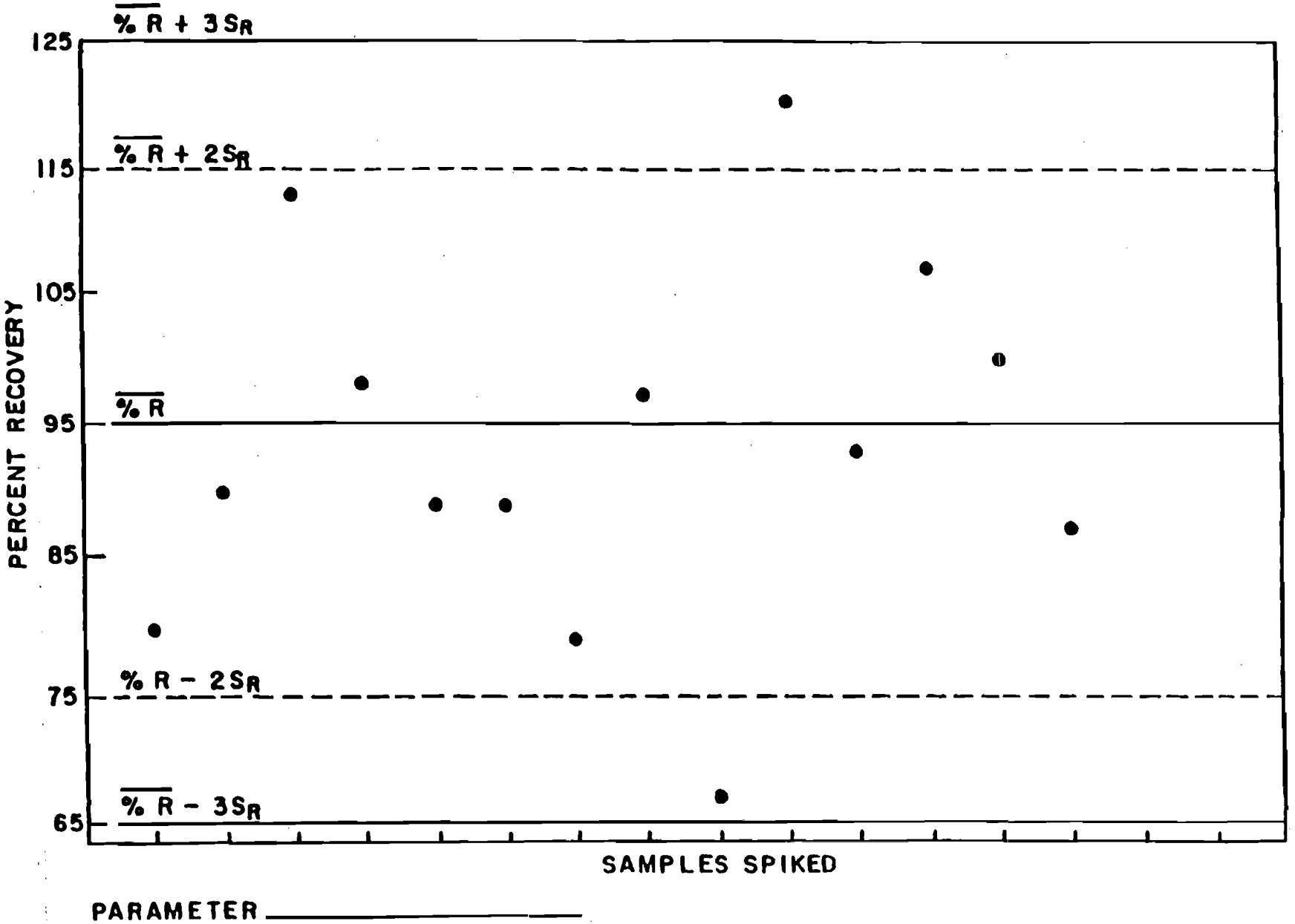


FIGURE 10-1

EXAMPLE ACCURACY QUALITY CONTROL CHART

ACTIVITY	RESPONSIBILITY
Data Processing, Corrections Reviewed, Results Amended	Analyst
Checking of Data Processing; Back check with originator	Independent Analyst or Group Leader
Preparation of Data Summaries and Draft Reports	Group Leader
Checking of Draft Data Report	Group Leader or Analyst
Final Review	Operations Manager
Approval for Issue	Laboratory Manager

FIGURE 10-2

DATA VALIDATION PROCESS

TABLE 10.1A: HISTORICAL PRECISION AND ACCURACY DATA/WATER [a]

LEVEL III ANALYTICAL TECHNIQUES - METHODS OTHER THAN CLP BAS METHODS

ANALYTES	METHOD (TECHNIQUE)	CONCENTRATION RANGE	PRECISION % RSD	ACCURACY % BIAS
BENZENE	624 (GC/MS)	11 ug/l	16	0
		480 ug/l	21	-16
	8240 (GC/MS)	5-100 ug/l	21	12
BROMODICHLOROMETHANE	624 (GC/MS)	8 ug/l	28	-8.6
		480 ug/l	18	-6.7
	501.1 (PURGE & TRAP GC)	0.9 ug/l	66	0
		550 ug/l	34	-3.8
	501.2 (EXTRACTION GC)	1.6 ug/l	61	33
		170 ug/l	23	-19
BROMOFORM	624 (GC/MS)	9 ug/l	32	-23
		400 ug/l	30	10
	501.1 (PURGE & TRAP GC)	4.6 ug/l	44	-27
		550 ug/l	41	7.5
	501.2 (EXTRACTION GC)	6 ug/l	14	-23
		170 ug/l	15	1.0

BASE SYSTEM

TABLE 1[illegible] PRECISION A[illegible] ACCURACY DATA/WATER [a]
(continued)

LEVEL III ANALYTICAL TECHNIQUES - METHODS OTHER THAN CLP RAS METHODS

ANALYTES	METHOD (TECHNIQUE)	CONCENTRATION RANGE	PRECISION % RSD	ACCURANCY % BIAS
METHYLENE CHLORIDE	624 (GC/MS)	7.2 ug/l	78	-17
		480 ug/l	52	-25
TOLUENE	624 (GC/MS)	13.5 ug/l	19	15
		600 ug/l	31	-14
	8240 (GC/MS)	25 ug/l	19	-10
		75 ug/l		
TRICHLOROETHENE	624 (GC/MS)	5.4 ug/l	48	44
		360 ug/l	39	-2.3
	8240 (GC/MS)	25 ug/l	24	5
		75 ug/l		
LEAD	200.7 (ICP)	42 ug/l	34	31
		47.7 ug/l	5	4.4
	239.1 (FLAME AA)	12 ug/l	5.9	17
		105 ug/l	6.7	-1.9
	239.2 (FURNACE AA)	10 ug/l	53	-.22
		234 ug/l	19	-3.1

a. Source: Draft Compendium of Information and Performance Data on Routinely Used Measurement Methods (RUMM) - Pilot Phase, RTI/3087/03, prepared for EPA Quality Assurance Management Staff, January 1986. This document should be consulted for more information on individual analytes.

TABLE 10.18: HISTORICAL PRECISION AND ACCURACY DATA/WATER

LEVEL III ANALYTICAL TECHNIQUES - SW-846 METHODS

Method Number	Method Name	Data Source	Range of Recovery (%)	Precision (%)	MDL (mg/l)
8310	Polynuclear Aromatic Hydrocarbons (HPLC) (Capillary)	SW 846	78 - 116	7.3 - 12.9	0.03 - 2.3
INORGANICS:	Metals (ICAP)	EPA 200.7	NA	3 - 21.9 (RSD)	1.3 - 75 Mg/l
	Metals (FLAME) 7000 Series	EPA 200	NA	NA	0.01 - 5
7000 Series	Metals (FLAME LESS/GF)	EPA 200	NA	NA	0.001 - 0.2 Mg/l
7470	Metals (MERCURY)	EPA 245.2	87 - 125	0.9 - 4.0	0.0002
9010	Cyanides	EPA 335.2	85 - 102	0.2 - 15.2	0.02 Mg/l
9030	Sulfides	EPA 376.1	NA	NA	1 Mg/l

a. For water only

b. NA Not Available

NOTES: Method Detection Limit (MDL) as listed on this table is the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.

Accuracy, presented as an average percent recovery, was determined from replicate (10-25) analyses of water and wastewater samples fortified with known concentrations of the analyte of interest at or near the detection limit. In most cases this was less than 10 times the MDL.

Precision data are used to measure the variability of these repetitive analyses reported as a single standard deviation or, as a percentage of the recovery measurements. For presentation purposes accuracy, precision and MDL information is presented as an average range of individual values for every analyte covered by the procedure. If specific information on a particular compound is required, the specific analytical method cited should be consulted.

TABLE 10.1C: HISTORICAL PRECISION AND ACCURACY DATA/WATER

LEVEL IV ANALYTICAL TECHNIQUES - CLP RAS METHODS

ANALYTES	TECHNIQUE	CONCENTRATION RANGE	PRECISION % RSD	ACCURACY % Bias
Semivolatiles[d]	GC/MS	N.A.[c]		
4-Methylphenol			33	-36
N-Nitroso-di-n-propylamine			31	+0.3
Nitrobenzene			32	-23
Isophorone			23	-0
2-Nitrophenol			30	-21
bis(2-Chloroethoxy)methane			34	-2.6
2,4-Dichlorophenol			29	-20
1,2,4-Trichlorobenzene			30	-47
Naphthalene			44	-30
4-Chloro-3-methylphenol			26	-32
2,4,6-Trichlorophenol			25	-17
2-Chloronapthalene			24	+3.4
Acenapthene			28	-12
2,4-Dinitrophenol			24	-23
2,4-Dinitrotoluene			34	-33
2,6-Dinitrotoluene			25	-40
4-Chlorophenyl-phenylether			34	+12
Fluorene			25	-24
4,6-Dinitro-2-methylphenol			30	-13
4-Bromophenyl-phenylether			32	-0.1
Hexachlorobenzene			36	-42
Pentachlorophenol			31	-24
Phenanthrene			21	-20
Fluoranthene			42	-15
Benzo(b)fluoranthene			39	-10
Benzo(a)pyrene			42	-29

TABLE 10.10: HISTORICAL PRECISION AND ACCURACY DATA/SOILS[a]

LEVEL IV ANALYTICAL TECHNIQUES - CLP RAS METHODS

ANALYTES	TECHNIQUE	CONCENTRATION RANGE	PRECISION % RSD	ACCURACY % Bias
Volatiles[b]	Purge & Trap GC/MS	N.A.[c]		
Chloroform			8.0	-0.1
1,2-Dichloroethane			13.1	+11.1
Dibromochloromethane			35.0	-12.0
Benzene			32.1	-10.3
Bromoform			16.6	-12.1
2-Hexanone			16.6	-45.5
Toluene			13.8	+13.7
Chlorobenzene			21.2	+13.2
Semivolatiles[d]	GC/MS	N.A.[c]		
1,4-Dichlorobenzene			27	-51
Nitrobenzene			21	-48
Isophorone			24	-47
2-Nitrophenol			35	-36
2,4-Dichlorophenol			31	-59
1,2,4-Trichlorobenzene			28	-43
Penta Chlorophenol			17	-40
Pyrene			25	-15
2-Methylnaphthalene			26	-42
bis-(2-Ethylhexyl)phthalate			33	-2
Phenol			38	-27
Acenaphthylene			26	-27
Diethyphthalate			16	-20
Dioxin[e]				
2,3,7,8-TCCD		1-10 ug/kg	15	-11.5

TABLE 10.2A: HISTORICAL PRECISION AND ACCURACY DATA/SOILS

LEVEL I FIELD SCREENING TECHNIQUES

MEASUREMENT	INSTRUMENT (TECHNIQUE)	INSTRUMENT RANGE	INSTRUMENT PRECISION [b]	INSTRUMENT ACCURACY [c]
RESISTIVITY	Bison 2390 T/R (Resistivity meter)	0-1999 millivolts	at 1% range setting, 0-5% of full scale	2% of measured value
TERRAIN CONDUCTANCE	EM 31 (conductivity)	0-1000 millimhos/meter	2% of full scale	5% at 20 millimhos/meter
TERRAIN CONDUCTANCE	EM 34-3 (conductivity)	0-300 millimhos/meter	2% of full scale	5% at 20 millimhos/meter
Magnetic Field Intensity	EDA - Omni IV (Magnetometer)	18000-110000 gammas	0.02 gamma	1 gamma at 50000 gammas at 23oC
Subsurface Lithology Changes	SIR-8 (Ground Penetrating Radar)	1-81 dielectric constant	N/A[d]	N/A[d]
Subsurface Lithology Changes	EG+G 1225 (Seismograph)	0-2000 milliseconds	N/A[d]	0.01%

TABLE 10.2B: HISTORICAL PRECISION AND ACCURACY DATA/AIR[a]

LEVEL I FIELD SCREENING TECHNIQUES[b]

ANALYTES	INSTRUMENT (TECHNIQUE)	INSTRUMENT RANGE	INSTRUMENT SENSITIVITY[c]	INSTRUMENT PRECISION[c]
Organics	Century OVA-128 (Flame Ionization)	0.1 - 1000 ppm Methane	0.1 ppm Methane	N.A.[d]
Organics	HNu PI-101 (Photoionization)	0.1 - 2000 ppm Benzene	0.1 ppm Benzene	± 1% of full scale deflection
Organics	AID - 710 (Flame Ionization)	0.1 - 2000 ppm Methane	0.1 ppm Methane	N.A.[d]
Organics	PhotoVac (GC-Photoion-ization)	N.A.	0.001 ppm Benzene	N.A.[d]

a. Source: Manufacturers' manuals unless otherwise cited. Mention of specific models does not constitute an endorsement of these instruments.

b. It is difficult to differentiate between Level I and Level II techniques and instrumentation. Several instruments may be used at both levels.

c. Sensitivity and precision refer to instrument specifications.

d. N.A. - Not Available.

TABLE 10.2C: HISTORICAL PRECISION AND ACCURACY DATA/SOIL[a]

LEVEL II/FIELD TECHNIQUES

ANALYTES	INSTRUMENT (TECHNIQUE)	FIELD RESULTS IN ppm (x)	CLP RESULTS IN ppm (y)	ACCURACY[b] % BIAS
PCBs	HNu 301 (GC/ELECTRON CAPTURE)	6.0	22.0	−72.7
		6.0	6.1	−1.6
		6.0	510.0	−98.8
		9.0	3.9	+56.7
		13.0	3.0	+333.3
		14.0	3.1	+351.6
		14.0	23.5	−40.4
		21.0	8.1	+159.3
		35.0	7.7	354.5
		41.0	2.1	+1,852
		46.0	11.0	+336.3
		50.0	460.0	−89.1
		65.0	23.1	+181.4
		67.0	18.7	+258.3
		92.0	75.0	22.7
		95.0	30.0	+216.7
		11	12.3	−10.6
		202	99.0	+104.0
		269	370.0	−27.3
		286	80.5	+255.3
		1215	640.0	+90.0
		1647	1040.0	+58.4
		3054	9,300	−67.2.

a. Source: CDM Project files.

b. Source: Accuracy calculated by assuming that CLP results on the same samples were completely accurate. % Bias = 100 $\frac{(x-y)}{y}$

11.0 DATA REPORTS

The format and content of a data report are dependent upon project needs, such as: whether or not explanatory text is required, client or contract requirements, and government agency reporting formats. The ITAS Quality Assurance Program does not specify a report format; however, the following are applicable to data presentation:

- The final data presentation shall be checked in accordance with data verification requirements of Section 10.2.2 and approved by the Laboratory Manager.

- Data are presented in a tabular format whenever possible.

- Data are generally formatted as a Certificate of Analysis such as shown on Figure 11-1, or formatted as an IT Corporation memorandum if issued within IT. Figure 11-2 is an example of a computer-generated report from the ITAS Sample Management System (SAM).

- Each page of data is identified with the project number and name; date of issue; and, if appropriate, client name.

- Data presentation includes:

  - Sample identification number used by the ITAS laboratory and/or the sample identification provided to the laboratory, if different than identification used in the laboratory

  - Chemical parameters analyzed, reported values, units of measurement, and analytical method used for the types of analysis specified

  - Detection limit of the analytical procedure if less than the detection limit is reported (e.g., <DL (10) where 10 is the detection limit)

  - Data for a chemical parameter reported with consistent significant figures for all samples (See Guidance, ASTM E29-67(80) and Standard Methods 103)

  - Results of Quality Control samples analysis if appropriate (See Section 8.3, Quality Levels)

- Achieved accuracy, precision, and completeness of data if appropriate

- Footnotes referenced to specific data if required to explain reported values.

- Data should be released from the laboratory only by the Laboratory Manager or Operations Manager.

If explanatory text is not issued with the analytical results, it is recommended that:

- A letter of transmittal be included for external clients

- A memorandum be included for work performed within IT.

As necessary, the letter of transmittal/memorandum should include:

- Person(s) receiving the data

- Person transmitting the data

- Document if the chain of custody was not provided or correct, if any samples were damaged in shipment, if sample containers were inappropriate for analysis, or if volume provided was inadequate for proper analysis

- Brief discussion of samples analyzed and the analytical program

- Discussion of any apparent data anomalies

- Discussion of any analytical difficulties.

Any analytical results verbally communicated are considered preliminary until data are presented in hard copy, with a formal letter of transmittal or memo accompanying the results.

IT ANALYTICAL SERVICES

CERTIFICATE OF ANALYSIS

FIGURE 11-1

EXAMPLE - CERTIFICATE OF ANALYSIS

IT Santa Clara ANALYTICAL REPORT

Page 1

COMPANY
FACILITY

REPORT
TO

ATTEN

PREPARED BY INTERNATIONAL TECHNOLOGY CORPORATION
397 Mathew Street
Santa Clara, CA 95050
PHONE (408)727-4277
CONTACT JIM

WORK ID
LABORATORY WORK ORDER # 87-06-157
LABORATORY JOB #
RECEIVED 06/19/87 REPORTED 06/26/87

SAMPLES 5 Waters
P.O. #
CERTIFIED BY

Duplicate of report of 06/25/87.

TABLE OF CONTENTS FOR ANALYTICAL RESULTS

PAGES	LABORATORY #	SAMPLE IDENTIFICATION
	S7-06-157-01	D4316 TRIP BLANK(6/15)6/18
2	S7-06-157-02	D4661 VINION DOM
2	S7-06-157-03	D4662 ITM7C
2	S7-06-157-04	D4314 ITM9C
2	S7-06-157-05	D4315 VINION DOM

FIGURE 11-2

EXAMPLE - COMPUTER GENERATED REPORT

&F0008H.PRN
06/26/87

IT Santa Clara ANALYTICAL RESULTS

COMPANY
FACILITY

WORK ID
LABORATORY WORK ORDER # S7-06-157
LABORATORY JOB #
RECEIVED 06/19/87 REPORTED 06/26/87

SAMPLE / TEST	D4661 VINION DOM S7-06-157-02	D4662 ITM7C S7-06-157-03	D4314 ITM9C S7-06-157-04	D4315 VINION DOM S7-06-157-05	UNITS	NORMAL METHOD LIMIT
1,1,-dichloro-ethane	nd @ 0.2	nd @ 0.5	nd @ 0.2	nd @ 0.2	ug/L	
1,1,-dichloro-ethylene	nd @ 0.1	nd @ 0.2	nd @ 0.5	nd @ 0.1	ug/L	
1,2,-dichloro-ethane	nd @ 0.2	nd @ 0.5	nd @ 0.5	nd @ 0.2	ug/L	
Benzene	nd @ 0.2	nd @ 0.5	nd @ 0.5	nd @ 0.2	ug/L	
Ethyl Benzene	nd @ 0.2	nd @ 0.5	nd @ 0.5	nd @ 0.2	ug/L	
Tetrachloro-ethylene	nd @ 0.2	nd @ 0.5	nd @ 0.5	nd @ 0.2	ug/L	
1,1,1-Tri-chloroethane	nd @ 0.2	nd @ 0.5	nd @ 0.5	nd @ 0.2	ug/L	
Trichloroeth-ylene	nd @ 0.2	nd @ 0.5	nd @ 0.5	nd @ 0.2	ug/L	
Toluene	nd @ 0.2	nd @ 0.5	nd @ 0.5	nd @ 0.2	ug/L	
Total Xylene	nd @ 1.0	nd @ 1.5	nd @ 1.5	nd @ 1.0	ug/L	

FIGURE 11-2 continued

EXAMPLE - COMPUTER GENERATED REPORT

12.0 RECORDS MANAGEMENT

The ITAS Quality Assurance Program has been developed to provide analytical results of known quality. To demonstrate that quality has been achieved, each ITAS laboratory maintains a records management system that includes documents which demonstrate the analytical performance of the laboratory.

Laboratory records are maintained in two broad categories:

- Documents which are specific to a project or a group of samples within an ongoing project, such as chain of custody and raw analytical data.
- Documents which demonstrate overall laboratory operation, such as instrument log books and control charts. These records will directly affect the data for a specific project, but in general their applicability is not limited to one project.

Following is a description of the records system used as the model within ITAS Laboratories. If it is necessary for a specific laboratory to deviate from this system, the laboratory shall maintain a description of their system as a Laboratory-Specific Attachment to the Quality Assurance Manual. Also, ITAS recognizes that specific regulatory or contractual demands may require deviation from the system described herein. In these instances, records shall be maintained as externally required.

The criteria for an acceptable records management system are that the data must be secure, retrievable, and complete. All laboratory records from time of sample receipt through reporting and disposal must be available and stored in a manner that safeguards their integrity from tampering or physical damage and loss. Any documentation that bears on the reported results must be available if requested by the client or an authorized regulatory agency or court. This includes operational and project-specific data. Data may be stored in "real-time" as it is

produced, or filed in a manner to allow prompt retrieval and assembly into a complete project file as described in the following section. Laboratory-Specific Attachments to this Quality Assurance Manual must describe therein (or by reference to SOPs provide the details) how the complete set of documentation is compiled, including the flow of data forms, locations, responsibilities, and checks on the records management system implemented.

12.1 PROJECT RECORDS

Separate record packages are maintained for each project or are retrievable so that packages can be assembled properly. Filing of records for a specific project shall be by the unique project identification number assigned by the laboratory for that project. Within a project file, categories of information are filed separately.

Table 12.1 presents the categories used within a project file. It is not expected that all categories will be applicable for every project. However, in all project files, specific information will be filed in accordance with the category designation provided. Following is a brief discussion of each category:

Project Index

Each project file shall have an index that lists each record unit within the project file.

Figure 12-1 presents an example of a project index page. An individual index file page should be used for each category applicable to a project. Within a specific category, individual files should be given a unique file number and be listed on the file index page. For example, if a testing program involves distinct groups of samples over a period of time, file number B1 should contain the chain of custody from the first group, B2 the chain of custody for the second group, etc. A brief description of the file contents should be made. Such as for the chain

of custody records for a group of samples, the description could be the dates the samples were collected.

A - Correspondence

All correspondence pertinent to the analytical program shall be maintained. This includes letters to and from clients and internal memorandums. Correspondence should be filed chronologically. Data reports and analysis requests are maintained in separate categories. The correspondence file should be updated as necessary. Each item of correspondence should not be entered in the project index. Rather, the index should list blocks of dates, such as for file A1, the description could be July 30, 1982 to January 10, 1983; for file A2, the description could be January 11, 1983 to September 17, 1983, etc.

B - Chain of Custody

Chain of custody records shall be maintained by the laboratory. The chain of custody forms should be filed for groups of samples as received and should be placed in the project file immediately after they are signed by the Quality Control Coordinator. When the samples are disposed of, this entry shall be made on the appropriate chain of custody form. Chain of custody records for samples sent to other laboratories must be maintained so the custody chain is unbroken.

C - Field Records

All field records supplied by field personnel pertinent to the analytical program shall be maintained. This could include the results of field tests or logs of sample collection. It is the responsibility of field personnel to provide records to the laboratory.

D - Request for Analysis

Analysis requests provided by field personnel are maintained in this file. Also, any changes or additions to the analytical program should be documented in this file.

E - Calibration Records

In general, calibration records are maintained with laboratory operation records. However, if an analytical program requires calibration which is performed solely for a project, the records shall be maintained in this file. If calibration is performed as an integral part of the analytical process; the calibration records should be maintained with the analytical data.

F - Analytical Data

Analytical data files should be complete for a group of samples in accordance with the needs of the project. The file should contain raw analytical data, processing of the data and/or data reduction, and data validation. It should be possible to use a data file to completely demonstrate that the data have been adequately obtained, processed, and reviewed. It is recommended that data be filed in accordance with the type of analysis performed. Suggested subcategories for data are presented in Table 12.1.

G - Quality Control Samples

If Quality Control samples, such as field blanks, are processed for a specific project, the data shall be maintained with the project file. The results of Quality Control samples processed on a general basis are included in the laboratory operations files. Statistical evaluation of Quality Control sample data for a project shall also be maintained in this file.

If Quality Control samples are processed as an integral part of a group of samples such that the data cannot be readily separated, the Quality Control sample data can be stored with the analytical data.

H - Data Reports

Complete copies of all reports issued by the laboratory shall be kept in the project file. With the reports shall be the review copy checked to

verify the data. If the report provides copies of data packages which are stored in Category F - Analytical Data, a memorandum should be attached to the report copy stating which specific files were included.

I - Project-Specific Requirements

If a project requires analytical procedures other than these adopted in the ITAS Quality Assurance Program, the requirements shall be included in this file. Specific requirements may be due to government regulation, specific contracts, or project need. Changes from stated practice can be, for example, frequency of QC sample analysis, test method, statistical data evaluation, and reporting format.

J - Nonconformances

Nonconformances and subsequent corrective actions which are specific to a project are included in this file. The record should be in the form of a memorandum (or copy of other records discussed in this manual) with the nonconformance stated, how it was corrected, and the approval for the correction. A separate file for each incidence is not required, the file should be maintained chronologically. A copy should be printed for the master nonconformance record (Section 12.2).

K - QA Plans

If specific Quality Assurance Plans, and revisions, are prepared for a project, they shall be stored in this file.

L - Miscellaneous

The miscellaneous file includes all records not applicable to the previous categories. Each distinct record(s) in this file should be entered in the project index.

M - Method Description

A written statement identifying the method(s) used for the project sample analyses, including the reference to approved methods and any approved modifications to the methods.

If it is necessary to adopt a new analytical procedure, a procedure different than conventionally used, or alter an existing procedure, the method used for the project must be documented. If the analytical procedure is developed by an ITAS laboratory as part of the analytical program, the procedure shall be documented and included. If an existing procedure is altered, the Analyst or Group Leader shall prepare a memorandum to the project file stating what the changes were and the justification for change.

N - Work Order Records

Internal tracking documents used by the laboratory (e.g., computer-generated daily status reports, etc.) must be included as part of the traceability of the project samples through the laboratory. If a duplicate manual log is maintained, traceability of the systems is required to show the same information is contained and accessible in both.

O - Subcontractor Records

Correspondence and reports and raw data (samples, calibration, and QC) must be received from the subcontractor following analysis reporting. Arrangements to retrieve these data are required in the ITAS analytical subcontracting policy, and must be followed to achieve complete project files.

12.2 GENERAL LABORATORY OPERATIONS RECORDS

General laboratory records document overall laboratory performance and operation. These records are filed separately from project records and will be maintained so they can be referenced to project records if necessary. Examples of general records pertinent to project records are instrument log books and computer software verifications.

There are two types of general laboratory records:

- Documents which demonstrate laboratory performance
- Reference documents for laboratory operations.

Records which demonstrate laboratory performance shall be filed in categories in a manner similar to project files. Reference documents are not indexed and their usage is not controlled.

Table 12.2 recommends the laboratory performance records to be maintained and the category system for their maintenance. Also shown in this table is the manner in which the files can be correlated to project files, if required. Indexing of files should be as described in Section 12.1.

Many of the laboratory operations records are in daily use, such as the Master Sample Log Book, instrument calibration logs, and control charts. It is not intended that the records be stored daily while they are in use. However, when individual log books, etc., are filled, they shall be placed in the files. For a book that is in use, it should be designated by a file index code and entered into the index. For example, considering the Master Sample Log books, the book for the period August 30, 1983 to April 4, 1984 is designated A1. Because it is complete, it is stored. Book A2 starts on April 5, 1984 and is still in use; therefore, the index would list A2, April 5, 1984 to _____. The completion date would be entered when book A3 is started.
Following is a brief discussion of the categories:

Index File

The index should be maintained as discussed in Section 12.1.

A - Master Sample Log Book

The master sample log books chronologically record all samples entering the laboratory, independent of project designation.

B - Instrument Calibration Logs

All calibration performed independent of a specific project shall be recorded by instrument. A separate file should be maintained for each instrument subject to calibration.

C - Instrument Maintenance Logs

Separate maintenance files should be kept for each instrument incorporated in the preventive maintenance program. The file shall include records of maintenance performed in-house or by outside groups. Service contracts should be included in the file for the applicable instrument.

D - Computer Software Verification

Copies of the data used to verify performance of software shall be maintained. For software that is periodically reverified with standard problems, the results of the performance shall be maintained. A separate file for each software package shall be indexed.

E - Performance Evaluation Records

Laboratory participation in Performance Evaluation Programs shall be documented in this category. If performance standards are analyzed as part of the overall Quality Control sample program, the results should be included in Category G.

F - Certification Program Records

If the laboratory participates in Certification programs, such as the AIHA program, the results shall be maintained in this category. Records should include all correspondence, analytical data, agency results, and certificates of performance.

G - QC Sample Analysis

This file should include the results of all QC samples inserted into the sample stream by the QC Coordinator. It is recommended that the files be maintained chronologically for each parameter included in the QC program.

H - Control Charts

Control charts should be filed chronologically for each parameter monitored.

I - Purchased Material Certificates

All information which verifies that purchased materials meet the requirements of the laboratory should be maintained. Certification may be supplied by a vendor or from in-house verification analysis. Separate files should be kept for chemicals, gases, water, glassware, etc.

J - QC Coordinator Reports

This file includes the monthly reports prepared by the QC Coordinator. Required signatures must be included. The file should be maintained chronologically.

Table 12.3 lists reference documents which should be available within the laboratory. These documents need not be included in the laboratory record system and may be kept where used. Reference documents shall be revised and updated as necessary to maintain them as "currently applicable documents."

K - Audit Records

Formal audit reports of internal system and performance audits are to be filed in this category, by date and topic (items audited). Where the audit is project-specific, it should be cross-referenced to the project audited (see Project Records Filing Category I). If the audit was performed by an external agent, a copy of the audit report should be

included in this category; if the audit was project-specific, it should be filed in the Project Records Filing Category I by date and project number and/or client.

L - Personnel Files

Training files of individuals are to be maintained current in this filing category. Records of SOP training received, proficiency tests passed, external training obtained, etc. are documented in this category chronologically by individual and by topic.

M - SOPs

A listing of the current laboratory Standard Operating Procedures (SOP) is maintained as an index, together with a complete collection of approved SOPs, their issue dates, and individuals authorized to perform the SOPs (see category L above); if SOP manuals are issued, the distribution records are maintained therein, by number and topic.

N - Methods

Those methods used by the laboratory are summarized, indexed, and compiled to provide a complete reference collection of the analytical procedures used. Method references are listed by reference method name and number and source, within the laboratory groups or classes of instruments tested (GC, GC/MS, AA, ICP, Wet Chemistry, etc.).

O - Subcontractor Records

Data received from subcontractors from analysis of audit (PE) samples, routine QC samples, blind samples submitted to them, and other information establishing the laboratory's performance levels are maintained in this category by subcontractor and project number.

P - Master Nonconformance Record

As described in Section 12.1, a copy of project-specific (and other) nonconformances and corrective actions is to be supplied to the Quality Control Coordinator. This Master Record will be periodically examined and reports issued describing systematic trends which in turn require investigation and correction by responsible Group Leaders, the Technical Director, the Operations Manager, or the Laboratory Manager. Group Leaders are encouraged to maintain Group Records to perform such evaluations regularly.

12.3 RECORD CONTROL

The Laboratory Manager shall designate an individual responsible for the records management system. This person shall:

- Initiate new project files including the project index
- Add new records to existing files, initiate new files within a category, and update the index
- Assist laboratory personnel in withdrawing and returning records.

To maintain control of records within the laboratory, a master sign-out sheet should be maintained. The sign-out sheet should indicate:

- Project from which file is borrowed
- File designation, such as E13, etc.
- Date and person borrowing record
- Date returned to the record system.

12.4 RECORD RETENTION

ITAS will maintain records associated with specific projects for the following time periods after completion of the project:

- Analysis performed as part of site mitigation activities - 10 years

- Records associated with facilities governed by the Resource Conservation and Recovery Act (RCRA) - 5 years after closure, if the analysis was performed prior to closure or for the 30-year monitoring period following closure, if the analysis was performed for the purpose of closure monitoring

- Conventional analysis - 7 years.

If necessary, pertinent supplemental records in the laboratory operations record system will be added to the affected project file.

If a specific contractual requirement, project demand, or government regulation requires that records be maintained for a longer period of time, project files will be kept as required. For projects that must be kept beyond the periods stated above, the project index shall be marked to indicate the required retention period.

Prior to scheduled record destruction, records shall be reviewed to determine if the holding period should be extended.

12.5 SAMPLE STORAGE

Analytical samples will be routinely stored (regardless of reason for analysis) by an ITAS laboratory, after submittal of a data report, for six weeks prior to disposal or return to client.

Samples will be stored for different periods if specified by project or contractual requirements.

Table 12.1
EXAMPLE PROJECT RECORDS
FILING CATEGORIES

CATEGORY	RECORD DESCRIPTION
-	Project Index
A	Correspondence
B	Chain of Custody
C	Field Records
D	Request for Analysis
E	Calibration Records
F	Analytical Data
Example: F-1	- GC Data
Example: F-2	- GC/MS Data
Example: F-3	- Metals
Example: F-4	- General Chemistry
Example: F-5	- Soils Chemistry
Example: F-6	- Other
G	QC Samples
H	Data Reports
I	Project-Specific Requirements
J	Nonconformances
K	QA Plans
L	Miscellaneous
M	Methods Descriptions
N	Work Order Records
O	Subcontractor Records

Table 12.2
EXAMPLE LABORATORY PERFORMANCE RECORDS
FILING CATEGORIES

CATEGORY	RECORD DESCRIPTION	CORRELATION TO PROJECT FILES
-	Index File	--
A	Master Sample Log Books	By date of arrival and sample number
B	Instrument Calibration Logs	By date, instrument serial number, and analyst
C	Instrument Maintenance Logs	By date and instrument serial number
D	Computer Software Verification	By date and software designation
E	Performance Evaluation Records	By date and sample designation
F	Certification Program Records	By name of program, date, and sample designation
G	QC Sample Analysis	By date and sample number
H	Control Charts	By chemical parameter, date, and sample number
I	Purchased Material Certificates	By date and parameter
J	QC Coordinator Reports	By date
K	Audit Records	By date and topic
L	Personnel Files	By individual, chronologically
M	SOPs	By numbered topic
N	Methods	By method and group
O	Subcontractor Records	By subcontractor and project
P	Matter Nonconformance Record	By group, chronologically

Table 12.3
REFERENCE DOCUMENTS

- Instrument Manuals
- Computer/Software Instruction Manuals
- Analytical Procedures
- QA Manual/Laboratory-Specific Attachments
- Manuals of Practice

PROJECT INDEX

PROJECT NUMBER _____
PROJECT NAME _____

FILE CATEGORY _____

FILE DESIGNATION	DESCRIPTION

FIGURE 12-1
EXAMPLE
PROJECT INDEX FILE

13.0 NONCONFORMANCES AND CORRECTIVE ACTION

A deficiency is an unauthorized deviation from documented procedures, practices, or standards, or a defect in an item, that could lead to degradation of quality.

A nonconformance is a deficiency in characterization documentation, or procedure sufficient to render the quality of an item unacceptable or indeterminate or any event which is beyond the limits documented and established for laboratory operation. Non-conformances may be caused by non-Laboratory operations (e.g., field collection paperwork not complete) and be identified at the Laboratory.

Nonconformances may include (but are not limited to) the following:

- Failure of an instrument to work properly
- Sample receiving documentation not correct
- Sample condition on receipt not acceptable
- Sample holding time exceeded
- Sample storage conditions outside criteria
- Incorrect sample preparation/analysis procedures used
- QC sample data (blank, spike, duplicate, surrogates, etc.) outside limits
- Calibration requirements not met
- Data recording errors, transcription errors, or failure to document
- Data validation errors
- A recovery or RPD result that is out of control (e.g., more than three standard deviations from the weighted mean for that month)
- Relative standard deviation for response factors greater than accepted limits

- Positive blanks
- Any situation or result which might affect the quality of data.

A nonconformance memorandum is formal documentation of a nonconformance that includes a description of the problem, the corrective action taken, the individual recognizing the problem and the date discovered, the sample analyses affected (if any) and the initials of appropriate reviewers and the Quality Control Coordinator (QCC) (see examples in Figure 13-1 through 13-3).

A corrective action is an appropriate measure applied to correct a deficiency and minimize the possibility of recurrence.

Corrective action will include, but not necessarily be limited to:

- Recalibration of instruments, using freshly prepared calibration standards
- Reanalysis of samples
- Replacement of lots of solvent or other reagents that give unacceptable blank values
- Additional training of laboratory personnel in correct implementation of sample preparation and analytical methods
- Reassignment of personnel, if necessary, to improve the overlap between operator skills and method requirements
- Communication with the clients to determine the appropriate action (e.g., insufficient sample remaining for reanalysis).

13.1 RESPONSIBILITIES

All employees of the Laboratory staff are responsible for reporting non-conformance they observe/identify.

Employees identifying problems that might affect quality are responsible for reporting them to the appropriate supervisor and signing nonconformance memos.

Each laboratory group leader is responsible for documenting and correcting problems that might affect quality in accordance with the requirements of this procedure. The supervisor is responsible for preparing and signing nonconformance memos, issuing nonconforming item tags, stopping work in the event of out-of-control situations, and notifying the QCC of nonconforming items. With the cognizant QCC, the supervisor is responsible for determining whether reported problems are nonconformances, concurring with proposed corrective action, and notifying the QCC that corrective action has been completed.

The QCC is responsible for reviewing nonconformance memos, recommending or approving proposed corrective actions, maintaining an up-to-date nonconformance log, verifying that corrective action has been completed, releasing nonconforming item tags, distributing and filing nonconformance memos, and assisting in resolving disagreements. With the lab management and group leaders, the QCC also is responsible for determining whether reported problems are nonconformances, whether operations need to be stopped, and establishing schedules for completion of corrective action. The QCC is responsible for assisting in resolving disagreements and quality problems, and performing audits of all affected organizations for compliance with the requirements of this procedure.

13.2 GENERAL PROCEDURE

The corrective action process is considered internal when the deficiency is identified by a laboratory employee, whether that employee works in the responsible group or is independent of it (the QCC, for example). External corrective action results when a deficiency is identified by an external organization, particularly through quality assurance functions such as audits.

13.3 INTERNAL NONCONFORMANCE CORRECTIVE ACTION PROCEDURE

Any laboratory employee noticing a deficiency suspected of being a nonconformance shall report the deficiency to the responsible supervisor and to the QCC on a nonconformance memo form.

If the QCC and the responsible supervisor determine that quality is affected, the deficiency becomes a nonconformance and the remainder of this procedure shall be implemented.

If the nonconformance is hardware/equipment related, the item shall be labeled with a nonconforming item tag (Figure 13-4) and segregated if possible so that it is not used until corrective action has been taken. Either the responsible supervisor or the QCC may issue a nonconforming item tag.

If analytical data (chromatograms, spectra, calculation sheets, etc.) that have not been completely processed or reported to other organizations are determined to be in error because of equipment malfunction, defective material, failure to follow procedures, anomalies in the data, process errors, quality control sample failure, or other errors, and the non-conforming data are to be maintained for historical purposes, a nonconformance memo shall be initiated describing the error, listing and attached to the affected data sheets. After confirmation of the problem as a nonconformance by the responsible supervisor and/or the QCC, each page of affected data is lined through, marked as rejected data, initialed and dated to prevent subsequent use in processing. A copy of the completed nonconformance memo is filed with the rejected data in the appropriate analytical data category of the project file. If there is justification for disposition of rejected data (the responsible supervisor and/or the QCC shall make this decision), a record of rejected data disposal and brief explanation shall be documented in the analytical run log.

Examples of corrective actions are the modification of nonconforming procedures, repair or replacement of deficient equipment, training or replacement of unqualified personnel, and reanalysis of any affected samples.

The Quality Control Coordinator shall maintain a log of nonconformances that includes a description of the problem and corrective action, and lists the responsible manager, the affected projects and sample numbers, and initial and closeout dates.

Once the corrective action has been taken, the QCC shall verify, through a special surveillance or audit, that the problem has been corrected. The QCC shall document that the corrective action has been completed satisfactorily (closed out) by signing the nonconformance memo and making an entry in the nonconformance log (Figure 13-5) for filing in the Master Nonconformance Record. The QCC can then remove the nonconforming item tag from out-of-service equipment.

The QCC shall file a copy of all records pertaining to the nonconformance with the nonconforming item records (i.e., specific project files or procurement files). The nonconformance log maintained by the QCC shall contain a reference to the file location.

13.4 EXTERNAL NONCONFORMANCE CORRECTIVE ACTION PROCEDURE

Reports of quality assurance functions by external organizations and reports of compulsory proficiency tests are considered to be externally generated.

External reports should be sent to the affected group supervisors and the QCC.

If the report identifies deficiencies that require corrective action, the QCC shall generate a nonconformance memo describing the particular deficiency and make an entry in the nonconformance log. The QCC shall verify that action is taken and that a response or notification of unavoidable delay is generated in time to meet the due date to the client.

The QCC shall verify that the problem has been corrected. With the responsible supervisor, the QCC shall prepare a formal written response to the external organization, if required. The Laboratory Manager shall transmit the

response to the external organization, with copies to other cognizant managers, as deemed necessary.

Upon notification by the external organization of acceptance of the corrective action, the QCC shall notify the responsible supervisor, the Laboratory Manager, and other cognizant managers. If corrective action is not acceptable, the QCC shall start the process over until the problem is satisfactorily resolved, at which time the nonconformance closeout is documented and dated in the nonconformance log.

The QCC will file the records pertaining to the nonconformance with the external audit documents and reference the file location in the nonconformance log.

Nonconformances and required corrective action can also result from the ongoing laboratory review of the QCC and audits performed by the ITAS Quality Assurance/Control Director. These activities are discussed in Section 14.0.

Written laboratory nonconformances shall be reported monthly to the ITAS Quality Assurance/Control Director by the Quality Control Coordinator. Corrective actions for nonconformances that are detected <u>after</u> data have been reported must also be reported to the Director. Figure 13-1 through 13-3 are example forms used to document nonconformance and corrective actions taken. The form is filed in the associated project file(s) and in the QCC's master nonconformance record.

FIGURE 13-1

EXAMPLE NONCONFORMANCE MEMO

SAMPLE RECEIVING

DATE _____
LIMS CODE _____
FILED BY _____

SAMPLE NO.(s) _____
_____

NONCONFORMANCE: [Check applicable item(s)]:

_____ (1) Not enough ample sent for proper analysis.
_____ (2) Sample bottle received broken and/or cap not intact.
_____ (3) Paperwork went to one lab; sample sent to another.
_____ (4) Samples received without proper refrigeration, when it has been deemed necessary.
_____ (5) Illegible sample numbers or label missing from bottle.
_____ (6) Numbers on sample not the same as numbers on paperwork.
_____ (7) Incomplete instructions received with sample(s), i.e., no Request for Analysis, no Chain-of-Custody, incomplete billing instructions, no due dates, etc.
_____ (8) Samples received in improper container.
_____ (9) Physical characteristics different than those on sampling sheets, i.e., two phases.
_____ (10) Rush samples put on hold because of incomplete paperwork.
_____ (11) Standard operating procedure not adhered to (specify) _____
_____ (12) Other (specify) _____

CORRECTIVE ACTION TAKEN: [Check applicable item(s)]:

_____ (1) Sample processed "as is."
_____ (2) Resampling requested.
_____ (3) Sample "on hold" until further notice.
_____ (4) Other (specify) _____

ROUTING

Title	Initials	Date	Check if Corrected
Coding Specialist			
QC Coordinator			
Lab Supervisor			

FIGURE 13-2

EXAMPLE NONCONFORMANCE MEMO

EXTRACTIONS

DATE _____
LIMS CODE _____
FILED BY _____

SAMPLE NO.(s) _____

NONCONFORMANCE: [Check applicable item(s)]:

_____ (1) Method development or modification, to include any extraction or cleanup sequence not currently used on a regular basis in the extractions lab. (Requires QA approval.) PLEASE SPECIFY _____

_____ (2) Sample matrix not as described on paperwork, i.e., supposed to be organic but is actually aqueous, nonhomogenous, etc. PLEASE SPECIFY _____

_____ (3) Reprep caused by: _____
(a) Error in spiking or surrogating
(b) Lost extract
(c) Lost sample
(d) Contamination
(e) Low recovery
(f) Other _____

_____ (4) Standard operating procedure not adhered to (SPECIFY) _____

_____ (5) Other (PLEASE SPECIFY) _____

CORRECTIVE ACTION TAKEN: [Check applicable item(s)]:

_____ (1) Error corrected by analyst.
_____ (2) Situation noted on sample tracking sheet and appropriate lab personnel notified.
_____ (3) Sample processed "as is."
_____ (4) Reextraction or resampling requested/performed.
_____ (5) Sample put "on hold" until further notice.
_____ (6) Client informed verbally.
_____ (7) Client informed by memo/letter.
_____ (8) Other (specify) _____

ROUTING

Title	Initials	Date	Check if Corrected
Analyst			
Group Supervisor			
QC Coordinator			
Lab Manager			

DATA REVIEW - GC/MS

DATE _____
LIMS CODE _____
FILED BY _____

SAMPLE NO.(s) _____
_____

NONCONFORMANCE: [Check applicable item(s)]:

_____ (1) Data missing.
_____ (2) Data improperly filled out.
_____ (3) Analysis incomplete or not meeting client request.
_____ (4) Daily tune nonconformance.
_____ (5) Initial calibration nonconformance.
_____ (6) Continuing calibration nonconformance.
_____ (7) Method blank nonconformance.
_____ (8) Sample nonconformance.
_____ (9) Additional QC nonconformance.
_____ (10) Other (specify) _____
_____
_____
_____

Explanation of Above (if needed): _____
_____
_____
_____

CORRECTIVE ACTION TAKEN: [Check applicable item(s)]:

_____ (1) Project returned to analyst(s) for rework
_____ (2) Reanalysis requested
_____ (3) Reprep and reanalysis requested
_____ (4) Data accepted "as is"
_____ (5) Client informed verbally
_____ (6) Client informed by memo/letter
_____ (7) Project "on hold" until further notice
_____ (8) Other (specify) _____
_____

ROUTING

Title	Initials	Date	Check if Corrected
Analyst	_____	_____	_____
Lab Supervisor	_____	_____	_____
QC Coordinator	_____	_____	_____
EPA Project Manager (if necessary)	_____	_____	_____

IT CORPORATION

CAUTION

NONCONFORMING ITEM

Tag No _____ Affected Item _____

_____

_____

Problem _____

_____

_____

_____

☐ Work may continue on this item subject to the following limitations

_____

_____

_____

☐ Work may not proceed on this item until this nonconformance has been dispositioned.

_____ _____

QC Coordinator Date

FIGURE 13-4

EXAMPLE - NONCONFORMING ITEM TAG

FI 13-5

EXAMPLE NONCONFORMANCE LOG

Date Issued	Responsible Manager	Description	Corrective Action Completion Date		Verified Date	Reference File Index
			Target	Date		

14.0 QUALITY ASSURANCE/QUALITY CONTROL AUDITS

Audits of an analytical laboratory are described as:

- Performance audits conducted on an ongoing basis within the laboratory by the Quality Control Coordinator. These audits are reported to the Laboratory Manager and the ITAS QA/C Director. They include: monthly surveillances, and semiannual internal audits.

- System audits performed on a scheduled, periodic (semi-annual) basis by the ITAS Quality Assurance/Control Director. These audits are external to the laboratory and are reported to the Vice President, Analytical Services and the Director, Quality Assurance and Discipline Management, Environmental Projects Group.

- Data audits performed on respresentative projects from a laboratory. These audits are conducted on a project-specific basis by the ITAS QA/C Director or his designee.

Audits of the laboratories are performed for the following reasons:

- To determine that contractual and regulatory obligations are fulfilled

- To determine that IT procedures and standards are being followed. These audits may include the ITAS QA Manual, Laboratory-Specific Attachments, project-specific QA Project Plans, Chain-of-Custody forms, Sample Collection Logs, etc.

- To establish that Quality Assurance objectives are met, including holding times, reporting turnaround times, use of approved analytical methods, and stated objectives for precision, accuracy, representativeness, completeness, and comparability (PARCCs)

- To serve as a management tool to evaluate appropriateness of Quality Assurance policies

- To identify potential or actual deficiencies for the purposes of evaluating compliance with requirements and providing the means for correction.

- To determine that records are prepared/maintained as required.

Audits are <u>not</u> conducted to assign blame.

The content and conduct of the audits are discussed below.

14.1 PERFORMANCE AUDITS

As stated in Section 2.1, the Quality Control Coordinator is responsible for the preparation of Quality Control samples, insertion into the sample stream, and analysis of the results. The samples are analyzed on a daily, ongoing basis and provide the means for demonstrating data quality by statistical analyses.

To complete internal laboratory reviews, the Quality Control Coordinator shall provide ongoing monitoring of laboratory operations. The review is conducted on behalf of the Technical Director and the Laboratory Manager to verify that the laboratory Quality Assurance Program is implemented and functioning on a daily basis.

Reviews may be of two kinds: surveillances or semiannual internal audits. Surveillances are detailed inspections of specific areas of a laboratory and its QA program, performed on a monthly basis by the QCC. Surveillances do not require as extensive planning and preparation as do audits, and prior communication with the surveyed group or personnel is not necessary. The QCC shall observe the activity of interest while it is in process and/or review objective evidence. A predesigned checklist based on the applicable documents and criteria will be used for this review. The semiannual internal audit covers the same criteria that the semiannual ITAS Quality Assurance Systems Audit does, namely the laboratory's QA Program. The audit is intended to be a spot check and should include:

- Sample maintenance
  - Are stated temperatures for sample storage provided?
  - Are samples processed and tested within prescribed holding times?
  - Are samples properly logged in?

- Calibration
  - Are calibrations performed as required?
  - Are they properly documented in instrument log books, or as part of project data if required?
  - Do calibration results indicate a trend in instrument performance?
- Preventive maintenance
  - Are adequate spare parts available?
  - Do specific instruments have repeated maintenance problems?
  - Is preventive maintenance performed and properly documented?
- Receipt and storage of standards, chemicals, and gases
  - Are all reagents, chemicals, and gases purchased for use in the laboratory of adequate grade for the intended use?
  - Are certifications of material compositions provided when required?
  - Are materials adequately stored to prevent degradation?
  - Are materials kept beyond stated shelf life?
  - Are internal standards properly prepared and stored?
  - Are internal standards kept beyond stated shelf life?
- Analytical Methods
  - Are the methods used appropriate for project requirements?
  - Are alternate methods approved for use?
- Data verification
  - Are data processed and validated as prescribed?
- Records management
  - Are the records of analyses complete and properly identified?

- Are documents submitted to the record system in a timely manner and are they properly maintained?

Nonconformances observed by the QCC shall be reported to the Group Leaders and Operations Manager for corrective action, or the Technical Director, Laboratory Manager, or the ITAS Quality Assurance/Control Director, if necessary, for corrective action to be taken. The QCC shall keep a log of nonconformances. The log shall document the nonconformance, date of occurrence, reason for occurrence if known, date of corrective action, and corrective action taken.

14.2 SYSTEM AUDITS

System audits shall be conducted by the ITAS QA/C Director or designee. These audits shall be on a semi-annual basis and provide a thorough overview of implementation of the Quality Assurance Program within the laboratory. If project-specific audits are performed because of project requirements, the audit shall focus only on the performance of the laboratory for the project.

System audits will review operation of the laboratory and resulting documentation, including all items reviewed by the Quality Control Coordinator. Particular emphasis will be placed upon implementation of the Quality Control sample program and nonconformance log. Review of these aspects of the laboratory Quality Assurance Program should indicate trends adverse to data quality.

Audits by the ITAS QA/C Director shall be performed in the following manner:

- An audit plan shall be prepared which considers the activities of the specific laboratory. The Quality Assurance Manual and Manuals of Practice are the sources of generic requirements for the audit. Laboratory-Specific Attachments are the source documents for specific laboratory activities. The audit plan should be reviewed and updated for every semiannual audit, with information gained during previous audits considered. The audit plan shall be the basis for the audit and should define: participating auditors, applicable documents, schedule, scope of laboratory activities. (See Figure 14-1 for an example form for an Audit Plan.)

A lead auditor will be responsible for the audit planning and performance. As necessary, technical specialists will assist the audit team in preparing and conducting of the audit. All persons participating in the audit team will be independent of the laboratory audited.

- Based on the audit plan, detailed checklists of questions to be asked during the audit will be prepared. The checklists will provide adequate means for indicating whether the question is satisfactorily answered, or if it is not applicable, and for comments.

- The audit team will meet at the beginning of the audit with the Laboratory Manager to discuss the laboratory operations to be audited. The QCC should be available to the audit team throughout the audit.

- At the close of the audit, the audit team will meet with the Laboratory Manager to discuss the audit findings. As necessary, the Operations Manager, Group Leaders, Technical Director, and Quality Control Coordinator should attend the audit closure to discuss the findings. The lead auditor can close a finding during this discussion if the laboratory staff can satisfactorily demonstrate that the finding is inappropriate. Also during the audit-closure meeting, the means for corrective action and verifying correction will be discussed. If corrective action can be initiated immediately after the audit closure, the action should be taken.

- An audit report will be prepared by the lead auditor and will discuss the following:

  - Date and location of audit

  - Audit team members and persons contacted in the laboratory

  - Laboratory operations audited

  - Description of items requiring corrective action and, if possible, the means for correction

  - Due date for completion of corrective actions

  - Means for verifying completion of corrective action

  - Review of the Quality Assurance Program.

  The audit report shall be issued as soon as possible after completion of the audit (required within 30 days).

- The Laboratory Manager is responsible for responding to the audit report. Response shall be in writing to the lead auditor and shall

state the corrective action taken or the action underway. If correction can be verified through correspondence, the Laboratory Manager shall attach documentation of corrective action to the audit response.

- Upon receipt of the audit response, the lead auditor must verify completion of the corrective action.

- After verification of corrective action, the lead auditor will issue a closure statement stating that all corrective action has been completed and the audit is closed. All audits must be closed.

During the course of the semiannual system audits, the ITAS Quality Assurance/Control Director and/or lead auditor should be cognizant of recurring nonconformances in a laboratory or trends which will affect quality. Recurring nonconformances and trends should be addressed in the audit report. Correction for such events may require a review of the adequacy of the Quality Assurance Program. If the inherent problem lies within the Program, the Program shall be amended through appropriate revision of Quality Assurance documents.

14.3 DATA QUALITY AUDITS

Data quality audits are designed to address the precision, bias (accuracy), representativeness, and completeness of the data. Representative projects performed by the Laboratory are evaluated periodically by the ITAS QA/C Director or his designee. USEPA guidelines and project-specific checklists are used as criteria during the audits. Results are reported to the laboratory QC Coordinator, Laboratory Manager, and the Project Manager.

INTERNATIONAL TECHNOLOGY CORPORATION

QUALITY ASSURANCE AUDIT PLAN

PROJECT NAME	DEVELOPED BY
PROJECT NO.	DATE

AUDIT TITLE

AUDIT LOCATION	AUDIT SCHEDULE	CHECKLISTS TO BE USED
	FROM _____	GENERIC • • • • • ☐
	TO _____	AUDIT SPECIFIC • • ☐

AUDIT PERSONNEL

LEAD AUDITOR _____
QA STAFF _____
_____
TECHNICAL SPECIALISTS _____
_____
_____

GROUP OR ORGANIZATION TO BE AUDITED

IT GROUP _____
_____
SUBCONTRACTOR _____
_____
OTHER EXTERNAL ORGANIZATION _____
_____

AUDIT TYPE

PREQUALIFICATION • • • • • • • • ☐
FIELD OPERATIONS • • • • • • • • ☐
LABORATORY • • • • • • • • • • ☐
IN-PROCESS PROJECT • • • • • • • ☐
PROJECT AND REPORT • • • • • • • ☐
PROJECT FILE • • • • • • • • • • ☐
SOURCE • • • • • • • • • • • • ☐
OTHER • • • • • • • • • • • • ☐
_____
_____

APPLICABLE DOCUMENTS

IT QA MANUALS • • • • • • • • • ☐
_____
PROJECT QA PLAN • • • • • • • • ☐
CONTRACT/PROCUREMENT DOCUMENT • ☐
SPECIFIC/GENERIC PROCEDURES • • • ☐
_____
_____

ACTIVITIES AND MATERIALS TO BE AUDITED

SUBCONTRACTOR CAPABILITIES • • • • • ☐
QA/QC PROGRAMS • • • • • • • • • • ☐
FIELD ACTIVITIES & IN-PROCESS RECORDS • • ☐
IDENTIFICATION & CONTROL OF SAMPLES • • ☐
LABORATORY ACTIVITIES & IN-PROCESS RECORDS • ☐
EQUIPMENT CALIBRATION & RECORDS • • • ☐
PRODUCTION ACTIVITIES & IN-PROCESS RECORDS • ☐
IN-PROCESS NUMERICAL ANALYSES & DESIGN • ☐
COMPUTER PROGRAM VERIFICATION & RECORDS • ☐
COMPLETED RECORDS AND CHECKPRINTS.
SUBCONTRACTOR • • • • • • • • • ☐
FIELD • • • • • • • • • • • • ☐
LABORATORY • • • • • • • • • • ☐
NUMERICAL ANALYSES & DESIGN • • • • ☐

TRANSMITTAL OF INFORMATION TO IT • • ☐

TRANSMITTAL OF INFORMATION FROM IT:
DATA & RECORDS • • • ☐
COMPUTER ANALYSES • • ☐
SAR SECTIONS • • • • ☐
LICENSE/PERMIT APPLICATION • • • • ☐
RI/FS • • ☐
REPORT • • ☐
OTHER • • ☐
_____
_____

RECORD CONTROL & RETENTION • • • • ☐
OTHER • • • • • • • • • • • • ☐
_____
_____

FIGURE 14-1

QA AUDIT PLAN FORM

15.0 QUALITY REPORTS TO MANAGEMENT

Both the Quality Control Coordinator and the ITAS Quality Assurance/ Control Director are responsible for preparing reports to management indicating effectiveness of the Quality Assurance Program.

15.1 PERFORMANCE AUDIT REPORTING

The internal laboratory review activities of the Quality Control Coordinator shall be summarized in a monthly surveillance report to the ITAS QA/C Director. The report can be in the form of a checklist, with brief narratives as required, describing the activities reviewed. A copy of the nonconformance log for the month shall be attached. The monthly report shall emphasize ongoing or recurring problems. To demonstrate review, the Laboratory Manager shall sign and date the report. The signed copy shall be maintained by the Quality Control Coordinator for a period of two years after the date of the audit.

15.2 SYSTEM AUDIT REPORTING

Audit reports prepared from the semiannual system audits shall be distributed to the Vice President, Analytical Services; Laboratory Manager; Regional Director Analytical Operations; and the ITAS QA/C Director (if not the lead auditor); and the Environmental Projects Group Director, Quality Assurance and Discipline Management. The ITAS QA/C Director shall sign and date the reports and shall maintain copies for two years after the date of the audit.

15.3 NONCONFORMANCE/CORRECTIVE ACTION RESOLUTION FROM SYSTEM AUDITS

The Laboratory Manager and ITAS Quality Assurance/ Control Director shall seek to resolve directly all differences concerning cited nonconformances, requests for corrective action, or the completion of corrective action. If the difference cannot be resolved, the Vice President, Analytical Services shall be the arbiter. The decision of the Vice President is final.

15.4 MANAGEMENT REVIEW OF THE QUALITY ASSURANCE PROGRAM

Review of the appropriateness and adequacy of the Quality Assurance Program is ongoing. At any time, the Laboratory Manager should present recommended changes to the ITAS QA/C Director.

During system audits, the Quality Assurance Program should be discussed. The audit report will document recommendations made by either the Laboratory Manager or the audit team for revision.

In addition to these reviews, the Vice President, Analytical Services shall conduct an annual review of the Quality Assurance Program considering:

- Results of system audit reports, such as, are undesirable trends occurring?
- Is the present status of Quality Assurance documents adequate? Should manuals be revised; are new manuals needed?
- Are audits fulfilling their purpose?

The Vice President will consult with the Laboratory Managers and the ITAS QA/C Director, and the Environmental Projects Group Quality Assurance Director during the review as deemed appropriate. To document the review, the Vice President will issue a memorandum to the ITAS Quality Assurance/Control Director, the Environmental Projects Group Director, Quality Assurance and Discipline Management, and Laboratory Managers stating the extent of the review and recommendations.

16.0 TRAINING

All quality related activities performed by ITAS shall be accomplished by personnel qualified on the basis of education, experience, and training.

The following definitions are relevant to the discussion of training in this section:

- Training - In-depth instruction to develop proficiency in the application of requirements, methods, and procedures. Such instruction may be internal or external classroom sessions, courses, or informal on-the-job assignments.
- Indoctrination - To instruct in fundamentals so as to provide understanding of principles involved.
- Qualification (Personnel) - The characteristics or abilities gained through training or experience or both, that enable an individual to perform a required function.
- Certification - The action of determining, verifying and attesting, in writing, to the qualifications of personnel or material.
- Orientation - The act or process of acquainting individuals with the existing situation, environment or condition.

16.1 QUALIFICATIONS

Each laboratory shall have job descriptions for all positions affecting data quality. These job descriptions shall provide the minimum qualifications in terms of education and experience, knowledge, and skills necessary for an analyst to carry out work in the laboratory. During an annual performance review, the laboratory supervisors shall compare each analyst's performance with the qualifications established in his/her job description.

ITAS normally expects necessary knowledge and fundamental chemical laboratory skills to have been demonstrated by formal academic training to include course work in general chemistry, qualitative analysis, quantitative analysis, and instrumental analysis.

16.2 PROFESSIONAL STAFF, TRAINING, AND QUALIFICATIONS

Laboratory staffs are composed primarily of professional personnel who are scientists. Such personnel shall be assigned duties within the capabilities of their education and experience by the Laboratory Manager or the Operations Manager. Qualifications of all professional personnel shall be documented by resumes which include academic credentials, employment history, experience, and professional registrations. Technicians and support personnel performing a technical function are qualified through experience and this will be indicated in their resumes and training files. These personnel shall also be assigned by the Laboratory Manager or the Operations Manager based on their capabilities. Technicians and support personnel shall be supervised in their activities by experienced personnel until in the opinion of the Laboratory Manager or Operations Manager, or the responsible Group Leader they are capable of independently performing their duties. This authorization to perform independently shall be documented in the training files.

16.2.1. Technical Training and Qualifications

An analyst hired to perform sample preparation procedures and/or analytical procedures shall receive direct instruction from a professional staff member. To become qualified on a particular procedure, the analyst must pass a specified qualifying step under an instructor's supervision. The qualifying is described in Laboratory-specific Attachments to this Manual or SOPs. Only upon acceptable completion of the appropriate qualifying step shall the analyst perform the procedure without assistance or supervision.

Some of the basic operations that should be periodically reviewed with laboratory personnel are: sample logging, sample handling, measuring, weighing, glassware preparation, use of instrumentation, data handling and reporting, quality control and safety.

Technicians or analysts who have not performed an analysis within a period of

one year shall be required to re-qualify before being allowed to perform the procedure again. Laboratory-specific criteria shall be set for maintaining qualifications.

The Laboratory Manager shall perform an annual review and, as appropriate, re-qualify each technician for performing specific analytical procedures. The QC Coordinator shall monitor the qualification status of each technician based on spike results and length of time since the analysis has been performed by each technician. A quarterly status report shall be provided to the Laboratory Manager.

Written procedures shall detail the qualification process and criteria to be used.

16.2.2 Quality Assurance Training and Qualifications

General training in the requirements of the ITAS Quality Assurance Program is required of all laboratory personnel. Formal training sessions and examinations will be conducted and documented by QA personnel. The training program shall address regulatory requirements as appropriate, basic quality control practices, responsibilities of the technical staff, responsibilities of each the QCC, the reporting of non-conformances, and the performance of audits. In addition, each laboratory analyst shall become familiar with the laboratory quality assurance program by reading quality control procedures and sections of the laboratory-specific attachment and the ITAS QA Manual appropriate to his/her position.

For initial quality assurance qualification, each new employee shall be given a quality assurance examination that will test the trainee's knowledge and use of the laboratory's quality assurance program. Passing this examination will demonstrate sufficient knowledge of the quality assurance program to perform work in the laboratory upon training in a specific technical area. Acceptance criteria for the QA examination are given in the laboratory-specific training procedures, which also denotes qualification of current employees. For

maintenance of QA qualification and retraining, the laboratory staff shall conduct periodic quality assurance training sessions for the laboratory analysts. Training shall be documented on a standard form (Figure 16-1) and maintained in a training file for each individual.

Training in the nature and goals of the Quality Assurance Program shall be provided at least once a year to all laboratory personnel by the Quality Control Coordinator.

16.3 QUALITY CONTROL COORDINATOR'S TRAINING AND QUALIFICATIONS

All Quality Control Coordinators shall receive training from the ITAS QA/C Director so that the QC Coordinators are proficient in the requirements of the IT Analytical Services Quality Assurance Program and its application. Training will emphasize the performance of audits and required reporting. This will include audit fundamentals, objectives, characteristics, organization, performance and results of quality auditing, and reporting and follow-up actions involved in conducting audits. Quality Control Coordinators must be capable of communicating effectively, in writing and orally, with all levels of the organization. Additional training may be required by on-the-job experience for practice and evaluation, or by classroom sessions such as workshops, speech-making, or management courses which stress public speaking. Related training should include statistical quality control, the writing of Standard Operating Procedures (SOPs), and the review of analytical data and QA Project Plans using standardized guidelines.

An examination of QC Coordinators will be conducted at the end of the training course. The examination may be either oral, written, practical, or a combination thereof to verify the QCC's comprehension of and ability to apply the prescribed body of knowledge and auditing skills. The examination will be documented, thus providing certification of the QCC's qualifications, and signed by the ITAS QA/C Director. The certification must include the QC Coordinator's name, date of certification, and date for recertification; the bases of certification, such as education, experience, skills, training, and

examination results; and the signature of the authorized certifying person.

The examination should cover applicable aspects of the IT Quality Assurance Program, regulatory requirements, and Quality Assurance auditing. The ITAS Quality Assurance/Control Director is responsible for preparing and administering the examination and evaluating the results.

Continued proficiency of Quality Control Coordinators shall be maintained through active participation in Quality Assurance audits and the preparation and the review of Quality Assurance documents. Evaluation of Quality Control Coordinator proficiency shall be conducted on a yearly basis by the Director, ITAS QA/C. If a previously certified Quality Control Coordinator fails to maintain proficiency through active participation as above, formal requalification by the ITAS QA/C Director shall be required. This requalification will consist of:

- Re-examination
- Satisfactory participation in at least one Quality Assurance audit of an IT laboratory or project
- Additional training and/or experience as deemed necessary by the ITAS QA/C Director.

Formal certification and the annual evaluation of QC Coordinators shall be documented and maintained by the Director, ITAS QA/C.

16.4 QUALIFICATION AND TRAINING RECORDS

Each laboratory employee shall have a Personnel Qualifications Record as shown in Figure 16-2. The record shall be documented with technical and quality assurance procedures for which the employee is qualified, the dates of qualification and renewal, and the approval signature. Other qualification and training documents include:

- Personnel resumes
- Record of observation on a sample preperation procedure

- Results of qualifying sample sets
- Quality assurance examinations
- Attendance records of training sessions
- Professional certificates
- Internal training certificates of qualifications.

The QC Coordinator is responsible for maintaining the personnel qualifications and training records as quality documents in the Quality/Operations record system Category N. The ITAS QA/C Director maintains the training file for all ITAS QC Coordinators.

Figure 16 - 1

EXAMPLE - INDIVIDUAL TRAINING RECORD

FOR: _____ HIRE DATE: _____

TITLE: _____ SUPERVISOR: _____

ACADEMIC TRAINING (to include College, Business, Trade or Technical School)

Name of Institution (including current enrollment)	City and State	Curriculum Major	Attendance	Title of Degree(s) Received or Expected	Date Degree Received or Expected

PROFESSIONAL CERTIFICATIONS

Certification	From	Valid from, to

SPECIAL SCHOOLING/TRAINING

Year	Course	Sponsored by/Location

ON THE JOB TRAINING

	Dates BASIC	Dates QUALIFIED	Action & Signature
Quality Assurance Manual			
Standard Operating Procedures			
Laboratory Documentation			
Receiving Inspection			
Specific Job Function			
New Employee Orientation			
Safety Orientation			

Documented On _____

By _____

QC Concurrence _____

Figure 16 - 2

EXAMPLE - PERSONNEL QUALIFICATION RECORD

NAME: _____ HIRE DATE: _____

TITLE: _____ SUPERVISOR: _____

PROCEDURE NAME, NUMBER	QUALIFY DATE	RENEW BY	APPROVAL

TRAINING SESSION	DATE	APPROVAL

ITAS QA MANUAL

GLOSSARY

This glossary is intended to provide standard definitions for terms in common use in ITAS Laboratories.

Absolute Method

Method in which characterization is based entirely on physical (absolute) defined standards.

Accuracy

The degree of agreement of a measurement (or an average of measurements of the same thing), X, with an accepted reference or true value, T, usually expressed as the difference between the two values, X - T, or the difference as a percentage of the reference or true value, 100(X - T)/T, and sometimes expressed as a ratio, X/T. Accuracy is a measure of the bias inherent in the system.

Aliquot

A measured portion of a sample taken for analysis.

Analysis Matrix Spike

A sample created by spiking target analytes into a prepared portion of a sample just prior to analysis.

Arithmetic Mean (Cm for populations, X for samples)

The most commonly used measure of central tendency, commonly called the "average". The sum of all the values of a set divided by the number of values in the set.

Audit

A documented activity performed in accordance with written procedures or checklists to verify, by examination and evaluation of objective evidence, that applicable elements of the quality assurance program have been developed, documented and effectively implemented in accordance with specified requirements. An audit should not be confused with surveillance or inspection.

Bias

A systematic (consistent) error in test results. The difference between the population mean and the true or reference value, or as estimated from sample statistics, the difference between the sample average and the

reference value.

Blind Replicate

A blind replicate sample is a duplicate sample which has been introduced as a separate sample by the Quality Control Coordinator during the log-in process or prior to analysis; it is not known to be a replicate sample when the analyst receives it (blind).

Bulk Sampling

Sampling of a material that does not consist of discrete, identifiable, constant units, but rather of arbitrary, irregular units (i.e., insulation material, asbestos tiles).

Calibration

Establishment of a relationship between various calibration standards and the measurements of them obtained by a measurement system, or portions thereof. The levels of the calibration standard should bracket the range of levels of which actual measurements are to be made; or the act of making a scheduled comparison of instrument performance against national standards for instruments which measure physical parameters, such as mass, time, and temperature. This type of calibration is independent of use in specific analyses and projects.

Calibration Check Compounds (CCC)

Target compounds used to evaluate the calibration stability (precision) of the GC/MS system. Maximum percent deviations of the CCCs are defined in the USEPA's Contract Laboratory Program (CLP) protocol.

Calibration Standard

A standard used to quantitate the relationship between the output of a sensor and a property to be measured. Calibration standards should be traceable to Standard Reference Materials or primary standard.

Certified Reference Material (CRM)

A reference material accompanied by a certificate issued by an organization generally accepted as technically capable to do so.

Chain-Of-Custody

A system of documentation to demonstrate physical custody and traceability of samples to provide defensible, scientifically sound data.

Certificate of Analysis

An ITAS form for reporting analytical results.

Checkprint

Copy of raw data or data report on which data is reviewed (checked), initialed or signed, and dated.

Check Standard Analyses

A midpoint standard or check standard analyzed with each group of samples to verify the standard curve.

Chemical Calibration

Relation of response of a measurement system to the concentration or amount of analyte of concern.

Coefficient of Variation (Relative Standard Deviation)

A measure of precision (relative dispersion). It is equal to the standard deviation divided by the mean and multiplied by 100 to give a percentage value.

$$CV\ (RSD) = \frac{s}{\bar{X}} \times 100 \text{ for a sample group}$$

Collaborative Tests (or Studies)

The evaluation of a new analytical method under actual working conditions accomplish by the participation of a number of typical or representative laboratories in analyzing portions of carefully prepared homogeneous samples.

Collocated Samples

Independent samples collected in such a manner that they are equally representative of the variable(s) of interest at a given point in space and time. The results will include sampling as well as anlaytical variability.

Comparability

A measure of the confidence with which one data set can be compared to another.

Comparative Method

Method in which characterization is based on chemical standards (i.e., comparison with such standards).

Completeness

The amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal operations, to fully evaluate and understand the system under optimum conditions; usually expressed as a percentage.

Composite

A sample composed of two or more increments.

Confidence Coefficient

The chance or probability, usually expressed as a percentage, that a confidence interval has of including the population value.

Confidence Interval

A value interval that has a designated probability (the confidence coefficient) of including some defined parameter of the population.

Confidence Limits

The outer boundaries of a confidence interval.

Continuing Calibration

Analytical standard run every 12 hours to verify the calibration of the GC/MS system.

Contract Laboratory Program CCLP

USEPA Superfund contract-required analytical methods and QC criteria.

Corrective Action

A measure taken to correct a deficiency and to minimize the possibility of recurrence.

Correlation Coefficient

A number between -1 and 1 that indicates the degree of linear relationship between two sets of numbers.

Data Quality Objectives

The precision, accuracy, representativeness, completeness and comparability goals to be achieved for a particular data set or project.

Data Validation

A systematic effort to identify any outliers and/or errors, and thereby cause deletion or flagging of suspect values for investigation. To provide valid data to the user and to demonstrate that numerical computation of data is correct and that it is correctly reported.

Data Verification

The steps taken within an analytical laboratory so that reported results correctly represent the analyses performed.

Decafluorotriphenylphosphine (DFTPP)

Compound chosen to establish mass spectral tuning performance for semi-volatile analysis by GC/MS.

Deficiency

An unauthorized deviation from accepted procedures, practices, or standards; or a defect in an item that could lead to degradation of quality.

Degrees of Freedom

The number of independent deviations used in calculating an estimate of the standard deviation.

Duplicate Sample Analyses

Duplicate analyses (different aliquots of the same sample) are performed to evaluate the precision of an analysis.

Error

The difference between an observed or measured value and its true value, or the probability interval that contains the systematic and random error with 1-Cs confidence.

External Reference Material (ERM)

A reference material provided by someone other than the end-user laboratory.

Field Blank

A blank that is prepared, handled, and analyzed in the same manner as normal carrying agents except that it is not exposed to the material to be selectively captured during sample collection.

Field Matrix Spike

A sample created by spiking target analytes into a portion of a sample in the field at the point of sample acquisition.

Geometric Mean

The nth root of the product of all values in a set of n values or the antilogarithm of the arithmetic mean of the logarithms of all the values of a set of n values. The geometric mean is generally used when the logarithms of a set of values are nearly normally (Gaussian) distributed, such as is the case of much pollution data.

Grade A Standard

Atomic weight standard.

Grade B Standard

Ultimate standard - a substance which can be purified to virtually Grade A.

Grade C Standard

Primary standard - commercially purified to a purity of 100 C= 0.02%.

Grade D Standard

Working standard - commercially available, purity of 100 C= 0.05%.

Grade E Standard

Secondary standard - of lower purity, standardized against Grade C material.

Gross Sample (also called Bulk Sample, Lot Sample)

One or more increments of material taken from a larger quantity (lot) of material for assay or record purposes.

Homogeneity

The degree to which a property or substance is randomly distributed throughout a material. Homogeneity depends on the size of the units

under consideration. Thus a mixture of two minerals may be inhomogeneous at the molecular or atomic level, but homogeneous at the particulate level.

Hazardous Substance List

A USEPA Superfund list of chemicals whose presence is to be tested in each sample received for CLP analysis. It includes the Priority Pollutants plus other commonly-occurring chemicals found at hazardous waste sites.

IDL

Instrument Detection Limit - the smallest signal an instrument can reliably detect.

Increment

An individual portion of material collected by a single operation of a sampling device from parts of a lot separated in time or space. Increments may be either tested individually or combined (composited) and tested as a unit.

Individuals

Conceivable constituent parts of the population.

Initial Calibration

Analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of an instrument to the target compounds.

Internal Reference Material (IRM)

A reference material developed by a laboratory for its own internal use.

Internal Standards

Compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for VOAs), and sample extract (for semi-volatiles) at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target compounds by GC/MS.

Laboratory Matrix Spike

A sample created by spiking target analytes into a portion of a sample when it is received in the laboratory.

Laboratory Sample

A sample, intended for testing or analysis, prepared from a gross sample or otherwise obtained. The laboratory sample must retain the composition of the gross sample. Often reduction in particle size is necessary in the course of reducing the quantity.

Laboratory-Specific Attachment

Documents supplementary or alternative implementing practices and services of a specific laboratory; for the specific laboratory, becomes incorporated with ITAS Quality Assurance Manual. Laboratory-Specific Attachments to the Quality Assurance Manual are prepared for three purposes:

- If an individual ITAS laboratory provides services that are unique to that laboratory, the service and necessary Quality Assurance requirements will be described in an attachment.
- Certain aspects of laboratory operations discussed in the Quality Assurance Manual are dependent upon laboratory facilities. Quality Assurance activities much as preventive maintenance, calibration, and data verification are functions of the equipment in a specific laboratory. Attachments will be prepared by each laboratory to document their specific operations. Laboratory-Specific Attachments are adopted as part of the Quality Assurance Manual for the preparing laboratory.
- A specific laboratory may perform certain actions in accordance with an alternate, approved technique to that presented in the ITAS QA Manual.

LOD

Limit of Detection - smallest concentration/amount of analyte that can be reliably reported as found/detected in a material/sample.

Lot

A quantity of bulk material of similar composition whose properties are under study.

Lot Size (N)

The number of units in a particular lot.

Lot, Batch

A definite quantity of samples collected under conditions that are considered uniform.

Macro Method

Method requiring more than milligram amounts of sample.

Manuals of Practice

Detailed discussions of specific technical subjects. Provides detailed information related to technical topics discussed in the Quality Assurance Manual. For example, a Manual of Practice for the field collection, preservation, and shipment of samples to ITAS laboratories provides specific uniform direction to IT personnel.

Matrix

The predominant material in which the analyte(s) of primary interest is found in the sample to be analyzed. A sample matrix is usually either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

Matrix Effect

Materials in a sample cause interferences in measurement of analytes and QC parameters which prevent the acquisition of acceptable results.

Matrix Spike

Aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix. The percent recovery for the respective compound(s) will then be calculated.

Matrix Spike Duplicate

A second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

MDL

Method Detection Limit - smallest concentration/amount of analyte a method can reliably detect, wherever located; when the sample containing the analyte is processed through the entire preparation and analysis procedure. The minimum concentration of an analyte that can be reported with 99% confidence to have a value that is above zero. The MDL is operationally defined as:

$$MDL = st_{(n-1,\ 1-\alpha\ =\ 0.99)}$$

Where s = the standard deviation of a minimum of 7 measurements of a solution containing the analyte at a concentration near the lowest standard recommended in the method and

$t_{(n-1, 1-\alpha = 0.99)}$ = the student's t value for a one-tailed test at the 99% confidence level and n-1 degrees of freedom.

Mean

The arithmetic mean is a measure of control tendency; it is equal to the sum of the observed values divided by the number of observations. Also call "average".

Measurement

The process or operation of ascertaining the extent, degree, quantity, dimensions, or capability with respect to a standard.

Median

The middle value of a set of data when the set of data are ranked in increasing or decreasing order. If there are an even number of values in the set, the median is the arithmetic average of the two middle values.

Method

An assemblage of techniques.

Method Blank

An analytical control consisting of all reagents, which may include internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background contamination. A method blank is a volume of deionized, distilled laboratory water for water samples, or a purified solid matrix for soil/sediment samples.

Micro Method

Method requiring milligram or smaller amounts of sample.

Mode

The value or values occurring most frequently in a set or sample of data.

Modified Method

A standard or reference method which has been changed to meet project or matrix requirements.

Nonconformance

Any event which is beyond the limits established for laboratory operation. A deficiency in characteristic, documentation, or procedure sufficient to render quality of an item unacceptable or indeterminate.

Observed value, Observation, or Variate (X)

The particular designated value of a characteristic; for example, X444C1555, X444C2555, X444C3555, and so on.

Operational Calibration

Routinely performed as part of instrument usage, such as the development of a standard curve for use with an atomic absorption spectrophotometer. Operational calibration is generally performed for instrument systems.

Outlier

An extreme value that statistically does not belong to the group of values with which it is associated.

Parameter

A constant or coefficient that describes some characteristic of a population (e.g., standard deviation, mean, regression coefficients). Also, a chemical being measured, i.e., an analyte.

Percent Difference

If two independent measurements of the same characteristics are available, it is convenient to use the percent difference instead of the coefficient of variation.

$$\text{Percent Difference} = \frac{|X - X|}{(X + X)/2} \times 100 = \frac{\text{Range}}{\bar{X}} \times 100$$

Percent Moisture

An approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at 105°C, including water.

Percent Recovery (%R)

The results of spiked samples are used to calculate this quality control parameter for accuracy evaluation.

$$\%R = \frac{\text{observed concentration spiked sample - sample concentration}}{\text{true spike concentration}} \times 100\%$$

Performance Audit

Planned independent sample checks of actual output made on a random basis to arrive at a quantitative measure of the quality of the output. Conducted on an ongoing basis within the laboratory by the Quality Control Coordinator. These audits are reported to the Laboratory Manager.

Periodic Calibration

Performed at prescribed intervals for equipment, such as balances and ovens. In general, equipment which can be calibrated periodically is a distinct, singular purpose unit and is relatively stable in performance.

Population

A generic term denoting any finite or infinite collection of individual things, objects, or events in the broadest concept an aggregate determined by some property that distinguishes things that do and do not belong.

Pre-Award Survey

On-site inspection, review, and discussions with prospective contractors at their facilities. Discussions would normally include, but not be limited to, the proposed project plan, personnel, procedures, schedule, and facilities.

Precision

A measure of mutual agreement (or variability) among individual measurements of the same property, usually under prescribed similar conditions. Precision is most desirably expressed in terms of the standard deviation but can be expressed in terms of the variance, range, or other statistic.

Preventive Maintenance

An organized program, within ITAS laboratories, of actions (such as equipment cleaning, lubricating, reconditioning, adjustment and/or testing) taken to maintain proper instrument and equipment performance

and to prevent instruments and equipment from failing during use.

Primary Standard

A material having a known property that is stable, that can be accurately measured or derived from established physical or chemical constants, that is readily reproducible and which can be accepted (within stated limits) and used without question to establish the same value of another substance or item.

Procedure

Detailed instructions to permit replication of a method.

Proficiency Testing

Special series of planned tests to determine the ability of field technicians or laboratory analysts who normally perform routine analyses. The results may be used for comparison against established criteria, or for relative comparisons among the data from a group of technicians or analysts.

Project-Specific Manuals

A manual that describes analytical and/or QA procedures required by a regulatory agency or by contract. It may supplement or change Quality Assurance and/or Quality Control practices for specific project.

Protocol

Methodology specified in regulatory, authoritative, or contractual situations.

Purge and Trap (Device)

Analytical techniques (device) used to isolate volatile (purgeable) organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

Qualification (Personnel)

The characteristics of abilities gained through education, training or experience, as measured against established requirements, such as standards or tests, that qualify an individual to perform a required function.

Quality

The totality of feature and characteristics of a product or service that bears on its ability to satisfy a given purpose. Absence of defects.

Quality Assurance

The overall controls imposed upon laboratory operations. The name given to procedures by which one ascertains that individual measurements are good enough for their intended purpose. Included are all actions taken by personnel, and the documentation of laboratory performance as specified in the Quality Assurance Program. A system for integrating the quality planning, quality assessment, and quality improvement efforts of various groups in an organization to enable operations to meet user requirements at an economic level.

Quality Assurance Manual

An orderly assembly of management policies, objectives, principles, and general procedures by which ITAS outlines how it intends to produce quality data. The Quality Assurance Manual defines the ITAS Quality Assurance Program. The Manual discusses all aspects for Quality Assurance and Quality Control, both administrative and technical. However, it is not intended that the Manual provide in-depth technical discussion. The Quality Assurance Manual has precedence in policy matters over all other ITAS quality-related documents. The Manual is applicable to and provides direction for all ITAS analytical laboratories.

Quality Assurance Project Plan

An orderly assembly of detailed and specific procedures by which an agency or laboratory delineates how it produces quality data for a specific project or measurement method. A USEPA-specified 16-element format is used to present the information for client and/or regulatory agency approval, prior to beginning field/laboratory work.

Quality Control

The daily, specific actions taken within the laboratory to verify sample integrity, performance of analyses, data processing, and record maintenance. A system of inspections, testing, and remedial actions applied to a process or operation so that, by inspecting a small portion (a sample) of the product currently produced, an estimate can be made of its quality and whether or not, or what if any, changes need to be made to achieve or maintain a predetermined or required level of quality.

QC Check Sample

A reference matrix containing known concentrations of parameters of

interest. If prepared in the laboratory, it is made using stock standard solutions independent of those used for calibration. If the results of these parameters do not meet acceptance criteria concentrations are taken.

Random Error

Variations of repeated measurements that are random in nature and individually not predictable.

Random Samples

Samples obtained in such a manner that all items or members of the lot, or population, have an equal chance of being selected in the sample.

Range (R)

The difference between the largest and smallest numbers in a set of numbers.

Range, Relative (RR)

The range divided by the mean of a particular set of numbers.

Raw Data

All documentation associated with the original recording of anlaytical results pertinent to a specific sample or set of samples. This may include laboratory worksheets, calculation forms, instrument-generated output, analyst notes, etc., from sample receipt through final reporting.

Reagent Blank

Sample composed of materials (water, etc.) which will be added to client samples and analyzed. If contaminants are found in the reagents at levels affecting these sample results, corrective actions must be taken.

Reagent Water

Water in which an interferent is not observed at or above the minimum quantitation limit of the parameters of interest. D1193 ASTM Type II reagent water specifications are:

Grade of Water	Maximum Total Matter (mg/l)	Maximum Electrical Conductivity at 25°C (mho/cm)	Minimum Electrical Resistivity at 25°C (M ohms/cm)	Minimum Color Retention Time of KMnO (min)
Type II	0.1	1.0	1.0	60

Reconstructed Ion Chromatogram (RIC)

A mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time.

Recovery

A determination of the accuracy of the analytical procedure made by comparing measured values for a fortified (spiked) sample against the known spike values. Recovery is determined by the following equation:

$$\%Rec = \frac{\text{measured spiked value - unspiked value}}{\text{known spiked value}} \times 100\%$$

Reference Material (RM)

Substance in which one or more properties are established for use to calibrate or verify a measurement.

Reference Method

A method of known and demonstrated accuracy.

Regression Coefficients

The quantities describing the slope and intercept of a regression line. Intercept (β_o for populations, b_o for samples), Slope (β_1 for populations, b_1 for samples).

Regression Line or Equation

The function that indicates the regression relationship. For example; $X = + T$ for populations, and $X = b + b T$ for samples.

Relative Error

An error expressed as a percentage of the true value or accepted reference value.

Relative Percent Different (RPD)

Statistic for evaluating the precision of a replicate set. For replicate results D_1 and D_2:

$$RPD\% = \frac{D_1 - D_2}{\frac{D_1 + D_2}{2}} \times 100\%$$

Relative Response Factor (RRF)

A measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RRF is determined by the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where A = area of the characteristic ion measured
C = concentration
is = internal standard
x = analyte of interest

Relative Standard Deviation

See Coefficient of variation.

Repeatability

The precision, usually expressed as a standard deviation, measuring the variability among results of measurements at different times of the same sample at the same laboratory. The unit of time should be specified, since within-day repeatability would be expected to be smaller than between-day repeatability.

Replicability

The precision, usually expressed as a standard deviation, measuring the variability among replicates. It could also be expressed as an upper confidence limit for the difference between two replicates as discussed above.

Replicated Sample

A sample that has been divided into two or more portions, at some step in the measurement process. Each portion is then carried through the remaining steps in the measurement process.

Replicates

Repeated but independent determinations of the same sample, by the same analyst, at essentially the same time and same conditions.

Representative Sample

A sample taken to represent a lot or population as accurately and precisely as possible.

Representativeness

The degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition.

Reproducibility

The precision, usually expressed as a standard deviation, measuring the variability among results of measurements of the same sample at different laboratories.

Resolution (aka Separation)

The separation between peaks on a chromatogram; calculated by dividing the height of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

Routine Method

Procedure used in measurement of an analyte. It must be qualified by other adjectives since no degree of reliability is implied.

Sample (Statistical)

A subset or group of objects or things selected from a larger set, called the "lot", or "population". It may consist of an individual or groups of individuals. A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

Sample Average

Same as arithmetic mean.

Sample Size (n)

The number of units in a sample.

Secondary Standard

A material having a property that is calibrated against a primary standard.

Segment

A specifically demarked portion of a lot, either actual or hypothetical.

Semi-volatile Compounds

Compounds amenable to analysis by extraction of the sample with an organic solvent. Used synonymously with Base/Neutral/Acid (BNA) compounds.

Spiked Sample

A normal sample of material (gas, solid, or liquid) to which is added a known amount of some substance of interest. The extent of the spiking is unknown to those analyzing the sample.

Split Sample

A sample divided into two portions, one of which is sent to a different organization or laboratory and subjected to the same environmental conditions and steps in the measurement process as the one retained in-house.

Standard Analysis

An analytical determination made with known quantities of target compounds; used to determine response factors.

Standard Deviation

The square root of the variance of a set of values:

$$s = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n}}$$

A measure of the dispersion about the mean of the elements in a population.

Standard deviation(s)

A measure of the dispersion about the average of the elements in a sample. An estimate of the standard deviation of a population.

Standard Method

A method of known and demonstrated precision issued by an organization generally recognized as competent to do so.

Standard Operating Procedures

Written, detailed documents describing the performance of routine laboratory tasks. They specify what is done, whose responsibility it is to perform tasks and whose to verify their correctness; they are sufficiently detailed to provide data of known quality and integrity, with a minimum loss of data due to out-of-control situations. They also provide for documentation to record the performance of all tasks and their results, and demonstrate the validation of the data each time the data are recorded, calculated, or transcribed. SOPs address the following major elements upon which analytical quality depends:

Organization and personnel
Facilities and equipment
Analytical methodology
Sample custody procedures
Quality control
Data handling.

Standard Reference Material (SRM)

A material produced in quantity, of which certain properties have been certified by the National Bureau of Standards (NBS) or other agencies to the extent possible to satisfy its intended use.

Standardization

The establishment of the value of a potential standard with respect to an established or known standard.

Statistic

A constant or coefficient that describes some characteristic of a sample. Statistics are used to estimate parameters of populations.

Statistical Control Chart

A graphical chart with statistical control limits and plotted values (usually in chronological order) of some measured parameter for a series of samples.

Strata

Segments of a lot that may vary with respect to the property under study.

Stratified Sample

A sample consisting of various portions that have been obtained from identified subparts or subcategories (strata) of the total lot, or population. Within each category or strata, the samples are taken

randomly to obtain a more representative sample than that which might otherwise be obtained by a completely random sampling.

Subsample

A portion taken from a sample. A laboratory sample may be a subsample of a gross sample; similarly, a test portion may be a subsample of a laboratory sample.

Surrogate Standard Analyses

A sample is fortified with surrogate spiking compounds before purging or extraction to monitor preparation and analysis of samples.

Surrogates (Surrogate Standard)

Compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labelled compounds not expected to be detected in environmental media.

Surveillance

A review (monitoring) or observation of ongoing work for the purpose of verifying that an action has been accomplished as specified.

System Audit

A systematic on-site qualitative review of facilities, equipment, training, procedures, recordkeeping, validation, and reporting aspects of total (quality assurance system to arrive at a measure of the capability and ability of the system. Performed on a scheduled, periodic basis by the Quality Control Coordinator or the ITAS Quality Assurances/Control Director.

System Performance Check Compounds (SPCC)

Target compounds designated to monitor chromatographic performance, sensitivity and compound instability or degradation on active sites. Minimum response factor criteria for the SPCCs are defined in the USEPA's CLP GC/MS protocols.

Systematic Error

The condition of a consistent deviation of the results of a measurement process from the reference or known level.

Target Analyte Spiking

Spiking with the analyte that is of basic interest in the environmental sample.

Technique

Physical or chemical principle for characterizing materials of chemical systems.

Tentatively Identified Compounds (TIC)

Compounds detected in GC/MS analysis of samples that are not target compounds, internal standards or surrogate standards. Up to 30 peaks (those greater than 10% of peak areas or heights of nearest internal standards) are subjected to mass spectral library searches for tentative identification.

Traceability of Data

The entire chain of acquired data from the original acquisition effort through to the final tabulation, synthesis, reduction and storage activity will be clearly documented in a fashion which will allow complete reconstruction.

Traceability of Instrumentation

All equipment used in the derivation of data during the conduct of project activities either in the field or during laboratory efforts shall be designated by a unique identification number. This number will in turn be indexed into a system which specifically designates all devices and instrumentation employed by a particular measurement unit and will in turn specify usage and performance periods associated with the identified instrument or device.

Traceability of Samples

During all environmental monitoring field efforts, acquired samples will be assigned specific and unique identification numbers, and such numbers shall be accompanied by documentation which clearly identifies all parameters associated with sample acquisition.

Traceability of Standards

All measurement activities which utilize standards for calibration purposes must have reliable and documented evidence as to the verifiability of standards used.

In this context, all calibration standards must be validated against primary standards such as those available from the National Bureau of

Standards (NBS) or from other primary sources.

Trip Blank Analyses

Trip blanks will be prepared by filling two VOA vials with organic-free water and shipping the blanks with each field kit (group of samples). Trip blanks accompany the sample bottles through collection and shipment to the laboratory and are stored with the samples. If the trip blanks indicate possible contamination of the samples, depending upon the nature and extent of the contamination, the samples may either be corrected for the trip blank concentration or the sources re-sampled.

Verification

An act of confirming and substantiating that an activity or condition has been implemented in conformance with the specified requirements.

APPENDIX B

IT ANALYTICAL SERVICES QUALITY ASSURANCE MANUAL

IT Mixed Waste Laboratory (Oak Ridge, Tennessee)

QUALITY ASSURANCE MANUAL

LABORATORY-SPECIFIC ATTACHMENT

ITAS-OAK RIDGE

MIXED WASTE LABORATORY

Approved for Issue:

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Laboratory Manager/Technical Director

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Corporate Director of Quality Assurance

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QC Coordinator

Controlled Copy No.:_____ Issued To:_____

STATEMENT OF MANAGEMENT POSITION

IT Corporation is committed to providing quality services for environmental management; services which meet the needs of our clients, satisfy regulatory requirements, and are commensurate with the current state of the art.

To satisfy our clients' quality objectives, to meet regulatory requirements, and to comply with IT Corporate-wide requirements, IT Analytical Services (ITAS) has adopted a comprehensive Quality Assurance Program. The principles and practices of the Program apply to every employee at every level within ITAS; they are fundamental to the way we do business and to the services we provide.

The ITAS Quality Assurance Manual (QAM), along with this ITAS Mixed Waste Laboratory Specific Attachment (LSA), defines the overall statement of Program policy for the Mixed Waste Laboratory. The ITAS QA Manual, this Specific Attachment and their companion Program documents provide both guidance to ITAS Mixed Waste personnel in fulfilling their responsibilities, and serve as a statement of the laboratory's commitment to quality to external parties.

Implementation of the Program is the responsibility of all ITAS Mixed Waste personnel. Management at every level has the duty and authority to insist that these responsibilities are met, and that the principles and practices of the Program are followed and implemented.

The Quality Control Coordinator (QCC) assures that the Program operates on a day-to-day basis as intended. The QCC has the authority and duty to stop work if and when necessary to satisfy Program requirements.

To verify that the Quality Assurance Program is successfully implemented, the laboratory is independently audited regularly by the ITAS Director of Quality Assurance and Compliance, who is directly responsible to the Vice President, Analytical Services. In addition, the laboratory is subject to additional audits by the corporate Director of Quality and Compliance, and by various regulatory authorities and other outside agencies.

Michael J. Smith
Vice President
Analytical Services

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MW1.0 INTRODUCTION

Criteria for Quality Assurance practices within International Technology Analytical Services (ITAS) laboratories are documented in the IT Analytical Services Quality Assurance Manual (QAM). The ITAS QAM was written to encompass all analytical laboratories within IT Corporation. The purpose of the ITAS QAM is to assure uniform Quality Assurance practices among all IT laboratories; individual laboratory practices may vary, but can never be less restrictive.

The ITAS Oak Ridge, Tennessee (Mixed Waste) Laboratory has written a "laboratory-specific" manual. This manual is an attachment to the ITAS QAM. As required by the ITAS Quality Assurance Program, this attachment is a supplement to, and is incorporated with, the ITAS Quality Assurance Manual to provide the Quality Assurance Program for the Oak Ridge (Mixed Waste) Laboratory operations (ITASMWL).

For the Mixed Waste laboratory operations, the ITAS Quality Assurance Manual and this attachment have been prepared to fulfill the policies of IT Corporation, the requirements of the US EPA, the State of Tennessee, and applicable sections of ANSI/ASME NQA-1 and US NRC Regulatory Guide 4.15.

MW1.1 Format of the Attachment

The laboratory-specific attachment is formatted in the same manner as the ITAS Quality Assurance Manual so that personnel in Mixed Waste laboratory operations can readily merge the requirements of the ITAS QAM and this document. Section headings

for both documents are the same with the exception that this attachment uses the section prefix MW so that attachment sections are clearly identified.

The attachment is not meant to be a self-standing document, the ITAS Manual must be included with the attachment to fully describe the laboratory-specific Quality Assurance Program. The attachment includes only those sections of the ITAS Quality Assurance Manual which must be supplemented for full documentation of a specific activity, or which provide for the parallel implementation of Quality Assurance practices. In the event that the attachment describes an alternative means for implementation, when compared with the manner prescribed in the ITAS Quality Assurance Manual, the implementation described in this attachment shall take precedence for Mixed Waste laboratory operations. Contractual obligations will, of course, supercede both the ITAS QA Manual and this supplement. Review and approval of the attachment, as prescribed in this Manual is in accordance with the requirements of the ITAS Quality Assurance Program.

Table MW1-1 lists the sections of the ITAS Quality Assurance Manual. The sections contained in this attachment are also identified along with the applicable sections of ANSI/ASME NQA-1 and US NRC Regulatory Guide 4.15. If a section is not contained in the attachment, the corresponding section in the ITAS Manual is adopted without supplement or change. Subsections are not listed in Table MW1-1, but are fully identified in the Table of Contents.

MW1.2 Document Control, Distribution, and Revision

The following are supplements to the discussions of Section 1.3 of ITAS Quality Assurance Manual, and specifics relating to Document Control, Distribution, and Revision.

Identified controlled documents shall be as follows: ITAS Quality Assurance Manual (prepared and controlled through the national ITAS Quality Assurance office), the ITAS-Mixed Waste Laboratory-Specific Attachment to the Quality Assurance Manual, internal Standard Operating Procedures, in-house prepared laboratory data forms, and project-specific manuals.

MW1.2.1 Responsibilities for Document Preparation and Approval

The Quality Control Coordinator shall be responsible for the format and content of the laboratory-specific attachment to the Quality Assurance Manual. Prior to initial issuance and any subsequent major revisions, the LSA shall be reviewed and approved at a minimum by the Laboratory Director and the Director, ITAS Quality Assurance and Compliance. The Quality Control Coordinator shall compile, issue, distribute, and maintain the LSA, record the distribution, and review the contents on an annual basis.

Each Group Supervisor shall create or approve all internally generated procedures and forms for his/her assigned area of work. The Group Supervisor shall be responsible for initially verifying the technical accuracy and adequacy of internal procedures and forms based on approved external methods and his/her technical expertise.

All Standard Operating Procedures and their subsequent revisions shall be reviewed and approved by the Laboratory Director, the Laboratory Manager/Technical Director, the Quality Control Coordinator, and the Director, ITAS Quality and Compliance. Each reviewer is responsible for assuring that the procedure or form is accurate and adequate based on his/her area of expertise. The QCC shall compile, issue, and distribute Standard Operating Procedures.

In-house data forms shall be reviewed and approved by the Group Supervisor (where applicable), the Laboratory Manager, and the Quality Control Coordinator. The QCC is responsible for maintaining a list of current forms.

Project-specific manuals shall be reviewed and approved by the Laboratory Manager, the Quality Control Coordinator, and the Project Manager if the laboratory is a part of a broad-based project and if required by contract.

MW1.2.2 Document Changes and Revisions

Changes to documents, other than those defined as minor changes, shall be considered as major changes and shall be reviewed and approved by the same organizations that performed the original review and approval unless other organizations are specifically designated. The reviewing organization shall have access to pertinent background data or information upon which to base their approval.

Minor changes shall be defined as those changes that do not affect the content or quality of the action being prescribed in the document, such as: punctuation or grammatical changes,

aesthetic changes or, in the case of data forms, small changes made strictly for the convenience of the user that do not affect the accuracy or integrity of the document.

A procedure change form, subject to all review and approval criteria as defined above, may be attached to a document to reflect policy and procedural changes which become effective between revisions. These changes will then be incorporated into the body of the document at the time of the next revision.

TABLE MW1-1

Contents of Laboratory-Specific Attachment
Mixed Waste Laboratory Operations and
the Applicable Regulatory Guide Requirements

Sections of ITAS Quality Assurance Manual	Content	Laboratory Specific Attachment Sections	ANSI/ASME NQA-1 Supplemental Requirements[a]	USNRC Regulatory Guide 4.15[a]
1.0	Introduction	MW1.0	2, 6, 6S-1	C.3
2.0	Organization	MW2.0	1, 1S-1	C.1
3.0	Standard Laboratory Practice	MW3.0	2	
4.0	Material Procurement and Control	MW4.0	4, 4S-1, 7, 7S-1, 8, 8S-1	
5.0	Sample Receipt & Initiation of Testing Program	MW5.0	8, 8S-1, 13, 13S-1	C.5
6.0	Calibration Practices	MW6.0	12, 12S-1, 14	C.6.1
7.0	Preventive Maintenance	MW7.0	14	
8.0	Analysis of Quality Control Samples	MW8.0	11	C.6.3
9.0	Analytical Procedures	MW9.0	9, 9S-1, 11, 11S-1	C.3
10.0	Data Verification	MW10.0	5	C.6.4, C.8
11.0	Data Reports	MW11.0		
12.0	Records Management	MW12.0	17, 17S-1	C.4

TABLE MW1-1
(Continued)

Sections of ITAS Quality Assurance Manual	Content	Laboratory Specific Attachment Sections	ANSI/ASME NQA-1 Supplemental Requirements[a]	USNRC Regulatory Guide 4.15[a]
13.0	Nonconformances & Corrective Action	MW13.0	14, 15, 15S-1, 16	
14.0	Quality Assurance/ Quality Control Audits	MW14.0	10, 10S-1, 18, 18S-1	C.9
15.0	Quality Reports to Management	MW15.0	17	
16.0	Personnel Training & Qualifications	MW16.0	2, 2S-1, 2S-3, 2S-4	C.2

[a]These columns list the sections and subsections of each regulatory guide corresponding to the sections of the Quality Assurance Manual and the attachment describing the appropriate implementation of the guidelines.

MW2.0 ORGANIZATION

The Mixed Waste laboratory conforms to the laboratory organization specified in the ITAS Quality Assurance Manual, with the exceptions noted. In addition, current organizational charts are maintained in the quality/operations files.

MW2.1 ITAS Quality-Related Responsibilities Within The Laboratory

* Regional Director
  - Reports directly to the Vice President, IT Analytical Services.
  - Provides resources for implementation of the Quality Assurance program at each laboratory.
  - Periodically determines the effectiveness of the QA Programs at each laboratory.
  - Recommends changes in the QA Program to the ITAS Director of Quality Assurance and Compliance.
* Laboratory Director
  - Reports directly to the Southern Regional Director of Analytical Operations.
  - Implements the Quality Assurance Program within the laboratory.
  - Periodically determines the effectiveness of the Quality Assurance Program in the laboratory.
  - Approves Laboratory-Specific Attachments to the Quality Assurance Manual and Project-Specific Manuals (QAPPs), SOPs, and revisions.
  - Recommends changes in the Quality Assurance Program to the ITAS Director of Quality Assurance and Compliance.
  - Issues laboratory reports.

- Maintains current laboratory organization chart.

- Supervises laboratory participation in inter-laboratory accreditation and proficiency programs.

* Laboratory Manager

  - Manages daily laboratory analytical operations and supervises Quality Control activities performed as part of routine analytical operations.

  - Supervises the preparation and maintenance of laboratory records.

  - Oversees the log-in of all samples received, and supervises sample storage facilities.

  - Defines the instrument preventive maintenance schedule and oversees preventive maintenance program.

* Technical Director

  - Provides technical overview of laboratory activities and serves as an "in-house" consultant for the applicability of general Quality Control practices to specific needs.

  - Leads the training of analysts in laboratory operations and analytical procedures.

  - Evaluates analytical techniques, procedures, instrumentation and Quality Control procedures, and provides recommendations to the Laboratory Manager.

  - Supervises the verification of software for data processing.

  - Recommends standards for Purchasing instrumentation, equipment, reagents, gases, and chemicals.

  - Defines the calibration program within the laboratory.

* Quality Control Coordinator

  - Coordinates preparation of Quality Control standards, arranges insertion of Quality Control samples into the laboratory sample stream, and reviews the results.

  - Performs statistical analyses utilizing results of QC samples analyses to verify that the laboratory is meeting stated Quality Control goals.

  - Informs the Operations Manager and Group Leaders of data which lies outside of acceptable limits.

  - Serves as the "focal point" for the reporting and disposition of nonconformances within the laboratory and verifies corrective actions for all nonconformances.

  - Notifies the Technical Director and Laboratory Manager of out-of-control situations.

  - Reports unresolved nonconformances to the ITAS Director of Quality Assurance and Compliance.

  - Stops production of data in a laboratory area where the review of quality control data or procedures shows significant problems.

  - Assists in the performance of Quality Assurance audits and performs Quality Control audits and surveillances.

  - Closes findings and recommendations of Quality Assurance audits.

  - Establishes and supervises the laboratory Quality Assurance training program.

  - Maintains current distribution lists for Laboratory-Specific Attachments, Project-Specific Manuals, and Standard Operating Procedures.

  - Reviews a minimum of 5% of all laboratory data after analyst cross-checks; if analyst cross-checks have not been performed, reviews a minimum of 20% of all laboratory data.

* Group Supervisors

  - Ensure calibration and preventative maintenance are performed as required.

  - Perform data validation and final raw data reviews.

  - Report out-of-control situations to the QC Coordinator.

  - Review and create in-house data forms.

  - Verify technical accuracy of internal procedures and forms.

  - Perform analytical procedures and data recording in accordance with accepted procedures.

* Analysts

  - Perform analytical procedures and data recording in accordance with accepted methods.

  - Perform and document calibration and preventive maintenance of instrumentation, as appropriate.

  - Perform data processing and validation.

  - Immediately report out-of-control situations, instrument malfunction, calibration failure, or other nonconformances to the Group Leader and Quality Control Coordinator, as appropriate.

  - Prepare data summaries for review by the Laboratory Manager.

MW3.0 LABORATORY PRACTICE

The Mixed Waste laboratory practices concerning the flow of analytical samples are described in Table MW3-1. The table shows the steps taken and the party responsible for sample movement throughout the laboratory system.

This section serves as an introduction to these functions at the ITAS Oak Ridge-Mixed Waste Laboratory and provides an overview of them. Each function is discussed in detail in the sections which follow (Sections 4.0 through 16.0, as denoted in Figure MW3-1).

TABLE MW3-1

Steps of Sample Analysis Flow
ITAS-Mixed Waste Laboratory

Step #	Description	Performed by	Documents Used
1	Mixed waste samples arrive at ITAS ORL	Client & ORL sample receptionist.	Sample Chain-of-Custody, Request for Analysis.
2	Outer sample container screened for radiation.	Sample handlers and sample receptionist.	Results recorded in logbook & on ORL worksheets.
3	Outer sample container is unpacked, sample(s) inspected & paper-work verified for completeness.	Sample custodian. ORL.	Chain-of-Custody, Request for Analysis
4	Individual sample containers screened for alpha & beta radiation.	Sample handler and sample receptionist. ORL.	Results recorded on ORL worksheets.
5	Sample categorized based on results of Activity 4	Health Physicist or Designee	Sample Chain-of-Custody & ORL worksheets.
6	Sample and documentation are transferred to MWL, all paperwork is reviewed for completeness.	Sample custodians. ORL	Sample Chain-of-Custody, Request for Analysis, ORL worksheets.

TABLE MW3-1
(Continued)

Step #	Description	Performed by	Documents Used
7	A project record file is prepared, job number assigned, all sample identification placed with sample in temporary storage (if necessary).	Sample custodian MWL	Above plus the master sample log & receiving form.
8	Project information is recorded into manual log & billing information is recorded.	Lab management	Billing information
9	Sample is transferred to proper containers (if appropriate), labeled & transferred to temporary storage area for Group Leader determination of analytical requirements; QC samples may be introduced into sample stream.	Sample handlers & QC Coordinator	Lab project file sample labels. QC Sample Tracking
10	LIMS Computer System is updated with project information.	Sample handlers	Manual or computer data system records.
11	Copies of job information are distributed to analytical Group Leaders for processing.	Analytical Group Leaders.	Lab project file. Request for Analysis.

TABLE MW3-1
(Continued)

Step #	Description	Performed by	Documents Used
12	Analysts collect samples from storage & perform tests; additional QC samples may be introduced into sample stream.	Analysts QC Coordinator	Internal custody area forms, analyst log, instrument maintenance log, raw data sheets.
13	Analytical data is reviewed & transcribed onto data reporting sheets for typing; unacceptable data will require corrective action.	Analysts or Group Leaders.	Instrument calibration log, QC log, raw data package.
14	Analytical results to Group Leaders on completion of testing protocol; second data verification; unacceptable data will require corrective action.	Group Leaders.	Copies of data reporting sheet, analytical data, QA/QC results.
15	Computer is updated new information & paperwork is placed in project file.	Group Leaders.	Computer data system entry, paperwork to project file.
16	When job is complete, job is logged off of active computer job file and data is entered in project file.	Data Management	Data sheets.
17	>5% of Analytical data for report is reviewed.	Quality Control Coordinator.	Raw data summaries and checklists.

TABLE MW3-1
(Continued)

Step #	Description	Performed by	Documents Used
18	Analytical report is prepared.	Data management	Final report
19	Analytical data report reviewed & signed by Lab Manager.	Lab management.	Final Report.
20	Sample is retained for six weeks & then properly disposed of, or sent back to source.	Sample management	Request for hazardous chemical lab disposal.

MW4.0 MATERIAL PROCUREMENT AND CONTROL

The methods of procurement, receipt, storage, identification, and use of quality related purchased materials, and the methods of procurement document control, shall be documented in laboratory operating procedures. The procedures shall apply to the following categories of purchased materials: chemicals, reagents and solvents, analytical glassware, gas chromatography columns, analytical instruments and replacement parts, instrument gases, laboratory containers, sample preparation equipment, and standard reference materials procured for GC/MS, GC, or inorganic analysis calibrations.

The quality level for each purchased item shall be determined by its eventual use. The Standard Operating Procedure, analytical method or client contract describing an item's use shall be referenced prior to its procurement to determine the specified quality level. Quality related items shall be procured from suppliers with the appropriate quality documentation concerning each purchased item. Catalog numbers referencing the item description in the supplier's catalog provide sufficient quality documentation for commercial grade items. All purchase orders for quality related items will be initialed by the Quality Control Coordinator or designee prior to purchase and upon receipt.

Upon receipt of purchased materials, receiving personnel shall verify: that the items received are those specified on the procurement documents; that the quality level designated on the item labels or shipping documents does agree with those specified on the procurement documents; and, that no damage to the items has occurred during shipment. These documents are maintained in

purchasing files. Any nonconformances of purchased materials shall be documented with a nonconformance memo. (See Section MW13.0 for a discussion of nonconformance and corrective action procedures.)

Quality-related items shall be stored according to supplier, operating procedure, analytical method and/or client contractual requirements in order to best maintain the quality and integrity of the item. Each item shall be labeled with receipt dates such that older items are used first. Items of quality level below that required in the procedure shall not be used without written approval and explanation from Quality Assurance personnel.

The requirements for acceptance of reagents, solvents, and gases described in Section 4.1 of the ITAS Manual shall be followed with the following additions:

MW4.1.3 Organic Chemical Analysis

Pesticide-quality solvents: methylene chloride, hexane, acetone, methanol and ether used for sample extractions will be analyzed to determine the extent of impurities present in each. Any lot of solvents showing significant interferant peaks shall be eliminated from consideration for use by this laboratory. Acetone is the only exception since there are always at least three peaks present due to self-condensation products of acetone. Each lot of 1,1,2-trichlorotrifluorethane shall be checked for impurities prior to its use in total petroleum hydrocarbon or oil and grease analysis.

MW4.1.4 Water

Water used for the analysis of trace metals shall be purified by reverse osmosis/deionization to a resistivity of not less than 10 megaohm - centimeters. Water for organic determinations should be deionized and then further purified with activated carbon. Water for organic analysis also should be checked for methylene chloride throughout the day whenever purge and trap methods are used.

Water blanks shall be analyzed at least 5% of the time, or once per batch, whichever is more frequent.

MW5.0 SAMPLE RECEIPT AND INITIATION OF TESTING PROGRAM

MW5.1 Chain-of-Custody (Form)

As stated in the ITAS QA Manual, ITASMWL shall not accept samples collected by IT personnel for analysis without correctly prepared Chain-of-Custody and Request for Analysis forms.

MW5.2 Field Collection and Shipment

Prior to collection, the collection team must consider the analyses to be performed so that the sample containers and shipping containers can be assembled and the proper preservatives added.

MW5.3 Laboratory Receipt

MW5.3.1 Initial Laboratory Receipt

Samples received by the Mixed Waste laboratory are first screened for activity categorization. Written procedures detail screening activities and sample categories which may be transferred to ITASMWL. As soon as a shipment designated for mixed waste analysis arrives at ORL, the sample receptionist shall notify the ITASMWL QC Coordinator who will verify that the shipment is expected.

MW5.3.2 Receipt by ITAS-Mixed Waste

Upon completion of radiological screening, categorization, and corresponding paperwork, the custody of each sample shall be transferred to the ITASMWL QC Coordinator or designee for entry

into the mixed waste analysis stream. The initial Chain-of-Custody form shall be signed and shall remain with the samples during the transfer. If, however, based on radiological categorization, a group of samples must be divided for special treatment, a new Chain-of-Custody will be generated to designate the transfer of each sample.

Upon receipt of samples by ITASMWL, the following items shall be checked:

- The sample containers are checked for damage such as leaks, spills, broken glass, or unusual odors. Evidence of any damage is reported to laboratory management immediately.
- Sample identification on the sample containers is compared to that on the Chain-of-Custody. Discrepancies are documented on a nonconformance memo and reported to the Laboratory Manager and/or QC Coordinator for corrective action.
- The seals and tapes on sample containers for chemical analysis are intact.
- The number of individual sample containers received is equal to the number of samples listed on the Chain-of-Custody or any deviation is documented and traceable.
- The sample custodian shall determine if temperature requirements have been met.
- The sample custodian shall ensure that sufficient volume of sample for all analyses, QC samples, and potential retests were supplied.
- A nonconformance memo is completed and given to the QC Coordinator to document any nonconformances and the corresponding corrective action during sample receipt.

One sample or a set of samples grouped by a common receipt time and common field origin or client will be identified by giving each a project code composed of a unique, client-specific three or four character code (e.g., "EPAL" for USEPA, Las Vegas), followed by a five digit number, sequentially assigned. Individual samples will be assigned sequential four digit numbers, preceded by one or two character prefixes. Triplicate labels, using prenumbered tape, will be affixed to each sample, two of which may be transferred to subsequent aliquots for use in the laboratory.

Sample information shall be recorded in the Master Sample logbook and on the Sample Receiving form. Deadlines for extraction and analysis, in order to meet holding times and due dates, will be determined by the Group Supervisors. Samples will be placed in temporary laboratory storage and their location documented on the Receiving form.

MW5.4 Laboratory Storage of Samples

The laboratory stores current samples and past samples which may or may not be classified as "Hazardous". Hazardous samples will be stored in accordance with site services policy. The duration of sample storage will be six weeks after release of analytical results, unless otherwise obligated by contract or agreement.

MW5.4.1 Preanalysis Storage

Personnel from organic and inorganic sections shall be notified of samples received and logged. These personnel shall be responsible for picking up samples, specific to their group, from

the designated storage area when analysis is to be performed. Internal Chain-of-Custody forms shall be used when required by contract.

Methods of storage are intended generally to:

1. Retard biological action.
2. Retard hydrolysis of chemical compounds and complexes.
3. Reduce volatility of constituents.
4. Reduce absorption/adsorption effects.

Preservation methods are generally limited to pH control, chemical addition, refrigeration, and freezing.

MW5.4.2 Post-Analysis Storage

Anticipation of reanalysis requires proper environmental control for stored samples. If reanalysis is not anticipated, environmental conditions will not be observed, unless required by contract, and the samples are stored at room temperature. Due to limited current storage space at the Oak Ridge facility, samples will be returned to the client after analysis, if possible.

MW5.4.3 Sample Extract Storage

During analysis and prior to data reporting, analytical laboratory groups are responsible for proper storage of sample extracts based on the analysis requested. After release of the analytical report, GC/MS BNA and GC Pesticide/PCB extracts will be routinely stored at 4°C. Inorganic extracts will be maintained at room temperature unless specified by contract, or unless reanalysis is anticipated.

Sample extracts shall be stored for a minimum of six weeks after release of results unless otherwise obligated by contract or agreement.

MW5.5 Sample Disposal

The sample custodian will transfer samples from temporary storage to the archive storage area as analyses are completed. Storage time shall be six weeks after release of analytical results unless:

1. Specified otherwise by the customer,
2. Sample is involved in litigation, in which case it is stored until notified by the client.

MW6.0 CALIBRATION PRACTICES

This section of the ITASMWL attachment to the QA Manual prescribes the specific calibration practices used by the Mixed Waste Laboratory. Only those procedures that vary from, or are in addition to, those described in Section 6.0 of the ITAS QA Manual are described.

MW6.2 Operational Calibration

MW6.2.2 GC/MS Calibration Procedure

MW6.2.2.1 Tuning and GC/MS Mass Calibration

Each GC/MS system used for HSL compound analysis shall be tuned as described in the ITAS QA Manual using DFTPP or BFB with one exception: the ion abundance criteria for both volatile and semivolatile/pesticide analyses must be demonstrated every twelve (12) hours during sample analysis rather than daily.

MW6.2.2.2 Calibration of the GC/MS System

Internal Standard Calibration

A minimum of five (5) concentrations of internal standards shall be used for the initial calibration of volatile HSL compounds. For all but ten (10) of the semivolatile HSL compounds, the initial calibration shall be performed at five (5) concentrations. Nine of the ten compounds (the ten compound names are listed in the "Semivolatile and Pesticide HSL Compounds" subsection of the

QA Manual) shall use a four-point initial calibration while benzidine will use a three-point initial calibration at 80, 120, and 160 total nanograms.

Calibration Check Compound (CCC) Response

The initial calibration curves of both volatile and semivolatile HSL compounds shall be checked as described in the QA Manual (i.e., % RSD must be < 30%). See Table 6.5 of the QA Manual for the complete list of CCCs.

System Performance Check Compound (SPCC) Response

The system performance check shall be performed on the initial calibration and in continuing calibrations as described in the QA Manual with two exceptions: (1) In the performance check of the volatiles calibration, the minimum acceptable average (or daily) response factor for bromoform will be 0.250; (2) for semivolatile analysis, benzidine will not be included in the group of SPCCs.

Continuing Calibration

A daily standard containing a midrange concentration of all volatile or semivolatile HSL compounds and the required surrogates and internal standards shall be analyzed every 12 hours during sample analysis.

The daily RF values for each CCC will be compared to the average RF values from the initial calibration by calculating a percent difference (% D) using the following equation:

$$\% D = \frac{\text{daily RF} - \text{avg RF}}{\text{avg RF}} \times 100$$

The % D of each CCC must be less than 25 percent for the daily standard to be accepted prior to sample analysis. A system performance check shall be performed as described above, using the daily standard data. If the CCC and SPCC criteria are met, then RFs and % Ds will be calculated and reported for all compounds in the daily standard. If any of the CCC or SPCC criteria are not met, the system will be evaluated and corrective action shall be taken; corrective action may involve running a new initial calibration.

The daily calibration standard may be combined with the mass calibration standard (BFB or DFTPP) and the two calibrations performed simultaneously.

GC/MS Calibration Records

Data from each tuning and mass calibration, initial calibration, and continuing calibration will be recorded on calibration summary forms. The initial calibration form and raw calibration data will be filed in the calibration section of the project files. The tuning and mass calibration and continuing calibration data and summary forms shall be cross referenced to the initial calibrations by date and filed with each associated project in the project files as described in Section MW12.0.

MW6.2.3 Calibration of the Gas/Liquid Chromatograph

The gas chromatography laboratory will perform instrument calibration by two basic procedures depending on the nature of the project: project specific procedures and routine calibration procedures for batch work. The calibration procedures for a specific project's analyses may deviate from the described procedures if the contact specifies special requirements.

MW6.2.3.1 Project Specific Procedures

Initial Linearity

Each time a new column is installed into a specific gas chromatograph, or the chromatographic conditions are changed, i.e., change of flow rates, detectors, electronics, etc., response linearity shall be established for each GC analyte at these conditions. Three different concentrations of each parameter in a set of noninterfering mixed standard solutions will be analyzed over a 24 hour period. Calibration factors will be calculated for each pesticide and PCB. If the linearity for the calibration factors is $\leq$ 10%, the samples analyzed on that gas chromatograph can be directly quantitated from the standard calibration factors. If the linearity is $\geq$ 10%, a calibration curve shall be generated for each compound to be quantitated.

Retention Time Window Determination

Retention time windows shall be developed and used for the identification of sample extract components. After the establishment of linearity (or simultaneously using the same standards) a second 24 hour run will be performed with a minimum

of three (3) concentrations of each pesticide and PCB standard. Retention time windows will be developed from the three concentration standards of each component that give the greatest deviation of retention times. The range will be determined by calculating three (3) times the standard deviation of the three (3) retention times for the individual compounds, and applying it to the daily retention time for that same component.

Each standard, sample, and method blank will be spiked with a surrogate compound. Percent difference in retention time shift for the spiked surrogate will be calculated. A 2% difference in retention time over a 72 hour run is allowable.

Standard and QC Solutions

Component breakdown shall be monitored periodically by injecting a standard containing 4,4'-DDT and endrin and looking for their breakdown products: DDD, DDE for the former, and endrin aldehyde and endrin ketone for the latter. Percent breakdown shall not exceed 20 percent for either 4,4'-DDT or endrin or corrective action must be taken.

Calculations for percent breakdown are described in the method operating procedure.

To ensure continued linearity, a set of low, medium, and high concentration daily evaluation standards of a representative group of pesticides or PCBs shall be injected before any samples are injected. Calibration factors will be calculated for each

parameter; the % RSD must be ≤ 10% for the run to continue. After a specified number of samples, the mid-range evaluation mix and complete standard mixes will be injected to check daily retention times, component breakdown, and linearity for all PCBs and pesticides in question.

MW6.2.3.2 Routine Calibration Procedures

Initial Linearity

Standards shall be run at a minimum of three different concentrations. The resulting chromatograms should elicit all respective peaks on scale to allow for quantitation using either peak height or area. PCB standards may contain more than one Aroclor only if there are not interfering peaks between them.

A response factor (RF) will be calculated for each compound at each concentration. The percent relative standard deviation (RSD) of the RFs for each compound will be determined. The acceptable limits to establish linearity are: a maximum of 10% RSD, if the RSD is greater than 10% but less than 20%, standards must be graphed to establish linearity.

If standards are not within acceptable limits, the instrument should be checked, the source of the problem identified, and corrections shall be made. Standards (for those out of acceptable limits) will then be rerun.

Daily Standards

A daily quantitation standard shall be run for each analyte of interest. The percent difference between the daily response factor (RF_d) and the three-point standard curve mean response factor (RF_m) will be calculated as follows:

$$\% \text{ Difference} = \frac{RF_d - RF_m}{RF_m} \times 100$$

To retain linearity, the % Difference must be $\leq \pm$ 15%.

Sample quantitation will be based upon the daily RF when the daily linearity limit is attained and the RSD of the three-point standard set is less than 10%. However, sample calculations will be based on the graphed calibration curve when the initial linearity RSD is greater than 10% but less than 20 and daily linearity is retained.

If the daily quantitation standard does not meet acceptable limits, the instrument should be checked, the source of the gross deviation identified and corrections shall be made. A new three-point standard curve may be required depending on the action taken to correct the deviation. If changes are minor, another daily standard can be run. If the standard meets the acceptable limits, a new calibration curve will not be necessary. An additional standard check will be run every ten samples to assure GC conditions have not changed from the initial daily standard check. This standard will be compared to the respective daily quantitation standard for retention time, peak heights,

response factor, and any visible chromatographic abnormalities. If their is significant deviation in these characteristics, all samples analyzed after the last acceptable standard check and before the next acceptable standard check will be reanalyzed.

MW6.2.3.3 Calibration Records

The project specific linearity and retention time window data will be reported on GC standard summary sheets and are stored along with the chromatograms in the calibration section of the project file by instrument and date.

All three-point, nonproject specific calibration curves shall be filed in the calibration section of the project file. As a calibration curve is replaced by a new three point, it shall be marked as replaced, initialed, and dated in the top right corner and remain in the filing system.

The daily standards will be filed with respective projects and cross referenced with the applicable three-point chromatograms.

MW6.2.4 Atomic Absorption (AA) Spectrophotometer

The AA spectrophotometer shall be calibrated for the element of interest immediately prior to the analysis of a sample batch for that element. A calibration verification check standard containing the element of interest is analyzed at a frequency of 10% of a source different from that used in calibration, preferably from NIST, if available.

MW6.2.4.1 Calibration Standards

Stock standards shall be either prepared according to EPA methods from high purity metals, oxides or nonhygroscopic reagent grade salts or purchased as commercially available standard solutions. Working calibration standards will be prepared by diluting stock solutions to the appropriate concentration. Low concentration working standards, as used for graphite furnace AA, will be prepared at the time of analysis; other solutions will be prepared monthly or more frequently as needed unless otherwise specified by contract. To minimize matrix effects, the working standards will be prepared to closely match the type of acid or combination of acids and the concentration level in the samples following processing. Where the sample matrix is so complex that viscosity surface tension and components cannot be accurately matched with standards, the method of standard addition must be used. This technique relies on the addition of small, known amounts of the analysis element to portions of the sample - the absorbance difference between those and the original solution giving the slope of the calibration curve.

MW6.2.4.2 Calibration Procedures

The initial determination and continued verification of the baseline noise, sensitivity and linear working range of each element are the important instrument calibration and operation considerations. In operating an atomic absorption system, a number of variables must be held constant before the system is in statistical control. These variables include instrument warm-up, burner alignment, flame type, fuel, oxidant, lamp intensity, slit

width, wavelength, matrix effects, aspiration time and aspiration rate. The following shall be performed (with further detail to be found in the manufacturer's literature):

- Install the correct lamp, set the recommended current, wavelength and spectral bandpass.

- Light the burner, allow it to stabilize and adjust its position for maximum absorbance using a midscale standard.

- Prepare working standards in the recommended concentration range at a minimum of three levels bracketing the sample concentrations plus a standard blank.

- The sensitivity is the concentration of an element that will produce an absorbance of 0.0044.

  Example: Absorbance if 1 ug/ml = 0.060 then the sensitivity = $\frac{0.0044}{0.060} = 0.073$

- The AA system shall be warmed-up until the output is stable (usually about 30 minutes. The hollow cathode tube will be aligned to produce the maximum emitted light to the detector. In flameless AA, the inert gas flow inside the furnace must be optimized to provide maximum sensitivity.

- Aspirate the most concentrated calibration standard to be used and adjust the scale expansion to obtain full-scale output. Successively, aspirate the remaining standards (for a total of at least three) and the standard blank. Construct a calibration curve, from which the concentration of the samples is determined.

- The readout values obtained for the standard curve of each element should fall within a specified range. If readings are excessively low, the operator should check gas flow, burner or cell alignment, wavelength, slit width, photomultiplier voltage, and lamp intensity prior to analysis.

- Output from the instrument is to a suitable strip chart recorder or as a computer printout and provides hard copy of all raw data.

- Instrumental Detection Limits shall be determined by multiplying by 3, the average of the standard deviations obtained on 3 nonconsecutive days from the analysis of a standard solution (each analyte in reagent water) at a concentration of three to five times the expected instrumental detection limit with seven consecutive measurements per day. Each measurement must be performed as though it were a separate and distinct sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples).

- Any deviation from historical values indicates that some aspect of the instrument is malfunctioning or deteriorating (such as a lamp). Before proceeding, correction shall be made.

- For routine sample concentrations at levels greater than the highest standard of the analytical method, the sample should be diluted until the concentration falls within the calibration range.

- If large amounts of dissolved salts are present in the solution to be analyzed, the nebulizer will not handle the solution in the same manner as it will a solution with a small quantity of dissolved salts. Such an interference should be accounted for by adding a salt to the standards or by matching the density of the samples and standards or by use of the method of standard additions.

- Burner heads, nebulizers, quartz cells, and reduction flasks should be cleaned according to manufacturer instructions whenever excessive electronic noise is apparent or whenever indicated by visual inspection. Tygon tubing should be replaced when deterioration is apparent. Optical lenses should be cleaned on a periodic basis.

- Each sample digestate shall be analyzed as extracted and then with an addition of a known standard spike. The recovery value determined from the standard addition measurement is used to calculate the actual sample concentration from the calibration curve.

MW6.2.4.3 Calibration Records for AA

When three-point calibrations are performed daily for sample analysis, the standard raw data shall be copied and filed in the project files with all the corresponding sample data with the original being placed in the first file listed on the cover sheet. If calibration is performed for operational purposes or if the calibration curve is established for a longer period of time than one day, the raw data will be filed in all project files affected and the original being placed in the first file listed on the cover sheet. Check standard and calibration blank data will be filed with the sample data immediately prior to the standard or blank.

MW6.2.5 Inductively Coupled Argon Plasma (ICP) Spectrometer

The ICP spectrometer shall be calibrated daily for each element of interest. The ICP analyst shall follow the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects will be investigated and established for each individual analyte line on that particular instrument. All measurements must be within the instrument linear range where correction factors are valid. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain quality control data confirming instrument performance and analytical results.

MW6.2.5.1 Calibration Standards

Standard stock solutions shall be purchased or prepared from ultra high purity grade chemicals or metals. All salts will be dried for 1 hour at 105° unless otherwise specified. Mixed intermediate and working standards will be prepared by dilution of multiple stock standards such that the elements are compatible and stable. Fresh mixed standards will be prepared as needed; however, intermediate standards shall not be used for longer than three months and working standards not longer than thirty days. If available, a quality control sample obtained from an outside source will be used for the initial certification of the calibration standards. A fresh dilution of this sample shall be analyzed every week thereafter to monitor their stability. If the results are not within ± 5% of the true value listed for the control sample, a new calibration standard will be prepared and the instrument recalibrated. If this does not correct the problem, a new stock standard and a new calibration standard will be prepared and calibration is repeated.

MW6.2.5.2 Calibration Procedure

In addition to the instrument manufacturer's calibration requirements, the following steps are taken:

- The calibration blank shall be prepared at the same acid concentration as the calibration standards in a sufficient quantity to be used to flush the system between standards and samples.
- The calibration blank shall have a concentration of less than ± the contract required detection limit (CRDL) as specified by CLP protocol.

- The instrument check standard shall be prepared by combining compatible elements at a concentration equivalent to the midpoint of their respective calibration curves. It will be analyzed at a frequency of 10% to determine instrument drift.

MW6.2.5.3 Calibration Records for ICP

The ICP calibration data shall be maintained as described in Section MW6.2.4.3, "Calibration Records for AA".

MW6.2.6 Ultraviolet/Visible Light (UV/VIS) Spectrophotometry

MW6.2.6.1 Standard Calibration

The UV/VIS spectrophotometer shall be calibrated initially with a blank and a minimum of three standards containing the parameter of interest. One standard will be at or near the detection limit. The standards will be prepared to bracket the measured concentration of the samples. Calibration standards will be prepared according to the approved analytical procedure being followed for the parameter of interest. (Refer to Section MW9.0 for approved analytical methods.) Calibration verification standards shall be analyzed for every batch of samples analyzed or for every twenty samples, whichever is more frequent. The calibration standard must be within ± 15% of true value and should be midpoint on the calibration curve. If the midpoint calibration standard is not within ± 15% of the true value, then a new calibration curve must be generated. If the midpoint calibration standard is still out, then all samples associated with that midpoint calibration standard must be reprepared and reanalyzed.

MW6.2.6.2 Operational Considerations

Prior to initial use and periodically as needed, the spectrophotometer shall be checked for the following critical factors:

Wavelength accuracy - shall be checked using optical glass filters or chemical solutions which exhibit absorption spectra with strong, sharp peaks. Didymium glass has a strong absorption line at 585 nm. Nickel, cobalt and chromium solutions can also be used to check wavelength accuracy. Prepared solutions are also available for this purpose. Note that the absorption peaks may be shifted on instruments with wide band widths. If the wavelength accuracy checks do not meet specifications quoted for the instrument, it will be serviced. Wavelength accuracy shall be checked after installation or transportation of the instrument or if the exciter lamp has been disturbed or replaced.

Stray light - is defined as all undesired radiant energy reaching the detector. Stray light causes deviation from Beer's law. Stray light of 0.5 and 5.0 percent causes a deviation of 2 and 15 percent respectively, at an absorbance of one. To check for stray light problems, prepare a solution of 0.16g potassium chromate per liter in 0.05N potassium hydroxide. Set the instrument wavelength to 374nm and measure the transmittance of the solution; it should be less than 0.05 percent or within instrument specification. If it is greater, check for obvious leaks or have the instrument serviced.

Sample cell - The length and positioning of the sample cell can affect the absorbance measured. Do not assume that a group of cells have the same path length as their listing. A common

mistake is to develop a standard curve with one cell and then measure samples with another. Measure the absorbance of the cobalt solution described above at 510nm using all cells. All cells with absorbances that do not agree within 1 percent shall be removed from use. Cell windows must be clean, and the cells must be reproducibly placed in the spectrophotometer.

Photometric linearity - is measured by the adaptivity of absorbance values. Instrument linearity can be evaluated by measuring the absorbance of serial dilutions of chromium, cobalt and nickel solutions. Instrument linearity shall be checked after installation or transport and prior to use and again if the instrument fails to yield reliable data after the analytical method has been checked.

MW6.2.6.3 UV/VIS Calibration Records

UV/VIS spectrophotometer calibration data and graphs shall be maintained with the sample data in the project files if requested by contract or agreement.

MW6.2.7 pH Meter

Calibration will be performed daily or whenever sample analysis is required by using 2 commercial buffers that will bracket the expected pH, generally 4 and 7, and 7 and 10. A pH standard will then be read that is between the two used to standardize. This midrange pH will be recorded in a calibration logbook, signed and dated. The logbook shall be retained as a quality document.

MW6.2.8 Conductivity Meter

Calibration shall be performed using a 0.01N solution of KCl and water which has a conductivity of less than 1 umhos/cm. The solution will be equilibrated at 25°C, the electrode rinsed in the KCl solution, the conductivity measured, and recorded in the calibration logbook. The cell shall be replaced or new standards made if results are unacceptable. The calibration logbook shall be retained as a quality document.

MW6.3 Periodic Calibration

MW6.3.1 Balances

All balances shall be checked quarterly, at a minimum by Quality Assurance personnel, using NIST traceable Class S weights. Balances, in addition, shall be checked daily using three Class S weights covering the working range of the balances. If the checks are not acceptable (acceptance criteria shall be given in the operating procedure or the balance logbook), the balance will be removed from service and repaired as needed. Daily check results will be recorded in a bound logbook maintained near the instrument and retained as a quality document. Quarterly checks will also be recorded in bound logbooks and retained as quality documents by the Quality Control Coordinator.

MW6.3.2 Thermometers

Thermometers shall be directly traceable to NIST thermometers. All working thermometers shall be calibrated annually, at a minimum, against certified NIST traceable thermometers.

Each thermometer shall be labeled with its unique code and its corresponding calibration records shall be maintained as quality documents as described in Section MW12.0.

MW6.3.3 Pipettors

Automatic pipettors shall be gravimetrically calibrated before use and subsequently calibrated quarterly or more often as necessary. Calibration data worksheets for each pipettor shall be maintained in the Quality/Operations files. Each pipettor will be labeled with its unique code, calibration date, and the initials of the analyst performing the calibration.

MW6.4 Tag Out Procedure for Instruments

Instruments which continually fail to meet calibration requirements will be tagged as a nonconformance item. Corrective action will be or either permanently remove the instrument from operation or to have the instrument repaired. If an instrument is repaired, its reliability must be demonstrated through recalibration before the nonconformance can be closed.

MW6.5 Summary of ITASMWL Calibration Requirements

Requirements for ITASMWL instrument calibration are summarized on Table MW6-1. Section 9.0 contains other calibration result sheets that are combined with sample results.

TABLE MW6-1

Summary of Calibration Requirements
Mixed Waste Laboratory Operations

Instrument to be Calibrated	Standard Reference	Calibrations Technique	Acceptable Performance Specifications
Atomic Absorption Spectro-photometer/ Graphite Furnace	At least 3 levels, bracketing the sample concentration plus 1 blank. Certified standards from chemical supply are used.	Direct reading using serial dilution of commercial standard, which are checked using NIST SRM's when a new bottle is opened.	Specifications are to achieve theoretical sensitivity as specified by the manufacturer.
Analytical Balances	Class "S" weight check.	Daily checks at 3 masses out of house service to calibrate every 3 months or as needed.	Depends on precision required by analytical method.
Conductivity Meter	In house KCl Solution.	N/A	If unacceptable results, either clean the cell or replace it.
(GC) Gas Chromato-graphy	All standards referenced to EPA materials; 3 levels + blank	Establish linearity & retention time Windows	± 10% deviation of RFs 2% deviation of RTs (See Section MW6.2.3)
(GC/MS) Gas Chromato-graphy Mass Spectrometry	All in house solutions of EPA HSL compounds and DFTPP & BFB trace-able to EPA materials.	Initial 5-point, % D < 25% for check RSD of CCCs & minimum RFs of SPCCs; daily std mass calibration must meet prior to sample analysis.	daily std RFs; of SPCC 0.3 & 0.25.

TABLE MW6-1
(Continued)

Instrument to be Calibrated	Standard Reference	Calibrations Technique	Acceptable Performance Specifications
Inductively Coupled Plasma Spectro-photometer	Certified standards from chemical supply house, checked against NIST SRM's.	Serial dilutions of commercial standards. Direct readout.	Readout should comply with theoretical specifications by manufacturer.
pH Meter	Commercial Buffers	Bracket technique.	90% of slope.
UV/VIS Spectro-photometer	Three levels of in house standards + blank.	Standard Curve.	15% of original curve.
Thermometers	NIST thermometer	Compare measurements at 3 different temps	Depends on use of thermometer, max. deviation: ± 2°C.
Pipettors	Weight/calculated volume of pure water.	Gravimetric method - repetitive weighings of water.	Low vol.: 2% High vol.: 1%

MW7.0 PREVENTIVE MAINTENANCE

Two types of preventive maintenance logbooks shall be maintained for each instrument. Routine preventive maintenance activities (performed daily, weekly, or quarterly) will be documented in a "check-off" form logbook. The routine maintenance standard procedure will be bound with blank copies of check-off forms. When the routine maintenance for an instrument has been performed, the performing analyst shall initial the appropriately dated box.

Major/minor preventive maintenance activities will be described along with instrument repairs in individual instrument logbooks. The logbooks will be bound and labeled with the instrument name and kept beside the instrument while in use. If an instrument is inoperable or performing outside of the required specifications, it will be tagged with its operational status to prevent inadvertent use while corrective action is taken.

Table MW7-1 summarizes the preventive maintenance requirements for ITASMWL. Instrument identification, items to be checked or serviced and the frequency in which each operation shall be performed are listed.

When maintenance logbooks and check forms are no longer in active use, they shall be retained in the laboratory operations file by instrument and date. Table MW7-2 presents a list of instruments which shall have assigned maintenance logbooks and the type of logbook assigned.

TABLE MW7-1

Preventive Maintenance Requirement
Mixed Waste Laboratory Operations

Instrument	Items Checked/Serviced	Minimum Frequency
Gas Chromatography	Replace column packing, clean detector, change glass wool plug, clean insert, replace septa, gas purity checks.	Determined by analyst so that the calibration is within required specifications.
Atomic Absorption Spectrophotometer	Burner head, Nebulizer Tygon Tubing	Daily Monthly
Analytical Balance	Internal weight, train, gears, electronics.	Quarterly
Inductively Coupled Plasma Spectro-photometer	Sample introduction system pumps.	Daily Weekly
GC/MS	GC/MS maintenance is the same as GC with the following additions:	
	1) For model 4000 & 4500	
	DP Oil	Bi-Weekly
	Mech Oil	Quarterly
	Power Con. Air Filter	Bi-Weekly
	QEM Filter	Bi-Weekly
	Water Bay Filter	Bi-Weekly
	2010 Interface Box	Bi-Weekly
	Vacuum Chaff Filter	Monthly
	2) For OWA's	
	Water Filter (if applicable)	Observe & change as needed
	Computer Air Filter	Monthly
	Card Cage Air Filter	Monthly
UV/VIS Spectro-photometer	Lamp, Wavelength checked, Serviced	As needed During calibration steps, As needed.

TABLE MW7-2

Instrument and Equipment Maintenance Logbooks
Mixed Waste Laboratory

	Instrument	Type of Logbook: Preventative	Type of Logbook: Major/Minor
1.	Gas Chromatographs	X	X
2.	GC/MS	X	X
3.	AA Spectrophotometer	X	X
4.	ICP Spectrophotometer	X	X
5.	UV/Vis Spec.		X
6.	pH Meter	Combined Log	
7.	Analytical Balances	X	
8.	Refrigerators (temperature check log)	X	As necessary
9.	Freezers (temperature check log)	X	As necessary
10.	Oven (temperature check log)	X	As necessary
11.	Water Purification System	X	As necessary

MW8.0 ANALYSIS OF QUALITY CONTROL SAMPLES

Section 8.0 of the ITAS QA Manual discusses samples which are routinely added to the normal laboratory sample stream to demonstrate that the laboratory is operating within prescribed requirements for accuracy and precision.

ITASMWL shall follow the ITAS QA sample program summarized in Table 8.1 of the manual with the amendments described below.

MW8.1 Types of Quality Control Samples

MW8.1.1 Reagent Blank Analyses

In addition to the procedure described in the ITAS QA Manual, an aliquot of each new reagent lot used for one analysis shall be tested for the parameters of interest. Only after a reagent lot is shown to be free of contamination (less than the required detection limit for any parameter) shall it be used for analysis.

MW8.1.2 Method Blank Analyses

For volatile organic analysis by GC/MS, a method blank shall be comprised of organic-free, deionized water spiked with three (3) internal standard compounds and three (3) surrogate compounds. One method blank will be run at least once every 12 hours.

A method blank for the analysis of base, neutral, and acid extractable compounds by GC/MS shall involve the addition of six (6) surrogate compounds to deionized water followed by the appropriate extraction, then the addition of six (6) internal standard compounds prior to analysis. One method blank shall be

prepared with each group of samples extracted together. These two types of method blanks are used to determine cumulative interferences as well as instrument performance and stability.

After an acceptable method blank is run, sample analysis may begin with no question of laboratory contaminants harming the integrity of the samples.

MW8.1.3 Matrix Spike Duplicate Pair Analyses

For inorganic and organic analyses, the analysis of a duplicate pair of matrix spikes may replace the analysis of a duplicate sample and a matrix spike sample as described in the ITAS QA Manual. One matrix spike and matrix spike duplicate shall be analyzed for every 20 samples per project, per matrix type, per concentration level, except where regulatory protocol dictates spike and duplicate. The results of the duplicate spike analyses will be used to determine both accuracy and precision. Percent recoveries on both spikes will be calculated.

MW8.1.4 Check Standard Analyses

Midpoint check standards shall be analyzed at a minimum frequency of one per twenty (20) inorganic and GC sample analyses. Continuing calibration standards shall be analyzed every twelve hours during GC/MS sample analysis. (See Section MW6.2.2).

MW8.1.5 Surrogate Standard Analyses

Surrogate standard determinations may also be performed on all samples and blanks for GC and GC/MS analyses when specified by contract. The GC surrogate recovery data will be summarized with the GC/MS surrogate recovery data of a particular project on the appropriate QC summary forms. The GC surrogate compound will also be used to evaluate retention time shift.

MW9.0 ANALYTICAL PROCEDURES

The methods currently adopted for the chemical analysis of mixed waste samples at ITASMWL are presented in Tables 9.1, 9.2, and 9.3 in the ITAS QA Manual. Copies of references cited shall be available in the laboratory. Methods not listed but approved by EPA and the latest revision of the ITAS QA Manual may be adopted for use upon client request.

MW9.1 Variance From Stated Analytical Methods

If used routinely, adaptations of methodology in tables cited above, shall be documented in Standard Operating Procedures (SOP) which shall be maintained in the laboratory. Each analytical SOP shall contain the points delineated in the QA Manual for inclusion in an analytical procedure. For project specific deviations to the cited methodology, the actual method used shall be documented in the project records.

MW9.2 Analytical Data Sheets

Prepared data sheets for recording raw data shall be formatted to meet the documentation requirements described in Section 9.0 of the QA Manual.

MW10.0 DATA VERIFICATION

ITASMWL shall follow the recommendations for verification of reported results discussed in Section 10.0 of the ITAS QA Manual with the following exceptions or additions.

MW10.1 Processing of Quality Control Data

As a part of the analytical quality control program, ITASMW shall determine precision and accuracy for each type of analysis (when applicable) by calculating RPD and percent recovery values as described in the QA Manual. These statistics will be tabulated by parameter each month. When sufficient new data exist, updated warning and control limits for each parameter will be calculated based on the latest statistical summaries of QC results. For analytical methods covered under CLP protocol, established limits will be utilized for flagging, but data falling outside CLP control limits will still be utilized to generate control charts (per EPA Manual 600/4-79-019). That is, data will be flagged in data packages, but results will still be utilized in laboratory-wide control charts.

MW10.2 Data Validation

MW10.2.1 General Data Validation Process

Figure MW10-1 depicts the data validation process performed at ITASMWL. A brief discussion of each step in the validation process is given.

1. Data Processing - The first step will include manual computations on data sheets, acquisition and processing of raw data by computer and may be performed by one or more analysts. Processed data for one project shall be compiled and transferred to the reviewer for Step Two.

2. Review of Data Processing - This step shall be performed as described in Section 10.2.1 ("original check") of the QA Manual on a minimum of 20 percent of all data. The checker must sign and date each document reviewed. All entries and calculations that the checker reviews shall be marked in black ink with a check mark. If errors are found in the data package being reviewed, they shall be returned to analyst for correction.

3. Summary of Results - After data review, each Group Supervisor shall tabulate the results of analyses performed in his/her action (i.e., GC, GC/MS, Metals). After tabulation, the Group Supervisor shall check the work for transcription errors.

4. Draft Report and Checking - The Laboratory Manager or designee shall check each results package (contains results summaries from all lab sections for one group of samples from a client) against the Request for Analysis to ensure appropriateness and completeness of analyses. He/she shall also check for the appropriateness and completeness of analyses. He/she shall also check for the appropriateness of: detection limits reported, report format, significant digits, and for transcription errors, and obvious calculation errors. The related QC data will be checked to ensure all statistics are within the designated control limits. After checking, a draft final report will be written, signed (full signature), and dated, ready for typing.

5. 5% Data Review - No less than five percent of all analytical data shall be reviewed by the QC Coordinator. This review will ensure that the quality control procedures at each step in the analytical process are being performed and documented as described in the ITAS QA Manual and this attachment. He/she will take corrective action and proceed further with an investigation if protocol has not been followed.

6. Final Report - The final report will be typed and proofed against the handwritten draft report by word processing personnel. Proofing shall be performed by a person independent of the report typist.

7. Report Approval and Issuance - The Laboratory Manager is ultimately responsible for assuring that the report going to the client is accurate according to the data provided. He/she shall give final approval for release of the report by signing and dating it for issuance. The Lab Manager has the authority to designate specified personnel to manage this responsibility.

MW10.2.2 Data Validation for Specific Analyses

Analytical data may be generated by the GC/MS computer software; the GC computer; the ICAP computer; AA; and other laboratory instrumentation. Outputs will be in graphic form (chromatograms) bar graph (spectra), and printed tabular form, and in standard formats specified for each analysis. The validity of instrument generated data shall be supported by the maintenance of the following records:

- Description of the calibration performed;

- Description of routine instrument checks (noise levels, drift, linearity, etc.);

- Documentation of the traceability of instrument standards, samples and data;

- Documentation of analytical methodology of instrument standards, samples and data;

- Description of the controls taken to determine and minimize interference contaminants in analytical methods (use of reference blanks and check standards for method accuracy and precision);

- Description of routine maintenance performed, and

- Documentation of sample preservation and transport when shipped elsewhere.

Specific checks will be initiated also to ensure the accuracy of instrument compound identification in analytical samples.

MW10.2.2.1 GC/MS Compound Identification Criteria

For positive identification, all ions present in the standard mass spectrum of a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum. The relative intensities of ions specified must agree within plus or minus 20% between the standard and sample spectra.

Ions greater than 10% in the sample spectrum, but not present in the standard spectrum, must be considered and accounted for by the analyst making the comparison. When GC/MS computed data processing programs are used to obtain the sample component spectrum, (i.e., spectrum enhancement) both the process and raw spectrum must be evaluated for compound identification.

MW10.2.2.2 GC Compound Identification Criteria

In gas chromatography, compounds are identified on the basis of retention times. Because component retention time reproducibility is rarely exact, retention time windows shall be developed and used for identification. (Refer to Section MW6.2.3.1.) The sample component's retention time must fall within the standard retention time window for tentative

identification to be established. The sample extract shall be then analyzed on a second GC column for confirmation. For a positive result to be reported, a peak must also elute within the retention time window of the tentatively identified component on the confirmation column. Quantitation shall be performed on the column with the best calibration performance.

MW10.3 Verification of Software

All computer software used to acquire, process, or report data at ITASMWL shall be verified upon initial use and reverified after any modification. Depending upon the purpose of the software, either test problems or reference standards will be processed by the software and the results compared to the true results (hand calculated) or certificates for the reference materials.

All paperwork generated during software verification such as computer printouts and supporting calculations shall be documented with the following information and filed in the laboratory operations file as discussed in Section MW12.0:

- Software name
- Instrument or analysis associated with software
- Date of verification
- Signature of verifier

TABLE MW10-1

Data Validation Process

Activity	Responsibility
Data Processing	Analyst
Checking > 20% of Data Processing	Independent Analyst or Group Leader
Results Summary for Each Group	Group Leader
Compiling and Checking Draft Report/Sign-off of Draft Report	Laboratory Manager or designee
> 5% Data Review	QC Coordinator
Report - Typing and Proofing	Word Processing Personnel
Final Report Approval for Issue, Sign-off	Laboratory Manager or designated personnel

MW11.0 DATA REPORTS

MW11.1 Format and Content of Data Reports

The content of the data report will vary with client needs and specifications. The format, however, will conform to the following specifications at ITASMWL:

* The final data presentation shall be checked in accordance with the data verification requirements outlined in Section MW10.0 and will be approved by the Laboratory Manager or designee.
* Data will be presented in a tabular format whenever possible.
* Data will be formatted as a Certificate of Analysis.
* Each page of data shall be identified with the project number (Job Number) and name, date of issue, and page number.
* Data presentation shall include, at a minimum, client and laboratory identification, sample descriptions, preparation dates, dates of analysis, and associated blank data, if applicable.
* The narrative portion of the Certificate shall include, at a minimum, the following sections:

  Introduction
  Analytical Results/Methodology
  Sample Summary Table

  Other sections may be added at the discretion of the client or Laboratory Manager.

MW11.2 Corrections to Data Reports

Corrections to data reports may be initiated either internally or externally. The course of action to be followed will depend on

whether or not the erroneous data was found externally or internally. Before any corrective action can be taken, however, the suspect data must be determined to be erroneous. The suspect Certificate will be compared against all pertinent project records necessary to judge the adequacy of the Certificate. If no erroneous information can be found then no corrective action is mandated.

MW11.2.1 External Corrections

If a Data Report is found by the client to include erroneous information or data, corrective action shall include:

* Marking the affected data or information and making any necessary corrections in red pen. Corrections will include initials of the person making the corrections and the date of the corrections.
* Generating a nonconformance describing the affected data or information and corrections to be made.
* Reprocessing the Certificate with flagged data corrected.
* Review of the reprocessed Certificate to ensure that corrections have been made properly.
* Reissuance of the Certificate of Analysis stipulating that a correction has been made to the original report.

When a Certificate of Analysis has been found by the client to contain erroneous information, both the original and the corrected Certificate of Analysis will be maintained in the Project File.

MW11.2.2 Internal Corrections

If a Data Report is found by laboratory personnel to include erroneous information or data, and the report has not yet been issued, corrective action shall include:

* Marking the affected data or information and making any necessary corrections in red pen. Corrections will include the initials of the person making the corrections and the date of the corrections.
* Reprocessing the Certificate with flagged data corrected.
* Review of the reprocessed Certificate to ensure that corrections have been made properly.
* Destruction of the erroneous report to preclude accidental submittal to the client.

If a data report is found by laboratory personnel to include erroneous information or data, and the report has already been issued to the client, corrective action shall be as in Section MW11.2.1. In addition, the client shall be notified that the report contains erroneous information. Both the original and corrected Certificate of Analysis shall be maintained in the Project File.

MW12.0 RECORDS MANAGEMENT

The following is a description of the records system used within ITASMWL. If it is necessary for a specific field project to deviate from this system, the laboratory shall maintain a description of the project-specific system within the project file.

MW12.1 Classification

As stated in the ITAS QA Manual, two broad categories exist for laboratory records:

Project records - Documents which are specific to a project or customer.

Quality/Operations Records - Documents which demonstrate overall laboratory performance.

ITASMWL records are maintained in accordance with the ITAS QA Manual with the exceptions and additions described in the sections below.

For the purpose of determining record retention requirements, records are further classified as "Permanent" ("Lifetime") or "Nonpermanent". Further discussion of these classifications is provided in Section 12.5 on Record Retention.

MW12.2 Project Records

For this description, it is necessary to distinguish a "laboratory project" from a "field/client project". A laboratory

project shall be defined as a group of samples received by the laboratory from one client on the same date and time. As described in Section 5.0, this group of samples shall be coded into the analytical system under a unique three or four-letter, five digit "project" code. This number shall be written on all records and data associated with the sample group. A "field/client project", on the other hand, will generally refer to work associated with one client over a longer period of time under one contract. The field/client project may involve work by groups other than IT Analytical Services, such as IT Engineering Services or IT Remediation. A field/client project will be identified by a project name and job number used by all IT groups associated with the contracted work. Within ITASMWL, one field/client project may include multiple laboratory projects. The cross-link between a field/client project and its associated laboratory projects will be the ITAS job number coded into the computer system with every project entry.

ITASMWL shall use the following indexed categories within a project file:

A-Correspondence

This category includes any letters, notes memos, phone dialogues, purchase orders, quotes, or other appropriate communications with the client or within the laboratory that have bearing upon the samples covered by that project code.

B-Tracking Records

Tracking records include chains-of-custody, requests for analysis forms, sample receipt and coding checklists, screening results,

field records, air bills, and other associated paperwork. Internal chains-of-custody and sample receiving forms shall also be included in this category.

C-Calibration/Analytical Data - Organics

This section includes raw data from GC, GCMS, and organic prep to provide documentation of the conditions under which the data were acquired.

D-Calibration/Analytical Data - Inorganics

This section includes raw data from metals, wet chemistry, and inorganic prep to provide documentation of the conditions under which the data were acquired.

E-Data Reports - Electronic Data Transfer

This section includes results sheets from the various groups, QC information necessary to complete a project, Certificates-of-Analysis, and electronic data transfer records, where applicable.

F-Nonconformances

Included are all original nonconformance memos originated in each group for that project code with corrective action and verification of corrective action indicated.

G-QAPP Summary

This section is applicable to those clients whose projects require a summary of the QA project plan submitted by the QA

Coordinator and, if applicable, specific methods and/or requirements submitted by the client for use on his samples.

MW12.3 Quality and Operations Records

ITASMWL shall follow the operations filing system described in the ITAS QA Manual with the following clarifications and additions. Table MW12.2 presents the category list for the quality/operations files. General laboratory quality/operations records will be filed separately from project records and maintained so they can be referenced to project specific records. All quality/operations records shall be legible, appropriately filled-out, and adequately identified to the item involved. The Quality Control Coordinator shall be responsible for verifying the implementation of the Quality/ Operations Records System. The following is a description of those categories not included or different from those detailed in the ITAS QA Manual.

A-Personnel Qualifications and Training

Records documenting the qualifications and training of all laboratory personnel shall be maintained as quality documents. This file will include personnel qualification records, results of qualification sample analysis, completed quality assurance examinations, professional certification documents, attendance records of internal training sessions, letters of qualification, and other training records as appropriate, as described in Section MW16.0.

B-Job Descriptions

This file contains a job description for each position at the Mixed-Waste Laboratory.

C-Master Resume File

This file contains a current resume for each active employee at ITASMWL.

D-Organizational Charts

Current organizational charts for the Mixed-Waste Laboratory, the ITAS-Oak Ridge Laboratory, and the Southern Regional Operations are maintained.

E-Inactive Employee File

All training and personnel records of past and current employees are retained in this category.

F-Computer Software Validation

Included in this file is software validation for instrumentation, hand-held calculations, and spreadsheets.

N-QA/QC Data

Results of blind performance evaluation samples in all lab areas and QA/QC samples and control chart information are kept in this file.

V-Miscellaneous Quality Documents

This category allows for the maintenance of quality documents not related to previous categories and not requiring a separate new category. The category index can be reference for an up-to-date listing of the category contents.

MW12.3.1 Quality Document Categorization

Records that furnish documentary evidence of quality in the overall performance and operation of ITASMWL shall be considered quality documents. Project-specific records will be considered quality documents only if the client contract specifies them as such.

MW12.4 Record Control

MW12.4.1 Project Record Handling and Control

Laboratory-generated project records will be delivered to the records room for categorization and filing. The project records custodian (the QCC or designee) shall:

* Write the category codes and file description of each group of records on the category index lists.
* Prepare a file folder for each code number. The file folder tab shall be documented with the category letter-number code, the file description and the associated laboratory project number if the category is subdivided by laboratory project.
* Place the files in the appropriate storage location.

To maintain control of project records, sign-out sheets shall be placed in the location from which a project record is removed. The sign-out sheet shall indicate:

* Project file number which was borrowed
* Date and person borrowing the record
* Date returned to the record system

When the project record is returned, the sign-out sheet will be removed from the file cabinet and kept for reuse.

Active files shall be maintained in locking metal file cabinets located in the office area of the laboratory. Only authorized personnel shall have access to the files. Authorized personnel include, but are not limited to, QA personnel, Laboratory Director, Laboratory Manager, Group leaders, Project Managers, and coding personnel. Other personnel may be authorized on a case-by-case basis. The file drawers shall be locked when not in use. Smoking, eating, and the use of chemicals are prohibited in the records retention area.

Project file documents may be designated as quality records by the client or the Quality Control Coordinator. In such cases, the Project files shall be stored as in Section MW12.4.2. They shall be maintained in two-hour rated Class B file containers meeting the requirements of NFPA 232-1975, or they shall be duplicated with one copy stored in the ITASMWL records room and one copy stored at a remote location.

It shall be the responsibility of the Quality Control Coordinator to oversee and implement the procedures for project files maintenance.

MW12.4.2 Quality Operations Record Handling and Control

Quality and operations records that are in daily use, such as the master sample logbook and current instrument logbooks shall be assigned a file index code which is recorded on the logbook and entered into the index. The logbooks shall be stored at the location of use until completed, at which time they shall be placed in the files for retention.

Active files shall be maintained in two-hour Class B file containers meeting the requirements of NFPA 232-1975 located in the Quality Assurance office or in dual files at remote locations. The file drawers will be kept locked when not in use.

Only authorized laboratory personnel shall have access to the files. Authorized personnel include Quality Assurance personnel and laboratory management.

File sign-out sheets shall be maintained to control removal and return of records. The sign-out sheets will indicate the file index code and abbreviated file description, date, person borrowing the record, and the date returned.

The ITASMWL Quality Control Coordinator is responsible for verifying the implementation of the Quality/Operations Record System.

MW12.5 Records Retention

MW12.5.1 Project Retention

Project records shall be classified as Permanent or Nonpermanent by the Quality Control Coordinator. Permanent records shall be maintained by ITASMWL for at least as long as the life of the project or nuclear facility plus ten years, or they may be transferred to the owner of the facility.

Nonpermanent records shall be maintained for the following time periods after completion of the project:

* Analysis performed as part of site mitigation activities - 10 years.
* Records associated with facilities governed by the Resource Conservation and Recovery Act (RCRA) - 5 years after closure, if the analysis was performed prior to closure or for the 30 year monitoring period following closure, if the analysis was performed for the purpose of closure monitoring.
* Conventional analysis - 7 years.

MW12.5.2 Quality/Operations Records Retention

Quality/Operations files will be maintained for retrievability during active use. Once per year the Quality/Operations files will be reviewed to determine those records that are no longer required for current operations and quality assurance documentation. These records will be transferred to permanent storage. The storage date of each record shall be documented on the appropriate index. A separate index shall be generated indicating the storage date and location. A copy of this index

is maintained in the active index file and the original is placed in an index file at the storage location. Quality/Operations records shall be maintained as long as the longest retained project-specific records associated with them, generally classified as Permanent.

Records shall be maintained for permanent storage in facilities which provide a suitable environment to minimize deterioration or damage, prevent loss, and facilitate retrieval without undue delay. The facilities shall meet the requirements of NQA-1 Supplement 17S-1, "Supplementary Requirements for Quality Assurance Records".

MW12.5.3 Permanent Records

The following is a list of "Permanent" records and shall be retained for the life of the project or nuclear facility plus 10 years.

* Laboratory procedures and revisions to procedures
* Laboratory calculation sheets
* Raw data generated by instrumentation including calibration and analytical data
* Instrument logbooks
* Sample logbooks
* Calibration certificates and reference certificates
* Disposal records
* Results of analysis of standards, blanks, spikes, QC samples, and internal cross-checks

* Nonconformance logbook and project specific nonconformances
* Training records
* Audits/Surveillance reports and responses

MW12.5.4 Nonpermanent Records

The following is a list of records classified as "Nonpermanent" and while they shall be retained for a minimum of seven years, shall not be retained for the life of a project or nuclear facility.

* Audit plans and logs
* Surveillance plans and logs

Prior to record destruction, the QC Coordinator shall review records to determine if the retention time should be extended.

TABLE MW12-1

Project Records
Filing Categories

Category	Record Description
-	Project Index
A	Correspondence
B	Tracking Records/ Screening Records
C	Organic Analytical Data
D	Inorganic Analytical Data
E	Data Reports/Electronic Data Transfer Records
F	Nonconformance
G	QAPP/Project Specific Requirements

TABLE MW12-2

Laboratory Quality/Operations Records
Filing Categories

Category	Record Description
-	Index File
A	Personnel Training & Qualifications
B	Job Descriptions
C	Master Resume File
D	Organizational Charts
E	Inactive Employee File
F	Computer Software Validation
G	Certificates
H	Certifications
I	Equipment Maintenance & Cal.
J	Master Sample Logbook
K	Blind PE Sample Logbook
L	Nonconformance Logbook
M	Instrument Calibration Data
N	QA/QC Data
O	Nonconformances: Monthly Breakdown
P	Audit Information

TABLE 12-2
(Continued)

Category	Record Description
Q	SOP File
R	QA Manual File
S	Procedures and References
T	QCC Reports
U	Subcontractor Records
V	Miscellaneous

MW13.0 NONCONFORMANCES AND CORRECTIVE ACTION

All conditions identified as being adverse to quality or rendering quality indeterminate will require corrective action. Identification of suspected adverse conditions can be made by lab employees in the course of their work (internal) or by individuals outside ITASMWL (external) through the process of review, surveillance, audit, or proficiency testing.

A summary of the procedures that shall be established to identify, document, and correct nonconformances within ITASMWL follows; the details of implementation shall be documented in laboratory operating procedures.

MW13.1 Identification and Documentation of Nonconformances

MW13.1.1 Internal

Any lab employee noticing a deficiency suspected of being a nonconformance shall report the deficiency to the responsible supervisor and to the Quality Control Coordinator on a nonconformance memo. The nonconformance memo shall be signed and dated by the generating employee and then routed to the QC Coordinator and Laboratory Director.

The responsible supervisor and the QC Coordinator shall determine whether a deficiency will affect the quality of analytical data, thus becoming a nonconformance. The QC Coordinator is responsible for maintaining a log of nonconformances and dates of corrective action and verification for each.

MW13.1.2 External

A laboratory nonconformance may be identified by an external organization through quality assurance functions such as an audit, surveillance, or proficiency testing. The report generated by the external QA function shall serve as documentation of the nonconformance. The QC Coordinator may generate a nonconformance memo for specific items cited for ease of circulation and for documentation of corrective action. Externally identified deficiencies recognized as laboratory nonconformances shall be recorded in the QC Coordinator's nonconformance log for tracking and follow up actions.

MW13.2 Corrective Action

MW13.2.1 Corrective Action

Corrective action shall include proposed corrective action, underlying root cause, and actions to prevent recurrence.

MW13.2.2 Follow-Up

The corrective action taken shall be documented on the nonconformance memo. The person performing the corrective action shall sign and date the nonconformance memo. Laboratory Management is responsible for ensuring that corrective is taken and completed. Verification of corrective action is the responsibility of the QC Coordinator.

MW13.3 Nonconformance Close-Out

The QC Coordinator shall record the dates of corrective action and verification in the nonconformance log. The original completed nonconformance memo shall be filed in Section F of a project file or with procurement documents in the purchasing files. The reference file index location code shall be recorded in the nonconformance log for ease of tracking.

MW14.0 QUALITY ASSURANCE/CONTROL AUDITS

The ITAS Oak Ridge-Mixed Waste Laboratory audit program shall consist of monthly surveillances and two types of audits, each performed biannually, resulting in quarterly audits.

* System audits shall be performed by the ITAS Quality Assurance staff and reported to the Vice President, Analytical Services.
* Performance audits shall be performed by the laboratory Quality Control Coordinator and reported to the Laboratory Manager/ Technical Director, the Laboratory Director, and the ITAS Director, Quality Assurance and Compliance.
* Surveillances shall be performed on a monthly basis (months not scheduled with an audit) within the laboratory by the Quality Control Coordinator and reported to the Laboratory Manager.

The content and conduct of the audits and surveillances are discussed below.

MW14.1 Performance and System Audits

The contents of performance and system audits may differ as described in the ITAS QA Manual; however, the conduct and documentation requirements of both types shall follow the same general guidelines.

MW14.1.1 Auditor Independence

The audit shall be performed by personnel who are independent of any direct responsibility for performance of the activities being

audited. Audit personnel shall have sufficient authority and freedom to make the audit process meaningful and effective.

MW14.1.2 Scheduling

The Quality Control Coordinator and Laboratory Director shall prepare a master schedule reflecting all audits to be performed in a calendar year. The schedule shall include the biannual systems audits previously scheduled by the Quality Assurance staff, the biannual performance audits on alternate quarters, and the monthly surveillances performed on months not scheduled with an audit. The schedule shall be used as a guide and may be revised and updated as the year progresses.

MW14.1.3 Audit Preparation

The audit team, designated by the type of audit being performed, may be made up of one or more members and shall be directed by a qualified lead auditor. The audit team shall develop and document an audit plan to include: the scope of the audit, the audit team members, the activities to be audited, personnel to be notified, the schedule, required documents, and checklists as each is applicable. The team leader should outline the tasks assigned to each auditor. Each auditor should become familiar with his/her assigned area.

MW14.1.4 Audit Performance

Each audit shall consist of the following general functions: 1) the preaudit conference in which the audit scope is briefly described and introductions are made; 2) the examination of evidence; 3) data taking, the completion of checklists is one

form of data taking; 4) recording of potential findings and observations; 5) post audit critique in which findings and observations are discussed and consolidated among the auditors, and; 6) the post audit conference in which the results of the audit are presented briefly to the audited group.

MW14.1.5 Audit Report

The lead auditor shall prepare the audit report based on the notes, checklists, and comments of the audit team members. The audit report shall consist of the following general features:

1. Transmittal letter
2. Description of the audit - will include names of audited groups, scope of the audit, and summary of audit activities
3. Audit findings in appropriate format
4. Audit observations and recommendations
5. Response date specified

The audit report should be issued as soon as possible but not more than thirty (30) days following the audit.

MW14.1.6 Corrective Action and Follow-Up

The lead auditor is responsible for audit follow-up and verification of corrective action. All audit findings shall be documented in the Quality Control Coordinator's nonconformance log for follow-up within the laboratory. A response to the audit report from the audited organizations should be submitted to the lead auditor within the specified time limit. The response shall

state the planned or accomplished corrective action and target dates for completion. After acceptance of the written response, the lead auditor shall verify corrective action through written communication, review of revised documents, reaudit, or other appropriate means.

MW14.1.7 Audit Records

All records associated with performance audits shall be considered quality documents and maintained as described in Section MW12.0. These records consist of:

* the audit plan
* checklists
* nonconformance log
* the audit report
* audit finding reports and responses
* other pertinent correspondence

Any subset of those records listed above received by the laboratory concerning the conduct and reporting of a system's audit shall be retained by the laboratory as quality documents.

MW14.2 Surveillances

Surveillances of a specific areas of ITASMW shall be performed by the Quality Control Coordinator or designee on a monthly basis. Surveillances do not require as extensive planning and preparation as do audits, nor prior communication with the surveyed organization or personnel.

The Quality Control Coordinator or designee shall observe the activity of interest while it is in process and/or review objective evidence using a predesigned checklist based on the applicable documents.

Nonconformances observed during a surveillance shall be documented in a nonconformance memo (Section MW13.0) and recorded into the Quality Control Coordinator's nonconformance log for follow-up and corrective action. The Laboratory Management is responsible for ensuring that corrective action is taken. The Quality Control Coordinator is responsible for insuring that the surveillance is "closed out". The date closed is recorded in the nonconformance logbook.

A copy of all surveillance records shall be maintained in the Quality/Operations audit files.

MW15.0 QUALITY REPORTS TO MANAGEMENT

The Quality Control Coordinator is responsible for preparing reports to management indicating effectiveness of the Quality Assurance Program. The Quality Control Coordinator will prepare monthly updates, summarizing current quality related activities to be given to the ITAS Director Quality Assurance and Compliance. The monthly quality report shall include:

1) Audits

   * Internal Audits/Surveillances
   * External Audits
   * Subcontractor Audits

2) Performance Evaluation Samples

   * PE Samples in house
   * PE Samples Submitted/Pending
   * PE Results Received

3) Certification

   * Received
   * Initiated

4) Nonconformance Summary

   * Tabulated by area

5) Holding Time Violation Summary

6) QC Data/Control Chart Summary

7) SOPs Issued/Revised

8) Training Received/Conducted

9) QAPPs Reviewed/Received

10) Miscellaneous

MW16.0 PERSONNEL TRAINING AND QUALIFICATIONS

The ITAS Mixed Waste laboratory personnel training and qualifications program shall be developed to ensure that all laboratory employees in quality related positions have been trained and qualified in their specific area of work and in the associated quality assurance procedures and to ensure that the training and qualification data are current and properly documented.

MW16.1 Qualifications

ITASMWL shall have detailed job descriptions for all laboratory positions. These job descriptions shall provide the minimum qualifications in terms of education and experience, duties, knowledge, and skills necessary for an analyst to carry out work in the Mixed Waste laboratory. During an annual performance review, the laboratory supervisors shall compare each analyst's performance with the qualifications established in his/her job description.

ITASMWL normally expects necessary knowledge and fundamental chemical laboratory skills to have been demonstrated by formal academic training which includes course work in the following areas: general chemistry, qualitative analysis, quantitative analysis, and instrumental analysis, or demonstrated through extensive laboratory training.

MW16.2 Training

MW16.2.1 Technical Training

An analyst hired to perform sample preparation procedures and/or analytical procedures shall receive one-on-one instruction from a professional staff member or a qualified analyst for each procedure. To become qualified on a particular procedure, the analyst must pass a specified qualifying set. The qualifying step for each sample preparation procedure shall be an independent performance of the procedure while being observed by the instructor. The qualifying step for each analytical procedure shall be an acceptable analysis of an unknown sample set (acceptance criteria shall be detailed in the laboratory training procedure). Only upon acceptable completion of the appropriate qualifying step shall the analyst perform the procedure without assistance or supervision.

MW16.2.2 Maintenance of Technical Qualification and Retraining

An analyst's qualification on a technical procedure shall be reviewed annually by the appropriate supervisor. The supervisor may extend qualification or provide retraining based on the analyst's observed performance of the procedure, results of qualification samples, or results of internal or external performance evaluation samples.

MW16.2.3 Quality Assurance Training

MW16.2.3.1 For Laboratory Analysts

Each laboratory analyst shall become familiar with the laboratory quality assurance program by reading quality control procedures and sections of the laboratory-specific attachment and the ITAS QA Manual appropriate to his/her position.

For initial quality assurance qualification, the trainee shall be given a quality assurance written exam that will test the trainee's knowledge and use of the laboratory's quality assurance program. Passing this exam will demonstrate sufficient knowledge of the quality assurance program to perform work in the Mixed Waste laboratory upon training in a specific technical area. Acceptance criteria for the QA examination will be given in the laboratory training procedure. The examination shall be maintained as a quality record. For maintenance of QA qualification and retraining, maintained as a quality record. For maintenance of QA qualification and retraining, the laboratory staff shall conduct periodic quality assurance training sessions for the Mixed Waste laboratory analysts.

MW16.2.3.2 For the Quality Control Coordinator

Quality assurance training for the laboratory Quality Control Coordinator is the responsibility of the ITAS Director of Quality Assurance and Compliance. This training shall consist of workshops and training sessions presented by the ITAS QA office and external training sessions presented by reputable quality

assurance organizations. Copies of the QC Coordinator's QA training records shall be maintained in the Mixed Waste laboratory's Quality/Operations files.

MW16.3 Qualification and Training Records

Each laboratory employee in a quality related position shall have a Personnel Qualification Record. The record shall be documented with technical and quality assurance procedures for which the employee is qualified, the dates of qualification and renewal, and the approval signature. Other qualification and training documents include:

- personnel resumes
- record of observation on a sample prep procedure
- results of qualifying sample sets
- quality assurance examinations
- attendance records of training sessions
- professional certificates, and
- internal training certificates of qualification

The QC Coordinator is responsible for maintaining the personnel qualification and training records as quality documents in the Quality/Operations record system, Category A.

APPENDIX C

IT ANALYTICAL SERVICES QUALITY ASSURANCE MANUAL

IT Radiological Sciences Laboratory (Oak Ridge, Tennessee)

QUALITY ASSURANCE MANUAL
LABORATORY - SPECIFIC ATTACHMENT

IT RADIOLOGICAL SCIENCES LABORATORY

Approved for Issue:

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Prepared By:

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Controlled Copy No: _____

Issued To: _____

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IT/Radiological Sciences Laboratory
Laboratory Specific Attachment
Revision Log

Revision No.	Date of Revision	Pages Affected	Description of Change

IT RADIOLOGICAL SCIENCES LABORATORY

GENERAL INFORMATION

Location:

1550 Bear Creek Road
Oak Ridge, Tennessee

Mailing Address:

P.O. Box 549
Oak Ridge, Tennessee 37831

Telephone:

(615) 482-9707

Licenses:

Radioactive Material License Nos. R-0143-L88 and N-0142-L8

IT RADIOLOGICAL SCIENCES LABORATORY

Policy Statement

It is the policy of IT Radiological Sciences Laboratory to provide quality radioanalytical services that meet the customer, corporate, and industry requirements. The Quality Assurance Program, as prescribed in this and the ITAS manual, is designed to satisfy the requirements of the ANSI/ASME NQA-1 Standard, where determined to be applicable, and the USNRC Regulatory Guide 4.15. When appropriate, detailed procedures are used to implement the policies of this manual.

It is the objective of this Quality Assurance Program to assure that a controlled quality system is maintained over all aspects of the laboratory which effect the quality of the data and to assure that data submitted to the customer conform to all requirements.

All levels of lab personnel are directed to perform all work in compliance with the requirements of this Quality Assurance Program.

The above Quality Assurance policy and the Quality Assurance Program as described in this manual and the ITAS Quality Assurance Manual, has the unqualified support of our corporation and management.

John A. Auxier
Laboratory Director

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RS1.0 INTRODUCTION

The International Technology Radiological Sciences Laboratory (IT/RSL) is one of several laboratories that comprise the IT Analytical Services (ITAS) division of the IT Corporation (IT). Provided in the ITAS Quality Assurance Manual is the overall Quality Assurance Program to which all the ITAS laboratories adhere. Each laboratory is required to supplement the ITAS manual with a Laboratory-Specific Attachment that further details the implementation of the quality assurance program and expands the program, where necessary, to meet the requirements of the individual laboratory's work. This document is the Laboratory-Specific Attachment for IT/RSL which, together with the ITAS QA Manual, form the QA/QC program for this laboratory. It is the expressed purpose of these documents and the overall program to provide a comprehensive plan for producing data of known quality.

As noted in the ITAS QA Manual, it is recognized that all laboratory personnel affect the quality of the data. This manual has been prepared in order that staff members will be cognizant of the procedures adopted by IT/RSL for the production of analytical data, and so they will be aware of their responsibilities. To assist in the incorporation of the two manuals, the material in this attachment has been presented in the same format and topics all covered in the same order as the ITAS Manual. The prefix RS distinguishes the RSL Manual sections from those of the ITAS Manual and other Laboratory-Specific attachments. Where sections found in the ITAS Quality Assurance Manual are not covered in this Manual, they are incorporated from the ITAS Manual by reference.

As in the ITAS Quality Assurance Manual, the terms Quality Assurance and Quality Control are used in this manual as follows:

- Quality Assurance - The overall controls imposed upon laboratory operations. Included are all actions taken by personnel, and the documentation of laboratory performance as specified in the Quality Assurance Program.

- Quality Control - The daily, specific actions taken within the laboratory to verify sample integrity, performance of analyses, data processing, and record maintenance.

RS1.1 OBJECTIVES OF THE QUALITY ASSURANCE DOCUMENTS

The objectives of the IT/RSL Quality Assurance Program and the approach to accomplishing these goals are the same as those of ITAS and are listed in Section 1.1 of the ITAS Quality Assurance Manual. In addition to stated objectives, IT/RSL has written this attachment to meet the requirements particular to the nuclear industry and the U.S. Nuclear Regulatory Commission.

RS1.2 IT/RSL QUALITY ASSURANCE DOCUMENTS

IT/RSL maintains several types of supplementary documents to provide a comprehensive Quality Assurance Program. Listed below are the various controlled documents and a brief discussion of the purpose of each.

- Quality Assurance Manual - The Quality Assurance Manual provides the ITAS Quality Assurance policy. The manual discusses all aspects of Quality Assurance and Quality Control, both administrative and technical. However, it is not intended that the manual provide in-depth technical discussion. The Quality Assurance Manual has precedence in policy matters over all other ITAS quality-related documents. The manual is applicable to and provides direction for the Radiological Sciences Laboratory.

- Laboratory-Specific Attachment - The Quality Assurance Manual Attachment specific to the radiological laboratory is prepared for two purposes:

  - The Radiological Sciences Laboratory provides services that are unique to this laboratory and the Quality Assurance requirements specific to that work are described in the attachment.

- Certain aspects of laboratory operations discussed in the Quality Assurance Manual are dependent upon laboratory facilities. Quality Assurance activities such as preventive maintenance, calibration, and data verification are functions of the equipment in a specific laboratory. The Laboratory-Specific Attachment has been prepared to document the specific operations of IT/RSL and is adopted as part of the Quality Assurance Manual.

- Project-Specific Manuals - Occasionally, contractual and regulatory demands, or uniqueness of a project's scope of work, require the preparation and implementation of a Project-Specific Quality Assurance Program and Manual. If such a manual and implementation is required, full documentation will be provided in a Project-Specific Manual. The requirements of the project will take precedence over conventional ITAS Quality Assurance practices for that work.

- Standard Operating Procedures - Standard Operating Procedures are developed to provide specific instruction for analytical and support activities which are necessary for the performance of quality work and production of quality data. These include standard methods approved for use and methods developed within the laboratory to meet specific client needs.

RS1.3 DOCUMENT CONTROL, DISTRIBUTION, AND REVISION

All Quality Assurance Documents are controlled documents and must be approved by designated personnel. The approvals required for issuing and revising the various documents follow:

- ITAS Quality Assurance Manual

  - Vice President, IT Analytical Services
  - Quality Assurance/Control Director, ITAS
  - Director, Quality Assurance, Environmental Projects Group

- Laboratory-Specific Attachment

  - Laboratory Manager
  - Laboratory Director
  - Quality Assurance/Control Director, ITAS
  - Director, Quality Assurance, Environmental Projects Group

- Project-Specific Manuals

  - Laboratory Manager
  - Laboratory Director
  - Laboratory Quality Control Coordinator
  - Quality Assurance Manager
  - Project Manager (if analytical services are part of a broader-based project, and is required by contract).

- Standard Operating Procedures

  - Laboratory Director
  - Laboratory Manager
  - Quality Assurance/Control Director, ITAS
  - Laboratory Quality Control Coordinator

Approval of manuals shall be denoted by a signature-and-date page in each manual which includes the above and the manual author. Responsibilities for control of documents, revisions and document receipt are addressed in ITAS QA Manual, Section 1.3.

RS2.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES

The Director of IT/RSL has the overall responsibility for the establishment and implementation of the Quality Assurance Program described in this and the ITAS Manual. The organizational chart, Figure RS2-1, illustrates the specific lines of authority and communication for the management, direction, and execution of the Quality Assurance Program.

Quality Assurance is sufficiently independent from the pressures of laboratory activities and from cost and schedule considerations. The Q.A. Coordinator has direct access to the Director when appropriate action is required and has the responsibility and authority to stop work when deemed necessary.

RS2.1 IT/RSL QUALITY-RELATED RESPONSIBILITIES WITHIN THE LABORATORY

This section identifies the various positions within the laboratory and the specific responsibilities of each for the implementation of the Quality Assurance Program and the completion of Quality Control activities.

- Laboratory Director shall:

  - Report directly to the Vice President, IT Analytical Services
  - Periodically determine the effectiveness of the Quality Assurance Program in the laboratory
  - Approve the Laboratory-Specific attachment to the Quality Assurance Manual, Project-Specific Manuals and Standard Operating Proecedures and Procedures and revisions.

- Laboratory Manager shall:

  - Approve the Laboratory-Specific Attachment to the Quality Assurance Manual, Project-Specific Manuals and Standard Operating Procedures and all revisions
  - Implement the Quality Assurance Program within the laboratory
  - Supervise routine Quality Control activities within the laboratory
  - Recommend to the ITAS QA/QC Director changes in the Quality Assurance Program.
  - Approve all reports issued by the laboratory
  - Maintain current laboratory organization chart

- Manage laboratory daily analytical operations
- Supervise sample storage facilities

- Technical Director shall:

  - Provide technical overview of laboratory activities
  - Define and oversee training program
  - Serve as an "in-house" consultant for the applicability of general Quality Control practices to specific needs.
  - Evaluate new analytical techniques, procedures, instrumentation and Quality Control procedures and provide recommendations to the Laboratory Manager.
  - Define calibration program for instrumentation within the laboratory
  - Define and oversee instrument preventive maintenance program and schedule.
  - Supervise the log-in of all samples received, completion of chain of custody, and maintenance sample log book
  - Supervise the verification of software for data processing
  - Recommend standards for purchasing instruments, equipment, reagents, gases and chemicals.

- Quality Control Coordinator shall:

  - Prepare Quality Control standards, insert Quality Control samples into the laboratory sample stream, and review results
  - Perform statistical analyses utilizing results of QC sample analyses
  - Notify the Laboratory Manager of out-of-control or biased situations
  - Report nonconformances to the ITAS Quality Assurance/Quality Control Director if the situation is not corrected within the laboratory
  - Assist in the performance of Quality Assurance audits and perform Quality Control Audits
  - Establish and supervise the laboratory QA training program
  - Supervise the maintenance of laboratory records
  - Monitor implementation of QA program
  - Perform annual review of QA manual and procedures
  - Serve as focal point "for the reporting and disposition of nonconformances

- Group Leaders shall:

  - Serve as the Lead Analyst within the laboratory group to which they are assigned (group meaning a division of people in the lab who perform work of a similar nature or in a specific area)
  - Organize and schedule the analytical testing program with consideration for sample-holding times

- Review all analytical data and submit to the Laboratory Manager for approval
- Implement data verification procedures
- Assign analysts for data processing and validation activities
- Evaluate instrument performance and supervise instrument calibration and preventive maintenance programs
- Report out-of-control or nonconforming situations to the Laboratory Manager and Quality Control Coordinator
- Stop production of data in laboratory area where review of Quality Control data or procedures show significant problems
- Resolve ongoing and recurring nonconformances within the laboratory
- Recommend corrective actions for resolutions of nonconformances
- Review statistical data to verify the laboratory is meeting stated Quality Control goals
- Maintain current distribution lists for Laboratory-Specific Attachments, Project Specific Manuals and Standard Operating Procedures
- Close findings and recommendations of Quality Assurance Audits

- Technicians shall:

  - Perform analytical procedures and data recording in accordance with accepted methods
  - Perform and document calibration and preventive maintenance of instrumentation, as required in associated procedures
  - Perform data processing and validation
  - Immediately report out-of-control situations, instrument malfunction, calibration failure, or other nonconformances to the Group Leader and Quality Control Coordinator, as appropriate

RS2.2 IT/RSL QUALITY ASSURANCE RESPONSIBILITIES OUTSIDE OF THE LABORATORY

Ultimate responsibility for the Quality Assurance lies with the Vice President, IT Analytical Services. To fulfill this responsibility, the Vice President, has assigned responsiblity within the laboratory to the Laboratory Director and external review of the quality performance is assigned to the Quality Assurance/Quality Control Director, IT Analytical Services.

The ITAS QA/QC Director reports directly to the Vice President and is independent of laboratory management. This independence allows the QA/QC Director to assess laboratory quality performance without being influenced by schedule and cost considerations. Responsibilities of the QA/QC Director shall be:

- Approve all ITAS Quality Assurance documents
- Perform audits of ITAS laboratories to verify compliance with the Quality Assurance Program
- Verify completion of corrective action cited in audits
- Act as the collection point for proposed changes in the Quality Assurance Program and initiate changes in the program
- As necessary, discuss with the Laboratory Manager and the Vice President unresolved nonconformances brought to the QA/QC Director's attention by a Quality Control Coordinator

The Quality Assurance Director, Environmental Projects Group, reports directly to the Vice-President, Environmental Projects Group and has responsibility for review of the Quality Assurance Program and periodic audits of the program implementation.

LABORATORY ORGANIZATIONAL CHART

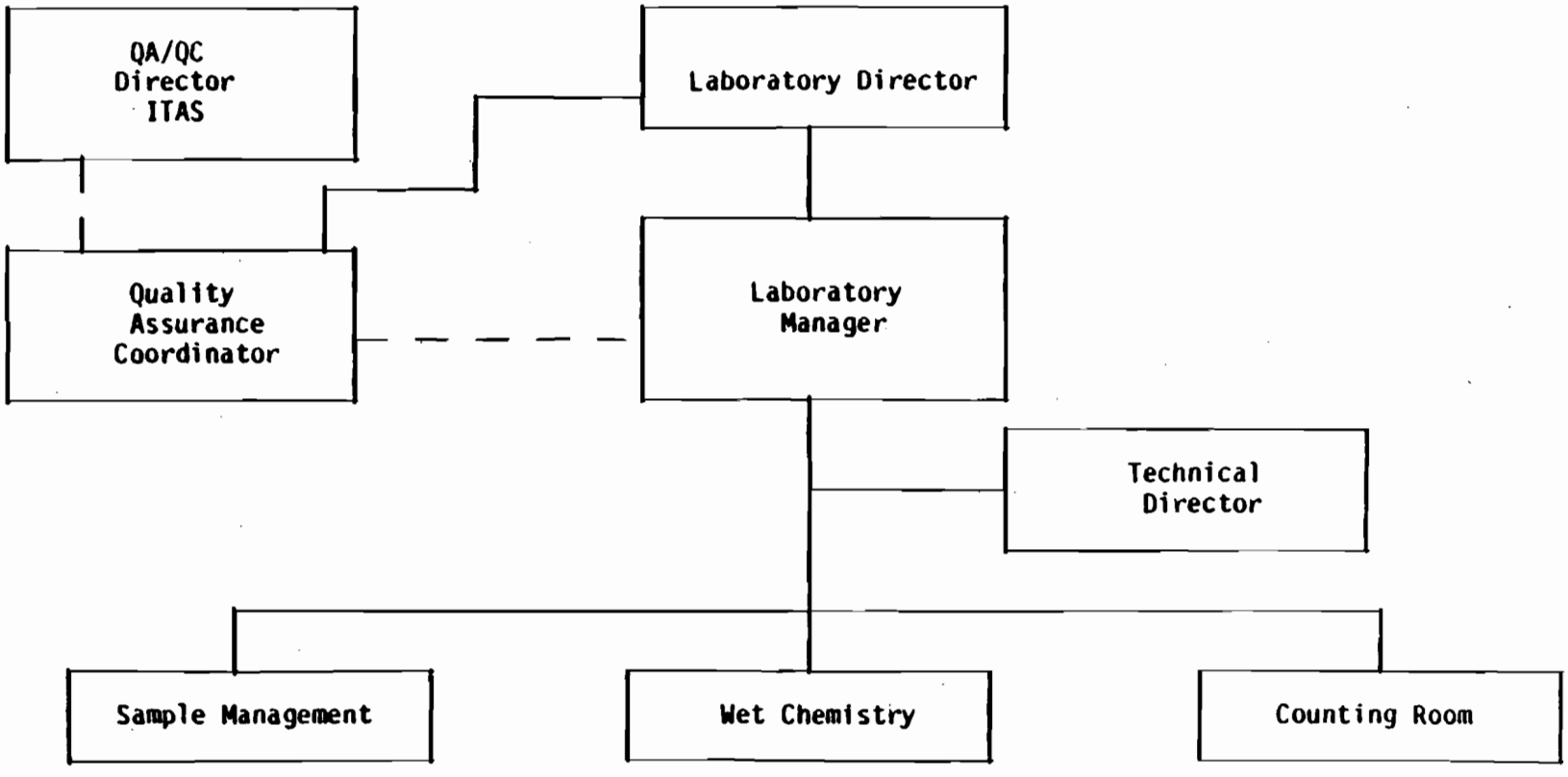

FIGURE RS2-1

RS3.0 STANDARD LABORATORY PRACTICE

The radioanalytical work and associated activities at IT/RSL shall be performed in accordance with documented, approved procedures. The following sections of this manual attachment outline the requirements of quality-related activities and, therefore, requirements for the procedures associated with each.

Table RS3-1 provides a description of functions for laboratory analyses. The functions outlined in this Table shall have procedures which prescribe the approved method by which these activities will be accomplished. Figure RS3-1 provides a flow chart for laboratory analysis and data.

RS3.1 RESPONSIBILITY FOR STANDARD OPERATING PROCEDURES

The Technical Director shall be responsible for generating, issuing, and revising standard operating procedures for all laboratory activities. These procedures shall include all analytical methods and any activities necessary for the successful completion of the analysis and the accurate reporting of the data generated.

The Quality Control Coordinator shall be responsible for the preparation, issuing, and revising of procedures related to quality activities.

RS3.2 STANDARD PROCEDURE FORMAT

Procedures shall have a cover page with all signatures required for approval and which identifies the procedure by title, identification number, and revision number and date (Attachment RS3-1). All procedures will be printed on standard procedure forms which will also include the procedure title, identification number, date of issuance, and revision number (Attachment RS3-2).

Procedures shall be written in the following format:

1.0 Purpose
2.0 Scope
3.0 Responsibilities
4.0 References
5.0 Requirements
6.0 Procedure
7.0 Precision and Accuracy
8.0 Quality Assurance Provisions
9.0 Calculations
10.0 Appendices

RS3.3 ISSUANCE OF STANDARD OPERATING PROCEDURES

Upon issuance of a procedure, all effected personnel shall be trained to the specifics of the instructions. Training of effected personnel shall also be performed for all changes made in a revision. Training and documentation requirements are discussed in Section 16.0 of this manual. Revisions, except non-material, editorial corrections, require the same review and approval as the original document.

Table RS3-1

Laboratory Function Descriptions

FUNCTION	DESCRIPTION	DOCUMENTS USED	PERFORMED BY
Receipt of Samples	Samples are unpacked, inspected & screened. Chain of Custody is reviewed for completeness and signed.	Chain of Custody	Sample Custodian
	Sample identification & information is entered into sample management system; lab ID assigned; labeling performed.	Chain of Custody Request for Analysis Form Sample labels	Sample Custodian
	Sample placed in appropriate holding storage area.		
Initiation of Testing Program	Laboratory work sheet generated for each analysis requested; work sheet filed in proper analysis file.	Request for Analysis Form Lab Work Sheet	Sample Custodian
Analysis	Samples are retrieved from storage and required preparation performed.	Lab Work Sheet Standard Operating Procedures	Laboratory Technician
	Required tests performed on prepared sample.	Standard Operating Procedures	Laboratory Technician
	Completed sample is counted on appropriate instrument.	Lab Work Sheet Calibration logbook Instrument printout SOPs	Laboratory Technician

TABLE RS3-1 (Continued)

Laboratory Function Descriptions

FUNCTION	DESCRIPTION	DOCUMENTS USED	PERFORMED BY
Data Processing & Validation	Analytical data are reviewed; calculations checked.	Lab Work Sheet Calibration logbook QC sample data	Group Leaders/ Laboratory Technician
	Data are reviewed for completeness & reasonableness; Approval given for reporting or retest initiated.	Lab Work Sheet QC Sample data Customer information	Laboratory Manager
Reporting	Analytical data are entered into computer data base; report is generated & verified.	Lab Work Sheet Client data report	Data Base Manager
	Verified client report is approved & signed.	Client data report	Laboratory Manager
Records	Customer or project files are updated	Customer forms Lab Work Sheets Instrument Print Outs Data Report QC Sample Data	Records Custodian

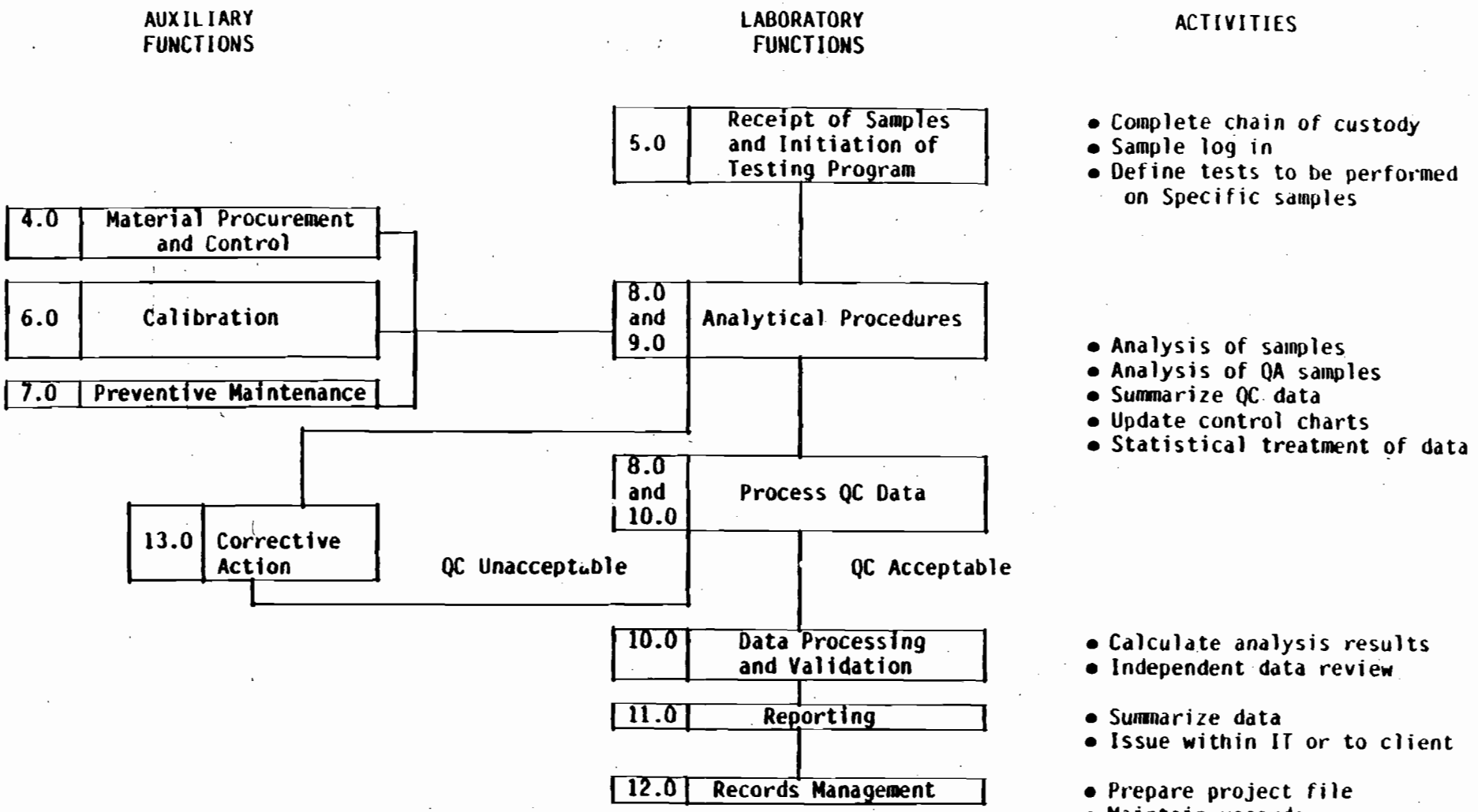

FIGURE RS3-1

LABORATORY ANALYSIS FLOW CHART

ATTACHMENT RS3-1

Laboratory Procedure Cover Page

INTERNATIONAL TECHNOLOGY CORPORATION

RADIOLOGICAL SCIENCES LABORATORY

NO.: _____

RADIOANALYTICAL LABORATORY PROCEDURE

TITLE: _____

APPROVED: _____ DATE: _____
Laboratory Manager

APPROVED: _____ DATE: _____
ITAS QA Director

CONCURRED: _____ DATE: _____
RSL Director

COPY NO.: _____ ISSUED: _____

REVISION:				
DATE:				

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ATTACHMENT RS3-2

Laboratory Procedure Standard Form

TITLE:	NO:	PAGE:
	REVISION:	DATE:

RS4.0 MATERIAL PROCUREMENT AND CONTROL

Materials purchased by IT/RSL for analytical work shall be commercial grade and meet the requirements denoted in specific analytical procedures in which they are to be used. Purchase order requisitions shall be approved by the Laboratory Manager or a designee. Radioactive reference standards orders shall be reviewed by the Health Physics Services Supervisor to assure license compliance. Upon receipt of ordered item, a person designated by the Laboratory Manager shall perform an inspection which shall determine the following:

1. Damage was not sustained during shipment;
2. The item received was the item ordered;
3. Documentation, as applicable to the item, was received and is acceptable;

If any of these requirements are not met, the item shall not be accepted and shall be returned to the supplier.

RS4.1 REQUIREMENTS FOR REAGENTS, SOLVENTS, AND GASES

If the analytical method does not specify the purity of materials, ACS grade shall be used, if it exists. Prior to performing an analysis, the label of the materials container should be checked by the technician to verify that the material purity meets the needs of the analysis. The preparation of fresh material should be as often as indicated by its stability and should be specified in the analytical method.

RS4.1.1 Water

Deionized/demineralized water shall be used for dilution and preparation of reagent solutions. Water quality shall be determined daily by measuring specific conductance and shall be recorded in a log book or, if automatic print-out, filed appropriately. A specific conductance less than 0.5 umho/cm at 25 degree C is required. This is equivalent to less than 0.5 μg/l of ionized material.

RS4.1.2 Compressed Air

Compressed air must be free of oil, water, and dirt. If produced in the laboratory, appropriate filters shall be used at the compressor to prevent moisture from entering the piping system.

RS4.2 REQUIREMENTS FOR LABORATORY CONTAINERS

Sample containers may be plastic or glass and may be used as received from commercial supplier. Containers may not be cleaned and reused but shall always be disposed of after use. Specifications for containers to be used in an analysis should be identified in the analytical method. General requirements for material composition and volumetric tolerances are discussed below.

RS4.2.1 Material Composition of Laboratory Vessels

The general use glassware should be chemically resistant borosilicate glass unless otherwise noted in the test procedure. The use of plastic vessels, containers, and other apparatus made of Teflon®, polythylene, polystyrene, and polypropylene may be used for certain specified applications.

The following guidelines should be followed when selecting the material composition of laboratory vessels:

- Borosilicate or polyethylene bottles are to be used for the storage of reagents and standard solutions, unless otherwise specified.
- Plastic bottles of polyethylene and Teflon® are satisfactory, in general, for the shipment of water samples. However, strong mineral acids, such as sulfuric acid, and organic solvents readily attack polyethylene.
- Borosilicate glassware is not completely inert, particularly to alkalis. Standard solutions of silica, boron, and the alkali metals should be stored in polyethylene bottles.

RS4.2.2 Volumetric Container Specifications

IT/RSL shall use glassware of sufficient accuracy as required in the analytical procedure for the measurement of sample or reagent volumes. Less accurate types of glassware have specific uses when less exact volumes are permitted by the procedure. In general, volumetric containers will not be calibrated by IT/RSL unless required by a specific analytical method. However, where specified, calibrated glassware requirements shall be strictly adhered to.

RS4.3 STORING AND MAINTAINING REAGENTS AND SOLVENTS

The following requirements shall apply for storing and maintaining reagents and solvents:

- All containers shall be properly labeled for contents and dated on date received or date of dilution.
- Standard reagents and solvents are stored in accordance with manufacturer's recommendations
- Light sensitive standard reagents or solvents are stored in dark bottles as required by specific procedures
- When fresh stock solutions are required, dilutions of the old and new standard are compared to determine consistency

RS4.4 RADIOACTIVE REFERENCE STANDARDS

IT/RSL shall use only radioactive reference standards which are traceable to the National Bureau of Standards (NBS). Certification of traceability shall be provided by the supplier. If this quality of standard is not commercially available, the best available for that isotope shall be used. The documentation received with each standard should include the following information:

- radionuclide identification, activity and error
- source identification number

- mass and volume of standard
- date of assay
- half-life of the radionuclide(s)

Detailed procedures shall be used for the receipt, storage, use, control, and disposal of radioactive standards. Attachment RS4-1 shall be used to record information about the original standard, the stock solution, and uses of the stock solution. Attachment RS4-2 shall be used to record the identification and uses of working solutions. Attachment RS4-3 shall be used to record calculations for the dilutions of each stock or working solution. The disposal of each standard and all dilutions shall be recorded on the form provided in Attachment RS4-4. All records of use and disposal shall be maintained in the Radioactive Material Inventory logbook . Completed records for standards which have been disposed of or depleted, shall be transferred to the quality assurance files. Standards shall be stored in a lead-lined, secured cabinet and shall be maintained no longer than five half-lives or ten years or as recommended by supplier, which is the shortest period of time.

The Laboratory Manager shall have the responsibility for the acquisition, receipt, storage, and control of all radioactive reference standards used in the laboratory.

The Health Physics Services Supervisor shall be responsible for the proper disposal of all radioactive reference standards.

RS4.5 GLASSWARE CLEANING REQUIREMENTS

Glassware to be used in radiochemical analyses should be well cleaned to insure that cross-contamination does not occur. As good practice, all containers should be rinsed with tap water as soon as possible after use. Glassware should then be soaked in hot tap water and a commercial detergent, scrubbed, rinsed in tap water followed by a thorough rinse with demineralized water. Glassware used with samples which are determined to be above levels usually seen in the environmental lab should be segregated and cleaned separately. Inspection for cracks and etching should be made before glassware is stored for reuse.

ATTACHMENT RS4-1

IT/RADIOLOGICAL SCIENCES LABORATORY
RADIOACTIVE MATERIAL INVENTORY CONTROL

Date Received: _____ Vendor ID: _____ Log#: _____

Form: Solid Liquid Gas

Isotope(s)	Amount	Concentration	Assay Date: _____

STOCK SOLUTION ID given: _____

Diluted with: _____ Amount: _____

USE:

DATE	AMOUNT TAKEN	FOR USE IN	ACTIVITY	INITIALS

ATTACHMENT RS4-2

IT/RADIOLOGICAL SCIENCES LABORATORY
WORKING SOLUTION LOG

Log# _____

Form: Solid Liquid Gas

Isotope(s)	Amount	Concentration

Assay Date: _____

WORKING SOLUTION ID given: _____

Diluted with: _____ Amount: _____

USE:

DATE	AMOUNT TAKEN	FOR USE IN	ACTIVITY	INITIALS

ATTACHMENT RS4-3

DILUTION CALCULATION

Log#: _____ Date: _____

Calculations made by: _____

Verified by: _____

ATTACHMENT RS4-4

DISPOSAL RECORD

Total volume used _____ Amount Remaining _____

Reason for disposal: _____

Method of disposal: _____

Final disposal approved by: _____

Removed from inventory and disposed by: _____

RS5.0 SAMPLE RECEIPT AND INITIATION OF TESTING PROGRAM

This section establishes and assigns responsibilities for receipt and handling of samples and the initiation of a testing program. Procedures written for these activities should be designed to maintain the sample integrity and individual sample identity; provide good health physics and industrial hygiene practices during inspection and screening; provide holding times and appropriate storage practices for particular types of samples; provide guidelines for assuring customer analysis request is properly addressed; and list the minimum requirements for sample documentation.

RS5.1 FIELD COLLECTION AND SHIPMENT

A discussion of the requirements to be addressed by International Technology collection teams prior to actual sample collection can be found in the ITAS QA Manual, Section 5.2. EPA recommendations for preservatives and containers to be used with water samples for radiological analyses are given in Table RS5-1.

RS5.2 CHAIN OF CUSTODY FORM

Documentation of sample collection, shipment, laboratory receipt, and laboratory custody until disposal is accomplished by the use of a Chain of Custody form when samples are collected by International Technology personnel. IT/RSL shall not accept samples collected by IT personnel which do not have a correctly prepared Chain of Custody. The approved IT Chain of Custody form is provided in Attachment RS5-1 and is discussed in detail in the ITAS QA Manual in Section 5.1.

RS5.3 REQUEST FOR ANALYSIS FORM

Request for Analysis form is a required document for samples collected by International Technology personnel. When samples collected by IT are received at IT/RSL without the Request for Analysis form correctly prepared, the samples

will be held until proper documentation of required analysis is received. Attachment RS5.2 shows the approved IT Request for Analysis form. If samples without Request for Analysis are external to IT Corporation, the customer services representative shall contact the client to determine the testing program required.

RS5.4 LABORATORY SAMPLE RECEIPT

When advance information of samples being shipped is provided, the person receiving the information should notify the Laboratory Manager and Sample Custodian of the incoming samples.

The Sample Custodian is responsible for the receipt and log-in of samples. Procedures specific to these activities shall be written and shall include the following:

- Examine shipping container for damage.
- Check with survey instrument to determine external activity and smear surfaces for removable contamination.
- Examine all samples and determine if samples have been damaged during shipment. Any sample affected by a damaged sample shall be considered damaged also.
- Compare samples received against those listed on the chain of custody.
- Note on the chain of custody form the condition of the samples and give the identification of any damaged samples. Sign and date the chain of custody form and attach the waybill to the form.
- Place samples in the appropriate storage area.
- Enter samples into the laboratory log-in system, providing all required information.
- Notify the Laboratory Manager of the arrival of the samples.
- Place completed chain of custody in the project file.

- Notify customer if any discrepancy in documentation or damage to samples is found.

If samples collected by IT personnel arrive without a correctly prepared Chain of Custody or with broken custody seals, the Sample Custodian shall notify the QC Coordinator. The QC Coordinator shall follow steps outlined in section 5.3 of the ITAS QA Manual.

RS5.5 LABORATORY STORAGE OF SAMPLES

The primary considerations for the determination of the appropriate storage and test schedule for a sample are the following:

- Maintenance of prescribed temperature where required
- Extracting and/or analyzing sample within the prescribed holding time for the parameters of interest

The Sample Custodian is responsible for placing samples in the proper storage environment and for notifying the Laboratory Manager of any sample which requires immediate analysis due to an expiring holding-time.

RS5.6 INITIATION OF TESTING PROGRAM

At the time of log-in, information from the client concerning the samples and the required testing will be entered into the computer data base. Laboratory work sheets for each analysis will be completed with the information and then put into the appropriate work-pending file in the laboratory.

The Laboratory Manager is responsible for setting the priorities of these samples on the basis of holding times and required reporting times.

RS5.7 SAMPLE DISPOSAL

Samples may be disposed of by any of the following methods:

- The sample may be completely consumed during analysis.

- Sample may be returned to the client or the location of sampling.
- Other samples shall be disposed of appropriately, based on measured activity of individual sample and according to disposal procedures.

The Chain of Custody shall be completed to indicate the disposition of samples. The Health Physics Services Supervisor shall be responsible for the sample disposal and shipping of radioactive and hazardous waste.

TABLE RS5-1

Water Sample Preservatives and Containers

PARAMETER	PRESERVATIVE[1]	CONTAINER[2]
Gross Alpha	Conc. HCl or HNO_3 to pH<2[3]	P or G
Gross Beta	Conc. HCl or HNO_3 to pH<2	P or G
Strontium-89	Conc. HCl or HNO_3 to pH<2	P or G
Strontium-90	Conc. HCl or HNO_3 to pH<2	P or G
Radium-226	Conc. HCl or HNO_3 to pH<2	P or G
Radium-228	Conc. HCl or HNO_3 to pH<2	P or G
Cesium-134	Conc. HCl or HNO_3 to pH<2	P or G
Iodine-131	Formaldehyde (~ 10 ml/l)	P or G
Tritium	None	P or G
Uranium	Conc. HCl or HNO_3 to pH<2	P or G
Photon emitters	Conc. HCl or HNO_3 to pH<2	P or G

1 It is recommended that the preservative be added to the sample at the time of collection unless suspended solids activity is to be measured. However, if the sample must be shipped to a laboratory or storage area, acidification of the sample (in it original container) may be delayed for a period not to exceed 5 days. A minimum of 16 hours must elapse between acidification and analysis.

2 P = Plastic, hard or soft; G = Glass, hard or soft.

3 If HCl is used to acidify samples which are to be analyzed for gross alpha or gross beta activities, the acid salts must be converted to nitrate salts before transfer of the samples to planchets. For this reason, HNO_3 is the preferred preservative.

INTERNATIONAL TECHNOLOGY CORPORATION

CHAIN-OF-CUSTODY RECORD

R/A Control No. _____

C/C Control No. _____

PROJECT NAME/NUMBER _____ LAB DESTINATION _____

SAMPLE TEAM MEMBERS _____ CARRIER/WAYBILL NO. _____

Sample Number	Sample Location and Description	Date and Time Collected	Sample Type	Container Type	Condition on Receipt (Name and Date)	Disposal Record No

Special Instructions: _____

Possible Sample Hazards: _____

SIGNATURES: (Name, Company, Date and Time)

1. Relinquished By: _____
   Received By: _____
2. Relinquished By: _____
   Received By: _____
3. Relinquished By: _____
   Received by: _____
4. Relinquished By: _____
   Received By: _____

WHITE - To accompany samples
YELLOW - Field copy

Request for Analysis Form

INTERNATIONAL TECHNOLOGY CORPORATION

REQUEST FOR ANALYSIS

R/A Control No. 030449

C/C Control No. _____

PROJECT NAME _____

PROJECT NUMBER _____

PROJECT MANAGER _____

BILL TO _____

PURCHASE ORDER NO. _____

DATE SAMPLES SHIPPED _____

LAB DESTINATION _____

LABORATORY CONTACT _____

SEND LAB REPORT TO _____

DATE REPORT REQUIRED _____

PROJECT CONTACT _____

PROJECT CONTACT PHONE NO. _____

Sample No	Sample Type	Sample Volume	Preservative	Requested Testing Program	Special Instructions

TURNAROUND TIME REQUIRED: (Rush must be approved by the Project Manager.)

Normal _____ Rush _____ (Subject to rush surcharge)

POSSIBLE HAZARD IDENTIFICATION: (Please indicate if sample(s) are hazardous materials and/or suspected to contain high levels of hazardous substances)

Nonhazard _____ Flammable _____ Skin Irritant _____ Highly Toxic _____ Other _____ (Please Specify)

SAMPLE DISPOSAL (Please indicate disposition of sample following analysis. Lab will charge for packing, shipping, and disposal.)

Return to Client _____ Disposal by Lab _____

FOR LAB USE ONLY

Received By _____ Date/Time _____

WHITE - Original, to accompany samples
YELLOW - Field copy

Attachment RS5-2

RS6.0 CALIBRATION PRACTICES

All counting instrumentation and measuring and test equipment used at IT/RSL shall be controlled by a formal calibration program. This program verifies that instruments used in activities affecting quality are of the proper type, range, accuracy, and precision to provide data that meets specified requirements. All instruments and equipment which measure a quantity, or whose performance is expected at a stated level, are subject to calibration. This requirement is not intended to imply a need for calibration or control of measurement tools if normal commercial practices provide adequate accuracy.

RS6.1 CALIBRATION SYSTEM

Development and documentation of the IT/RSL calibration program is the responsibility of the Technical Director. Implementation of the program is the responsibility of the Group Leaders and the analysts. The Quality Control Coordinator shall review the implementation of the program as prescribed in Section RS14.0. The various elements of the calibration program are discussed below.

RS6.1.1 Calibration Procedures

Written procedures shall be developed by IT/RSL for all instruments and equipment subject to calibration as described in this manual. As a minimum, the procedures shall include:

- Equipment to be calibrated
- Reference standards used for calibration
- Acceptable performance tolerances
- Frequency of calibration
- Calibration documentation required

RS6.1.2 Equipment Identification

Equipment that is subject to calibration shall be uniquely identified so that calibration records can be designated with the equipment's specific identification number. Equipment shall be identified by the manufacturer's serial number or by the IT inventory control number.

RS6.1.3 Calibration Frequency Determination

Laboratory instruments shall be calibrated at prescribed intervals and/or as part of the operational use of the equipment. Frequency shall be based on the type of equipment, stability characteristics, manufacturer's recommendations, intended use, required accuracy, effect of error on the process, and prior experience. Whenever accuracy is suspect, a special calibration will be performed.

The Technical Director shall maintain a current listing of laboratory instruments and their minimum calibration frequencies in a memo to the Quality Assurance files. The original memo and each update requires the approval signatures of the Laboratory Manager, and the Quality Assurance/Quality Control Director, ITAS. The memo shall include the following information:

- Each type of instrument in use
- Minimum calibration frequency for each
- Calibration methodology

RS6.1.4 Calibration Reference Standards

IT/RSL shall use reference materials from, or traceable to, the National Bureau of Standards, where they exist, as prescribed in the ITAS QA Manual, Section 6.1.4. If no national standard exists, the basis for the reference standard shall be documented.

RS6.1.5 Calibration Failure

Equipment that fails calibration or becomes inoperable during use shall be removed from service by tagging with a "Nonconforming Item" tag (shown in figure RS6-1) to indicate it is out of calibration and to prevent inadvertent use. When a piece of equipment fails, a "Notice of Equipment Calibration Failure" (Attachment RS6-2) form shall be completed and submitted to the QC Coordinator by the technician determining the instrument to be out of calibration. The QC Coordinator shall file this form in the equipment calibration file for that instrument.

It is the responsibility of the laboratory staff to be sure that the equipment being used is functioning properly. If an equipment malfunction is suspected or an electrical interruption is experienced, the equipment shall be recalibrated immediately or tagged and removed from service until recalibration can be performed. If it fails recalibration, it shall be repaired and satisfactorily recalibrated before being returned to use. Samples counted on that instrument between the calibration failure and the last satisfactory calibration shall be evaluated to determine acceptability of data.

RS6.1.6 Calibration Records

Calibration records shall be prepared and maintained for each piece of equipment subject to calibration. Records demonstrating accuracy of reference standards shall also be maintained.

Records shall include the following, as appropriate:

- Identification number and type of instrument
- Calibration frequency and acceptable tolerances
- Identification of calibration procedure used
- Date calibration was performed

- Identity of IT/RSL personnel
- Reference standard used for calibration
- Calibration data
- Certificates or statements of calibration provided by external agencies, and traceability to national standards
- Information regarding calibration acceptance or failure and any repair of failed equipment

RS6.2 OPERATIONAL CALIBRATIONS

Operational calibration frequency, as previously discussed, is dependent upon the stability characteristics of the specific type of instrument, intended use, and required accuracy. The calibration frequency shall be provided in each analytical procedure for which the instrument is required. A list of all equipment that is calibrated on an operational basis shall be maintained in the laboratory operational files also. The calibration data for each instrument shall be maintained in a bound logbook at the location of the instrument. All entries shall be initialed and dated by the technician performing the calibration. Each page of the logbook should be copied when it is filled and submitted to the QC Coordinator for filing. Each instrument shall have an individual folder in which calibration records shall be maintained. Each logbook shall be filed in the associated file when it is completely filled or replaced.

RS6.3 PERIODIC CALIBRATIONS

Periodic calibration shall be performed for equipment such as radiation survey instruments, balances, thermometers, ovens, and furnaces that are required in analytical methods, but which are not routinely calibrated as part of the analytical procedure. Records for periodically calibrated equipment, which have a frequency of quarterly or longer, shall be maintained in the laboratory operational quality records files by the QC Coordinator and shall not require a logbook. Each instrument shall have an individual folder in which calibration

certificates will be kept. A summary of calibrations will be compiled on the "Equipment Calibration Record" (Attachment RS6-3) and kept in the front of each folder. A calibration sticker shall be affixed to the instrument to indicate current calibration status. Figure RS6-4 provides an example of calibration sticker to be used for radiation measurement equipment calibrated by IT/RSL.

RS6.3.1 Balances

Each balance shall be checked daily before use with weights designated for that purpose for each balance. A full range calibration shall be performed monthly. Weights used shall be, or standardized against, certified Class S weights. The log-book for each balance should have a schedule to indicate required weights. Data shall be recorded on the "Balance Calibration" form (Attachment RS6.5) and plotted on the appropriate control chart. External certification of balances shall be required annually with documentation including verification of weights used. IT/RSL Class S calibration weights shall be recertified every three years.

RS6.3.2 Thermometers

A certified, or reference, thermometer shall be maintained for use in calibrating working thermometers. Reference thermometers shall be provided with NBS traceability for initial calibration and shall be recertified every three years with equipment directly traceable to the National Bureau of Standards.

Working thermometers shall be compared with the reference thermometers every 12 months. In addition, working thermometers should be visually inspected for damage by laboratory personnel prior to use. Calibration temperatures and acceptance criteria shall be based upon the working range of the thermometer and the accuracy required for its use. Data shall be recorded on the "Thermometer Calibration" form (Attachment RS6-6) and submitted to the QC Coordinator for proper filing.

ATTACHMENT RS6-1

C A U T I O N

NONCONFORMING TAG

Affected Item: _____

_____

I.D. No.: _____

Description of Nonconformance:

_____

_____

_____

_____

____ Work may continue with this item subject to the following limitations:

_____

_____

_____

_____

____ Work may not proceed with this item until the disposition of the nonconformance.

_____ _____

Signature Date

ATTACHMENT RS6-2

NOTICE OF EQUIPMENT CALIBRATION FAILURE

EQUIPMENT NUMBER: _____

EQUIPMENT NAME: _____

REASON FOR FAILURE TO PASS CALIBRATION: _____

_____

_____

WAS EQUIPMENT REPAIRED OR REPLACED: _____

_____

_____

IF REPAIRED, WAS THE CORRECTIVE ACTION ADEQUATE: _____

_____

_____

_____

_____

SIGNED: _____

ATTACHMENT RS6-3

LABORATORY

EQUIPMENT CALIBRATION RECORD

EQUIPMENT NUMBER: _____

EQUIPMENT NAME: _____

_____

REQUIRED CALIBRATION PERIOD: _____

Date Calibration Performed	Calibration Performed By	Date Calibration Performed	Calibration Performed By

ATTACHMENT RS6-4

Instrument Calibration Sticker

INTERNATIONAL TECHNOLOGY CORPORATION

Date _____ Make _____

Model _____ Serial _____

THIS INSTRUMENT HAS BEEN CALIBRATED IN ACCORDANCE WITH TN REGULATIONS FOR RADIATION CONTROL.

Signed

R-73010-L88

License Number

IT Corporation is a wholly owned subsidiary of International Technology Corporation

120A-10-85

ATTACHMENT RS6-5

BALANCE CALIBRATION

EQUIPMENT NUMBER: _____

EQUIPMENT NAME: _____

DATE: _____ DATE LAST CALIBRATED: _____

CALIBRATION PERIOD: _____

Weights Applied to Balance	Balance Reading	Does Balance Meet Standard

Signed: _____

THERMOMETER CALIBRATION

EQUIPMENT NUMBER: _____

EQUIPMENT NAME: _____

DATE: _____ DATE LAST CALIBRATED: _____

CALIBRATION PERIOD: _____

Temperature According To Reference	Thermometer Being Calibrated

WORKING RANGE: _____

ACCEPTANCE CRITERIA: _____ °C

Signed: _____

RS7.0 PREVENTIVE MAINTENANCE

The preventive maintenance program for instrumentation and equipment at IT/RSL is based on the instrument type and usage. Counting equipment is not included in this program due to the nature and usage of this instrumentation. The performance of each instrument is monitored under the calibration program at a frequency appropriate for the characteristics and stability of the individual instrument. If the instrument is found to be performing inadequately, as defined in the calibration procedure for the instrument, it will be removed from service until it has been repaired.

Some other types of laboratory equipment are also under the calibration program and do not require a preventive maintenance schedule. Included in this group are balances and pipettes. When balances are found to be out of calibration, they are repaired and recertified by an external contract service. If pipettes are determined to be out of calibration, they are discarded.

Certain laboratory equipment does require a preventive maintenance program. The schedule for the preventive maintenance of each piece of equipment is based on the equipment and the individual parts which are subject to wear, deterioration, or other change in operational characteristics without periodic maintenance. Included in this group are centrifuges and vacuum pumps.

A listing of the instruments and equipment that are included in the program and the frequency of maintenance shall be recorded on a "Preventive Maintenance Schedule" (Attachment RS7-1). A preventive maintenance record shall be kept on each piece of equipment for all maintenance performed, including external agencies when required. (Attachment RS7-2). (Items to be checked and/or serviced during maintenance are as directed in the equipment manual.)

The Technical Director is responsible for preparation and documentation of the program. Group leaders shall implement the program, and the QC Coordinator shall perform audits and surveillances to verify compliance.

ATTACHMENT RS7-1

PREVENTIVE MAINTENANCE SCHEDULE

Instrument/Equipment	Serial Number	Maintenance Frequency	Maintenance Required	To be Performed By

ATTACHMENT RS7-2

Preventive Maintenance Record

INSTRUMENT: _____

SERIAL NUMBER: _____

MAINTENANCE FREQUENCY: _____

Maintenance Performed by	Date	Comment

TABLE R5/-1

Preventive Maintenance Requirements

Instrument/Equipment	Maintenance Frequency	Items Checked/Serviced	Reference
Manual Alpha/Beta Counter	Monthly	Clean Fan Filter	Manufacutrer's Manual
Gamma Spec System MCA Equipment Rack	 Monthly Monthly	 Clean Fan Filter Check For Operation	 Manufacturer's Manual
Vacuum Pump	Monthly Annually	Clean Fan Filter Change Pump Oil	Manufacturer's Manual
Germanium Detectors	Weekly	Fill LN_2 Dewars	Manufacturer's Manual
Alpha Scintillation Counters	Quarterly	Electronic Check	Manufacturer's Manual

RS8.0 ANALYSIS OF QUALITY CONTROL SAMPLES

This section discusses the analytical quality control program which shall be used to demonstrate that IT/RSL is operating within prescribed requirements for accuracy and precision. Quality control samples of known activities shall be analyzed for each analysis type performed to determine accuracy and precision. This data shall be used in the preparation of control charts for each type of analysis and evaluated as discussed in Section RS10.1. The quality control samples should be analyzed within the normal sample stream. In no case shall the levels of activity used in blind or known spiked samples compromise the health and safety of laboratory personnel nor cause deterioration of the low-level detection capability of counting equipment.

RS8.1 INTRALABORATORY QUALITY CONTROL SAMPLES

Following is a discussion of the types of internal quality control samples used at IT/RSL. QC samples shall be analyzed as described below unless analytical procedures prescribe specific sample analysis be performed. In such case, the procedural requirements will be met.

The intralaboratory quality control samples are summarized in Table RS8-1. Included in the table are:

- Type of sample.
- Purpose of the sample.
- Frequency with which the sample is to be analyzed.
- Whether the sample is used for the statistical evaluation of accuracy and/or precision.
- Person responsible for introducing the quality control sample into the sample stream.

If the sample is introduced by the Quality Control Coordinator, the radionuclide content and the activity are unknown to the technician. Samples unknown to the technician provide independent verification of laboratory operation.

RS8.1.1 Method Blank

A method blank is a volume of demineralized water for liquid samples, or an appropriate solid matrix for soil/sediment samples, carried through the entire analytical procedure. The volume or weight of the blank must be approximately equal to the volume or weight of the sample processed. Analysis of the blank verifies that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware are known and minimized. Results of method blank analyses shall be maintained with the corresponding analytical data in the client file and copies maintained in a QC Master file.

RS8.1.2 Duplicate

A duplicate is a second aliquot of a sample which is approximately equal in mass or volume to the first aliquot and is analyzed for the same parameters. The laboratory performs duplicate analyses to evaluate the precision of an analysis. A QC Duplicate Analysis data sheet (Attachment RS8-1) shall be initiated when the sample is entered into the sample stream. This data shall be submitted with the associated client data to the laboratory review staff for evaluation of the client data and filed in the customer/project file. Copies shall be maintained in the QC master file.

RS8.1.3 Matrix Spike

A matrix spike is an aliquot of a sample which is spiked with a known concentration of the analyte of interest. The purpose of analyzing this type sample is to evaluate the effect of the sample matrix upon the analytical methodology. A QC Spike Analysis data sheet (Attachment RS8-2) shall be initiated when the sample is spiked and shall be submitted with the associated

client data to the laboratory review staff for evaluation of client data and filed in the customer/project file. Copies shall be maintained in the QC master file.

RS8.1.4 Blind Replicate

A blind replicate is a duplicate sample unknown to the technician which has been introduced into the laboratory as a separate sample. These samples are used for the verification of analytical precision.

RS8.1.5 Verification/Reference Standard

A verification or reference standard is a prepared sample of known concentration of a purchased standard reference material. These samples are analyzed in triplicate and the results are used to verify accuracy and precision of the procedure.

RS8.1.6 Blind Replicate Standard

A blind replicate standard is a prepared sample of known concentration of a purchased standard reference material submitted in duplicate as blind samples into the laboratory. These samples verify accuracy and precision of the procedure and technician.

RS8.2 INTERLABORATORY VERIFICATION SAMPLES

It is the policy of IT/RSL that the laboratory participate in laboratory cross-check programs for the independent verification of the laboratory's analytical accuracy and precision. IT/RSL shall participate in the EPA Radiation Quality Assurance Program. The laboratory shall participate in each test as frequently as samples are provided. The laboratory shall participate in other interlaboratory programs where they are deemed meaningful and useful to the scope of the laboratory work being performed.

The QC Coordinator shall be responsible for coordinating interlaboratory programs and reporting results of the associated analyses to the issuing agency.

It shall be the responsibility of the Laboratory Manager to ensure that the laboratory performs the required analyses in a timely manner and that results are reported on time.

Timely performance of the required analyses so that results may be reported on time shall be the responsibility of the laboratory technicians.

RS8.2.1 Data Evaluation

Data returned from EPA or other programs shall be reviewed and evaluated by tne QC Coordinator and the Laboratory Manager on a routine basis. If reported values fall outside two standard deviations, as determined by the issuing agency, the Laboratory Manager shall investigate the analysis in question. If reported values fall outside three standard deviations, an investigation of the analysis shall be performed by the Laboratory Manager and a written explanation of any corrective action deemed necessary shall be sent to the QC Coordinator. A non-conformance report shall be written if the values reported for two consecutive reports of the same analysis and the same matrix fall outside the three standard deviation limit. If such a situation occurs, the Laboratory Manager shall instruct the laboratory staff to discontinue that particular analytical procedure until the non-conformance has been investigated and the corrective action has assured compliance. Other quality control data shall be evaluated to determine the extent of the problem, whether customer data has been effected, and what remedial action including client notification, will be required.

Table RS8-1

INTRALABORATORY QUALITY CONTROL SAMPLE SUMMARY

Type	Purpose of Sample	Frequency	Accuracy & Precision Applicaiton	Introduced by
Reagent Blank	Determine the background of each reagent/solvent to be used in an analysis. Must use identical conditions to actual analysis including detection system. Background must not interfere with intended analysis.	With each new batch of reagent/solvents.	No	Analyst
Method Blank	The analysis is performed using only the reagents and solvents used in the method. Determines cumulative interference. If cumulative interference cannot be eliminated, it must be taken into account when computations are done.	With each group of samples, 1 out of 20 samples, or daily, whichever is more frequent.	Yes	Analyst
Blind Repeat	An aliquot of a sample, unknown to analyst at time of sample log in, is introduced by QC Coordinator (known to QC coordinator)	1 out of 100 or at least once a month.	Yes	QC Coordinator
Duplicate (Replicate)	An aliquot of a sample known to analyst. Calculate rate of percent difference.	1 out of 20 samples	Yes	Analyst
Spiked Samples (Matrix Spikes)	A known concentration of a specific parameter is added to an aliquot of a sample with the matrix of interest. Percent recovery is determined and spike is compared against an unspiked aliquot.	1 out of 20 samples	Accuracy only	Analyst
Standard Reference Materials	Standards prepared by recognized external agency (e.g., NBS) and used to determine performance of entire system instrumentation and analysis.	Scheduled periodic basis per specific lab procedures.	Accuracy only	Analyst or QC Coordinator
Blind Replicate Standard	A replicate standard of fixed concentration added by QC Coordinator, unknown to analyst.	Monthly	Yes	QC Coordinator

ATTACHMENT RS8-1

INTRALABORATORY QUALITY CONTROL DATA SHEET

Duplicate Analysis Data

Project: _____ Sample Type: Water _____

_____ Soil _____

Analytical Method: _____ Sediment _____

Instrument Used: _____ Other _____

(Analytical results reported in d/m or pCi/unit)

SAMPLE#	INIT'LS	DATE ANALYZED	1st ANALYSIS	DATE ANALYZED	2nd ANALYSIS

COMMENTS:

ATTACHMENT RS8-2

INTRALABORATORY QUALITY CONTROL DATA SHEET

Spike Analysis Data

Project: _____ Sample Type: Water _____

_____ Soil _____

Analytical Method: _____ Sediment _____

STANDARD NUMBER: _____ Other _____

SAMPLE #	DATE SPIKED	INIT'LS	(d/m or pCi/unit) ADDED	OBSERVED	DEVIATION

COMMENTS:

RS9.0 ANALYTICAL PROCEDURES

A written operating procedure is the standard against which the quality of the laboratory work is judged. Current, validated, complete procedures shall be maintained and used in the analysis of samples and operations of instruments. These procedures shall be clear and complete written instructions. They shall include the following:

- Prescribed method for sample preparation and holding time, where applicable
- Instrument standardization, including calibration
- Analytical techniques to be used in processing the sample
- Method for computation of analytical results
- Criteria for assessing the quality of the work.

RS9.1 DATA SHEETS

A standard data sheet format for recording raw data shall be prescribed and shall include the following:

- Identification of the customer
- Identification of sample
- Identification of each technician performing work and date performed
- Identification of the person performing data validation and date performed
- Raw data resulting from the analysis
- Calculations performed on raw data.

RS9.2 ANALYTICAL METHODS

Analytical methods are incorporated into Standard Operating Procedures (SOPs) at IT/RSL. Procedures reference source documents published by agencies such as the U.S. Environmental Protection Agency (USEPA), American Public Health Association (APHA) and American Society for Testing and Materials (ASTM) and incorporates the various methods, as applicable, to the particular types of samples analyzed by IT/RSL. These procedures are verified by interlaboratory comparison programs where they exist, and by the IT/RSL QA/QC program. As required by contract, specific methods shall be used.

RS9.3 VARIANCE FROM PROCEDURES

Analyses are performed in accordance with the methods cited in the IT/RSL Standard Operating Procedures Manual unless specific project requirements or needs dictate adoption of an alternate method or modification of the cited methods. Such alternate methods and modifications shall be documented in the customer records.

Any unapproved deviation from approved procedures is a serious breach of laboratory policy and shall require a nonconformance report be written in accordance with Section 13.0 of this manual.

RS10.0 DATA VERIFICATION

As defined in the ITAS QA Manual, data verification is the steps taken within the analytical laboratory to assure that reported results represent the analyses performed. The two basic verification activities are:

- The processing of quality control sample results to demonstrate that analyses are within laboratory-prescribed limits for accuracy, precision, and completeness.
- The validation of data to demonstrate that numerical computation of data is correct and that it is correctly reported.

This section establishes the steps and methods by which both means of verification shall be accomplished at IT/RSL. The responsibilities associated with these activities shall also be discussed.

RS10.1 PROCESSING OF QUALITY CONTROL DATA

This section discusses the analytical treatment of the quality control data resulting from the quality control samples discussed in Section RS8.0. The purposes of these analyses are to determine precision, accuracy, or possible contamination of a sample through the addition of substances required for the process. This information shall then be used to determine the validity of each set of customer sample results.

RS10.1.1 Specific Routine Procedures Used to Assess Data Precision and Accuracy

Following are the samples used for evaluating the precision and accuracy of all data generated within IT/RSL.

- A method blank is prepared and analyzed with each set of samples to determine possible contamination.
- One sample in every twenty or one in each batch is analyzed in duplicate to determine precision.

- One sample in every twenty or one in each batch is spiked and analyzed for the analyte(s) of interest to determine accuracy.

- A blind replicate is introduced by the Quality Control Coordinator at least once monthly to verify precision.

- Standard Reference Materials (SRMs) are introduced periodically into the sample stream by the Quality Control Coordinator to evaluate the testing procedure and the technician's performance.

- Blind replicate standards are introduced into the sample stream by the Quality Control Coordinator to verify precision and accuracy.

When the analyses of sample sets are completed, the results will be reviewed and evaluated to assess the validity of the data set. Review shall be based on the following criteria:

- Method Blank Evaluation - The method blank results are evaluated for high readings characteristic of background contamination. If high blank values are observed, laboratory glassware and reagents should be checked for contamination and the analysis halted until the system can be brought under control. A high background is defined as one which is sufficient to result in a difference in the sample value greater than or equal to the smallest significant digit known to be true.

- Replicate Sample Evaluation - Duplicate sample analysis for the sample set is used to determine the precision of the analytical method for the sample matrix. If the precision value exceeds the warning limit for the given parameter, the Laboratory Manager shall be notified. If the precision value exceeds the control limit, the Laboratory Manager and the Quality Control Coordinator must be notified and the sample set must be reanalyzed.

- Matrix Spike Evaluation - The known value of the spike and the recovered value of the spike are used to calculate the accuracy value expressed as a normalized deviation. If the accuracy value exceeds the warning limits, the Laboratory Manager shall be notified. If the accuracy value exceeds the control limits, the Laboratory Manager and Quality Control Coordinator shall be notified and the sample set must be reanalyzed.

RS10.2 STATISTICAL EVALUATION OF QUALITY CONTROL DATA

IT/RSL quality control data analysis for the assessment of the accuracy and precision of laboratory results shall be an ongoing process administered by the QC Coordinator or designee, using the standard statistical radioanalytic process control techniques recommended by the Environmental Protection Agency.

RS10.2.1 Precision Determination by Range Analysis

Statistical Range Analysis is used to calculate the expected range (Mean Range) and control limits for a replicate result and assess whether a lab replicate result is "in control" or not. A replicate result is "in control" if it lies within the Mean Range + 3 Sigma. Range Analysis is used to determine the precision, or reproducibility, of lab results.

RS10.2.2 Accuracy Determination

Laboratory accuracy is determined by calculating the Expected Precision for a known value (either a Reference Standard Sample or Matrix Sample) and assessing the divergence of the recovered sample result from the Known in terms of statistical control. The Expected Precision is based on the activity level of the known value. IT/RSL uses those limits recommended by US-EPA Los Vegas. Statistical Control Bounds are defined as the "Mean", or Known value, +/- 3 Sigma. A result >3 Sigma or <-3 Sigma is considered to be out of control.

RS10.2.3 Normalized Scaling of QC Results

To expedite the timely assessment of QC data, IT/RSL shall use "normalized" range and deviation statistics for the evaluation of laboratory process control:

1. Normalized Range Analysis Statistics are obtained by scaling the statistical output to a Normalized Mean Range of 1 and a Sigma of 1. A replicate result is said to be "in control" if it lies between 0 and +4 (Mean + 3 Sigma).

2. Normalized Deviation Statistics are obtained by scaling the statistical output to a Normalized Mean of 0 and a Sigma of 1. A deviation from Known value is said to be "in control" if it lies between -3 and +3 (Mean +/-3 Sigma).

The use of normalized scaling allows the QC analyst to track analytic process control on a uniform scale as each QC sample result becomes available. IT/RSL uses statistical data normalization procedures outlined in EPA-600/4-81-004.

RS10.2.4 Control Charts

Laboratory QC results shall be evaluated on a point-by-point basis in the following manner:

- Where the QC result is that of a replicate analysis with no Spike, or Known Value given, a Normalized Range Analysis statistic shall be computed for each result. This statistical value shall be plotted on a scatterplot scaled from zero to positive N, with Normalized Mean Range=1, Upper Warning Limit (UWL)=3 and Upper Control Limit (UCL)=4. The lower bound of zero indicates that the replicate results are identical, and normalized values >4 indicate "outlier" values.

- Where the replicate results are those emerging from a Reference Standard sample, a Normalized Deviation statistic shall be computed and the resulting value plotted on a scatterplot with Mean=0, UWL=+2, UCL=+3, LWL=-2, and LCL=-3. Normalized Deviation values falling outside the Upper and Lower Control Limits (+/-3 Sigma) indicate "outlier" data values. Reference Standard samples shall be analyzed in triplicate, and Normalized Range Analysis Statistics shall also be computed and applied in the same manner as outlined in the preceeding paragraph.

- A Matrix Spike sample result shall be assessed as a "single determination" Normalized Deviation statistic to be plotted on a scatterplot with Mean=0, UWL=+2, UCL=+3, LWL=-2, and LCL=-3. Again, the Control Bounds are from -3 to +3.

The Range Analysis and Normalized Deviation control charts shall be assembled for each type of analysis.

Range Analysis and Normalized Deviation statistical output for QC data shall be preserved on a data form containing the following information in addition to the statistical results:

- Date
- Sample ID
- Analyst ID
- Instrument ID
- Analysis
- Matrix

RS10.2.5

The QC Coordinator or designee shall monitor the ongoing output of individual QC data results for conformance to predetermined acceptance criteria with respect to precision (Range Analysis) and accuracy (Normalized Deviation from Known). Additionally, for the monitoring of process control trends, QC data results shall be analyzed statistically in batch with the accumulation of a minimum of twenty (20) data points. Such data sets shall be processed to yield Means and Control Limits for the assessment of overall laboratory process performance with respect to both precision and accuracy objectives. QC data sets shall further be compared statistically over time to assess the presence or absence of any statistically significant changes in IT/RSL laboratory process control, using accepted radioanalytic process control computational procedures.

RS10.3 Data Validation

Data validation begins with the processing of data and continues through the review of the data and reporting of analytical results. Data processing can be performed by the technician who obtained data or another technician. Data review begins with a reviewer, who is independent of the data acquisition and processing, validating that data has been correctly calculated and appropriate and complete entries have been made. The review continues through the review and approval by the Laboratory Manager (or a designee) and the verification that reported analytical results correspond to the data acquired and processed. This procedure is outlined in Figure RS10-1.

As stated, the first step in validation is data processing. In general, data will be processed by a technician in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to data sheets
- Input of raw data for computer processing
- Direct acquisition and processing of raw data by a computer.

If data are manually processed by a technician, all steps in the computation shall be provided. If calculations are not performed directly on the data sheet, calculations shall be recorded on standard IT/RSL calculation paper and attached to the data sheet. The technician shall sign and date in black, indelible ink each page of calculations.

For data that are input by a technician and processed using a computer, a copy of the input shall be kept and uniquely identified with the sample numbers and other information as appropriate. The technician inputing the data shall initial and date the print-out generated.

If data are directly acquired from instrumentation and processed, the technician shall verify that the following are correct: project and/or sample numbers,

output parameters such as units and counting time. The technician shall initial and date associated sample data sheets.

RS10.3.1 Review of Data Processing

The following is a discussion of the method to be used for reviewing data generated. All data shall be checked in this manner.

- The technicians processing the data shall give the data package to the Laboratory Manager, or his designated reviewer. The package shall include all data sheets, computer print-outs and calculation sheets, as appropriate.

- The independent reviewer shall review the data for:

  - Appropriateneess of equations used
  - Correctness of numerical input
  - Numerical correctness of all calculations by re-performing numerical computations
  - Reasonableness of results for type of analysis performed

- All entries and calculations reviewed shall be marked by a check mark. If the checker disagrees with any part of the computations, a single line shall be drawn through the entry and the revised number written above it.

- Any changes made by the reviewer shall be back-checked by the originator. If the originator agrees with the change, no further action is necessary. However, if the originator disagrees and both parties can not agree, the Laboratory Manager will be required to review data and decide on the correct value.

- The checker shall initial and date the appropriate section of the work sheets and sign and date corresponding calculation sheets, as appropriate. Signing and dating indicates agreement with data as entered. All changes shall be initialed and dated by reviewer and originator.

- If the data have been processed by computer, the reviewer shall check each input entry. Agreement shall be indicated by a check mark. If the reviewer disagrees with the input, the number shall be marked through with a single line and the corrected number entered above it. Corrections must be back-checked by the originator as discussed above.

- If an input error is identified and correction can not be made, the data must be reprocessed. In this event, the checker shall mark the second set of input to indicate agreement with the changes. The checker shall sign and date the computer input to indicate agreement.

RS10.3.2 Data Approval

The Laboratory Manager, or a qualified designee, must approve all sample data including quality control data, before results may be reported. After the data review by the independent reviewer, the Laboratory Manager, or designee, shall review data sheets for the following:

- Completeness and reasonableness of data for type of analysis performed.
- Check appropriate sections for required initials and dates.
- Check that specification and contract requirements have been met.

If the data sheet is complete and data is acceptable, the approval section shall be initialed and dated. The approved data is then submitted to the Data Base Manager for report generation.

If the data is rejected, the work sheets shall be filed in the appropriate customer file for void or rejected data. Notification of the customer and/or re-analysis of the sample shall be initiated by the Laboratory Manager as appropriate.

RS10.3.3 Review of Data Reports

Review of data reports is required to verify that information reported by IT/RSL corresponds with the processed analytical results. Review is only required of the data as it is presented for issuance. The draft report shall be checked against the reviewed data so that transcription errors are corrected, when they occur. This checking process is as follows:

- Using the draft report, data entries are checked against lab work orders and data sheets for customer information and sample identification, analytical results, and reporting units (e.g., pCi/l or µCi/ml).

Incorrect entries shall be circled in red ink with correct information entered beside it. If no corrections are to be made, the reviewer shall sign draft report and return to Data Base Manager for issuance of final report. If corrections are needed, the revised report should be reviewed as described above.

After the checking of the data report is complete, it is submitted to the Laboratory Manager for final review and approval for issue is indicated by signature.

RS10.4 VERIFICATION OF SOFTWARE

If computer software is used to acquire, process, or report data, it is necessary to demonstrate that the software correctly performs its intended function. The methods and requirements of this verification is discussed in section 10.3 of the ITAS Quality Assurance Manual.

DATA VALIDATION PROCESS

Activity	Responsibility
Data Processing	Technician
Checking of Data Processing	Independent Reviewer Designated by Laboratory Manager
Review and Approval of Data	Laboratory Manager or Designee
Review of Draft Report	Data Base Manager or Designee
Final Review and Approval for Issue	Laboratory Manager or Designee

RS11.0 DATA REPORTS

Data reports provided by IT/RSL are generated from a computer data base which contains information on all samples received at the laboratory. The primary software currently in use offers three standard formats from which most client and project needs can be met. However, when the needs are such that the primary program can not acccomodate the requirements, secondary programs are available. In all cases, the reports shall meet the ITAS QA Manual specifications for data report presentation. The reports include the following:

- The final data reports shall be checked in accordance with data verification requirements of Section RS10.0 and shall be approved by the Laboratory Manager.
- Data are presented in tabular format whenever possible.
- Each report is identified with the client name, date of issue, and, if appropriate, the project number and name.
- Data presentation includes:
  - Sample identification number used by IT/RSL and the sample identification provided to the laboratory;
  - Parameters analyzed, reported values with error limits, and units of measure;
  - Detection limit of the analytical procedure, if the reported value is less than the detection limit, unless contract requires less than result with error limit;
  - Data for a given parameter are reported with consistent significant figures for all samples;
  - Results of Quality Control sample analysis, if required;
  - Footnotes referenced to specific data if required to explain reported values.

RS11.1 TRANSMITTAL DOCUMENTS

Data shall be transmitted from the laboratory only by approval of the Laboratory Manager or his designee. If explanatory text is not issued with the analytical results, a letter of transmittal from the Laboratory Manager, or his designee, shall be included which should explain anomalous results.

As necessary, the letter should include:

- Person(s) receiving the data
- Person transmitting the data
- State if the chain of custody was not provided or correct, if any samples were damaged in shipment, if sample containers were inappropriate for analysis, or if volume provided was inadequate for proper analysis
- Brief discussion of samples analyzed and the analytical program
- Discussion of any apparent anomalies
- Discussion of analytical difficulties
- Discussion of any nonconformances which effect data.

RS12.0 RECORDS MANAGEMENT

This section establishes requirements for the records management system at IT/RSL. All records are to be legible, complete, and adequate for the identification of the item involved. Records shall be considered valid only if initialed, signed or otherwise authenticated and dated by appropriate personnel.

Records shall be maintained in facilities which provide a suitable environment to minimize deterioration or damage, prevent loss, and facilitate retrieval without undue delay.

Each file shall have a file index which shall list each record unit within the file. The form to be used for indexing the file is shown as Attachment RS12-1.

RS12.1 CLASSIFICATION

As stated in the ITAS QA Manual, two broad categories exist for laboratory records:

Project Records - Documents which are specific to a project or customer.

Laboratory Operations Records - Documents which demonstrate overall laboratory performance.

IT/RSL records are maintained in accordance with the ITAS QA Manual with the exceptions and additions described in the sections below.

For the purpose of determining record retention requirements, records are further classified as "Permanent" ("Lifetime") or "Nonpermanent". Further discussion of these classifications are provided in Section RS12.5 on Record Retention.

RS12.2 PROJECT AND CUSTOMER RECORDS

When work is performed in support of a project, records are maintained by project. However, most of the work at IT/RSL is not project specific but is long term support of our customers' environmental and personnel monitoring programs. Therefore, files are set up by customer and are often referred to as customer records in various documents. The terms are interchangeable for these purposes as the files are set up identically and either term applies to the same records.

The categories specified in the ITAS QA Manual for project records are used. However, the analytical data is filed in numerical order of the laboratory identification numbers or as required by contract.

Individual file categories are added only as they become necessary. Table RS12-1 provides a list of categories and associated records.

RS12.3 GENERAL LABORATORY OPERATIONS RECORDS

IT/RSL uses a log-in sheet for recording sample receipt which is filed in Section A of these records as each sheet is filled. This log sheet contains sample identification and information provided by the customer and the identification assigned by the laboratory.

All other sections outlined in the ITAS QA Manual are maintained as described. The following sections outline supplemental categories which have been added to these files and the documents which are included in each. Table RS12-2 lists the categories and the means of correlation to the customer files.

RS12.3.1 K - Audit Reports

A separate file shall be maintained for each performance and system audit performed by IT/RSL, ITAS or any customer or outside agency. These files shall include all correspondence, meeting attendance sheets, audit reports, and corrective actions associated with the given audit.

RS12.3.2 L - Controlled Documents

The master copy of each controlled document and each revision issued shall be maintained in this file. A list shall be kept which has the name of each person issued a controlled copy and the name and mailing address of each person's organization. A signed record of receipt shall be maintained for each controlled copy issued. Each revision issued shall have a separate folder in this section. Documents which require controlled issue are listed in Section RS1.0 of this manual.

RS12.3.3 M - Equipment Inventory

A current copy of the equipment inventory list shall be maintained in the file. The list shall include the calibration frequency of each piece of equipment, as appropriate.

RS12.3.4 N - Qualification and Training Records

This file shall include a job description for each type of position in the laboratory, position qualification requirements, individual employee resumes, and training records for each employee. Also included shall be training and qualification status matrices.

RS12.4 REFERENCE DOCUMENTS

Table RS12-3 lists the documents which shall be available in the laboratory. These documents are not included in the laboratory records system and should be

kept where they are used. Reference documents shall be revised and updated as necessary to maintain them as "currently applicable documents".

RS12.5 RECORD RETENTION

As discussed above in section RS12.1, IT/RSL records are classified as "Permanent" or "Nonpermanent" and shall be retained in accordance with their classification. Each record index shall be marked to indicate the classification of the file. Both types of records and their required retention times are discussed below:

- "Permanent" ("Lifetime") records are those which must be maintained for the lifetime of the project or as long as required by contract. These records are to be forwarded to the customer for retention by the customer's records keeping system upon request or at the end of the contract period.
- "Nonpermanent" records are those required to show evidence that an activity was performed in accordance with the applicable requirements but need not be retained for the life of the project. IT/RSL shall maintain these records for seven years or until applicable regulatory requirements are met and the customer's requirements as specified by contract or purchase order are satisfied, where such requirements exist.

RS12.5.1 Permanent Records

The following is a list of "Permanent" Quality Assurance records and shall be retained for the life of the project (Lifetime of plant or nuclear power plants).

- Laboratory procedures and revisions to procedures
- Laboratory calculation sheets
- EPA cross-check worksheets and results
- Gamma Spectra printouts
- Documentation of computer programs writing to the laboratory data base

- Instrument logbooks and control charts
- Field sheets
- Sample receipt logbooks
- Calibration curve preparation material
- Radioactive standard calibration certificates, logbooks, and disposal records
- Results of analysis of standards, blanks, spikes, QC samples, and internal cross-checks

RS12.5.2 Non-Permanent Records

The following is a list of records classified as "non-permanent" and while they shall be retained for a minimum of seven years, shall not be retained for the life of the project or nuclear plant.

- Audit plans, reports, responses, logs, and corrective action documentation
- Nonconformances logs and reports
- Surveillance plans, logs, and reports
- Personnel training records

Prior to record destruction, the QC Coordinator shall review records to determine if the retention time should be extended.

ATTACHMENT RS12-1

FILE INDEX

RECORD CLASSIFICATION: _____
PROJECT NUMBER: _____
PROJECT NAME: _____
CATEGORY: _____

FILE DESIGNATION	DESCRIPTION

TABLE RS12-1

PROJECT RECORDS
FILING CATEGORIES

CATEGORY	PROJECT DESCRIPTION
-	Project Index
A	Correspondence
B	Chain of Custody
C	Field Records
D	Request for Analysis
E	Calibration Records
F	Analytical Data
G	QC Samples
H	Data Reports
I	Project-Specific Requirements
J	Nonconformance
K	QA Plans
L	Miscellaneous

TABLE RS12-2

LABORATORY PERFORMANCE RECORDS
FILING CATEGORIES

Category	Record Description	Correlation To Project Files
--	Index File	--
A	Master Sample Log Books	By date of arrival and sample number
B	Instrument Calibration Logs	By date, instrument serial number, and analyst
C	Instrument Maintenance Logs	By date and instrument serial number
D	Computer Software Verification	By date and software designation
E	Performance Evaluation Records	By date and sample designation
F	Certification Program Records	By name of program, date, and sample designation
G	QC Sample Analysis	By date and sample number
H	Control Charts	By chemical parameter, date, and sample number
I	Purchased Material Certificates	By date and parameter
J	QC Coordinator Reports	By date
K	Audit Reports	By date
L	Controlled Documents	By date
M	Equipment Inventories & Records	By date and instrument serial number
N	Qualifications & Training Records	By name of position, name of personnel, and date

TABLE RS12-3

REFERENCE DOCUMENTS

- Instrument Manuals
- Computer/Software Instruction Manuals

RS13.0 NONCONFORMANCES AND CORRECTIVE ACTIONS

Any condition determined to be adverse to quality shall be subject to corrective action. This includes, but is not limited to, deficiencies and nonconformances. These terms and the method of controlling the corrective actions associated with each are discussed in the following sections.

RS13.1 DEFICIENCIES

A deficiency is an unauthorized deviation from the accepted procedures, practices, or standards, or a defect in an item that could lead to degradation of quality. All laboratory employees are responsible for reporting any observed deficiency suspected of being a nonconformance. A nonconformance report form (Attachment RS13-1) shall be initiated by the employee and given to the QC Coordinator and a copy to the Laboratory Manager. The responsible supervisor(s) shall be notified of the deficiency and shall be responsible for identifying the source of the deficiency and proposing corrective action.

The QC Coordinator shall be responsible for determining if the deficiency is a nonconformance.

RS13.2 NONCONFORMANCES

A nonconformance is a deficiency in characteristic, documentation, or procedure that renders the quality of an item or service unacceptable or indeterminate. Nonconformances can be due to data which outlie accepted bounds for accuracy and precision, improper equipment calibration or maintenance, or improper data verification. Any activity in the laboratory which adversely affects data quality can result in a nonconformance.

The nonconformance control program at IT/RSL covers all lab activities including receiving, sample preparation and analysis, equipment calibration control, records, or any activity that is covered by the requirements of the Quality Assurance Program or contract documents.

The QC Coordinator is responsible for maintaining a nonconformance log. This log shall list each nonconformance along with the proposed and actual dates that the corrective action is completed and verified. Attachment RS13-2 presents an example of the nonconformance log. A copy of the nonconformance and the corrective actions shall be filed in the customer file when it is specific to an individual customer(s).

Nonconforming equipment or samples will be controlled by marking and physical segregation. Where physical segregation is not practical, nonconforming items may be tagged, marked, or identified as nonconforming by other means.

RS13.3 CORRECTIVE ACTIONS

A corrective action is a measure taken to correct a deficiency and to minimize the possibility of recurrence. Corrective actions shall be instituted for any situation identified as a condition adverse to quality and shall be documented and reported to appropriate levels of management. Procedures will assure that conditions adverse to quality such as failures, malfunctions, deficiencies, deviations, and nonconformances are promptly identified and corrected.

RS13.4 DEFECTS AND NONCOMPLIANCE PER 10 CFR PART 21

Each employee of IT/RSL is responsible for notifying appropriate management of conditions which the employee perceives as potential defects or noncompliances as identified by 10 CFR 21. This notification shall be made by completing a "Notice of Anomaly" form (Attachment RS13-3). The report shall be given to the Laboratory Manager, who shall submit the report to the Laboratory Director, or his designee. The Laboratory Director or a designated representative shall verbally inform the customer of the possible defect or non-compliance within twenty-four (24) hours and in writing within five (5) days of its identification.

The customer is responsible for evaluating the report and, if appropriate, notifying the Nuclear Regulatory Commission.

The QC Coordinator shall post in a conspicuous location(s) Section 206 of the Energy Reorganization Act of 1974 and a notice which describes the regulations/procedure and indicates where they may be examined. It should also state to whom reports may be made. A copy of the posting is shown in Attachment RS13-4. A flow chart of the noncompliance reporting sequence is provided in Figure RS13-5.

ATTACHMENT RS13-1
Nonconformance Report
(Service and Activities)

Service or Activity:_____ NRC No.:_____

Date Discovered: __/__/__
Location: _____

Nature of Nonconformance: Description of Nonconformance and Apparent Cause:

( ) Procedural Deficiency _____
( ) Data Deficiency _____
( ) Instrumentation Problem _____
( ) Documentation Problem _____
( ) Material Problem _____
( ) Other_____ _____

Responsible Manager	Originator/Date	Quality/Date
Disposition: ( ) Accept Deviation (Use as is) ( ) Modify Plan/Procedure ( ) Repeat Service/ Activity ( ) Terminate ( ) Reject ( ) Waiver ( ) Rework ( ) Repair ( ) Conditional Acceptance* *State Conditions		

Underlying Cause:

Cause Code [ ][ ]

Action to Prevent Recurrence:

01 Medical Failure
02 Personnel Failure
03 System Failure
04 Material Failure
05 Process Failure

Corrective Action Verifiction:
( ) Verified (Note any appropriate conditions): _____

( ) Cannot Verify (Note reasons for lack of verification): _____

QA Coordinator

Date: ___/___/____

(Use space below for comments or extensions to the above topics.)

ATTACHMENT RS13-2

NONCONFORMANCE LOG

NUMBER	DATE ISSUED	RESPONSIBLE UNIT/MGR.	CORRECTIVE ACTION COMPLETION DATE		VERIFICATION DATE	
			TARGET	ACTUAL	TARGET	ACTUAL

ATTACHMENT RS13-3

NOTICE OF ANOMALY	DATE:

NOTICE NO:_____ P.O. NO:_____ CONTRACT NO:_____

CUSTOMER:_____ JOB NO:_____

NOTIFICATION MADE TO:_____ NOTIFICATION DATE:_____

NOTIFICATION MADE BY:_____ VIA:_____

CATEGORY: ____SPECIMEN ____PROCEDURE ____TEST EQUIPMENT DATE OF ANOMAL:_____

SAMPLE:_____ SAMPLE NO:_____

TEST:_____ I.D. NO:_____

SPECIFICATION:_____ PARA NO:_____

REQUIREMENTS:

DESCRIPTION OF ANOMALY:

DISPOSITION - COMMENTS - RECOMMENDATIONS

TECHNICIAN:_____ VERIFICATION: _____

LABORATORY MANAGER: _____

LABORATORY DIRECTOR:_____

ATTACHMENT RS13-4

FEDERAL LAW Concerning the Reporting of
NONCOMPLIANCES AND DEFECTS
in Products/Services Subject to Regulation
by the NUCLEAR REGULATORY COMMISSION

Section 206 of the Energy Reorganization Act of 1974, as amended, is as follows:

(a) Any individual director, or responsible officer of a firm constructing, owning, operating, or supplying the components of any facility or activity which is licensed or otherwise regulated pursuant to the Atomic Energy Act of 1954, as amended, or pursuant to this Act, who obtains information reasonsbly indicating that such facility or activity or basic components supplied to such facility or activity -

(1) Fails to comply with the Atomic Energy Act of 1954, as amended, or any applicable rule, regulation, order, or license of the Commission relating to substantial safety hazards, or

(2) Contains a defect which could create a substantial safety hazard, as defined by regulations which the Commission shall promulgate, shall immediately notify the Commission of such failure to comply, or of such defect, unless such person has actual knowledge that the Commission has been adequately informed of such defect or failure to comply.

(b) Any person* who knowingly and consciously fails to provide the notice required by subsection (a) of this section shall be subject to a civil penalty in an amount equal to the amount provided by section 234 of the Atomic Energy Act of 1954, as amended.

(c) The requirements of this section shall be prominently posted on the premises of any facility licensed or otherwise regulated pursuant to the Atomic Energy Act of 1954, as amended.

(d) The Commission is authorized to conduct such reasonable inspections and other enforcement activities as needed to insure compliance with the provisions of this section.

Regulation 10CFR21 for implementing the foregoing requirement of the law was promulgated by the Nuclear Regulatory Commission on June 6, 1977. This regulation sets forth the kinds of non-compliance or defect situations which suppliers must report to the Commission, and requires suppliers to institute various measures in order that the Commission can effectively enforce the reporting requirement.

ATTACHMENT RS13-4
(Continued)

One such requirement is that each supplier subject to the regulation must establish a Procedure for dealing with deviations, and for informing its responsible officer of any resulting defect or failure to comply.

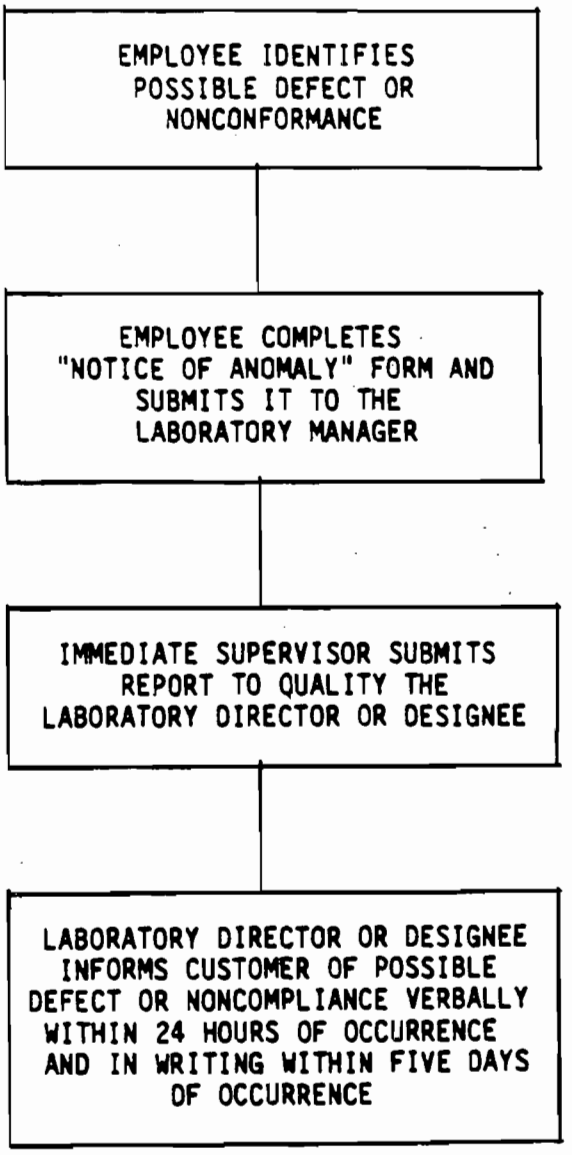
A copy of the full text of Regulation 10CFR21, and of the Procedure to be followed in this facility pursuant thereto, is available for examination by any employee in the Quality Assurance Office, Building 4.

ANY EMPLOYEE WHO HAS REASON TO BELIEVE THAT GOODS OR SERVICES SUBJECT TO REGULATION BY THE NUCLEAR REGULATORY COMMISSION HAVE BEEN DELIVERED FROM THIS FACILITY WHICH ARE NONCOMPLIANT OR DEFECTIVE, AS DEFINED IN SECTION 206 ABOVE, OR REGULATION 10CFR21, IS HEREBY INSTRUCTED TO SO INFORM THE LABORATORY MANAGER.

* "Any person" in this paragraph refers to the director or responsible officer identified in Paragraph (a).

FIGURE RS13-1

PROCEDURE FOR REPORTING OF DEFECTS AND NONCOMPLIANCES PER 10 CFR PART 21



RS14.0 QUALITY ASSURANCE/QUALITY CONTROL AUDITS

The IT Analytical Services' Quality Assurance Manual requires that two types of audits be performed at each laboratory. These audits are:

- Performance audits - conducted on an ongoing basis within the laboratory by the Quality Control Coordinator. These audits are reported to the Laboratory Manager and the ITAS Quality Assurance/Quality Control Director.
- System audits - performed on a scheduled, periodic basis by the ITAS Quality Assurance/Quality Control Director. These audits are external to the laboratory and are reported to the Vice President, IT Analytical Services, and Laboratory Manager.

RS14.1 PERFORMANCE EVALUATIONS

The Quality Control Coordinator is responsible for the performance audits of IT/RSL. In addition to preparation and evaluation of quality control verification samples, the QC Coordinator shall perform ongoing surveillances and audits.

RS14.1.1 Surveillances

Monthly surveillances shall be performed according to a semiannual schedule which shall encompass all areas of laboratory activities prescribed in the IT/RSL Laboratory Specific Attachment for which the QC Coordinator is not directly involved. Surveillance checklists, as shown in Attachment RS14-1, shall be completed and maintained in the quality records files. Any deficiency observed during a surveillance shall be reported to the appropriate laboratory supervisor and documented per Section RS13.0 of this manual attachment.

Surveillance schedules shall be maintained in the quality records files. A Surveillance Schedule form is shown in Attachment RS14-2. This schedule is intended to be a guide for covering all areas of laboratory operations and may be adjusted as necessary to monitor areas where deficiencies are observed.

RS14.1.2 Audits

Internal audits shall be performed to provide for a full audit every two months. An annual audit schedule shall be maintained in the quality records files. An audit schedule form is provided in Attachment RS14-3. As much as applicable, the audit system described in Section 14.2 of the ITAS QA Manual shall be followed for the preparation, performance, reporting and follow-up of the IT/RSL internal audits. This shall include the preparation of an audit checklist and formal audit report. Objective evidence will be examined for compliance with the Quality Assurance Program requirements. Attachment RS14-4 is a sample cover sheet for the audit checklist.

Any deficiency discovered during an audit shall be identified to the Laboratory Manager during the close of the audit and shall be written as an audit finding which shall require a written response describing the proposed corrective action. Findings shall be written on a standard Audit Action Report form as shown on Attachment RS14-5. Subsequent surveillances shall monitor the area of the deficiency to assure effectiveness of the corrective action. A follow-up to corrective actions shall be included in the next scheduled audit. The audit report shall follow a standard format which is provided in Attachment 14-6.

RS14.2 SYSTEM AUDITS

System audits shall be conducted by the ITAS Quality Assurance/Quality Control Director or designee. The audits shall be on a semiannual basis and provide a thorough overview of the implementation of the Quality Assurance Program. On an annual basis the Quality Assurance Program shall be evaluated for effectiveness and completeness. The system audit program is discussed in detail in Section 14.2 of the ITAS Quality Assurance Manual.

ATTACHMENT RS14-1

Reviewer:_____

Date:_____

Page:_____

IT/Radiological Sciences Laboratory

Quality Assurance

Surveillance Checklist

Attribute or Condition Checked	N/A	Satisfactory	
		YES	NO

ATTACHMENT RS14-2

IT/Radiological Sciences Laboratory

Surveillance Schedule

Calendar Year:_____ Prepared by:_____

Date Issued:_____ Q.A. Coordinator

Date Revised:_____

Surveillance Number	Subject & Scope	Dates		Performed by
		Target	Actual	

ATTACHMENT RS14-3

YEARLY AUDIT PLAN AND SCHEDULE

IT/Radiological Sciences Laboratory
Audit Plan and Schedule

Calendar Year: _____	Prepared by: _____ Quality Assurance
Date Issued: _____	
Date Revised: _____	Approved by: _____

Audit Number	Subject and Scope	Auditor(s)	Dates		
			Target	Actual	Report

ATTACHMENT RS14-4

INTERNAL AUDIT CHECKLIST

IT/RADIOLOGICAL SCIENCES LABORATORY

SHEET 1 of _____

INTERNAL AUDIT CHECKLIST

1.0 AUDIT SCOPE: _____

_____

2.0 AUDIT PURPOSE: _____

_____

3.0 APPLICABLE DOCUMENTS: _____

_____

4.0 AUDIT CHECKLIST: Prepared By: _____ Date: _____

Approved By: _____ Date: _____

QUESTIONS	AUDITOR'S COMMENTS

ATTACHMENT RS14-5

AUDIT ACTION REPORT

Audit Number:_____
AAR Number:_____

To:_____ Title:_____

Organization:_____

Description of Finding Discovered by Audit Team:

Signature:_____ _____
Lead Auditor - Date Quality Coordinator

Complete Items 1, 2, & 3 Within ____ Days and Return to Lead Auditor.

1) Root Cause For Discrepancy:

2) Corrective Action Plan (Including Action to Prevent Recurrence):

3) Corrective Action
Will Be Completed By: _____ Signature:_____
Cognizant Mgr. - Date

Evaluation of Proposed Corrective Action:

Signature:_____ _____
Lead Auditor - Date ITAS QA/C Director - Date

Follow-Up and Final Close-Out:

Signature:_____ _____
Lead Auditor - Date ITAS QA/C Director - Date

ATTACHMENT RS14-6
AUDIT REPORT FORMAT
(TYPE OF AUDIT PERFORMED) (AUDIT NUMBER)

1.0 OBJECTIVE

Short paragraphs that outline the objectives of the audit.

2.0 SCOPE

A short paragraph that states when the audit was performed, who performed it, what areas were reviewed, and the names of the key persons contacted.

3.0 CONCLUSIONS

3.1 A sentence or short paragraph, separately numbered for each major conclusion. The conclusions shall directly relate to the objective(s) of the audit as stated in "1.0 OBJECTIVE".

3.2 A sentence or short paragraph detailing the status (if applicable) of corrective/preventive action of previous audits.

3.3 A short sentence detailing how many attributive checks were made and how many were found to be nonconforming.

4.0 OBSERVATIONS

The following statement will be entered in this section of the report:

"An audit report is attached describing each finding"

Audit Performed By:

Name:_____

Title:_____

Lead Auditor:

Name:_____

Approved By:

Name:_____

Title:_____

RS15.0 QUALITY REPORTS TO MANAGEMENT

Reports to various levels of management are required for the verification of the implementation of the Quality Assurance Program and as an indication of the effectiveness of the overall program. These reports are described in the following sections.

RS15.1 PERFORMANCE AUDIT REPORTS

A monthly report of quality related the activities at IT/RSL will be submitted to the ITAS Quality Assurance/Quality Control Director. This report shall include monthly surveillance summaries, nonconformance reports, and quality control data summaries. The Laboratory Manager shall receive a copy of this report also. A copy of this report shall be maintained in the quality records files.

The audit reports for internal performance audits shall be submitted for approval to the ITAS Quality Assurance/Quality Control Director. Upon approval, the report shall be distributed to the Laboratory Manager and the Laboratory Director. The Laboratory Manager shall sign and date his copy to demonstrate review, and the copy shall then be maintained in the quality records files.

RS15.2 SYSTEM AUDIT REPORTS

Audit reports received from the ITAS semiannual audits shall be issued and maintained according to Section 15.2 of the ITAS QA Manual.

RS15.3 NONCONFORMANCES/CORRECTIVE ACTION RESOLUTION

Differences concerning cited nonconformances, requests for corrective action, or the completion of corrective action which can not be resolved directly between the Laboratory Manager and the Quality Assurance Coordinator shall be decided by the Laboratory Director.

RS16.0 PERSONNEL QUALIFICATION AND TRAINING REQUIREMENTS

An important factor affecting all laboratory activities is the training and qualification of those doing the work. The qualification requirements for each technical position at IT/RSL shall be stated in an approved job description. Job descriptions shall be reviewed annually by the QC Coordinator to assure currency of the document and compliance to requirements for each position. Training shall be provided both on the job and in more formal settings. The latter is provided through such means as seminars, vendor's instrument training, in-house courses, and company funding of accredited courses after hours.

RS16.1 QUALITY ASSURANCE TRAINING

Orientation and indoctrination to the IT/RSL Quality Control Program shall be provided to all technical and management level personnel hired at IT/RSL. This training shall be the responsibility of the Quality Assurance Coordinator. Training shall be documented on a standard form (attachment RS16-1) and maintained in a training file for each individual.

Training in the nature and goals of the Quality Assurance Program shall be provided at least once a year to all laboratory personnel by the Quality Assurance Staff. Attachment RS16-2 is the Attendance Record form which shall be used to document the in-house personnel training.

The Quality Assurance staff at IT/RSL shall receive training prescribed by the ITAS QA/QC Director. Documentation of this training shall be maintained in the personnel training files.

RS16.2 TECHNICAL TRAINING AND PROCEDURAL QUALIFICATION

An annual training schedule shall be prepared by the Technical Director to establish a training plan for laboratory personnel. This schedule may be for both in-house and external training.

Training shall also be performed for all procedures which an individiual will need in order to perform a job assignment properly. These procedures shall include general laboratory operations and analytical and counting methodology. Each technician shall meet basic criteria and be qualified by the Laboratory Manager before being permitted to perform activities without supervision. The laboratory qualification process includes:

- Observing procedure being performed by a trained technician;
- Satisfactorily performing work under the supervision of a trained individual;
- Successfully completing analysis of spiked samples for results to fall within two standard deviations of the mean determined for the method;
- Periodic rechecks by analyzing spiked samples.

Technicians who have not performed an analysis within a period of one year, shall be required to re-qualify through spiked sample analysis before being allowed to perform the analytical work again. Criteria shall be set for maintaining qualification based on spike sample results.

The Laboratory Manager shall perform an annual review and, as appropriate, re-qualify each technician for performing specific analytical procedures. The QC Coordinator shall monitor the qualification status of each technician based on spike results and length of time since the analysis has been performed by each technician. A quarterly status report shall be provided to the Laboratory Manager.

Written procedures shall detail the qualification process and criteria to be used.

ATTACHMENT RS16-1

Quality Assurance Orientation Session Checklist

Name: _____
Job Title: _____
Date of Hire: _____

Quality Assurance Program Section	Reviewed
Statement of Management Position	
Quality - Related responsibilities for job title: Section or Topic _____ _____	_____ _____
Nonconformances and Corrective Action (including 10 CFR 21)	
Data Entries and Corrections	

I attended the session covering the Quality Assurance sections or topics as described above.

_____ Employee Signature _____ Date

_____ Q.A. Coordinator Signature _____ Date

Recheck on _____ Date indicates

☐ Training was adequate.

☐ Further Training is needed.

_____ Q.A. Coordinator Signature

Follow-up Training Session Covered:

_____

_____

_____ Employee Signature _____ QC Coordinator Signature _____ Date

ATTACHMENT RS16-2

ATTENDANCE RECORD

PERSONNEL DEVELOPMENT SESSION

Date: _____ Time: _____

Title of Session: _____

_____

Description of Session: _____

_____

Session Conducted By: _____

_____

Name	Signature	Name	Signature

Approved: _____ Date: _____

GLOSSARY OF TERMS

Included below are definitions of certain quality-related terms used in this document.

Acceptance Criteria - Specified limits placed on characteristics of an item, process or service defined in codes, standards or other requirement documents.

Anomaly - A deviation from established requirements, methods or sequence, malfunction or other conditions which may influence test data or test performance.

Approval - An act of endorsing or adding positive authorization or both.

Audit - A documented activity performed in accordance with written procedures or checklists to verify, by examination and evaluation of objective evidence, that applicable elements of the quality assurance program have been developed, documented and effectively implemented in accordance with specified requirements. An audit should not be confused with surveillance or inspection for the sole purpose of process control or product acceptance.

Certificate of Conformance - A document signed by an authorized individual certifying the degree to which items or services meet specified requirements.

Condition Adverse to Quality - An all inclusive term used in reference to any of the following: failure, malfunctions, deficiencies, and nonconformances. A significant condition adverse to quality is one which is generic in nature to a large number of items or is a deficiency in the quality program.

Corrective Action - A measure taken to correct a deficiency and to minimize the possibility of recurrence.

Deficiency - An unauthorized deviation from accepted procedures, practices, or standards; or a defect in an item that could lead to degradation of quality.

Deviation - A departure from specified requirements.

Document - Any written or pictorial information describing, defining, specifying, reporting, or certifying activities, requirements, procedures or results. A document is not considered to be a Quality Assurance Record until it satisfies the definition of a Quality Assurance Record.

Inspection - The act of examining, observing or measuring the degree of conformance of materials, supplies, components, parts, appurtenances, systems, processes or structures to approved quality requirements.

Measuring and Test Equipment (M&TE) - Devices or systems used to calibrate, measure, gage, test or inspect in order to control or to acquire data to verify conformance to specified requirements.

Nonconformance - A deficiency in characteristic, documentation or procedure that renders the quality of an item or activity unacceptable or indeterminate.

Procedure - A document that specifies or describes how an activity is to be performed. It may include methods to be employed, equipment or materials to be used and sequence of operations.

Qualification (Personnel) - The characteristics of abilities gained through education, training or experience, as measured against established requirements, such as standards or tests, that qualify an individual to perform a required function.

Quality Assurance (QA) - All those planned and systematic actions necessary to provide adequate confidence that an item will perform satisfactorily in service.

Quality Assurance Record - A completed document that furnishes evidence of the quality of items and/or activities affecting quality.

Quality Control - Those quality assurance actions which provide a means to control and measure the characteristics of an item, process, or facility to established requirements.

Raw Test Data - Materials such as engineering logs, oscillograph and strip charts, computer printouts or other materials which were generated because of a test, qualification or analysis program but which, from a good engineering standpoint, need not be included in a report of results to satisfy customer requirements.

Safety Related - The name applied to structures, systems, components, materials, services or activities within a nuclear power plant to assure: the integrity of the reactor coolant pressure boundary; the capability to effect a safe reactor shutdown; the capability to prevent or mitigate an accident with potential for exposure; and the operation of the facility within technical specification limits or Federal Regulations.

Special Process - A process, the results of which are highly dependent on the control of the process or the skill of the operators, or both, and in which the specified quality cannot be readily determined by inspection or test of the product.

Standard - The result of a particular standardization effort approved by a recognized authority.

Surveillance - A review (monitoring) or observation of ongoing work for the purpose of verifying that an action has been accomplished as specified.

Testing - Verification of the capability of an item to meet specified requirements by subjecting it to a set of physical, chemical, environmental or operating conditions.

Verification - An act of confirming, substantiating and assuraing that an activity or condition has been implemented in conformance with the specified requirements.

Waiver - Documented authorization to depart from specified requirements.

APPENDIX D

IT ANALYTICAL SERVICES QUALITY ASSURANCE MANUAL

IT Analytical Laboratory

Knoxville (Middlebrook Pike), Tennessee

INTERNATIONAL TECHNOLOGY CORPORATION

Quality Assurance Manual
Laboratory - Specific Attachment

IT ANALYTICAL SERVICES - KNOXVILLE
(MIDDLEBROOK PIKE)

Approved for Issue:

Elizabeth Towery
ITASK QC Coordinator

Kerry Klemm
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Corporate Director of
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Nancy Seabolt
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Southeastern Regional Director

Linda Pocratsky Reeves, Ph.D.
ITAS Director of Quality and
Compliance

Copy No. Uncontrolled

ABBREVIATIONS

ITASK - IT Analytical Services, Knoxville (Middlebrook Pike)

CLP - USEPA's Contract Laboratory Program

SOP - Standard Operating Procedure

TABLE K1-1

Contents of Laboratory-Specific Attachment
Knoxville Laboratory Operations

Sections of ITAS Quality Assurance Manual	Content	Attachment Sections
1.0	Introduction	K1.0
2.0	Laboratory Organization	K2.0
3.0	Standard Laboratory Practice	K3.0
4.0	Material Procurement and Control	K4.0
5.0	Sample Receipt and Initiation of Testing Program	K5.0
6.0	Calibration Practices	K6.0
7.0	Preventive Maintenance	K7.0
8.0	Analysis of Quality Control Samples	K8.0
9.0	Analytical Procedures	K9.0
10.0	Data Verification	K10.0
11.0	Data Reports	K11.0
12.0	Records Management	K12.0
13.0	Nonconformances and Corrective Action	K13.0
14.0	Quality Assurance/Quality Control Audits	K14.0
15.0	Quality Reports to Management	K15.0
16.0	Training	K16.0

K1.0 INTRODUCTION

Criteria for Quality Assurance practices within International Technology Analytical Services laboratories are documented in the IT Analytical Services Quality Assurance Manual. The ITAS Manual was written to encompass all analytical laboratories within IT Corporation. The purpose of the ITAS Manual is to assure uniform Quality Assurance practices among all (IT) laboratories; however, individual laboratory practices will vary from laboratory to laboratory.

The ITAS Knoxville, Tennessee (Middlebrook Pike) laboratory has written a "laboratory-specific" manual. This manual is an attachment to the ITAS Manual. As required by the ITAS Quality Assurance Program, this attachment is a supplement to and is incorporated with the ITAS Quality Assurance Manual to provide the Quality Assurance Program for the Knoxville laboratory operations (ITASK). Refer to the ITAS Quality Assurance Manual for specifics on issues not addressed in this attachment.

For the Knoxville laboratory operations, the ITAS Quality Assurance Manual and this attachment have been prepared to fulfill the policies of IT Corporation, the requirements of the US EPA and the State of Tennessee.

K1.1 Format of the Attachment

The laboratory-specific attachment is formatted in the same manner as the ITAS Quality Assurance Manual so that personnel in Knoxville laboratory operations can readily merge the requirements of the ITAS Manual and this document. Section headings for both documents are the same with the exception that this attachment uses the section prefix K so that attachment sections are clearly identified.

This attachment is not meant to be a self-standing document, the ITAS Manual must be included with the attachment to fully describe the laboratory-specific Quality Assurance Program. The ITASK attachment includes only those sections of the ITAS Quality Assurance Manual which must be supplemented for full documentation of a specific activity, or which provide for the parallel implementation of Quality Assurance practices. In the event that the attachment describes an alternative means for implementation, when compared with the manner prescribed in the ITAS Quality Assurance Manual, the implementation described in this attachment shall take precedence for Knoxville laboratory operations. Contractual obligations will, of course, supersede both the ITAS QA Manual and this supplement. Review and approval of the attachment, as prescribed in this Manual is in accordance with the requirements of the ITAS Quality Assurance Program.

Table K1-1 lists the sections of the ITAS Quality Assurance Manual. The sections contained in this attachment are also identified. If a section is not contained in the attachment, the corresponding section in the ITAS Manual is adopted without supplement or change. Subsections are not listed in Table K1-1, but are fully identified in the Table of Contents.

TABLE K1-1

Contents of Laboratory-Specific Attachment
Knoxville Laboratory Operations

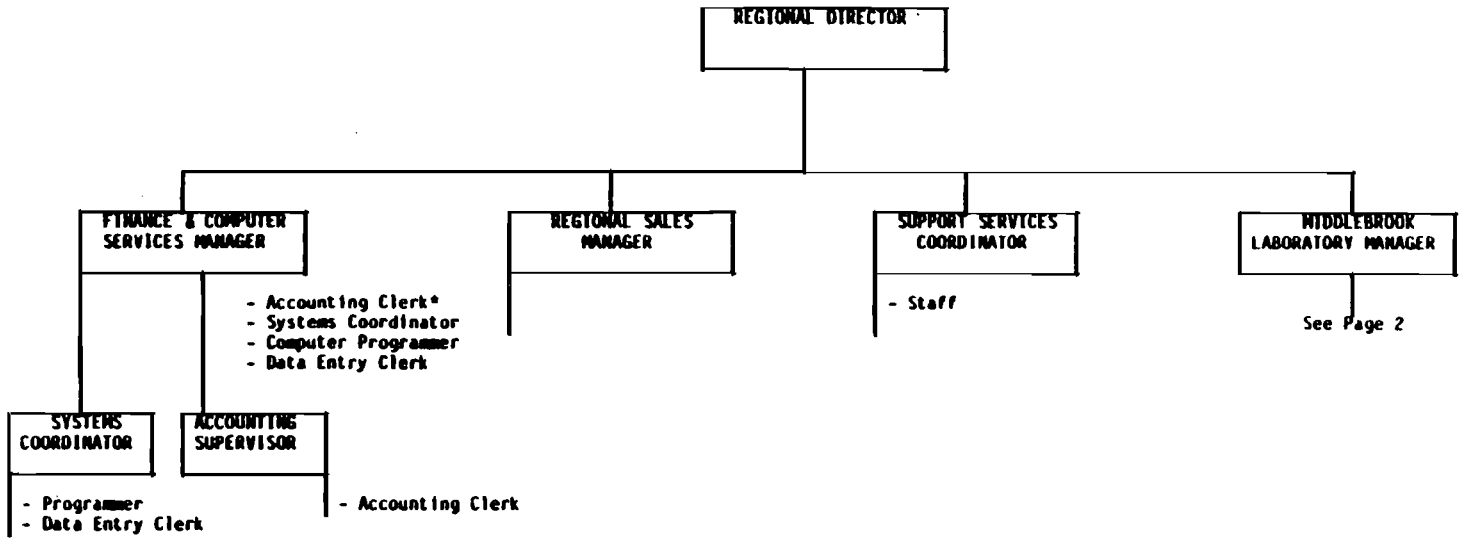
Sections of ITAS Quality Assurance Manual	Content	Attachment Sections
1.0	Introduction	K1.0
2.0	Laboratory Organization	K2.0
3.0	Standard Laboratory Practice	K3.0
4.0	Material Procurement and Control	K4.0
5.0	Sample Receipt and Initiation of Testing Program	K5.0
6.0	Calibration Practices	K6.0
7.0	Preventive Maintenance	K7.0
8.0	Analysis of Quality Control Samples	K8.0
9.0	Analytical Procedures	K9.0
10.0	Data Verification	K10.0
11.0	Data Reports	K11.0
12.0	Records Management	K12.0
13.0	Nonconformances and Corrective Action	K13.0
14.0	Quality Assurance/Quality Control Audits	K14.0
15.0	Quality Reports to Management	K15.0
16.0	Training	K16.0

K2.0 ORGANIZATION

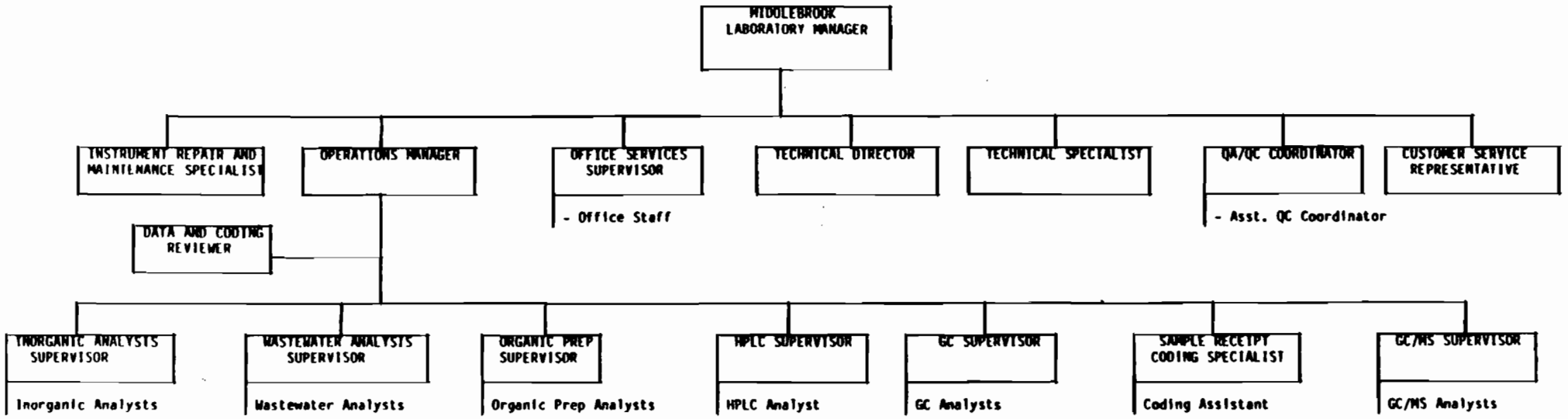
K2.1.1 Quality-Related Responsibilities

The Middlebrook Pike laboratory is part of the ITAS Analytical Services group in the Knoxville area. The organization of the Middlebrook laboratory is shown in Figure K2-1. The Quality Assurance responsibilities of the positions sited are as described in Section 2.1 of the ITAS Quality Assurance Manual.

IT ANALYTICAL SERVICES
KNOXVILLE
PC-4620



IT ANALYTICAL SERVICES
KNOXVILLE
PC-4620



K3.0 LABORATORY PRACTICE

Knoxville laboratory practices and operating procedures are described throughout the Knoxville specific QA Manual. Table K3-1 briefly describes the laboratory functions relating to the flow of samples through the laboratory. The table also indicates the party responsible for sample movement within the laboratory system. Figure K3-1 presents a flow chart of functions for laboratory analysis.

The following manual sections address specific laboratory procedures and practices.

TABLE K3-1

ANALYSIS FLOW CHART
KNOXVILLE LABORATORY OPERATION

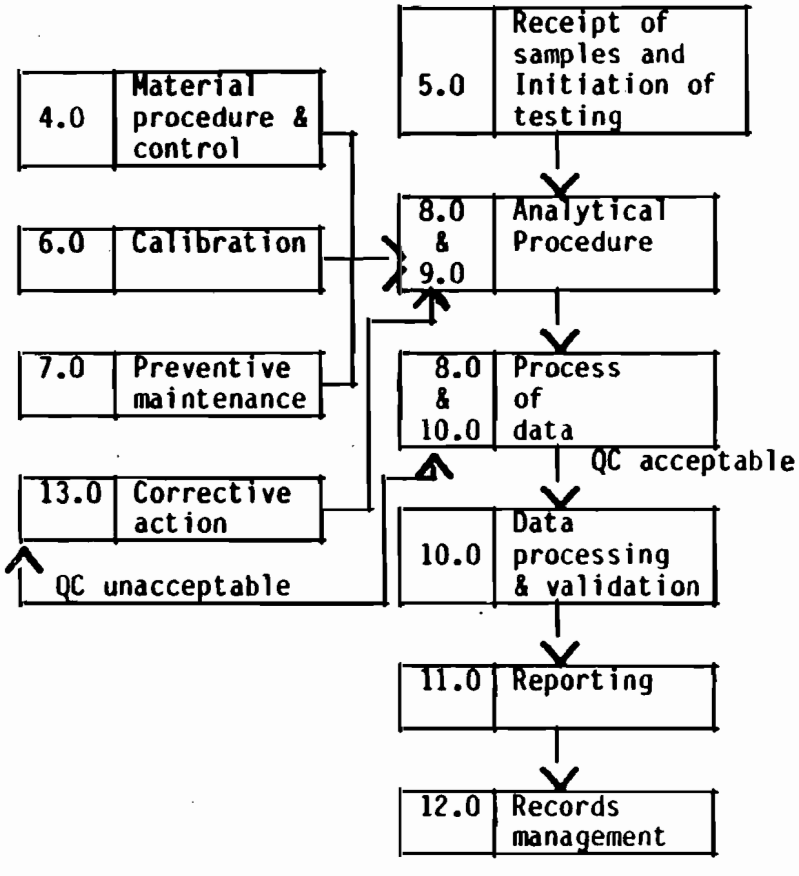
STEP #	DESCRIPTION	PERFORMED BY	DOCUMENTS USED
1	Samples sent to laboratory.	Client	Sample Chain-of-Custody, Request for Analysis
2	Sample is unpacked and inspected and paperwork verifying completeness.	Sample handlers and sample receptionist	Above plus any correspondence, quotations, etc.
3	A project record file is prepared, job assigned, all sample identification placed with sample in temporary storage.	Sample Custodian	Project file, samples labels
4	Project information is recorded into manual log and billing information is recorded.	Customer Service Representative	Billing information
5	Sample is placed in proper storage area with proper labels, and computer is updated with project information.	Sample Custodian	Computer data system records project file
6	Copies of job information are distributed to analytical group leaders for and test assignment processing.	Sample Custodian	Project file, Request for Analysis
7	Chemists collect samples from storage area and perform test.	Group Leaders/Analysts control removal and return	Analysis log, instrument maintenance log, data sheets

TABLE K3-1 (continued)

STEP #	DESCRIPTION	PERFORMED BY	DOCUMENTS USED
8	Analytical data is reviewed and transcribed onto data reporting sheets for typing.	Analysts or Group Leaders	Instrument calibration log, Q.C. log, data sheets
9	Analytical results to Group Leader on completion of testing protocol.	Analysts	Copies of data reporting sheet, analytical data, QA/QC results
10	Data is reviewed and approved and paperwork is placed in project file.	Group Leaders	Copies of data reporting sheet, analytical data, QA/QC results, project file
11	QC data is copied for QC Coordinator for entry into control charts.	Group Leader QC Coordinator	QA/QC results
12	Analytical data for the report is compiled and reviewed and contents of project file reviewed and approved.	Operation Manager or Project Manager	Project file
13	Analytical report is prepared.	Word processing or EDT	Final report
14	Final report is reviewed and signed by Lab Manager or Operations Manager.	Management	Signed report
15	All records and documents are maintained.	Management	All documents
16	Sample is retained for 2 weeks after report issued and then properly disposed.	Sample Custodian	Request for hazardous chemical lab disposal

FIGURE K3-1

LABORATORY ANALYSIS FLOW CHART
KNOXVILLE LABORATORY OPERATIONS



Activities	Applicable Steps For Analytical Samples
° Complete Chain-of-Custody	
° Sample log in	1,2,3,4
° Define tests to be performed on specific samples	5,6
° Define QC sample requirements	5,6
° Analysis of samples	7
° Analysis of QC samples	7
° Summarize QC data	11
° Update control charts	11
° Statistical treatment of data	10
° Calculate analysis results	7,8
° Independent data review	9,10,12
° Summarize data	10,12
° Issue within IT or to client	13,14
° Prepare project file	10,12,15
° Maintain records	15

NOTE: 1. See TABLE K3-1

K4.0 MATERIAL PROCUREMENT AND CONTROL

This section is to provide additional detail to the ITAS Manual Section 4.0.

The duties specified in the ITAS QA Manual concerning purchasing materials and controlling them in the laboratory are assigned to the Group Leaders. Each Group Leader is responsible for materials used in his/her area.

Routinely purchased materials are tracked by a computer inventory program. This program includes a list of materials, reorder points and order sizes. A vendor catalog number for each item is also included. The catalog number defines the grade or other specifications that must be met. Purchases are made by the purchasing department and tracked using this system.

Purchase requisitions are filled out for materials not in the inventory system. Material specifications, with Group Leader approval, are stated on the requisition. The material specifications may consist of a vendor catalog number. The requisition is approved by the appropriate Group Leader and then goes to the purchasing department. Orders are placed by the purchasing department.

When materials are received, they are checked by the appropriate Group Leader or designee. The person receiving the shipment signs and dates the packing slip indicating approval of the material. The packing slip is filed with the purchasing records.

K4.1.3 Organic Chemical Analysis

Pesticide-quality solvents, methylene chloride, hexane, acetone, methanol and ether are analyzed to determine the extent of impurities present in each. Any lot of

solvent showing significant interferent peaks is eliminated from consideration for use by this laboratory. Acetone is the only exception since there are always at least three peaks present due to self-condensation products of acetone.

The purity grade of GC and GC/MS carrier and detector gases must be sufficient to meet detector and method requirements. Instrument manufacturer literature is used as a guideline for determining the purity grade to use.

K4.1.4.1 Water

Water used for the analysis of trace metals should be purified by reverse osmosis/deionization to not less than 10 MΩ cm. Water for organic determinations should be deionized and then further purified with activated carbon. Water for organic analysis should be checked for methylene chloride throughout the day whenever purge and trap methods are used.

K4.3 Storing and Maintaining Reagents and Solvents

As an exception to the ITAS QA Manual, the organic reagent TBA solution is stored at room temperature as allowed by CLP protocol.

K4.4 Glassware Cleaning Requirements

Containers provided for laboratory samples as well as closure and sealing procedures are EPA approved. When vigorously cleaned sample containers are required, commercially available, precleaned, quality controlled containers (e.g., from I-Chem) may be used. Quality control certificates shall be maintained on file by the QC Coordinator.

Chromic acid washing of glassware is used only when it is found to be necessary; for example, when routine cleaning procedures have not been sufficient to eliminate interfering residue. Cleaned glassware is stored uncovered in closed cabinets or drawers. Glassware is solvent rinsed, with the extracting solvent, prior to use.

K5.0 SAMPLE RECEIPT AND INITIATION OF TESTING PROGRAM

K5.1 Field Sample; Receipt, Archiving, Collection and Shipment

Any samples that are damaged due to leaky containers, broken glass, or unusual odors that may be present are reported to Laboratory Management immediately.

At any time a sample is to be shipped by common carrier or sent through the United States mail, it must comply with the Department of Transportation (DOT) Hazardous Material Regulations (49 CFR "TRANSPORTATION").

Prior to collection, the collection team must consider the analyses to be performed so that the sample containers and shipping containers can be assembled and the proper preservatives added. See Table 5-1 in the ITAS QA manual. Preservatives are not added by the laboratory after receipt of samples.

K5.2 Chain-of-Custody (Form)

As stated in the ITAS QA Manual, ITASK shall not accept samples collected by IT personnel for analysis without correctly prepared Chain-of-Custody and Request for Analysis forms. Figure K5-1 is an internal Chain-of-Custody form, which is used as necessary, e.g., for analyses per CLP protocol.

K5.3 Laboratory Receipt and Entry of Sample

The sample is removed from the shipping container and the sample identification information contained on the sample bottles compared to that on sample packing lists, or included Chain-of-Custody documents. If discrepancies exist, appropriate notes (signed and dated) are made on the Chain-of-Custody document and the shipping and receiving supervisor is notified.

The following items are checked and noted on the Sample Receipt Log (Figure K5-2) upon receipt of samples.

A) The seals and tapes on the cooler are unbroken and uncut.

B) The sample containers in the cooler are intact.

C) The cooler contains ice. If not, measure the temperature of the water in the cooler.

D) The identification on the sample bottles correspond to the entries on accompanying forms.

E) The samples are of sufficient quantity to perform the requested analyses.

F) The number of sample containers received (i.e., bottles) is equal to the number of samples listed on the Chain-of-Custody or accompanying forms.

G) Projects are identified by giving each a unique, client-specific three or four character code (e.g., "EPAL" for USEPA, Las Vegas), followed by a five digit number, sequentially assigned. The label in Figure 5-6 is not used. Instead, individual samples are assigned sequential four digit numbers, preceded by one or two character prefixes. Triplicate labels, using prenumbered tape, are affixed to each sample, two of which may be transferred to subsequent aliquots for use in the laboratory.

H) Any nonconformances are noted on a nonconformance form (Figure K5-2).

Sample preservation is not checked at this time. The pH of samples for metals, wastewater, and TOC analyses is checked by the analyst just prior to analysis. If the samples are not at the proper pH, a nonconformance memo is filled out. The sample pH is adjusted by the analyst when required by the analytical method. If the pH measured does not correspond to the information on the Request for Analysis form, the client is notified in the analysis report.

K5.4 Laboratory Storage of Samples

This facility stores: Samples, and past samples, which may or may not be classified as "Hazardous". The duration of sample storage is now for 2 weeks after the report has been issued, unless otherwise obligated by contract or agreement.

K5.4.1 Pre-Analysis Storage

The samples are placed into appropriate storage by the sample custodian (or staff).

Samples are stored as prescribed in Section K5.4 of the ITASK Laboratory-Specific Q.A. Manual. The requirements for storage temperatures and holding times specified in the analytical methods are used (EPA and SW 846 methods). Note that the requirements in Table 5-1 of the ITAS QA Manual refer to water samples. The holding times may be different for other matrices.

K5.4.2 Post-Analysis Storage

Anticipation of reanalysis prescribes proper environmental control. If reanalysis is not anticipated, environmental conditions will not be observed, the samples are stored at room temperature.

K5.5 Initiation of Testing Program

After the analytical program has been entered into the laboratory computerized information management system, a coding package is produced.

The coding package contains a copy of the Chain-of-Custody and Request for Analysis forms as well as the analytical program and the computer generated test assignments.

This coding package then goes to the Group Leaders. The Group Leaders update the computer information by verifying test assignment and assigning any additional tests that are needed.

K5.6 Predisposal

K5.6.1 Sample Storage

The sample custodian will transfer samples from temporary storage to the archive storage area as analyses are completed. Storage time is two weeks after the report is issued unless:

1) Specified otherwise by the customer
2) Sample is known to be involved in litigation, in which case it is stored until the lab is notified by the client

The samples are categorized in storage according to their sample number, which in turn has traceability and directly corresponds to sequential dating.

K5.6.2 Disposal of Lab-Generated Waste

Disposal of laboratory generated waste is addressed in ITASK SOP No. MA850107.

Figure K5-1

ITAS-K-QA003R2

Project Code _____
EPA Case # _____
Sample No. Range _____

INTERNAL CHAIN-OF-CUSTODY FORM - ORGANICS
IT Analytical Services-Knoxville

Sample No.	Date	Extract* Type or Original Sample?	Moved From	Moved To	Reason	Signature

* O - Original Sample
A - Acid Extract
P - Pesticide/PCB Extract
E - BN/AE Extract
B - Base Neutral Extract
V - VOA Sample

Figure K5-2

SAMPLE RECEIPT LOG ITAS-K-CD01OR2

Project Code _____ Number/Type of Samples _____

Date Project Coded _____ _____

Date/Time Received _____ _____

********** For EPA-CLP Use **********

Case # _____
SDG # _____
Contract # _____

Forms	Received	
Federal Express Airbill	Yes	No
Traffic Report	Yes	No
Sample Tags	Yes	No

Custody Seals - present, intact, & properly placed? Yes ___ No ___ Comments _____
Sample Condition - intact & properly packed? Yes ___ No ___ Comments _____
Chain-of-Custody - present & correctly filled out ? Yes ___ No ___ Comments _____
Request/Analysis - present & correctly filled out ? Yes ___ No ___ Comments _____
Blue/Wet Ice - present Yes ___ No ___ Blue Ice Frozen Yes ___ No ___
Temperature of Wet Ice _____

Turnaround Time - ___Routine (10-15 working days) ___Other (specify) _____
Rush Turnaround - ___<48 hr ___ 48-96 hr ___5-10 days ___ Other _____
QC Level - ___I ___II ___III ___IV ___Other (specify)_____

Special Instructions: _____

Lab No.	Client Identification	Analysis	Container/Location

Page ___ of ___

FIGURE K5-3

ITAS KNOXVILLE LABORATORY

NONCONFORMANCE MEMO

PROJECT CODE: _____ FILED BY: _____ DATE: _____

SAMPLE(S) AFFECTED: _____

AREA: CODING _____ GC _____ GC/MS _____ HPLC _____ PREP _____ METALS _____ GEN CHEM _____ OT-EP _____

NONCONFORMANCE (Check appropriate item(s)):

____ Not enough sample received for proper analysis.

____ Sample received ☐broken ☐without proper preservative _____ ☐without proper refrigeration _____ ☐in improper container _____ ☐with custody seal missing or broken ☐ with illegible sample numbers or labels missing.

____ Sample received with incomplete paperwork: ☐Request for Analysis form missing ☐Chain-of-Custody form missing/unrelinquished ☐billing instructions incomplete ☐no due date or TAT specified ☐method for sample disposition not specified ☐numbers on paperwork different than numbers on bottles _____ ☐other _____.

____ Holding time exceeded ☐at receipt ☐in the lab by ____ days. Reason. _____.

____ Sample lost during extraction/analysis; no re-prep or re-analysis possible. Reason: _____.

____ QC sample data reported to client outside of ☐method ☐internal ☐QAPP ☐contract ☐regulatory limits. Reason: _____.

____ Incorrect preparation/analysis procedure(s) used; no re-prep or re-analysis possible. Reason: _____.

____ Samples analyzed and reported under an invalid calibration curve.

____ Incorrect/incomplete data reported to client. Specify: _____.

____ Blank contamination; no re-prep of associated samples done or possible. Reason: _____.

____ Reported detection limits higher than specified in ☐method ☐contract ☐QAPP, due to ☐matrix ☐insufficient sample ☐instrumentation ☐other _____.

____ Other (specify) _____.

CORRECTIVE ACTION:

____ Client (Name: _____) informed verbally on _____ by _____.
____ Client informed in writing on _____ by _____.
____ Sample(s) processed "as is".
____ Sample(s) preserved in laboratory (specify): _____.
____ Sample(s) on hold until _____.
____ Other _____.

Corrective action initiated/performed by _____ Date _____

Operations Concurrence: _____ Date _____

QC Concurrence: _____ Date _____

CORRECTIVE ACTION VERIFICATION

____ Verified

____ Cannot verify. Reason _____.

QC Coordinator: _____ Date _____

THIS DOCUMENT MUST BE RETAINED IN THE PROJECT FILE

K6.0 CALIBRATION PRACTICES

This section of the ITAS-K attachment to the QA manual prescribes the specific calibration practices used by the Knoxville laboratory. Only those procedures that vary from, or are in addition to, those described in Section 6.0 of the ITAS QA Manual are described.

K6.1.6 Calibration Records

The records prepared and the record maintenance procedures are described separately for each instrument type in Section 6.2 - 6.3.

K6.2 Operational Calibration

K6.2.1 GC/MS Calibration

K6.2.1.1 Tuning and GC/MS Mass Calibration

Each GC/MS system used for HSL compound semivolatile/pesticide analysis is tuned using DFTPP. Each GC/MS system used for HSL volatile analysis is tuned using BFB. The ion abundance criteria for both volatile and semivolatile/pesticide analyses must be demonstrated every twelve (12) hours during sample analysis. Specific ion abundance criteria are listed in the appropriate SOP and in Figures K6-1 and K6-2.

K6.2.1.2 Initial Calibration of the GC/MS System

GC/MS uses an internal standard calibration procedure. Specific methods may require modified procedures.

Each GC/MS system used for HSL compound analysis is initially calibrated using a minimum of 5 concentrations. Because of lower response, a four point calibration is

used for 9 of the semivolatile compounds and a three-point calibration is used for benzidine. Surrogates and internal standards are included in each standard. The average relative response factor (RRF) and % RSD (percent relative standard deviation) of each HSL compound is calculated and tabulated. The % RSD of the calibration check compounds (CCC) must be <30% and the average RRF of each performance check compound (SPCC) must be $\geq$ 0.300 (0.250 for Bromoform) for volatiles and $\geq$ 0.05 for semivolatiles/pesticides. See Figures K6-3 through K6-8 for CCC and SPCC details.

K6.2.1.3 Continuing Calibration

A daily standard containing 50 ng of all volatile or semivolatile HSL compounds and the required surrogates is analyzed every 12 hours during sample analysis.

The daily RRF values for each CCC are compared to the average RRF values from the initial calibration by calculating a percent difference (% D) using the following equation:

$$\% D = \frac{\text{daily RRF} - \text{avg RRF}}{\text{avg RRF}} \times 100$$

The % D of each CCC must be less than 25 percent for the daily standard to be accepted prior to sample analysis. A system performance check is also performed as described above, using the daily standard data. If the CCC and SPCC criteria are met, then RRF's and % Ds are calculated and reported for all compounds in the daily standard. If any of the CCC or SPCC criteria are not met, the system is evaluated and corrective action is taken; corrective action may involve running a new initial calibration.

The daily calibration standard may be combined with the mass calibration standard (BFB or DFTPP) and the two calibrations performed simultaneously.

6.2.1.4 GC/MS Calibration Records

Data from each tuning and mass calibration, initial calibration, and continuing calibration are recorded on calibration summary forms (Figures K6-1 through K6-8) and filed with the raw calibration data by instrument, analysis, and date in the calibration section of the laboratory operations files. These files are near the associated instrument. Copies of calibration data and summary forms are filed with the new data and data package for each associated project in the project files as described in Section K12.0.

K6.2.2 Calibration of the Gas/Liquid Chromatograph

The gas chromatography laboratory performs instrument calibration by two basic procedures depending on the nature of the project: project specific procedures and routine calibration procedures. The calibration procedures for a specific project's analyses may deviate from the described procedures if the contract specifies special requirements or if the analytical method specifies a different calibration procedure.

K6.2.2.1 Project Specific Procedures (EPA-CLP Procedures)

K6.2.2.1.1 Initial Linearity

Each time a new column is installed into a specific gas chromatograph, or the chromatographic conditions are changed, i.e., change of flow rates, detectors, electronics, etc., response linearity is established for each GC analyte at these conditions. Three different concentrations of each parameter in a set of noninterfering mixed standard solutions are analyzed. Calibration factors are then calculated for each pesticide and PCB. The linearity for the calibration factors is determined and recorded.

K6.2.2.1.2 Retention Time Windows

Retention time windows are used to tentatively identify pesticides/PCBs during sample analysis. Retention time windows are established by making three (3) injections of all pesticide/PCB standard solutions over a 72 hour period (each compound should be injected near the beginning, near the middle, and near the end of the 72 hour period). The retention time shift of the surrogate dibutyl chlorendate must be <2.0% difference for packed columns (<1.5% for mega bore column). If the dibutyl chlorendate retention time shift is acceptable, the retention time windows for the standards are calculated. The retention time window for each component is ± 3 times the standard deviation of the three absolute retention times for that component. If the calculated retention time window is less than 0.01 minutes, then the retention time window is calculated as ± 1% of the initial retention time of the component in the 72 hour sequence (± 0.75% for mega bore columns).

K6.2.2.1.3 Standard and QC Solutions

Component breakdown is monitored by injecting a standard containing DDT and endrin and looking for its breakdown products: DDD, DDE for the former, and endrin aldehyde and endrin ketone for the latter. The combined breakdown must be ≤ 20%. The breakdown check standard is injected at the beginning of a 72 hour (the second standard) run and after every tenth sample.

To ensure continued linearity, a set of low, medium, and high concentration daily evaluation standards of a representative group of pesticides or PCBs are injected before any samples are injected. Calibration factors are calculated for each parameter; the % RSD must be ≤ 10% for the run to continue. After a specified number of samples, the mid-range evaluation mix and a specified standard mix are injected to

check daily retention times, component breakdown, and calibration factor % differences. Calibration factor % differences must be ≤ 15% for quantification and ≤ 20% for confirmation.

K6.2.2.2 Routine Calibration Procedures

K6.2.2.2.1 Initial Linearity

Standards are run at a minimum of 3 to 5 different concentrations. The resulting chromatograms should elicit all respective peaks on scale to allow for quantitation using either peak height or area. PCB standards may contain more than one Aroclor only if there are no interfering peaks between them.

The results may be used to prepare a calibration curve for each analyte. Alternatively, the percent relative standard deviation (%RSD) of the RFs for each compound is determined. If the %RSD is < 20% over the working range, linearity through the origin can be assumed and the average response factor used in place of a calibration curve.

If standards are not within acceptable limits, the instrument should be checked, the source of the problem identified, and corrections made. Standards (for those out of acceptable limits) will then be rerun.

K6.2.2.2.2 Retention Time Windows

If SW-846 3rd edition methods are used, retention time windows will be established. The procedure is described in Section K6.2.2.1.2.

K6.2.2.2.3 Daily Standards

The working calibration curve or response factor must be verified on each working day by injecting a daily quantitation standard for each analyte. (More than one solution may be required to cover all the compound.) If the response for any analyte varies from the predicted response by more than ± 15%, a new calibration curve must be prepared. When comparison of the daily standard is made to the initial average response factor, the daily response factor is used for quantification. However, if comparison of the daily standard to the calibration curve is required, then the initial calibration curve is used for quantification.

If the daily quantitation standard does not meet the ± 15% difference limits, the instrument should be checked, the source of the gross deviation identified and corrections made. A new standard curve may be required depending on the action taken to correct the deviation. If changes were minor, another daily standard can be run. If the standard meets the acceptable limits, a new calibration curve will not be necessary.

An additional standard check will be run every ten samples to assure GC conditions have not changed from the initial daily standard check. This standard will be compared to the respective daily quantitation standard for retention time, response factors, and any visible chromatographic abnormalities. If retention times are outside the retention time windows or the response factor % difference is > 15% (>20% for confirmation), then all samples analyzed after the standard failing criteria and before the next good standard check will be reanalyzed.

K6.2.2.3 Calibration Records

The project specific linearity and retention time window data are reported on GC standard summary sheets and are stored along with the chromatograms in the calibration section of the laboratory operations file by instrument and date in the GC lab.

All nonproject specific initial calibration curves or data are filed in the GC lab in the calibration section of the laboratory operations file by instrument and then date, but distinguished from the project specific calibration data. When an initial calibration is replaced by a new initial calibration, it will be marked as deleted, initialed, and dated in the top right side and remain in the filing system.

The daily standards will be filed with respective projects and cross-referenced with the applicable initial calibration data.

K6.2.3 Atomic Absorption Spectrophotometer (AA)

K6.2.3.1 Calibration

Light source and optical system: The AA signal is the ratio between the initial source intensity and the intensity after the absorption process. The optimum initial source intensity for each element is provided as recommended lamp currents by instrument manufacturers.

Chemical and flame factors: Unless the fraction of atoms in both standard and samples that get aspirated into the flame are the same, inaccurate data will result. Matrix effects in samples are the primary cause of this difference. Matrix effects can be minimized by closely matching the solvent, acid concentration and the concentration of other major anions and cations in the standards with the samples.

Errors due to different matrices also can be minimized by using the standard addition method of calibration.

Because atoms absorb monochromatic light at the primary wavelength, any ionization during sample atomization will decrease sensitivity. This ionization effect, which is most pronounced for the alkali and alkaline earth elements, is minimized by adding an ionization buffer to all sample and standards.

The formation of refractory compounds in the flame also causes a loss of sensitivity. This problem can be minimized by the use of nitrous oxide-acetylene flame or using releasing agents. Method handbooks and instrument manufacturer instruction manuals provide guidance regarding the correct choice of fuel, oxidant, type of flame (rich or lean) and ionization buffers or releasing agents.

The initial determination and continued verification of the baseline noise, sensitivity and linear working range of each element are the important instrument calibration and operation considerations. In operating an atomic absorption system, a number of variables must be held constant before the system is in statistical control. These variables include instrument warm-up, burner alignment, flame type, fuel, oxidant, lamp intensity, slit width, wavelength, matrix effects, aspiration time and aspiration rate. The following applies (with further detail to be found in the manufacturer's literature):

- Install the correct lamp, set the recommended current, wavelength and spectral bandpass.
- Light the burner, allow it to stabilize and adjust its position for maximum absorbance using a midscale standard.
- Prepare standards in the recommended concentration range (see Table K6-1).

- The sensitivity is the concentration of an element that will produce an absorbance of 0.0044.

  Example: Absorbance if 1 μg/ml = 0.060 $\frac{0.0044}{0.060} = 0.073$

- The AA system shall be warmed-up until the output is stable (usually about 30 minutes. The hollow cathode lamp will be aligned to produce the maximum emitted light to the detector. In flameless AA, the inert gas flow inside the furnace must be optimized to provide maximum sensitivity.

- Aspirate the most concentrated calibration standard to be used and adjust the scale expansion to obtain full-scale output. Successively, aspirate the remaining standards (for a total of at least three) and the standard blank. Construct a calibration curve, from which the concentration of the samples is determined. A single standard addition is used on all samples, unless it has been determined to be unnecessary for a given sample matrix.

- The readout values obtained for the standard curve of each element should fall within a specified range. If readings are excessively low, the operator should check gas flow, burner or cell alignment, wavelength, slit width, photomultiplier voltage, and lamp intensity prior to analysis.

- Output from the instrument is to a suitable strip chart recorder or printer, which allows continuous monitoring of baseline noise, lamp stability, etc., and provides hard copy of all raw data.

- Any deviation from historical values indicates that some aspect of the instrument is malfunctioning or deteriorating (such as a lamp). Before proceeding, correction shall be made.

- If large amounts of dissolved salts are present in the solution to be analyzed, the nebulizer will not handle the solution in the same manner as it will a solution with a small quantity of dissolved salts. Such an interference should be accounted for by adding a salt to the standards or by matching the density of the samples and standards or by use of the method of standard additions.

K6.2.3.2 Record Storage

The charts for each instrument and each day contain calibration and sample raw data and associated calculations. Each chart is given a number. The charts are numbered sequentially and filed in the AA storage area. The charts are cross-referenced in the laboratory project files.

K6.2.4 Inductively Coupled Argon Plasma Spectrometer (ICAP)

K6.2.4.1 Initial Calibration

- A high level calibration standard of all elements is analyzed. (More than one solution may be required to cover all of the elements.)
- A calibration blank prepared at the same acid concentration as the calibration standards is analyzed.
- The ICAP computer does a linear regression using the standard and the blank and constructs the calibration curve for each element.

K6.2.4.2 Daily Calibration

An instrument check standard containing elements at a concentration equivalent to the midpoint of their respective calibration curves is analyzed. The standard(s) is prepared using standards from a different source than the calibration standards. The recovery of the daily standard must be within 90-110%.

K6.2.4.3 Record Storage

Each day's data consisting of calibration data and sample data is numbered. The charts are numbered sequentially and stored in ICAP storage areas. The charts are cross-referenced in the laboratory project files.

K6.2.5 TOC

This procedure is specific to the O.I. instrument used by ITASK.

- Set I.R. at zero so millivolt output is between 1-10 mv and fasten pump head.
- Analyze reagent blanks until the values for each type of analysis are consistent (± 0.5 mv) for four consecutive runs, average each value and enter average value in "calibration constants" display mode.
- Calculate the standard mass in µg of the calibrating, working standard of interest and enter the value in "calibration constants".
- Analyze the calibration standard at least four times. The standard deviation for three consecutive runs must be less than 5%; enter the average millivolt value in the"calibration constant" display mode.

- Analyze a water blank until all appropriate analyses are less than 1.0 ppm.
- Analyze standards of the other types of carbon analyses which are required for a project that are different than the calibration standard. If the average standard value for three consecutive runs of these standards falls within ± 10% of the true value, proceed with sample analysis.

Calibration data and charts are filed by project in the laboratory project files.

K6.2.6 High Pressure Liquid Chromatograph (HPLC)

K6.2.6.1 Initial Calibration

A minimum of three (3) concentrations are analyzed. The results may be used to prepare a calibration curve for each analyte. Alternatively, linearity through the origin may be assumed if the present relative standard deviation (%RSD) of the response factor is ≤ 20% and an average response factor used.

K6.2.6.2 Daily Calibration

A daily standard is analyzed for each analyte. The daily response must be within ± 15% of the predicted response using the initial calibration data.

K6.2.6.3 Standard Check

A standard check is run every ten samples to assure HPLC conditions have not changed from initial daily standard check. This standard is compared to the respective daily standard for retention time, peak heights, response factor, and any visible chromatographic abnormalities. If there is significant deviation in these characteristics, all samples analyzed after the last good standard check and before the next good standard check will be reanalyzed.

K6.2.6.4 Record Storage

Three-point calibration curves are filed by date with the project they were run with. The daily standards are filed with their respective projects and cross referenced with the applicable three-point chromatograms.

K6.2.7 Ion Chromatography

A calibration curve is prepared by injecting standards, (single anion or mixture). The order of retention is always F^-, Cl^-, NO_2^-, Br^-, NO_3^-, PO_4^{3-}, SO_4^{2-}. Inject at least three concentrations for each anion plus a blank. This will produce a calibration curve with a peak height vs. concentration per anion. If linearity is established for a given detector setting, (correlation coefficient > 0.995) it is acceptable to calibrate with a single standard. The daily calibration standard must be within ± 10% of the calibration curve. Calibration data is stored at the instrument in the instrument logbook.

K6.2.8 Ultra Violet/Visible Light UV/VIS Spectrophotometry

Wavelength accuracy - can be checked using optical glass filters or chemical solutions which exhibit absorption spectra with strong, sharp peaks. Didymium glass has a strong absorption line at 585 nm. Nickel, cobalt, chromium, and holmium oxide solutions can also be used to check wavelength accuracy. Prepared solutions are also available for this purpose. Note that the absorption peaks may be shifted on instruments with wide band widths. If the wavelength accuracy checks do not meet specifications quoted for the instrument, have it serviced immediately.

Stray light - is defined as all undesired radiant energy reaching the detector. Stray light causes deviation from Beer's law. Stray light of 0.5 and 5.0 percent causes a deviation of 2 and 15 percent respectively, at an absorbance of one. To check for stray light problems: Prepare a solution of 0.16g potassium chromate per liter in 0.05N potassium hydroxide. Set the instrument wavelength to 374nm and measure the transmittance of the solution. It should be less than 0.05 percent or within instrument specification. If it is greater, check for obvious leaks or have the instrument serviced.

Length and positioning of sample cells can affect the absorbance measured. Do not assume that a group of cells have the same path length as their listing. A common mistake is to develop a standard curve with one cell and then measure samples with another. Measure the absorbance of the cobalt solution described above at 510nm using all cells. Set aside all cells with absorbances that do not agree within 1 percent. Cell windows must be clean, and the cells can be reproducibly placed in the spectrophotometer.

Photometric linearity - is measured by the additivity of absorbance values. Instrument linearity can be evaluated by measuring the absorbance of serial dilutions of chromium, cobalt and nickel solutions. Calibration records are kept in a logbook at the instrument.

K6.2.9 pH Meter

Calibration is obtained by using 2 commercial buffers at concentrations to bracket the expected sample concentration range. Calibration records are kept in a logbook at the instrument.

K6.2.10 Conductivity Meter

Calibration is performed using a 0.01m solution of KCL and water with a conductivity of less than 1 μmhos/cm. Equilibrate the solution at 25°C. Rinse the electrode in the above KCL solution and measure the conductivity. The conductivity should be approximately 1400 μmhos/cm. The cell is replaced or new standards are made if results are outside 1400 ± 30 μmhos/cm. Calibration records are kept in a logbook at the instrument.

K6.3 Periodic Calibration

K6.3.1 Balances

Calibration: all balances are serviced quarterly under a maintenance contract with a balance service company. Using class "S" weights, the analytical balances are checked daily. The results are recorded in a logbook. For balances which are direct reading to 0.01 gram, the acceptance criteria for the calibration check is ± 0.01g for 0 to 100g and ± 0.1 percent of the applied weight over 100g. If the daily calibration check is not acceptable, the balance will be serviced as needed. The balances are calibrated to at least 95% capacity a minimum of once every 3 months. Calibration records are kept in the balance room.

K6.3.2 Thermometers

Thermometers are all directly tracable to NBS thermometers. All working thermometers are calibrated annually using NBS traceable thermometers.

K6.4 Summary of ITASK Calibration Requirements

Requirements for ITASK instrument calibration are summarized in Tables K6-1 and K6-2. Table K6-2 shows a list of instrument logs maintained by the laboratory.

Table K6-1. Summary of Operational Calibration Requirements

Instrument	Calibration Standards Used, Initial and Daily Minimum	Acceptance Limits	Corrective Actions	Reference[1]
Atomic Absorption Spectrophotometer	Initial: 3 levels + blank Daily: 1 check standard (midrange) per 10 samples;	Linear regression correlation coefficient >0.995; Daily check std 90-110% recovery	Make new standards or establish new calibration curve	1,2
Gas Chromatograph	Initial: 3-5 levels	Std curve or calibration factor (CF) if % RSD <20;	Make new standards or establish new calibration curve	1
	Daily: 1 level of check standard	CF < ± 15% of initial calibration		
	Std check every 10 samples	CF ± 15% of daily calibration (< ± 20% for confirmation). Retention times within retention time windows. (For methods using retention time windows.)		
Inductively Coupled Plasma Emission Spectrophotometer	Initial: high standard + blank Daily: Instrument check standard and calibration blank every 10 samples	Check std 90-110%	Recalibrate. Repeat twice; if outside control limit, then: recalibrate and make new stds if necessary	3
Ion Chromatograph	Initial: 3 levels + blank Daily: 1 level of check standards every 10 samples (midrange)	Linear regression correlation coefficient >0.995 Daily: ± 10% of original curve	Make new standards, recalibrate	4

Table K6-1. Summary of Operational Calibration Requirements (cont.)

Instrument	Calibration Standards Used, Initial and Daily Minimum		Acceptance Limits	Corrective Actions	Reference[1]
pH Meter	Daily:	2 levels	±0.05 pH unit Bracket sample range	Clean or replace electrod; recalibrate	1
UV-Visible Spectrophotometer	Initial: Daily: Monthly:	3 levels + blank Check standard Wavelength accuracy and photometric linearity	Std curve + 10% of initial calibration Manufacturer specifications ± 10% previous observations	Recalibrate Service Service	5
HPLC	Initial: Daily:	3 levels + blank Check standard and check std every 10 samples	Std curve or calibration factor % RSD <20% 15% of original curve	Make new standards and/or establish a new calibration curve.	
Total Organic Carbon	Daily:	1 level + blank	± 10% of true value	Make new standards	1
GC/MS	Tune: Initial: Daily:	BFB or DFTPP 5 levels + blank 1 level	Meets criteria %RSD <30% (CCC) ± 25% of initial curve (CCC) Retention time ± 30 sec for internal standards	Make new standard recalibrations	1
Conductivity Meter	Daily: (when used)	1 level	Direct reading approximately 1400 ± 30 µmhos/cm	Clean or replace cell	6

References:
1) EPA SW-846, "Test Methods for Evaluating Solid Waste"
2) USEPA Methods for Chemical Analysis of Water and Wastes, 1979
3) USEPA 200.7, 1982
4) USEPA 300.0, 1984
5) Directions for DIDYMIUM Calibrating Standard Coleman No. 6-400. Perkin Elmer 12/1972
6) Instrument Manual

Table K6-2. Summary of Periodic Calibration Requirements

Instrument	Calibration Frequency	Acceptance Limits	Corrective Actions	Reference
Analytical Balance	Daily: Sensitivity (with Class "S" weight)	0.001 gm Difference less than 0.1 mg	Adjust, sensitivity	QA 841214R1-3
Thermometers	Calibrate in constant temperature baths at two temperatures against precision thermometers certified by NBS annually	$\pm 0.5^{\circ}C$	Tag and remove from service, replace	QA 860626R0
Pipettors	Gravimetric check semi-annually	High volume (>100 μL): < 1.0% relative error and RSD Low volume (<100 μL): < 2.0% relative error and RSD	Service or replacement	QA 870624R0

TABLE K6-3

Table of maintained instrument log books at ITASK Laboratory (each log sheet includes: Date of Review, Initials of person entering data and/or observations).

A. Extraction Lab Logs (pipettors)

B. GC Analysis Logs

C. GC/MS Analysis Logs

D. GC Maintenance Logs

E. GC/MS Maintenance Log

F. Refrigerator Temperature Logs

G. Analytical Balance Check Logs

H. Freezer Temperature Logs

I. AA/ICAP Run Logs

J. AA/ICAP Maintenance Logs

K. Misc. Lab Logs

L. Misc. Lab Maintenance Logs

M. Conductivity Logs

K7.0 PREVENTIVE MAINTENANCE

The ITASK Preventive Maintenance Program was developed to meet and supplement the guidelines prescribed by the ITAS QA Manual. Routine preventive maintenance procedures are the responsibility of the individual Group Leaders. A department specializing in equipment and instrument maintenance has been created for this laboratory. The ITASK Instrument Maintenance Specialist performs in-house corrective maintenance and may perform the more difficult and intricate preventive maintenance procedures.

Table K7-1 summarizes the preventive maintenance requirements for the ITASK laboratory. The table lists the instruments and equipment included in the program, items to be checked/serviced and the frequency of the required maintenance procedure. Figures K7-1 through K7-15 are examples of the Preventive Maintenance Records maintained by the laboratory.

Preventive maintenance records are kept in each laboratory department either in a central file or in individual instrument/equipment logbooks. A copy of the preventive maintenance schedule is placed in the front of the respective instrument/equipment logbook. Documentation of the preventive maintenance procedures are recorded in the logbook as they are performed. Specifics for preventive maintenance are detailed in the respective instrument maintenance S.O.P.'s.

For reference, a central file listing the instruments and equipment covered by the Preventive Maintenance Program is kept in the Instrument Maintenance Shop. Maintenance frequency and items to be checked/serviced are listed (Table K7-1). Behind this table, filed by department, are examples of the preventive maintenance records and a listing of the spare parts routinely maintained by the laboratory.

TABLE K7-1

Preventive Maintenance Requirements
Knoxville Laboratory Operations

INSTRUMENT	ITEMS CHECKED/SERVICED	FREQUENCY
Inductively Coupled Argon Plasma (ICAP)	Replace Pump Tubing	As needed
	Inspect and Clean Sample Introduction System	As needed
	Drain Air Filters	Semi-weekly
	Check Drain Receptacle Level	Weekly
	Clean Torch	Monthly
AA 151B For Mercury Analysis (AVA)	Replace Check Valves	As needed
	Check Pump Delivery	As needed
	Check Tubing Integrity	As needed
	Clean Burner/Adsorption Cell Mount	Monthly
	Clean Adsorption Cell	Monthly
	Replace System Tubing	Semi-Annually
Atomic Absorption Spectrophotometer - Flame Units	Inspect Backflash Diaphram	As needed
	Check Drain Receptacle Level	As needed
	Replace Nebulizer Chamber O-rings	As needed
	Replace Beam Splitter	As needed
	Clean Nebulizer	Weekly
	Clean Burner Head	Weekly
	Clean Spray Chamber	Weekly
	Remove and Clean Burner Mount	Monthly

TABLE K7-1 (continued)

INSTRUMENT	ITEMS CHECKED/SERVICED	FREQUENCY
Graphite Furnace 951/655	Replace Graphite Cuvette	As needed
	Replace Dry-Rite and Glass Wool	As needed
	Check Drain Receptacle	As needed
	Replace Temperature Sensor	As needed
	Clean Electrodes	As needed
	Replace Graphite Inserts	As needed
	Replace Electrodes and Sensor Posts	As needed
	Check Graphite Insert	Weekly
	Check Recirculator Water Level	Weekly
	Replace Set Screw	Weekly
	Check Electrode Appearance	Weekly
	Clean Nebulizer and Spray Chamber	Monthly
	Clean Furnace Chamber and Windows	Monthly
	Flush Furnace Head with Acetic Acid	Semi-Annually
	Replace Recirculator Water	Semi-Annually
Graphite Furnace 12E/188	Check Drain Receptacle	As needed
	Clean Saphire Window	As needed
	Replace Graphite Cuvette	As needed
	Clean Cell Body and Windows	Weekly
	Replace Marble Chips	Weekly
	Check Recirculator Water Level	Weekly
	Clean Temperature Sensor Filter	Semi-Monthly
	Clean FASTAC Pump	Monthly
	Clean Nebulizer and Spray Chamber	Monthly
	Clean Air Filter	Monthly
	Flush Furnace Head with Acetic Acid	Semi-Annually
	Replace Recirculator Water	Semi-Annually

TABLE K7-1 (continued)

INSTRUMENT	ITEMS CHECKED/SERVICED	FREQUENCY
High Performance Liquid Chromatography (HPLC)	Degas Solvents	Daily when used
	Check for Leaks	Daily when instrument used
	Flush Column	Daily when instrument used
	Check Injector for Blockage/ Pressure Drop	Daily when instrument used
	Check Column Pressure	Daily when instrument used
	Replace Lamp	As needed
	Replace Guard Column	As needed
Organic Prep: Sonicator	Inspect Probe Tips for Etching/ Pitting	Daily when used
	Replace Probe Tip	As needed
	Disassemble and Clean Sonicator Probe Tips	Semi-Annually
	Tune Sonicator	Semi-Annually
pH Meters	Gel Filled Maintenance Free Electronics Checked	As needed
UV/VIS Spectrophotometer	Replace Lamp	As needed
	Check Wavelength	Monthly
Millipore Water System	Conductivity Checked	Daily
	Pressure Check	Daily
	Change Ion Exchange Bed and Filters	As needed
Analytical Balance	Internal Weight Train, Gears Electronics	Annually by Service Contract

TABLE K7-1 (continued)

INSTRUMENT	ITEMS CHECKED/SERVICED	FREQUENCY
Ion Chromatograph	Degas Pump Head	As needed
	Rebuild Pump Head	As needed
	Clean/Replace Cell Packing	As needed
	Change Column	As needed
	Check Plumbing	Daily when in use
	Check Bed Support by Monitoring Pressure	Daily when in use
	Replace Plunger Seals	Semi-Annually
	Clean Check Valve	Semi-Annually
	Oil Pumps	Semi-Annually
Refrigerators	Temperature Checked and Logged	Daily
Walk-in Coolers	Temperature Checked and Logged	Daily
Ovens	Temperature Checked and Logged	Daily

TABLE K7-1 (continued)

INSTRUMENT	ITEMS CHECKED/SERVICED	FREQUENCY
Gas Chromatograph	Inlet, Detector and Column Temperatures	Daily when instrument in use
	Gas pressures	Daily
	Column and Detector Flow Rates	Daily when instrument in use
	Check Zero and Noise Level	Daily when instrument in use
	Check Responses, Retention Times Using a Check Standard	Daily when instrument in use
	Check Autosampler for Bubbles, Flush Volume	Daily when instrument in use
	Check Electrolyte Level and Flow Rate (HALL only)	Daily when instrument in use
	Reactor Tube Temperature (HALL only)	Daily when instrument in use
	Check Pressure in Gas Tanks, Replace in <500 PSI	Weekly
	Clean Outside Surfaces of Instruments	Weekly
	Replace Septa	As needed
	Replace Column or Part of Column Packing	As needed
	Clean/Replace Detector	As needed
	Replace Glass Wool/Glass Insert	As needed
	Clean Injection Port	As needed
	Replace Reaction Tube (HALL only)	As needed
GC Purge and Trap GC Purge and Trap Autosampler	Check Zone Temperatures	Daily when in use
	Helium Tank Pressure (>500 PSI)	Daily when in use
	Purge and Trap Regulator Pressure (20 PSI)	Daily when in use
	Bake Trap	Daily when in use
	Replace Trap	As needed
	Acid Wash Purge Vessel	As needed
	Leak Test	As needed

TABLE K7-1 (continued)

INSTRUMENT	ITEMS CHECKED/SERVICED	FREQUENCY
Total Organic Carbon Analyzer	Gas Tank Pressures	Daily when in use
	N_2 and O_2 Regulator Pressures	Daily when in use
	Zone Temperature	Daily when in use
	Purge and Detector Flow Rates	Daily when in use
	Clean Port Valves	After 72 hrs of operation
	Purge Gas and Detector Leak Test	After 72 hrs of operation
GC/MS	Diffusion Pump Oil	Bi-Weekly
	Mechanical Pump Oil	Quarterly
	Power Con. Air Filter	Bi-Weekly
	QEM Filter	Bi-Weekly
	Water Bay Filter	Bi-Weekly
	Interface Box	Bi-Weekly
	Vacuum Chaff Filter	Monthly
	Turbo Pump Oil	Quarterly
	Water Filter (if applicable)	Observe and change as needed
	Computer Air Filter	Monthly
	Card Cage Air Filter	Monthly
	Source-Clean Ceramics, Polish Lenses	As needed
	Clean Poles and Ceramics on the Poles	As needed
	Clean Contacts on the Component Boards	As needed
	Vacuum the Component Boards	As needed
	Clean All Fan Screens	Weekly
	Vacuum Outside of Instrument	Weekly
	Clean Grob and Replace Quartz Insert	As needed
	Replace Septum	Daily (each shift)
	Injection Port Liner Checked	Daily
	Column maintenance	As needed
	Disk Drive (CMD only)	Semi-Annual (Service Engineer) or as needed
	Printer	Quarterly

Figure K7-1

Example - AA Preventive Maintenance Record - Week 1

PREVENTIVE MAINTENANCE - METALS
_____ - WEEK 1

ITEM (FREQUENCY)	INITIALS/DATE
General Laboratory	
Drain Air Compressor (W)	_____
Clean Lab Area (General Housekeeping) (W)	_____
Clean Fume Hood - Prep (W)	_____
Clean Fume Hood - Mercury (M)	_____
Clean Hot Plates (M)	_____
Dispose of Sample Digestates - Organize Shelves (M)	_____
Inductively Coupled Argon Plasma (ICAP) Unit	
Clean Air Filters (BW)	_____
Clean Air Filters (BW)	_____
Check Drain Receptacle Level (W)	_____
Clean Torch (M)	_____
Mercury Analysis - 151B with Automatic Vapor Accessory (AVA)	
Clean Burner/Absorption Cell Mount (M)	_____
Clean Absorption Cell (M)	_____
Flame Atomic Absorption Spectrophotometer Unit(s)	
Clean Nebulizer (W)	_____
Clean Burner Head (W)	_____
Clean Spray Chamber (W)	_____
Remove & Clean Burner Mount (M)	_____
Graphite Furnace 951/655	
Check Graphite Insert (W)	_____
Check Recirculator Water Level (W)	_____
Replace Set Screw (W)	_____
Check Electrode Appearance (W)	_____
Clean Nebulizer & Spray Chamber (M)	_____
Clean Furnace Chamber & Windows (M)	_____
Graphite Furnace 12E/188	
Clean Cell Body & Windows (W)	_____
Replace Marble Chips (W)	_____
Check Recirculator Water Level (W)	_____
Clean FASTAC Pump (M)	_____
Clean Nebulizer & Spray Chamber (M)	_____
Clean Air Filter (M)	_____
Clean Temperature Sensor Filter (BM)	_____

Figure K7-2

Example - AA Preventive Maintenance Record - Week 2

PREVENTIVE MAINTENANCE - METALS
_____ - WEEK 2

ITEM (FREQUENCY)	INITIALS/DATE
General Laboratory	
Drain Air Compressor (W)	_____
Clean Lab Area (General Housekeeping) (W)	_____
Clean Fume Hood - Prep (W)	_____
Inductively Coupled Argon Plasma (ICAP) Unit	
Clean Air Filters (BW)	_____
Clean Air Filters (BW)	_____
Check Drain Receptacle Level (W)	_____
Mercury Analysis - 151B with Automatic Vapor Accessory (AVA)	
Flame Atomic Absorption Spectrophotometer Unit(s)	
Clean Nebulizer (W)	_____
Clean Burner Head (W)	_____
Clean Spray Chamber (W)	_____
Graphite Furnace 951/655	
Check Graphite Insert (W)	_____
Check Recirculator Water Level (W)	_____
Replace Set Screw (W)	_____
Check Electrode Appearance (W)	_____
Graphite Furnace 12E/188	
Clean Cell Body & Windows (W)	_____
Replace Marble Chips (W)	_____
Check Recirculator Water Level (W)	_____

Figure K7-3

Example - AA Preventive Maintenance Record - Week 3

PREVENTIVE MAINTENANCE - METALS
_____ - WEEK 3

ITEM (FREQUENCY)	INITIALS/DATE
General Laboratory	
Drain Air Compressor (W)	_____
Clean Lab Area (General Housekeeping) (W)	_____
Clean Fume Hood - Prep (W)	_____
Inductively Coupled Argon Plasma (ICAP) Unit	
Clean Air Filters (BW)	_____
Clean Air Filters (BW)	_____
Check Drain Receptacle Level (W)	_____
Mercury Analysis - 151B with Automatic Vapor Accessory (AVA)	
Flame Atomic Absorption Spectrophotometer Unit(s)	
Clean Nebulizer (W)	_____
Clean Burner Head (W)	_____
Clean Spray Chamber (W)	_____
Remove & Clean Burner Mount (M)	_____
Graphite Furnace 951/655	
Check Graphite Insert (W)	_____
Check Recirculator Water Level (W)	_____
Replace Set Screw (W)	_____
Check Electrode Appearance (W)	_____
Graphite Furnace 12E/188	
Clean Cell Body & Windows (W)	_____
Replace Marble Chips (W)	_____
Check Recirculator Water Level (W)	_____
Clean Temperature Sensor Filter (BM)	_____

Figure K7-4

Example - AA Preventive Maintenance Record - Week 4

PREVENTIVE MAINTENANCE - METALS
_____ - WEEK 4

ITEM (FREQUENCY)	INITIALS/DATE
General Laboratory	
Drain Air Compressor (W)	_____
Clean Lab Area (General Housekeeping) (W)	_____
Clean Fume Hood - Prep (W)	_____
Inductively Coupled Argon Plasma (ICAP) Unit	
Clean Air Filters (BW)	_____
Clean Air Filters (BW)	_____
Check Drain Receptacle Level (W)	_____
Mercury Analysis - 151B with Automatic Vapor Accessory (AVA)	
Flame Atomic Absorption Spectrophotometer Unit(s)	
Clean Nebulizer (W)	_____
Clean Burner Head (W)	_____
Clean Spray Chamber (W)	_____
Graphite Furnace 951/655	
Check Graphite Insert (W)	_____
Check Recirculator Water Level (W)	_____
Replace Set Screw (W)	_____
Check Electrode Appearance (W)	_____
Graphite Furnace 12E/188	
Clean Cell Body & Windows (W)	_____
Replace Marble Chips (W)	_____
Check Recirculator Water Level (W)	_____

Figure K7-5

Example - AA Preventive Maintenance Record - Week 5

PREVENTIVE MAINTENANCE - METALS
JANUARY - WEEK 1

ITEM (FREQUENCY)	INITIALS/DATE
General Laboratory	
Drain Air Compressor (W)	_____
Clean Lab Area (General Housekeeping) (W)	_____
Clean Fume Hood - Prep (W)	_____
Clean Fume Hood - Mercury (M)	_____
Clean Hot Plates (M)	_____
Dispose of Sample Digestates - Organize Shelves (M)	_____
Inductively Coupled Argon Plasma (ICAP) Unit	
Clean Air Filters (BW)	_____
Clean Air Filters (BW)	_____
Check Drain Receptacle Level (W)	_____
Clean Torch (M)	_____
Mercury Analysis - 151B with Automatic Vapor Accessory (AVA)	
Clean Burner/Absorption Cell Mount (M)	_____
Clean Absorption Cell (M)	_____
Replace System Tubing (BA)	_____
Flame Atomic Absorption Spectrophotometer Unit(s)	
Clean Nebulizer (W)	_____
Clean Burner Head (W)	_____
Clean Spray Chamber (W)	_____
Remove & Clean Burner Mount (M)	_____
Graphite Furnace 951/655	
Check Graphite Insert (W)	_____
Check Recirculator Water Level (W)	_____
Replace Set Screw (W)	_____
Check Electrode Appearance (W)	_____
Clean Nebulizer & Spray Chamber (M)	_____
Clean Furnace Chamber & Windows (M)	_____
Flush Furnace Head with Acetic Acid (BA)	_____
Replace Recirculator Water (BA)	_____
Graphite Furnace 12E/188	
Clean Cell Body & Windows (W)	_____
Replace Marble Chips (W)	_____
Check Recirculator Water Level (W)	_____
Clean FASTAC Pump (M)	_____
Clean Nebulizer & Spray Chamber (M)	_____
Clean Air Filter (M)	_____
Clean Temperature Sensor Filter (BM)	_____
Flush Furnace Head with Acetic Acid (BA)	_____
Replace Recirculator Water (BA)	_____

Figure K7-6

EXAMPLE - HPLC PREVENTIVE MAINTENANCE RECORD

Maintenance Performed	Date/Initials				
1) Check column pressure & injector system for pressure drop					
2) Flush column (organic flush)					
3) Leak check					
4) Degas solvents					
5) Replace or change guard column or column					
6) Replace lamp					
Other (Specify) _____ _____ _____ _____					

Maintenance Frequency: Items 1-4 are performed daily when the instrument is in use. Other items are performed on an as needed basis.

Figure K7-7

EXAMPLE - SONICATOR PREVENTIVE MAINTENANCE RECORD

Maintenance Performed	Date/Initials				
Inspect probe tips for etching/pitting					
Replace probe tip					
Disassemble and clean probe tip					
Tune sonicator					
Other (Specify) _____ _____ _____ _____					

Maintenance Frequency:

Inspect probe tips daily when used

Replace probe tip as needed

Disassemble and clean probe tip semi-annually

Tune Sonicator semi-annually

Figure K7-8

Example - UV/VIS Preventive Maintenance Record

06

Date	λ of min. Trans.	Adjustment: Not Required	Adjustment: Required	Action Taken	Initials
4/5/85	585		✓	Reading unstady w/empty sample well; cleaned lamp — no help; cleaned bottom & blower — dust out — seems OK now	GMW
7/23/85	585	✓		Same thing, but this time new lamp & cleaning did not help — call PE	GMW
3/23/87	585	✓		Normal calibration test run	DFW
~~7/6/87~~ 7/10/87	585	✓		—	DFW.
8/8/87	585	✓		—	DFW
9/87	—	—	—	No Calib. check made	DFW
10/9/87	585	✓		No problems, no maintenance needed	DFW
11/13/87	585.	✓		No vis. problems, no main. required	DFW
12/11/87	585.	✓		No maintenance required	DFW
1/26/88	585.	✓		No maintenance required	DFW
2/15/88	585.	✓		No maintenance required	DFW
3/2/88	585.	✓		No maintenance required	DFW
4/15/88	585	✓		" " "	DFW
5/23/88	585	✓		—	DFW
6/9/88	585	✓		—	DFW

Figure K7-9

Example - UV/VIS Preventive Maintenance Record

	Exciter Lamp Changes: Date:	Analyst Initials:
1	2-4-81	DCH
2	5-4-81	DCH
3	3-2-83	KLD
4	12-29-83	DFW
5	7-22-85	DFW
6	8-21-85	KLD
7	9-30-85	DFW
8	11-11-85	KLD
9	1-23-86	KLD
10	3-20-86	DFW
11	4-16-86	KLD
12	5-15-86	LWC
13	8-19-86	LWC
14	11-10-86	DFW
15	2-5-87	KAK
16	5-11-87	DFW
17	7-6-87	KAK/DFW
18	12-11-87	D.F.W.
19	2-8-88	KAK
20	5-23-88	DFW
21	7-6-88	DFW
22	9-12-88	DFW

Figure K7-10

Example - Millipore Water System Preventive Maintenance Record

71

Year: 1988

Mo.	Day	Resistivity (megaOhms)	Time of Reading	(Pressure) PSI	Initials
July	11	-17-	08 30	10.5	DHW
"	12	-17-	09 10	10.5	DHW
July	13	No Reading Recorded This Date			
"	14	No Reading Recorded This Date			
July	15	-17-	16 00	12.5	DHW
"	18	-17- (Filter change)	14 00	20.5	DHW
July	19	-17-	10 30	19.5	DHW
"	20	-17-	08 30	16.5	DHW
July	21	-17-	10 45	15.5	DHW
"	25	-17-	14 05	15.5	DHW
July	26	No Readings Recorded This Date			
"	27	-17-	09 30	15.0	DHW
July	28	No Readings Recorded This Date			
"	29	-17-	08 45	15.0	DHW
August	1	-17-	1553	18.0	RSR
"	2	-17-	0905	15.0	RSR
August	3	-16-	0830	17.5	DHW
"	4	Changed filters -17-	10 00	15.0	DHW
August	5	-17-	1215	14.0	DHW
"	8	No Readings Recorded This Date			
"	9	-17-	1605	15.5	RSR
August	10	No Readings Recorded This Date			
"	11	-17-	08 30	14.5	DHW
August	12	-17-	09 20	14.5	DHW
August	15	-17-	0730	18.0	RSR
"	16	No Readings Recorded This Date			
"	17	No Readings Recorded This Date			
"	18	-17-	1449	15.5	RSR
Aug	19	No Readings Recorded This Date			
"	22	No Reading Recorded This Date			
Aug	23	-17-	10 15	14.0	DHW
"	24	-17-	08 05	14.0	DHW

Figure K7-11

Example - Ion Chromatograph Preventive Maintenance Record

INTERNATIONAL TECHNOLOGY CORPORATION

By _____ Date _____ Subject Ion Chromatograph Preventive Maintenance Sheet No. ____ of ____
Chkd. By ____ Date _____ INSTR. _____

1989	JAN.	FEB.	MAR.	APR.	MAY	JUN.	JUL	AUG.	SEP.	OCT.	NOV.	DEC.
1												
2												
3												
4												
5												
6												
7												
Semiannually												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												
21												
22												
23												
24												
25												
26												
27												
28												
29												
30												
31												

28-12-85

Figure K7-12

Example - Daily Temperature Log

DAILY TEMPERATURE LOG

I.D. Number										
Temp Range °C			4 ± 2	-10 ± 2	4 ± 2	20 ± 1	105 ± 2	35 ± 0.5		180 ± 2
Date	Time	Initials								

Figure K7-13

Example - Gas Chromatograph Preventive Maintenance Record

INTERNATIONAL TECHNOLOGY CORPORATION

By _____ Date _____ Subject Gas Chromatograph Daily and Weekly Preventive Maintenance Sheet No. ____ of ____

Chkd. By ____ Date _____ INSTR. _____

1989		JAN.	FEB.	MAR.	APR.	MAY	JUN.	JUL.	AUG.	SEP.	OCT.	NOV.	DEC.
	1												
	2												
	3												
	4												
	5												
	6												
	7												
weekly													
	8												
	9												
	10												
	11												
	12												
	13												
	14												
weekly													
	15												
	16												
	17												
	18												
	19												
	20												
	21												
weekly													
	22												
	23												
	24												
	25												
	26												
	27												
	28												
weekly													
	29												
	30												
	31												

08-10-85

Figure K7-14

Example - GC/MS Preventive Maintenance Record

GC/MS DAILY PREVENTIVE MAINTENANCE

	JANUARY	FEBRUARY	MARCH	APRIL	MAY	JUNE	JULY	AUGUST	SEPTEMBER	OCTOBER	NOVEMBER	DECEMBER	
1													1
2													2
3													3
4													4
5													5
6													6
7													7
8													8
9													9
10													10
11													11
12													12
13													13
14													14
15													15
16													16
17													17
18													18
19													19
20													20
21													21
22													22
23													23
24													24
25													25
26													26
27													27
28													28
29													29
30													30
31													31

Figure K7-15

Example - GC/MS Preventive Maintenance Record

GC/MS MONTHLY AND QUARTERLY PREVENTIVE MAINTENACE

JANUARY													
FEBRUARY													
MARCH													
APRIL													
MAY													
JUNE													
JULY													
AUGUST													
SEPTEMBER													
OCTOBER													
NOVEMBER													
DECEMBER													
TOTAL													

JANUARY													
FEBRUARY													
MARCH													
APRIL													
MAY													
JUNE													
JULY													
AUGUST													
SEPTEMBER													
OCTOBER													
NOVEMBER													
DECEMBER													
TOTAL													

K8.0 ANALYSIS OF QUALITY CONTROL SAMPLES

This section of the ITAS QA Manual discusses samples which are routinely added to the normal laboratory sample stream to demonstrate that the laboratory is operating within prescribed requirements for accuracy and precision. The following discussion supplements the ITAS Quality Assurance Manual, only those points of the ITASK routine program that vary from the ITAS QA Manual are addressed. As stated in the ITAS QA Manual, specific analytical procedures may prescribe different QC samples.

K8.1.4 Method Blank Analyses

A method blank is included with each group of samples or one per twenty whichever is more frequent. All GC/MS method blanks are spiked with surrogate compounds.

K8.1.7 Duplicate Sample Analysis

For organic analyses the duplicate sample may be a spiked duplicate. The frequency of analysis is one MS/MSD pair per twenty (20) samples or at least one per month.

K8.1.14 Blank Spike Analyses

If the results of MS/MSD sample analyses are acceptable, a blank spike analysis is not required.

K8.2 Performance Evaluation and Certification Programs

ITASK routinely participates in several interlaboratory certification and performance evaluation programs. A discussion of laboratory performance programs follows; a current summary of ITASK laboratory certifications is maintained by the Quality Control Coordinator in the Quality/Operations files.

K8.2.1 Laboratory Performance Programs

- The EMSL-LV (Environmental Monitoring Systems Laboratory-Las Vegas) Quarterly Organic Performance Evaluation Study. The evaluation consists of aqueous materials spiked with Hazardous Substance List pollutants, at environmentally representative levels. Samples for all laboratories are from the same homogeneous batch.

- The annual WSPE (Water Supply Performance Evaluation). Samples originate from EMSL-Cincinnati (Environmental Monitoring and Support Laboratory).

- The annual WPPE (Water Pollution Performance Evaluation Study). This study is a continuing effort to provide independent evaluations of the EPA Regional laboratories, State laboratories and selected laboratories for water pollution parameters.

- The NIOSH-Proficiency Analytical Testing (P.A.T.). Participation is required for all laboratories with AIHA Certification. Laboratories participating in P.A.T. must pass to be certified. Samples are received quarterly in two categories: metals and organics.

K9.0 ANALYTICAL PROCEDURES

Details of the procedures followed by ITASK for the analysis of samples are documented in the appropriate individual SOPs (Standard Operating Procedures). The SOPs used by ITASK are collected together into a laboratory Standard Operating Procedures Manual. Activities pretaining to calibration, preventive maintenance and quality control sample analyses are addressed in the appropriate section of the ITASK QA Manual as well as in the SOP Manual.

K9.1 Analytical Methods

Tables K9-1 and K9-2 list the analytical procedures normally used by ITASK for sample analysis. Parameters, approved methods and references are presented. If the laboratory has need to analyze for a parameter that is not listed in Table K9-1 or K9-2 but is included in the ITAS approved method list, then the ITAS approved method will be used. Any variations from approved methods, which are sometimes necessary due to limited sample quality, unusual sample matrix, etc., should be approved by the Laboratory Manager, or the Operations Manager and the Technical Director.

K9.2 Detection Limits

The ITASK method for reporting detection limits varies from the method described in the ITAS QA Manual. The quantitation limit or Contract Required Quantitation Limit (CRQL) is reported with a "U" qualifier following the value. For GC/MS and Metals CLP analyses the quantitation limit or CRQL is not the actual detection limit. The concentration of GC/MS analyte found below the quantitation limit but above the detection limit are reported with a "J" qualifier following the value. Metals data under CLP protocol uses a "B" qualifier instead of the "J" used by GC/MS. For GC data the quantitation limit or CRQL is assumed to also be the detection limit.

Usually the listed EPA method detection limits are used as the ITASK method detection limit. A higher quantitation limit may be used when the client does not need the lower EPA method detection limits. In other cases, depending upon client needs, detection limits lower than the published limits may be used. Lower detection limits are used only when it can be demonstrated that the reported limits are actually achievable.

Instrument detection limits, when required, are determined as appropriate to the specific analytical method being used.

TABLE K9-1

List of Approved Test Procedures
ITASK Laboratory Operations

Parameters	Approved Methods		
	SW-846	EPA 1983	Standard Method 16th Edition
1. Acidity		305.2	402
2. Alkalinity		310.1	403
3. Aluminum	7020	202.1 202.2	
4. Ammonia		350.2	
5. Antimony	7041 6010	204.1 204.2 200.7	
6. Arsenic	7060 6010	206.2 200.7	
7. Barium	7080	208.1 200.7	
8. Beryllium	7090 6010	210.1 200.7	
9. Biochemical Oxygen Demand		405.1	507
10. Boron	6010	200.7	
11. Bromide-Ion Chromatography			429
12. Cadmium	7130 7131 6010	213.1 213.2 200.7	
13. Calcium	7140 6010	215.1 200.7	

TABLE K9-1 (continued)

Parameters	Approved Methods		
	SW-846	EPA 1983	Standard Method 16th Edition
14. Chemical Oxygen Demand (COD)		410.2 410.1	
15. Chloride		325.3	
Ion Chromatography		300.0	429
16. Chlorine-Total Residual		330.5	
17. Chromium VI Dissolved			312B
18. Chromium-Total	7190 6010	218.1 200.7	
19. Cobalt-Total	7200 6010	219.1 200.7	
20. Color		110.2	
21. Copper-Total	7210 6010	220.1 200.7	
22. Cyanide-Total	9010	335.2	
23. Cyanide Amenable to Chlorination		335.1	412F
24. Fluoride-Total		340.2	
Ion Chromatography		300.0	
25. Gold-Total		231.1	
26. Hardness-Total		215.1 + 241.1 200.7	
27. Hydrogen Ion (pH)		150.1	
28. Iron-Total	7380 6010	236.1 200.7	
29. Kjeldahl Nitrogen-Total		351.3	

TABLE K9-1 (continued)

Parameters	Approved Methods		
	SW-846	EPA 1983	Standard Method 16th Edition
30. Lead-Total	7420 7421 6010	239.1, 239.2 200.7	
31. Magnesium-Total	7450 6010	242.1 200.7	
32. Manganese-Total	7460 6010	243.1 200.7	
33. Mercury-Total	7470, 7471	245.1, 245.5	
34. Molybdenum-Total	7480 6010	246.1 200.7	
35. Nickel-Total	7520 6010	249.1 200.7	
36. Nitrate (as N) Ion Chromatography		353.3 300.0	 429
37. Nitrate-nitrite		353.3 300.0	418C 418F
38. Nitrite (as N) Ion Chromatography		 300.0	419 429
39. Oil and Grease-Total recoverable	9071	413.1	503D
40. Organic Carbon-Total (TOC)		415.1	505
41. Organic Nitrogen (as N)			420
42. Orthophosphate (as P)		365.3	
43. Osmium-Total	7550 6010	252.1 200.7	303C
44. Oxygen, dissolved		360 405.1	421B 412F

TABLE K9-1 (continued)

Parameters	Approved Methods		
	SW-846	EPA 1983	Standard Method 16th Edition
45. Palladium-Total		253.1	303A 304
46. Phenols-Total		420.1	556
47. Platinum-Total		255.1	
48. Potassium-Total	7610 6010	258.1 200.7	
49. Residue-Total		160.3	209
50. Residue-filterable (TDS)		160.1	209
51. Residue-nonfilterable (TSS)		160.2	209
52. Residue-settleable		160.5	209
53. Residue-volatile (TVS)		160.4	209
54. Selenium-Total	7740 6010	270.2 200.7	
55. Silica-dissolved		200.7	
56. Silver-Total	7760 6010	272.1, 272.2 200.7	
57. Sodium-Total	7770 6010	273.1, 200.7	
58. Specific Conductance		120.1	
59. Sulfate (as SO^2) Ion Chromatography		375.4 300.0	
60. Sulfide (as S)	9030	376.1	
61. Sulfite (as SO^3)		377.1	428A
62. Surfactants		425.1 Hach*	512A

*Hach Crystal Violet Analytical Chem 38, 791 (1966)

TABLE K9-1 (continued)

Parameters	Approved Methods		
	SW-846	EPA 1983	Standard Method 16th Edition
63. Temperature, °C		170.1	212
64. Thallium-Total	7841 6010	279.2 200.7	
65. Tin-Total	7870 6010	282.1 200.7	
66. Titanium		283.1 200.7	
67. Vanadium	7910 6010	286.1 200.7	
68. Zinc-Total	7950 6010	289.1 200.7	

TABLE K9-1 (continued)

EPA Methodologies

Purgeable Halocarbons - Method 601 or 8010

Bromodichloromethane
Bromoform
Bromomethane
Carbon Tetrachloride
Chlorobenzene
Chlorethane
2-Chloroethylvinyl Ether
Chloroform
Chloromethane
Dibromochloromethane
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Dichlorodifluoromethane
1,2-Dichloroethane
1,1-Dichloroethane
trans-1,2-Dichloroethane
1,2-Dichloropropane
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
Methylene Chloride
1,1,2,2-Tetrachloroethane
Tetrachloroethene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Tetrachloroethane
Trichlorofluoromethane
Vinyl Chloride

Purgeable Aromatics - Method 602 or 8020

Benzene
Chlorobenzene
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Ethylbenzene
Toluene

Drinking Water VOC's - Method 502.2 or 524

TABLE K9-1 (continued)

Phthalate Esters - Method 606 or 8060

Bis(2-ethylhexy) phthalate
Butyl benzyl phthalate
Di-n-butyl phthalate
Diethyl phthalate
Dimethyl phthalate
Di-n-octyl phthalate

Organochlorine Pesticides and PCB's - Method 608 or 8080

Aldrin
α-BHC
β-BHC
γ-BHC
δ-BHC
Chlordane
4,4'-DDD
4,4'-DDE
4,4'-DDT
Dieldrin
Endosulfan I
Endosulfan II
Endosulfan Sulfate
Endrin
Endrin Aldehyde
Heptachlor
Heptachlor Epoxide
Toxaphene
PCB-1016
PCB-1221
PCB-1232
PCB-1242
PCB-1248
PCB-1254
PCB-1260

PAH's (PNA's) - Method 610 or 8100

Acenaphthene
Acenaphthylene
Anthracene
Benzo(a)anthracene
Benzo(a)pyrene
Benzo(b)fluoranthene
Benzo(g)perylene
Benzo(k)fluoranthene
Chrysene
Dibenzo(a,h)anthracene

TABLE K9-1 (continued)

Fluoranthene
Fluorene
Indeno(1,2,3-cd)pyrene
Naphthalene
Phenanthrene
Pyrene

Priority Pollutants 1984 October EPA 624
Volatile Organic Compounds (Method 624 or 8240 - Purge and Trap GC/MS)

Extractable Organic Compounds (EPA Method 625 or 8270 - Extraction GC/MS)

REFERENCES

1) Standard Methods for the Examination of Water and Wastewater, 16th Edition.

2) SW-846, Test Methods for Evaluation Solid Waste, Physical, Chemical Methods, 2nd Edition, USEPA, 1982.

3) SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, USEPA, 1986.

4) Annual Book of ASTM Standards 1983, 1984.

5) EPA-600/4-79-020 Methods for Chemical Analysis of Water and Wastes, Revised March 1983.

6) EPA-600/4-82-057 Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, March 1983.

K10.0 DATA VERIFICATION

The data verification steps described in Revision 1 of the ITAS QAM will be followed by the ITAS-Knoxville laboratory. The modifications used by the ITAS-Knoxville are described below:

K10.1.1. Specific Routine Procedures Used to Assess Data Revision and Accuracy

- The analysis of field, trip, bottle, and rinsate blanks as well as field spikes and duplicates, are performed upon client request.
- The frequency of analysis and criteria for acceptability of quality control samples are described in the appropriate methods or SOPs.
- Blind duplicates will be introduced by the QCC for one major analysis type (volatiles, semivolatiles, pesticides/PCBs, metals, explosives, or general chemistry) per month.

K10.2.1 Review of Data Processing

The "original check" procedure is used by the ITAS-Knoxville laboratory.

K11.0 DATA REPORTS

The format and content of a data report are dependent upon project needs, such as: whether or not explanatory text is required, client or contract requirements, and government agency reporting formats. The ITAS Quality Assurance Program does not specify a report format; however, the following are modifications to the data presentation recommendations in the QAM:

- The final data presentation shall be checked in accordance with data verification requirements of Section 10.2.2 and approved by the Laboratory Manager or Operations Manager.
- Data are generally formatted as a Certificate of Analysis as shown in Figure K11-1, or as the Certificate of Analysis shown in Figure K11-2 for intercompany projects.
- Explanatory text is issued with analytical results as appropriate, e.g., as a case narrative with CLP data packages, as required by the client, and to explain nonconformances/analytical difficulties that have an effect on data interpretation/usage.

Figure K11-1

INTERNATIONAL TECHNOLOGY CORPORATION

ANALYTICAL SERVICES

5815 Middlebrook Pike • Knoxville Tennessee 37921 • 615-588-6401

CERTIFICATE OF ANALYSIS

TO

DATE REPORTED
PROJECT CODE
ORDER NUMBER
PAGE _____ OF _____

PERFORMED BY

TITLE

Accredited by the American Association for Laboratory Accreditation, Accreditation of the Institute has been awarded in the current AALA Directory of Accredited Laboratories

Figure K11-2

INTERNATIONAL TECHNOLOGY CORPORATION

ANALYTICAL SERVICES

CERTIFICATE OF ANALYSIS

K12.0 RECORDS MANAGEMENT

The records management system followed by ITASK complies with the criteria listed in the QAM, in that:

- o Data are secure, retrievable, and complete,
- o Laboratory records are available from time of sample receipt through sample disposal,
- o Laboratory records are stored in a manner that safeguards their integrity,
- o Reported results are available upon client request, and
- o Data are stored in a manner that allows prompt retrieval and assembly into a complete profit file, as needed.

The following SOP's provide the details for compiling project records within the individual departments:

ITASK SOP No. WW870713R0, "Records Management - WW".

ITASK SOP No. AA870713R0, "Records Management - AA/ICP".

ITASK SOP No. OP870713R0, "Records Management - Organic Prep".

ITASK SOP No. GC840523R2, "Records Management - GC".

ITASK SOP No. M_870715R0, "Records Management - GC/MS".

K12.1 Project Records

Project files are established and maintained for each laboratory project (usually one shipment of samples for one client). Each file is assigned a unique alpha-numeric project code consisting of a 3-4 letter acronym for client name/location, followed by

K12.1 Project Records (cont.)

a sequentially assigned 5 digit number (e.g., EPAL 42051). Within a project file, categories of information are filed in accordance with the project index shown in Table K12.1. SOP No. QA870521R1, "Project Records Maintenance" describes the content of each of the project index categories.

K12.2 General Laboratory Operations Records

These records are filed as described in SOP No. QA870622R0, "Quality and Operations Records Retention."

PROJECT INDEX CHECKLIST

Project Code: _____

FILE DESIGNATION	RECORD DESCRIPTION	INCLUDED IN FILE	IF "NO" SPECIFY LOCATION OF FILE
A	Correspondence	YES___ NO___ N/A___	_____
B	Tracking Records/ Coding Information	YES___ NO___ N/A___	_____
C	Organic Analytical/ QC Information		
	- GC	YES___ NO___ N/A___	*
	- HPLC	YES___ NO___ N/A___	*
	- GC/MS	YES___ NO___ N/A___	*
	- Misc. Organics	YES___ NO___ N/A___	_____
	- Organic Prep	YES___ NO___ N/A___	_____
	- Subcontractor	YES___ NO___ N/A___	_____
D	Inorganic Analytical/ QC Information		
	- Metals	YES___ NO___ N/A___	*
	- Water/Wastewater	YES___ NO___ N/A___	_____
	- Subcontractor	YES___ NO___ N/A___	_____
E	Data Reports	YES___ NO___ N/A___	_____
F	Nonconformances	YES___ NO___ N/A___	_____
G	QAPP Summary/ Proj. Spec. Req.	YES___ NO___ N/A___	_____

*GC, HPLC, GC/MS, and metals raw data and data packages are filed in the respective labs.

Completed & Reviewed By: _____ Date: _____

K13.0 NONCONFORMANCES AND CORRECTIVE ACTION

The procedures for handling nonconformances and corrective actions will be followed as described in Revision 1 of the ITAS QAM. However, the nonconformance memo has been standardized for use within all groups in the laboratory (see Figure K13-1). The nonconformance memo will no longer be used to document routine re-extraction, reanalysis, and rework options which are handled at the bench-level or by operations. However, these bench-level/operations decisions will be documented either in case narratives or exception reports.

Corrective actions will be verified by the QCC or Assistant QCC when possible. The log used to track nonconformances and corrective actions is attached (Figure K13-2).

K13.3 Internal Nonconformance Corrective Action Procedure

The only exception to the internal nonconformance corrective action procedure described in the QAM involves the use of nonconformance equipment tags. Equipment/hardware failure will not result in an adverse effect on data quality, as such equipment is taken out of service and repaired as soon as possible. At such time when the laboratory becomes an around the clock operations, nonconformance tags may be used for communication to the 2nd and 3rd shifts.

K13.4 External Nonconformance Corrective Action Procedure

The intent of the external nonconformance corrective action procedure described in the QAM is followed by ITAS-Knoxville. However, the method for documenting such nonconformances is different; i.e., the memorandum form is used rather than a nononformance form.

FIGURE K13-1

ITAS KNOXVILLE LABORATORY

NONCONFORMANCE MEMO

PROJECT CODE: _____ FILED BY: _____ DATE: _____

SAMPLE(S) AFFECTED: _____

AREA: CODING _____ GC _____ GC/MS _____ HPLC _____ PREP _____ METALS _____ GEN CHEM _____ OTHER _____

NONCONFORMANCE (Check appropriate item(s)):

____ Not enough sample received for proper analysis.

____ Sample received ☐broken ☐without proper preservative _____ ☐without proper refrigeration _____ ☐in improper container _____ ☐with custody seal missing or broken ☐ with illegible sample numbers or labels missing.

____ Sample received with incomplete paperwork: ☐Request for Analysis form missing ☐Chain-of-Custody form missing/unrelinquished ☐billing instructions incomplete ☐no due date or TAT specified ☐method for sample disposition not specified ☐numbers on paperwork different than numbers on bottles _____ ☐other _____.

____ Holding time exceeded ☐at receipt ☐in the lab by ____ days. Reason. _____.

____ Sample lost during extraction/analysis; no re-prep or re-analysis possible. Reason: _____.

____ QC sample data reported to client outside of ☐method ☐internal ☐QAPP ☐contract ☐regulatory limits. Reason: _____.

____ Incorrect preparation/analysis procedure(s) used; no re-prep or re-analysis possible. Reason: _____.

____ Samples analyzed and reported under an invalid calibration curve.

____ Incorrect/incomplete data reported to client. Specify: _____.

____ Blank contamination; no re-prep of associated samples done or possible. Reason: _____.

____ Reported detection limits higher than specified in ☐method ☐contract ☐QAPP, due to ☐matrix ☐insufficient sample ☐instrumentation ☐other _____.

____ Other (specify) _____.

CORRECTIVE ACTION:

____ Client (Name: _____) informed verbally on _____ by _____.
____ Client informed in writing on _____ by _____.
____ Sample(s) processed "as is".
____ Sample(s) preserved in laboratory (specify): _____.
____ Sample(s) on hold until _____.
____ Other _____.

Corrective action initiated/performed by _____ Date _____

Operations Concurrence: _____ Date _____

QC Concurrence: _____ Date _____

CORRECTIVE ACTION VERIFICATION

____ Verified

____ Cannot verify. Reason _____.

QC Coordinator: _____ Date _____

THIS DOCUMENT MUST BE RETAINED IN THE PROJECT FILE

INTERNAL NONCONFORMANCE LOG

Page: _____

ITAS-K-QA035R0

Date of Entry	Date N/C was Initiated	Description of Problem	Responsible Group and Supervisor	Description of Corrective Action	Project(s) Affected	Sample Number(s) Affected	Close-Out Date	QCC Verification of Corrective Action

FIGURE K13-2

K14.0 QUALITY ASSURANCE/QUALITY CONTROL AUDITS

In addition to the performance audits conducted by the QC Coordinator and the systems audits conducted by the ITAS QA Manager, ITASK is frequently audited by various Federal and State agencies and other contractors. Results of such audits shall be reported to the Laboratory Director, the Laboratory Manager, the Technical Director, and the ITAS QA/C Director.

ITASK follows the ITAS QA Manual with the exception of the following changes and supplements.

The QCC monitors laboratory operations by conducting monthly surveillances and semi-annual internal audits. The QCC will observe the activity of interest or review objective evidence; checklists may be used for this review, if applicable.

K15.0 QUALITY REPORTS TO MANAGEMENT

ITASK follows the ITAS QA Manual with the exception of the following changes and supplements.

15.1 Performance Audit Reporting

The results of the monthly performance audit will be included in the QCC's monthly report to the QA/C Director. The format of the monthly performance audit report will vary depending on the activity audited. The Laboratory Manager will receive a copy of this report.

K16.0 TRAINING

This section supplements Section 16 of the ITAS Quality Assurance Manual. The general types of qualification procedures are addressed and the records maintained to document employee qualifications and training are described.

K16.2 Professional Staff, Training and Qualifications

Specific criteria for qualification in an area will be established by the area supervisor or Group Leader. The qualification procedures must receive approval from the Technical Director and the Laboratory Manager or Operations Manager. As criteria are established, the Group Leaders (Supervisors) will document the procedures in appropriate SOP's.

Training may include attendance of off-site and/or in-house (ITAS/ITASK) training courses appropriate to the employee's area. Observation of step performance and written tests will be included as appropriate to employee qualification.

At a minimum, initial qualification will require the acceptable performance on a Performance Evaluation sample. The Performance Evaluation (PE) sample will be an external SRM or PE sample, when available. The PE sample may be a laboratory generated sample.

Annual requalification may be demonstrated by acceptable performance on a PE sample.

Only the QC Coordinator and assistant will receive training in the performance of audits.

K16.0 TRAINING (continued)

K16.4 Qualification and Training Records

The QC Coordinator will maintain a qualification and training record for each technical employee. Each employee's file will contain:

1. Personnel resume
2. Results of qualifying sample sets (PE samples)
3. Quality Assurance examinations
4. Attendance records of training sessions
5. Internal training certificates of qualifications (Personnel Qualification Record).

The personnel resume will replace Figure 16.1.

ITASK has developed its own Personnel Qualification Record forms. Figure K16.1 - K16.14 are examples.

FIGURE K16.1

ITAS - MIDDLEBROOK PIKE
GC LAB ANALYST TRAINING LOG

Analyst's Name _____ Technician Level _____

Date Hired _____ Chemist Level _____

This form is to certify that the above named analyst has been instructed in and has successfully completed analyses using the following test methods. The GC Group Supervisor will date and initial to indicate which analyses the analyst has sufficient knowledge of in order to successfully perform sample analyses.

Methods:	Date	Initial
(EPA) 608		
Organochlorine pesticides in water	____	____
Polychlorinated biphenyls in water	____	____
(EPA) 617		
Organochlorine pesticides in city water	____	____
(SW-846) 8080		
Organochlorine pesticides in soil	____	____
Organochlorine pesticides in waste	____	____
Organochlorine pesticides in RCRA (EP Tox)	____	____
Polychlorinated biphenyls in soil	____	____
Polychlorinated biphenyls in waste	____	____
Polychlorinated biphenyls in surface wipes	____	____

Signatures: Analyst _____ Date _____

Group Supervisor _____ Date _____

QC Coordinator _____ Date _____

FIGURE K16.2

GC LAB ANALYST TRAINING LOG
(continued)

Analyst's Name _____

Methods:	Date	Initial
(SOP) GC870731R0		
PCB's in oil	____	____
(SW-846) 5503		
PCB's in air (Florisil tubes)	____	____
(SW-846) 8150		
Chlorinated herbicides in soil	____	____
Chlorinated herbicides in waste	____	____
Chlorinated herbicides in RCRA (EP Tox)	____	____
(SOP) GC860523R0		
Chlorinated herbicides in city water	____	____
(EPA) 604		
Phenols	____	____
(SW-846) 8015/8020		
BTXE/gasoline	____	____
Gasoline/diesel fuel	____	____
High boiler hydrocarbons	____	____
(EPA) 601/602	____	____

Signatures: Analyst _____ Date _____

Group Supervisor _____ Date _____

QC Coordinator _____ Date _____

FIGURE K16.3

GC LAB ANALYST TRAINING LOG
(continued)

Analyst's Name _____

Methods:	Date	Initial
(SOP) GC85062480 - Pest/PCB's by CLP	____	____
P • CAM 127 Industrial Hygiene (GC 851230R0)	____	____
(SOP) FA85080281 - Livingston	____	____
(SOP) GC85061980 - THM's	____	____
(SW-846) 9060		
Total organic and inorganic carbon in water	____	____
Total non-purgeable organic carbon in soils	____	____
Total inorganic carbon in soils	____	____
Total purgeable organic carbon in soils	____	____
(SOP) OM86102380		
Total organic halogen in water	____	____
(EPA) 502		
Volatile organic compounds	____	____

Signatures: Analyst _____ Date _____

Group Supervisor _____ Date _____

QC Coordinator _____ Date _____

FIGURE K16.4

ITAS-MIDDLEBROOK PIKE
WATER/WASTEWATER LABORATORY
ANALYST TRAINING LOG

Analyst's Name _____ Technician Level _____
Date Hired _____ Chemist Level _____

This form is to certify that the above named analyst has been instructed in and has successfully completed analyses using the following test methods. The Water/Wastewater Group Supervisor will date and initial to indicate which analyses the analyst has sufficient knowledge of in order to successfully perform sample analyses.

Methods:

Titrimetric		Date	Initials
Acidity	(EPA 305.2)		
Alkalinity	(EPA 310.1)		
COD	(EPA 410.1 & 410.2)		
Chloride	(EPA 352.3)		
Sulfide	(EPA 376.1)		
Sulfide	(SW-846, Meth. 9030)		
Sulfite	(EPA 377.1)		
Un-Ionized H_2S	(Std. Meth. 427E.)		
Iodide	(EPA 345.1)		
Turbidimetric			
Sulfate	(EPA 375.4)		
Turbidity	(EPA 180.1)		
Biological			
BOD	(EPA 405.1)		
Fecal Coliform	(Std. Meth. 909C)		
Total Coliform	(Std. Meth. 909A)		

Signatures: Analyst _____ Date _____
Group Supervisor _____ Date _____
QC Coordinator _____ Date _____

FIGURE K16.5

ANALYST TRAINING LOG (continued)

Analyst's Name _____

Methods:

Distillations/Digestions		Date	Initials
Ammonia	(EPA 350.2)		
COD	(EPA 410.1 & 410.2)		
Cyanide, Total	(EPA 335.2)		
CN^-, Free	(Std. Meth. 412H)		
CN^-, Amenable to Cl^-	(EPA 335.1)		
Fluoride	(EPA 340.1)		
Total Kjedahl Nitrogen	(EPA 351.3)		
Phenols, Total	(EPA 420.1)		
Phosphorus, Total	(EPA 365.3)		
Colorimetric			
Ammonia/TKN	(EPA 350.2 & 351.3)		
Bromide	(Std. Meth. 405)		
Color	(EPA 110.2)		
Cyanide	(EPA 335.1 & 335.2)		
	(Std. Meth. 412H)		
Iodine	(HACH DPD Method)		
Nitrate	(EPA 353.3 by HACH)		
Nitrite	(EPA 353.3 by HACH)		
Phosphorus, Total	(EPA 365.3)		
Ortho-Phosphate	(EPA 365.3)		
Residual (Free) Chlorine	(EPA 330.5)		
Hexavalent Chromium	(Std. Meth. 312B)		
Extractions			
Oil & Grease	(EPA 413.1)		
Oil & Grease	(Std. Meth. 503D)		
Oil & Grease	(SW-846, Meth. 9071)		
Phenols	(EPA 420.1)		
Surfactants	(EPA 425.1)		

Signatures: Analyst _____ Date _____

Group Supervisor _____ Date _____

QC Coordinator _____ Date _____

FIGURE K16.6

ANALYST TRAINING LOG (continued)

Analyst's Name _____

Methods:

Electrode		Date	Initials
Chloride	(Std. Meth. 407C)		
Fluoride	(EPA 340.2)		
pH	(EPA 150.1)		
pH	(SOP WW01-WW801119R1)		
Specific Conductance	(EPA 120.1)		
Residues			
Moisture	(*)		
Total Solids	(EPA 160.3)		
Total Dissolved Solids	(EPA 160.1)		
Total Suspended Solids	(EPA 160.2)		
Total Settleable Solids	(EPA 160.5)		
Total Volatile Solids	(EPA 160.4)		
Total Volatile Suspended Solids	(*)		
Miscellaneous			
Bicarbonates	(Std. Meth. 403/406 A or C)		
Carbonates	(Std. Meth. 403/406 A or C)		
Hydroxide	(Std. Meth. 403/046 A or C)		
Odor	(EPA 140.1)		
Ion Chromatography			
Anion Scan	(EPA 300.0)		
	(Std. Meth. 429)		
	($F^-/Cl^-/Br^-/NO_2/NO_3/PO_4/SO_4$)		
Other _____			
_____			

*SOP to be written

Signatures: Analyst _____ Date _____

Group Supervisor _____ Date _____

QC Coordinator _____ Date _____

FIGURE K16.7

ANALYST TRAINING LOG (continued)

Analyst's Name _____

Methods:

Other Test Methods	Date	Initials

Signatures: Analyst _____ Date _____

Group Supervisor _____ Date _____

QC Coordinator _____ Date _____

FIGURE K16.8

ITAS-MIDDLEBROOK PIKE
AA/ICP LABORATORY
ANALYST TRAINING LOG

Analyst's Name _____ Technician Level _____
Date Hired _____ Chemist Level _____

This form is to certify that the above named analyst has been instructed in and has successfully completed analyses using the following test methods. The AA/ICP Group Supervisor will date and initial to indicate the analyst has sufficient knowledge of each of the following test methods to successfully perform sample analyses.

FIGURE K16.9

Methods:

Sample Preparation	Date	Initials
Sample preservation to pH <2 with HNO_3		
Percent solids per CLP SOW		
Density ASTM D1475-60		
Dissolved Metals by Filtration 0.45 u		
EPA SW-846 Method 3005		
EPA SW-846 Method 3010		
EPA SW-846 Method 3020		
EPA SW-846 Method 3050 Flame		
EPA SW-846 Method 3050 Furnace		
EPA CLP Modified 3010		
EPA CLP Modified 3020		
EPA CLP Modified 3050 Flame		
EPA CLP Modified 3050 Furnace		
Total Metals in Air P:CAM 173		
Total Metals on Glass Fiber Fillers HNO_3 Leach		
Preparation PE/SRM Per Instructions		
EP Toxicity Extraction SW-846 1310		
pH MCAWW 150.1		

FIGURE K16.10

				Date	Initials
ICAP	Inductively Coupled Argon Plasma Spectroscopy				
	Methods for Chemical Analysis of Water: Wastes (MCAWW) 200.7				
	EPA-SW-846 Method 6010				
	EPA-CLP Modified 200.7				
GFAA	Graphite Furnace Atomic Absorption Spectroscopy				
	MCAWW	Antimony	204.2		
		Arsenic	206.2		
		Lead	239.2		
		Selenium	270.2		
		Thallium	279.2		
	SW-846	Antimony	7041		
		Arsenic	7060		
		Lead	7421		
		Selenium	7740		
		Thallium	7840		
	CLP-Mod	Antimony	204.2		
		Arsenic	206.2		
		Lead	239.2		
		Selenium	270.2		
		Thallium	279.2		

FIGURE K16.11

				Date	Initials
CVAA	Manual Cold Vapor Atomic Absorption Spectroscopy				
	MCAWW	Mercury in Liquids 245.1			
		Mercury in Sediments 245.5			
	SW-846	Mercury in Liquid Waste 7470			
		Mercury in Solid or Semisolid Waste 7471			
	CLP-Mod	Mercury in Water 245.1			
		Mercury in Soil/Sediment 245.5			
FAA	Flame Atomic Absorption Spectroscopy				
	MCAWW	Aluminum	202.1		
		Barium	208.1		
		Beryllium	210.1		
		Cadmium	213.1		
		Calcium	215.1		
		Chromium	218.1		
		Cobalt	219.1		
		Copper	220.1		
		Gold	231.1		
		Iron	236.1		
		Lead	239.1		
		Magnesium	242.1		
		Manganese	243.1		
		Molybdenum	246.1		
		Nickel	249.1		
		Osmium	252.1		
		Platinum	255.1		
		Potassium	258.1		

FIGURE K16.12

FAA	Flame Atomic Absorption Spectroscopy (continued)			Date	Initials
	MCAWW	Silver	272.1		
		Sodium	273.1		
		Tin	282.1		
		Titanium	283.1		
		Vanadium	286.1		
		Zinc	289.2		
	SW-846	Aluminum	7020		
		Barium	7080		
		Beryllium	7090		
		Cadmium	7130		
		Calcium	7140		
		Chromium	7190		
		Cobalt	7200		
		Copper	7210		
		Iron	7380		
		Lead	7420		
		Magnesium	7450		
		Manganese	7460		
		Molybdenum	7480		
		Nickel	7520		
		Osmium	7550		
		Potassium	7610		
		Silver	7760		
		Sodium	7770		
		Tin	7870		
		Vanadium	7911		
		Zinc	7950		

FIGURE K16.13

FAA	Flame Atomic Absorption Spectroscopy (continued)		Date	Initials
	CLP-Mod Aluminum	202.1		
	Barium	208.1		
	Beryllium	210.1		
	Cadmium	213.1		
	Chromium	218.1		
	Cobalt	219.1		
	Copper	220.1		
	Iron	236.1		
	Lead	239.1		
	Manganese	243.1		
	Nickel	249.1		
	Silver	272.1		
	Vanadium	286.1		
	Zinc	289.2		

FIGURE K16.14

Other	Date	Initials
SW-846 Chromium, Hexavalent Coprecipitation 7195		

Signatures: Analyst _____ / Date _____

Group Supervisor _____ / Date _____

QC Coordinator _____ / Date _____

APPENDIX E

OPERABLE UNIT 3 STANDARD OPERATING PROCEDURES

STANDARD OPERATING PROCEDURE 1.1

GENERAL INSTRUCTIONS FOR FIELD PERSONNEL

1. PURPOSE

To provide field personnel with instructions regarding activities to be performed before, during, and after field investigations.

2. DISCUSSION

The Field Sampling Plan (FSP) or Work Plan (WP) contains specific details about procedures and equipment for a given operation. Refer to the FSP or WP for the type of samples, measurements, and tests to be collected or performed. The collection and documentation of data should be performed as described in specific SOPs. These general instructions are intended to supplement the information supplied in the FSP or WP and associated SOPs and clarify the role of field personnel at remedial investigations. These instructions will ensure that field personnel take the proper precautions to understand the site, the objective and the schedule for the field program, their authority, and their responsibilities described in the FSP or WP.

This SOP is supported by others that describe procedures and rationale for performing reconnaissance geophysical and soil gas surveys; soil and rock boring; sample logging; soil and sediment sample collection; installation and operation of vadose-zone instruments and samplers; groundwater monitoring well installation, development, and sampling; operation of sampling equipment; performance of aquifer testing; collection, preservation, handling, packaging, and shipping of samples; decontamination procedures; health and safety monitoring; and radiological surveys.

3. PROCEDURES

3.1. Associated Procedures

Before every operation, a review of this SOP and SOPs 1.3-1.10 is required. In addition, a review of associated SOPs for each task is necessary. The associated SOPs are listed in Section 3.1 of each task SOP. Constant review of the SOPs will ensure that the work performed in the field is legally defensible, well documented, and cost-effective. The decontamination procedures are important for protecting the health and safety of workers.

3.2. Preparation

3.2.1. Office

A. Personnel should review the FSP or WP and associated documentation for a specific operation and obtain all information related to the purpose and intent of the field program. This may include (but is not limited to) the documents listed below.

1. The scope of work or work plan described in the FSP or WP

2. Previous reports related to the site

3. Reports related to the area

4. Site maps

5. Area maps

6. Access agreements

7. The subcontractor's work plan

8. Data collection and equipment checklists

9. Associated SOPs

B. Field personnel are expected to maintain a good working relationship with the client, community, and subcontractors. With this in mind, field personnel should contact installation staff, members of the community (in coordination with installation staff), and subcontractors before work is initiated. During the initial contact, permission to enter private property or security areas should be obtained.

C. Obtain and test all equipment needed for the task. See checklist in Appendix 5.1.

D. Most sample analyses must be performed within a stringent time period. In addition, laboratories are vulnerable to heavy overloads. Contact the laboratory before sampling activities begin to ensure that the personnel are aware of specific requirements for analyses and can complete the work quickly and efficiently.

E. Delays at the freight office can be eliminated by contacting the carrier before arrival with a shipment. The carrier can supply information on regulations and specifications for shipping, the address of the nearest delivery office, and the time of the next freight pickup in the area.

3.2.2. Documentation

A. Obtain a logbook and ER Program data collection forms. All measurements, observations, and instrument readings should be entered on the forms according to the instructions supplied. All entries should be made in black ink that is not water soluble (not a felt-tip pen). Make an entry in each blank. Where there is no data entry, enter UNK for Unknown, NA for Not Applicable, or NP for Not Performed. If any procedure was not performed as prescribed, give the reason for the change or omission on the form. To change an entry, draw a single line through it, add the correct information above it, and initial the change. Information that does not require data entry should be entered in the logbook. All logbooks are numbered, bound, and contain numbered pages for quality assurance/quality control purposes. Do not alter the logbook or data collection forms in any manner.

B. The information management codes and sample identification numbers used in data entry are assigned by the data administrator. This system is necessary to avoid duplication of site identifiers or inaccurate entries. Because the list of codes is continually being updated, SOPs cannot be revised each time a new list is produced.

C. Three days before leaving for any field trip, a previsit/pretravel report form must be submitted to DOE. The report contains information about dates of travel, mode of travel, hotel accommodations, and contact phone numbers. Arrangements for renting field vehicles should be made at this time.

3.2.3. Field

A. Check the condition and operation of all supplies and equipment at the site. Perform calibration checks specified in operators' manuals or appropriate SOPs.

B. Establish decontamination zones and barricades to public access.

3.3. Operation

A. The field personnel monitor and provide technical direction for the field work, log samples, take measurements, and sometimes pack and ship samples.

B. Under direction of the site manager, field personnel may designate sampling or hole locations, depth and completion zones, types and depths of sample, and approve and record procedures, materials, and all activities conducted in compliance with the FSP OR WP.

NOTE: Whenever a sample is collected, a custody record must be initiated on the Custody Transfer Record/Lab Work Request form and a label affixed to the sample container--either a Soil Sample Identification Label or a Water Sample Identification Label. SOP 1.3, Sample Control and Documentation, contains copies of these forms and instructions for completing the forms.

C. Additional duties that the field personnel may perform are described below.

1. Keep a logbook to record information concerning equipment, personnel, site visitors, and activities (start and stop times, supplies used, footage drilled/installed, and site observations), as well as weather or site conditions affecting the activities. The field personnel should note all relevant instructions and information. All information pertaining to a field activity should be entered in a bound book with consecutively numbered pages (see SOP 1.3, Sample Control and Documentation, for instructions on keeping the logbook). Subcontractors should sign/initial the daily log, thereby certifying that the account records agree with their estimates.

2. Telephone the site manager or office headquarters daily and provide a progress report.

3. Observe that the subcontractor complies with the FSP OR WP and all applicable permits and licenses.

4. Complete all data collection forms according to applicable instructions as work progresses.

5. Observe whether the subcontractor follows the applicable health and safety requirements. If violations occur, the field personnel should stop work and immediately notify the site manager or site health and safety officer.

6. Monitor air, personnel, and equipment for contamination and record results on appropriate forms or in the logbook.

7. Supervise decontamination of equipment and personnel. Record procedures used for decontamination in the logbook. Collect decontamination solutions in containers for appropriate disposal as specified in the FSP OR WP.

8. Notify the site manager of any modifications to the contract that may be appropriate. Work not defined in the FSP OR WP should not be initiated without the approval of the site manager.

3.4. Postoperation

3.4.1. Field

A. Ensure that all equipment is accounted for, decontaminated (see SOP 1.6, General Equipment Decontamination), and ready for shipment.

B. Restore the site to the presampling conditions as specified in the FSP OR WP. Restoration can include repair of damage to the land surface (tire ruts) or private property (fences), as well as restoration anticipated at the time the FSP OR WP was prepared (for example, revegetation or borehole abandonment).

C. Mark sampling locations or survey points with wooden lathe stakes, wooden survey pegs, or metal fenceposts. Write the location ID on the marker or survey flagging so that it is readily visible. Mark groundwater monitoring wells on the guard pipe and inside the casing cap. Use a black marker for wooden stakes, flagging, and the casing cap. Mark the guard pipes with welds or stencils and paint.

D. Shipping samples is the last task in most field operations. SOPs 1.3-1.5 should be used as guides to sample handling and transport. SOP 1.5, Guide to Handling, Packaging, and Shipping of Samples, is a summary of Department of Transportation regulations pertaining to the transport of hazardous substances most commonly sampled in the field. Use SOP 1.5 in conjunction with the appropriate Code of Federal Regulations and guidance supplied by the freight carrier to ensure that packages are documented and properly labeled.

3.4.2. Documentation

A. Record any restoration work in the logbook.

B. Record any uncompleted work in the logbook. This record may include sampling that could not be performed, damage that could not be repaired, or requirements for long-term monitoring (for example, the need to verify instrument readings at different times of the year).

C. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

D. Review data collection forms for completeness.

E. Submit a travel expense report.

3.4.3. Office

A. Deliver original forms and logbooks to the site manager for technical review. He/she will review, sign forms, and transmit to the document control officer (copies to the files) for eventual delivery to the Department of Energy.

B. Inventory equipment and supplies. Repair or replace all broken or damaged equipment. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

C. Contact the analytical laboratory to ensure that samples arrived safely and instructions for sample analyses are clearly understood.

4. SOURCES

None.

5. APPENDIX

5.1. Equipment and Supplies Checklist

APPENDIX 5.1

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Overshoes

_____ Work gloves (2 pairs)

_____ Acid (10% HCL) bottle

_____ Clipboard case

_____ Strapping tape

_____ Pin hammer

_____ Tape measure (tenths)

_____ Protractor

_____ Hat

_____ First aid kit

_____ Sun screen

_____ Thermoluminescent dosimeter (TLD)

_____ Safety shoes/boots

_____ Ziplock bags

_____ Preprinted labels

_____ Distilled (organic-free) water

_____ Methanol (Nanograde)

_____ Freight forms

_____ Telephone directories

_____ Chain-of-custody forms

_____ Hard hat

_____ Pieces of wood (2 inches x 2 inches x 8 inches) to indicate core loss intervals

APPENDIX 5.1, continued

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Stamped, addressed envelopes (large and regular sizes)

_____ Phone and gas credit cards

_____ Calculator

_____ Pens, pencils, and permanent markers

_____ Package cord

_____ Flagging

_____ Hand lens

_____ Tool box

_____ Rain suit

_____ Camera

_____ Ear plugs

_____ Stopwatch

_____ Cold-weather gear

_____ Alpha meter

_____ Safety glasses

_____ Kitchen screen (determine lithology)

_____ Ice chest

_____ Bound logbook

_____ Data collection forms

STANDARD OPERATING PROCEDURE 1.3

SAMPLE CONTROL AND DOCUMENTATION

1. PURPOSE

To define the steps necessary for sample control and identification, data recording, and chain-of-custody documentation.

2. DISCUSSION

The Field Sampling Plan (FSP) or Work Plan (WP) contains specific details about the procedures and equipment for this SOP. Refer to the FSP or WP for the type of samples to be collected and the destination of the collected samples. Collection and measurement of samples and the documentation of data will be performed as described in specific SOPs.

This SOP describes the methodology of sample control and documentation for all projects. Sample control and documentation are necessary to ensure the defensibility of data and to verify the quality and quantity of work performed in the field. Accountable documents include logbooks, data collection forms, correspondence, sample labels or tags, chain-of-custody reports, photographs, and analytical records. Waterproof black ink must be used in recording all data in documents bearing serial numbers.

The logbooks are under the supervision of the quality control officer. There may be several logbooks; for example, there may be a separate logbook for field activities, one for samples, and one for instruments. The quality control officer numbers the logbooks and assigns them to individuals designated for specific tasks of the project. All information pertinent to a field activity must be entered into a logbook. A record of uncompleted work is kept in a logbook. All project logbooks are turned over to the document control officer at the end of each work period and to a central file at the end of the field activity.

All logbooks are numbered and bound, and the pages are numbered. Waterproof black ink is used for recording all data. Logbook pages should never be removed, and no data should removed. To change an incorrect entry, the individual draws a line through the entry, writes the change above the entry, dates and initials the change. If anyone other than the person to whom the logbook is assigned makes an entry, that person dates and signs the entry.

Record all information pertinent to the sampling activity (for example, date, site, ID number, and location) in the logbook. Note the field conditions, weather conditions, and any unusual circumstances. Notes should be as descriptive and inclusive as possible. A person reading the entries should be able to reconstruct the sampling situation from the recorded information. Language should be objective, factual, and free of personal feelings and inappropriate terminology.

3. PROCEDURES

3.1. Associated Procedures

Before every operation, a review of the SOPs 1.1-1.10 is necessary. These SOPs contain information on the performance of field activities. They should be consulted for specific information about equipment and supplies; sample collection, preservation, packaging, and shipping; decontamination procedures; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.4	Sample Containers and Preservation
1.5	Guide to the Handling, Packaging, and Shipping of Samples

3.2. Preparation

3.2.1. Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Obtain appropriate permission for property access.

D. Assemble the equipment and supplies listed in Appendix 5.1. Ensure the proper operation of all sampling equipment.

E. Notify the analytical laboratory of sample types, the number of samples, and the approximate arrival date.

F. Contact the carrier that will transport samples to obtain information on regulations and specifications.

3.2.2. Documentation

A. Obtain a logbook from the QA officer.

B. Record results of the equipment check in the logbook.

C. Obtain a sufficient number of the appropriate ER Program data collection forms (see INDEX TO SOPs).

D. Consult the ER Program data administrator for a current list of information management codes, location IDs, and sample numbers used in the completion of data forms.

3.2.3. Field

Field preparation requires organizing sample bottles, sample labels, and documentation in an orderly, systematic manner that promotes consistency and traceability of all data. Appropriate items should be completed before a sample is collected.

A. Record all pertinent information (for example, date, site, ID number, and location) in the logbook. Note field conditions, unusual circumstances, and weather conditions.

B. Fill out information on the sample identification label and attach the label to a sample bottle.

C. Complete initial information required on data collection forms.

3.3. Operation

3.3.1. Logbook

Enter all information pertinent to a field activity in a bound logbook with consecutively numbered pages. Enter information that does not require data entry into the logbook. If not included on a data collection form, entries in the logbook should include at least the information listed below.

- Date and time of entry
- Purpose of sampling
- Name and address of field contact
- Site identification
- Type of process producing waste (if known)
- Type of sample (soil, water, sediment, etc.)
- Sample identifier and size of sample taken
- Type of analysis requested
- Description of sampling point or location number
- Date and time for collection of sample
- Collector's sample identification number(s) and/or name
- References of the sampling site (like maps or photographs)
- Field observations and sampling locations
- Associated field measurements
- Method of sample collection, preservation techniques, and any deviations or anomalies noted

- Transfer of a logbook to individuals designated for specific tasks of the project

- Any uncompleted work

For water samples, the TEGD suggests the following additional information.

- Presence of immiscible layers and detection method

- Collection method for immiscible layers

- Sample distribution and transporter

- Climatic conditions including air temp

- Internal temperature of field and shipping container

Because sampling situations vary widely, make notes as descriptive and inclusive as possible. A person reading the entries should be able to reconstruct the sampling situation from the recorded information. Use language that is objective, factual, and free of personal feelings or any other inappropriate terminology. If anyone other than the person to whom the logbook was assigned makes an entry, he/she should date and sign the entry. Never remove logbook pages. If a mistake is made, draw a single line through the mistake, write the new information above the line, and date and initial the change.

3.3.2. Photographs

Photographs provide the most accurate record of the field worker's observations. They can be significant during future inspections, informal meetings, and hearings. A photograph must be documented to be a valid representation of an existing situation. For each photograph taken, record the items listed below in the logbook and on the back of each processed photograph.

- Date and time

- Signature of photographer

- Name and identification number of site

- Type of camera, lens, f-stop, shutter speed, and film used

- General direction faced and description of the subject

- Distance from photographer to object

- Location at the site

- Sequential number of photograph and the roll number

Any remarks about the contents of a photograph could jeopardize its value as legal evidence, so limit comments to the photograph's location. Photographs should be taken with a perspective similar to that afforded by the naked eye. Telephoto or wide-angle shots cannot be used in enforcement proceedings.

3.3.3. Sample Labels

Use soil and water sample identification labels to tag or label sample containers. Seal each sample immediately after it is collected and labeled with waterproof black ink. Label tags may be filled out before collection to minimize the handling of the sample containers. Appendix 5.1, Soil Sample and Water Sample Identification Labels, provides examples of the common sample labels to be used. Instructions for completing the labels are included in Appendix 1.3, Data Form Completion.

When appropriate, use an etching tool to mark sample containers in the field, rather than immediately applying a sample label or tag. This avoids possible label contamination problems and subsequent decontamination difficulties. In this case, write the data intended for the sample label in the logbook and transcribe them onto the label after the sample containers have been decontaminated.

Firmly attach the labels to the sample containers. The containers must be dry enough for gummed labels to be securely attached.

3.3.4. Sample Collection and Inventory

The number of persons involved in collecting and handling samples should be kept to a minimum. Use the guidelines established in this SOP and SOP 1.5, Guide to the Handling, Packaging, and Shipping of Samples. Complete data collection forms at the time the sample is collected and have the sample collector(s) sign or initial them. Include the date and time. On liquid containers, mark the liquid level with waterproof black ink. This requirement is not necessary for completely filled volatile organics analysis (VOA) septum vials. If the volume received by the laboratory is different than when collected, the sample container may have leaked, been tampered with, or spilled hazardous materials. Use the Custody Transfer Record/Lab Work Request form, Appendix 5.2, to inventory all samples collected in the field. Instructions for the form are in Appendix 5.3, Data Form Completion.

3.3.5. Chain of Custody

A. Objectives

The primary objective of the chain-of-custody procedure is to create an accurate written record that can be used to trace the possession and handling of the sample from the moment of its collection through analysis and introduction as evidence. A sample is in someone's custody when one of the criteria listed below has been satisfied.

1. The sample is in one's actual possession.

2. The sample is in one's view after being in one's physical possession.

3. The sample is in one's physical possession and is then locked up so that no one can tamper with it.

4. The sample is kept in a secured area that is restricted to authorized personnel.

B. Transfer of Custody and Shipment

When transferring the samples, the transferee should sign and record the date and time on the Custody Transfer Record/Lab Work Request form. Custody transfers made to a sample custodian in the field should account for each sample, although samples may be transferred as a group. Every person who takes custody should fill in the appropriate section of the Custody Transfer Record/Lab Work Request form. To reduce the number of custody records, minimize the number of custodians in the chain of possession.

The field custodian is responsible for properly packaging and dispatching samples to the appropriate laboratory. This responsibility includes filling out, dating, and signing the appropriate portion of the Custody Transfer Record/Lab Work Request form. The chain-of-custody form is then placed in a sealed plastic bag and taped to the inside of the cooler lid, where it is immediately visible when the cooler is opened. Signed and dated chain-of-custody seals are also placed on the sample container lids to detect sample tampering.

In general, packaging of environmental samples will include the following: placement of sample containers in zip-lock plastic bags to reduce the chance of both breakage and release of sample if breakage does occur, and placement of the bagged sample containers in a cooler containing sufficient vermiculite to prevent breakage and to absorb any material that leaks. For water samples in 4L amber glass containers, the samples will be wrapped in "bubble wrap" to prevent breakage. Ice or blue ice is used to maintain sample temperatures at 4°C. If ice is used, it must be double-bagged in zip-lock bags. Experience has shown that fully 1/4 to 1/3 of the cooler volume must be ice, packaged around and over the sample containers, to maintain temperatures at 4°C.

Send all packages to the laboratory with the chain-of-custody record and other pertinent forms. Retain a copy of these forms at the originating office (either carbon or photocopy). Register mailed packages with a return receipt requested. For packages sent by common carrier, retain receipts as part of the permanent chain-of-custody documentation. Pack samples so that they do not break in shipment. Seal the cooler with chain-of-custody seals so that any tampering can be readily detected. SOP 1.5, Guide to the Handling, Packaging, and Shipping of Samples, describes these procedures in detail.

3.4. Postoperation

3.4.1. Field

A. Verify that all sample bottles have been correctly identified and labels have all necessary information (location, time, and date).

B. Cross-check filled sample bottles in possession against those recorded in the logbook. Maintain custody of filled sample bottles by keeping them in actual possession, within view, locked or sealed up to prevent tampering, or bringing them into a secure area.

C. Prepare samples for transport according to SOP 1.3, Sample Control and Documentation; SOP 1.4, Sample Containers and Preservation; and SOP 1.5, Guide to Handling, Packaging, and Shipping of Samples.

3.4.2. Documentation

A. Record data and any uncompleted work in the logbook.

B. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

C. Document the chain of custody on the Custody Transfer Record/Lab Work Request form.

D. Review data collection forms for completeness.

3.4.3. Office

A. Deliver original forms and logbooks to the site manager for technical review. He/she will review, sign forms, and transmit to the document control officer (copies to the files) for eventual delivery to the Department of Energy.

B. Inventory equipment and supplies. Repair or replace all broken or damaged equipment. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

C. Contact the analytical laboratory to ensure that samples arrived safely and instructions for sample analyses are clearly understood.

4. SOURCE

EPA. 1986. "RCRA Ground-water Monitoring Technical Enforcement Guidance Document." U.S. Environmental Protection Agency document. Washington, D.C.: U.S. Government Printing Office.

5. APPENDIXES

5.1. Soil Sample and Water Sample Identification Labels

5.2. Custody Transfer Record/Lab Work Request Form

5.3. Data Form Completion

APPENDIX 5.1

SOIL SAMPLE AND WATER SAMPLE IDENTIFICATION LABELS

SOIL SAMPLE IDENTIFICATION LABEL

FACILITY CODE _____ LOCATION ID _____

SAMPLE ID _____ LOG DATE _____

LAB CODE _____ LOG TIME _____

SAMPLER _____ LOGGER CODE _____

SAMPLE DEPTH INTERVAL FROM DATUM

BEGINNING DEPTH (FT FROM DATUM) _____

ENDING DEPTH (FT FROM DATUM) _____

ANALYSIS REQUESTED _____

COMMENTS _____

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WATER SAMPLE IDENTIFICATION LABEL

FACILITY CODE _____ LOCATION ID _____

SAMPLE ID _____ LOG DATE _____

LAB CODE _____ LOG TIME _____

SAMPLER _____ LOGGER CODE _____

ANALYSIS REQUESTED _____

PRESERVATION METHOD _____

COMMENTS _____

WSL-133

APPENDIX 5.2

CUSTODY TRANSFER RECORD/LAB WORK REQUEST FORM

WESTON

WESTON Analytics Use Only

Custody Transfer Record/Lab Work Request

Client _____
Work Order _____
Date Rec'd. _____ Date Due _____
RFW Contact _____
Client Contact/Phone _____

Refrigerator#											
#/Type Container											
Containers/Volume											
Preservative											
ANALYSES REQUESTED ➡											
WA Use Only Lab ID	Client ID/Description	Matrix	Date Collected								

Matrix:
S - Soil
SE - Sediment
SO - Solid
W - Water
O - Oil
A - Air
DS - Drum Solids
DL - Drum Liquids
F - Fish
X - Other

Special Instructions:

Item/Reason	Relinquished by	Received by	Date	Time	Item/Reason	Relinquished by	Received by	Date	Time

WESTON Analytics Use Only

Samples Were:
1 Shipped or Hand-Delivered
NOTES:

2 Ambient or Chilled
NOTES:

3 Received Broken/Leaking (Improperly Sealed)
Y N
NOTES:

4 Properly Preserved
Y N
NOTES:

5 Received Within Holding Times
Y N
NOTES:

COC Tape Was:
1 Present on Outer Package Y N
2 Unbroken on Outer Package Y N
3 Present on Sample Y N
4 Unbroken on Sample Y N
NOTES:

COC Record Was:
1 Present Upon Receipt of Samples Y N

Discrepancies Between Sample Labels and COC Record? Y N
NOTES:

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APPENDIX 5.3

DATA FORM COMPLETION

Use a pen with black ink that is not water soluble (not a felt-tip pen). Make an entry in each blank. Where there is no data entry, enter UNK for Unknown, NA for Not Applicable, or ND for Not Done. If any procedure was not performed as prescribed, give the reason for the change or omission on the form. To change an entry, draw a single line through it, add the correct information above it, and initial the change.

SOIL SAMPLE IDENTIFICATION LABEL

1. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

2. Sample ID. Four-digit number assigned to ensure that data collected retains uniqueness from other data collected at the same location ID.

3. Lab Code. Three-character code identifying the company responsible for performing the analysis of water, soil, biota, and air samples.

4. Sampler. Name of person(s) collecting sample.

5. Location ID. Four-character code assigned sequentially to each borehole, test pit, or surface location where chemical, biological, radiological, and other measurements are taken.

6. Log Date. The date the information recorded on the label was obtained in the format DD-MMM-YY (01-JAN-88).

7. Log Time. The time the sample was collected (HH:MM).

8. Logger Code. Three-character or four-character code identifying the company responsible for collecting the information recorded on the form.

9. Sample Depth Interval from Datum:

   a. Beginning Depth (Ft From Datum). Depth from the ground surface to the top of the sampling interval in the format of feet and tenths of feet.

   b. Ending Depth (Ft From Datum). Depth from the ground surface to the bottom of the sampling interval in the format of feet and tenths of feet.

10. Analysis Requested. Type of analysis requested.

11. Comments. Any additional information.

APPENDIX 5.3, Continued

WATER SAMPLE IDENTIFICATION LABEL FORM

1. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

2. Sample ID. Four-digit number assigned to ensure that data collected retains uniqueness from other data collected at the same location ID.

3. Lab Code. Three-character code identifying the laboratory responsible for performing the analysis.

4. Sampler. Name of person(s) collecting sample.

5. Location ID. Four-character code assigned sequentially to each borehole, test pit, or surface location where chemical, biological radiological, and other measurements are taken.

6. Log Date. The date the information recorded on the form was obtained in the format DD-MMM-YY (01-JAN-88).

7. Log Time. Time the sample was collected (HH:MM).

8. Logger Code. Three-character or four-character code identifying the company responsible for collecting the information recorded on the form.

9. Analysis Requested. Type of analysis requested from the laboratory.

10. Preservation Method. Type of preservative used.

11. Comments. Any additional information.

APPENDIX 5.3, Continued

CUSTODY TRANSFER/LAB WORK REQUEST FORM

1. Received By. Completed by lab.

2. Date. Completed by lab.

3. Assigned To. Completed by lab.

4. Client. Client name.

5. Client Contact. WESTON person who will be laboratory contact.

6. Phone. Phone number of WESTON contact.

7. RFW Contact. Laboratory contact.

8. Date Due. Date analysis is due from the laboratory.

9. Project Number. Project number under which work is billed.

10. Sample No. Completed by laboratory.

11. Client ID No. Three-part identifier consisting of facility code, location ID, and sample ID and separated by dashes (for example, KCP01-01-01 and PXT02-0002-0001). See the ER Program data administrator for conventions to identify field quality control samples.

12. Description. Any descriptive information about the sample.

13. Matrix. Matrix type for sample; see valid matrix codes on lower half of form.

14. Date Collected. Date the sample was collected in the format DD-MMM-YY (01-JAN-88).

15. Container/Preservative. Container size and type (500-ml glass).

16. Analysis Requested. The type of analysis requested for each sample. The column heading indicating the type.

    PCB=Polychorinated Biphenyl

    HE=High Explosive

    HSL=Hazardous Substance List

    EPTOX=Extraction Procedure Toxicity

    VOA=Volatile Organic Analysis

APPENDIX 5.3, Concluded

CUSTODY TRANSFER/LAB WORK REQUEST FORM

BNA=Base Neutral Acid

TCLD=Toxic Characterization Leach Procedure

PEST=Pesticides

MAJ=Major Cation/Anion

17. Matrix. Valid matrix codes.

18. Special Instructions. Any special instructions.

19. Items/Reason. The reason the custody is transferred for all or selected items of the shipment.

20. Relinquished By. Signature of person sending samples.

21. Received By. Person or (shipping company) who received samples.

22. Date. Date sample is sent.

23. Time. Time sample is sent.

STANDARD OPERATING PROCEDURE 1.4

SAMPLE CONTAINERS AND PRESERVATION

1. PURPOSE

To provide guidance in the selection of suitable containers for samples, container cleaning, required sample volumes, sample collection, holding times, and the recommended preservation techniques for water, wastes, sediments, sludges and soil samples.

2. GENERAL DISCUSSION

The Field Sampling Plan (FSP) or Work Plan (WP) provides information about the scope and details of a given operation and establishes the number, types, and analyses of field samples (including field analyses). Refer to the FSP or WP for the procedures and equipment to be used in collecting samples. Collection and measurement of samples and the documentation of data will be performed as described in the associated procedures.

In choosing a sample container, the ideal material should be nonreactive with the sample and the particular analytical parameter to be tested. Glass or Teflon containers must be used with samples analyzed for organic compounds to prevent the introduction of extraneous organic compounds, such as those that might be leached from plastic containers. The rigid plastic screw caps for the bottles must be Teflon lined to prevent contamination.

Once a sample has been collected, steps must be taken to preserve the sample's chemical and physical integrity during transport and storage before analysis is conducted. The type of sample preservation required will vary according to the sample type and the parameter to be measured.

3. PROCEDURES

3.1. Associated Procedures

Before every operation, a review of the SOPs 1.1-1.10 is necessary. These SOPs contain information on the performance of field activities. They should be consulted for specific information on equipment and supplies; sample collection, preservation, packaging, and shipping; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.3	Sample Control and Documentation
1.5	Guide to the Handling, Packaging, and Shipping of Samples
1.6	General Equipment Decontamination
2.8	Sampling for Volatile Organics

3.2. Preparation

3.2.1. Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Obtain appropriate permission for property access.

D. Assemble the equipment and supplies listed in Appendix 5.1. Ensure the proper operation of all sampling equipment.

E. Notify the analytical laboratory of sample types, the number of samples, and the approximate arrival date.

F. Contact the carrier that will transport samples to obtain information on regulations and specifications.

3.2.2. Documentation

A. Obtain a logbook from the QA officer.

B. Record results of the equipment check in the logbook.

C. Obtain a sufficient number of the appropriate ER Program data collection forms (see INDEX TO SOPs).

D. Consult the ER Program data administrator for a current list of information management codes, location IDs, and sample numbers used in the completion of data forms.

3.2.3. Sample Container Preparation

Sample containers will vary according to the matrix and nature of the sample to be collected. Wide-mouth containers are generally used for soils and wastes; narrow-mouth containers are used for water. Calculations should be made to determine the number and type of containers required for the sampling effort.

Procurement of containers should be initiated as early as possible to avoid unavailability and shipping delays and to satisfy cleaning requirements. Obtain the required number of sample bottles included in the FSP or WP of a type consistent with recommendations in EPA-600/4-79-020 (EPA 1979). Sample containers can usually be obtained directly from the laboratory performing the analyses.

A. Container Type

1. Identify the containers required for analysis by matrix as shown in Appendixes 5.2 through 5.7 (for example, amber glass, narrow-mouth bottles for PCB analysis of water samples). Confirm the container requirements with the analytical laboratory and the FSP or WP.

2. Calculate the number of each type of container required by including duplicates and blanks with the number of investigative samples specified in the FSP or WP.

B. Container Cleaning

Containers certified as being precleaned to EPA specifications will be obtained from the manufacturer through the laboratory. If certified containers are not obtained, follow the procedures outlined below to wash containers and caps. The person washing containers must wear gloves. A bottle blank must be taken from each cleaned batch to confirm the success of the cleaning procedure if precleaned, certified bottles are not used.

1. Inorganic and general parameters

   a. Wash containers, septa or liners, and closures in hot tap water with laboratory-grade, nonphosphate detergent.

   b. Rinse three times with tap water.

   c. Rinse three times with ASTM Type 1 deionized water.

   d. Oven dry containers, septa or liners, and closures.

   e. Remove containers, septa or liners, and closures from oven.

   f. Place liners in closures (Teflon side down) and place on containers. Containers should not be removed from the preparation room until sealed.

2. Trace metals

   For certain parameters, a special cleaning procedure is needed to avoid adsorption or contamination resulting from interaction with container walls. These procedures are outlined below.

   a. Wash containers, closures, and Teflon liners in hot tap water with laboratory-grade, nonphosphate detergent.

   b. Rinse three times with tap water.

   c. Rinse one time with 1:1 nitric acid.

   d. Rinse three times with ASTM Type 1 deionized water.

   e. Air dry in a contaminant-free environment.

   f. Place liners in closures and place closures on containers. Containers should not be removed from the preparation room until sealed.

3. Extractable Organics

   a. Wash glass bottles, Teflon liners, and caps with hot tap water using laboratory-grade, nonphosphate detergent.

   b. Rinse three times with tap water to remove detergent.

   c. Rinse three times with ASTM Type 1 organic-free water.

   d. Oven dry bottles, liner, and caps to 105° - 125° for one hour.

   e. Rinse with pesticide-grade hexane or pesticide-grade methylene chloride.

   f. Oven dry bottles, liner, and caps to 105° - 125° for one hour.

   g. Allow bottles, liners, and caps to cool to room temperature in an enclosed contaminant-free environment. Cap the containers after the glassware has been cooled.

   h. Store in a contaminant-free area.

4. Volatile Organics Samples Containers

   a. Wash glass vial, Teflon-based septa, Teflon liners, and caps in hot water using laboratory-grade, nonphosphate detergent.

   b. Rinse three times with tap water.

   c. Rinse three times with ASTM Type I organic-free water.

   d. Oven dry vial, caps, septa, and liner at 105° for one hour.

   e. Allow vial, caps, septa, and liners to cool to room temperature in an enclosed contaminant-free environment.

   f. Seal vials with septa (Teflon side down) and cap.

   g. Label each vial with lot number and pack in the case.

   h. Store in contaminant-free area.

5. Sterilization

   a. For microbiological analyses, sterilize the container and its stopper/cap by autoclaving at 121°C for 15 min or by dry heat at 180°C for 2 hrs. Heat-sensitive plastic bottles may be sterilized with ethylene oxide at low temperatures.

   b. The sample bottles can be wrapped with aluminum foil before sterilization. Remove the protective wrapping after the sample is taken to facilitate cleaning the bottle before shipment to the analytical laboratory.

c. An acceptable alternative for emergency or field use is the sterilization of containers by boiling in water for 15 min.

3.2.4. Sample Volume

The volume of sample collected should be sufficient to perform all the required analyses plus an additional amount to provide for any quality control needs, split samples, or repeat examination. The volumes listed in Appendixes 5.2 through 5.7 are intended as general guidelines. Specific volume requirements to be followed will be those specified in the appropriate Quality Assurance Project Plan, Work Plan, and/or Field Sampling Plan. The laboratory receiving the sample should be consulted for specific volume requirements, and these should be specified in the FSP or WP.

NOTE: A sufficient number of containers must be available to ship the proper sample volume. For example, Department of Transportation (DOT) and International Air Transport Association (IATA) regulations limit the size of a sample container to 16 oz if the contents may include hazardous materials. In this case, two 500-ml or four 250-ml containers would be required to ship a one-liter fluid sample. See SOP 1.5, Guide to the Handling, Packaging, and Shipping of samples for additional information.

3.2.5. Sample Preservation

Sample containers may arrive at the site with the proper type and amount of preservatives in them. If onsite preservation of the samples is necessary, the proper reagents should be provided for the field crew in an easily usable form that can be added at the time of sampling. Preservation required for the specific analyses requested may be determined by using Appendixes 5.2 through 5.7. The preservation requirements specified in the Quality Assurance Project Plan, Work Plan, and/or Field Sampling Plan will take precedence over Appendixes 5.2 through 5.7. Materials commonly needed for sample preservation are listed below.

1. Small bottles of pelletized NaOH
2. Ascorbic acid crystals
3. Lead acetate paper and pH paper
4. Calibrated sampling scoops
5. Reagent-grade acids (HNO_3, HCl, and H_2SO_4) in Safe-Kote bottles
6. Calibrated dispenser bottles (0.5 to 2 ml) for acids.

3.2.6. Field

The appropriate number and type of precleaned containers, along with preservatives, equipment, and packaging containers, should be stored in a facility that can be locked or guarded. The storage facility should be located near the site and decontamination staging area, but should also be accessible to freight trucks that will be delivering new container shipments and transporting samples to the laboratory.

3.3. Operation

A. Soils/Wastes Sample Collection

NOTE: All individuals in the sampling area must wear gloves appropriate to their tasks. Only the persons collecting samples and filling sample containers must discard their gloves between sampling tasks.

1. While wearing protective gloves, fill the bottle with the sample. Wet soils should have enough headspace to allow for expansion.

2. Take extreme care to avoid contamination of the bottles or caps. Remove the cap just before filling and replace it as soon as possible after filling. Avoid any personal contact with the inside of the bottle or cap.

3. Clean the exterior of the bottle with a wipe moistened with deionized water, followed by a wipe moistened with methanol. When appropriate, implement SOP 1.6, General Equipment Decontamination. Attach a completed sample label (according to SOP 1.3, Sample Control and Documentation) and cover with clear tape. The tape should extend at least 1/4 inch beyond the edges of the label. Wrap a strip of Parafilm around the junction of the bottle and cap.

4. Preservation of soil samples is usually accomplished by protecting the sample from UV light by using an amber bottle and keeping the sample cool.

5. If required, place the container in a cooler. Maintain the samples at a cool temperature with frozen packaged ice (for example, Blue Ice) or ice cubes sealed in two plastic bags. Avoid freezing the sample by packing to prevent contact between the coolant and the sample container.

6. If samples are not delivered to the laboratory on a daily basis, check ice chests and insulated boxes every 24 hrs and replace thawed ice or Blue Ice packs as needed.

7. Avoid exposing the sample to extreme hot or cold temperatures and intense sunlight, even if no specific preservation is recommended.

8. EPA guidance recommends that a 4°C temperature be maintained in the sample container before and during shipment. The temperature in the packed container should be confirmed to be 4°C before the samples are shipped. A temperature reading from the middle of the package near the containers is satisfactory for this purpose.

   The temperature should be checked again at the analytical laboratory. Record both temperatures in the special instructions section of the Custody Transfer Record/Lab Work Request form (see SOP 1.3., Sample Control and Documentation). If a continuous temperature record can be obtained during the shipment period, record the maximum temperature in the container on the custody transfer form.

B. Water Sample Collection

NOTE: All individuals in the sampling area must wear gloves appropriate to their tasks. Only the persons collecting samples and filling sample containers must discard their gloves between sampling tasks.

1. Before collecting samples for organics and CN, use the Hach Test Kit for residual chlorine and sulfides. If present, preserve samples according to instructions in Appendix 5.2, Recommendation for Sampling and Preservation of Water Samples According to Measurement (for example, $Na_2S_2O_3$ to organic samples).

2. Collect samples directly in the appropriate container, ensuring that the sampling flow rate, if applicable, does not exceed the flow rate used while purging. Collect samples in the order of their volatilization sensitivity (TEGD).

   1) Volatile organics

   2) Extractable organics

   3) Dissolved metals

   4) Total metals

   5) Cations

   6) Anions

   7) Radionuclides

3. Do not filter unless specified in the Sampling Plan.

4. Do not rinse the container.

5. Slowly fill each container almost full, except VOAs (see SOP 2.8, Sampling for Volatile Organics).

6. Add any prescribed preservative.

7. Cap the container, shake, and reopen it.

8. If using acid or base preservative, check the pH adjustment with pH paper.

9. If necessary, add more preservative.

10. Complete steps 3 and 5-8 from Section 3.3.A.

11. If an error was made in collection, discard the entire bottle and start with a new one.

C. Holding Time

In general, analyze samples as soon as possible after collection. Some parameters are required to be analyzed in the field (See Appendix 5.2). Allowable holding times are listed as guidelines. They represent the maximum times that samples are considered valid. The required holding times for the task performed will be those specified in the appropriate Quality Assurance Plan, Work Plan, and/or Field Sampling Plan and will take precedence over Appendix 5.2. There are instructions in the FSP or WP for delivering the samples to the laboratory as soon as possible. (See SOP 1.5, Guide to the Handling, Packaging, and Shipping of Samples).

3.4. Postoperation

3.4.1. Field

A. Store unused, clean sample bottles in a clean environment for later use.

B. Clean acid dispensers and store them dry for the next field operation.

C. Ensure that all equipment is accounted for, decontaminated (see SOP 1.6, General Equipment Decontamination), and ready for shipment.

D. Restore the site to presampling conditions as specified in the FSP or WP.

E. Make sure all wells are labeled, sampling locations are properly staked, and the location ID is readily visible on the guard pipe or location stake.

F. Prepare samples for transport according to SOP 1.3, Sample Control and Documentation; SOP 1.4, Sample Containers and Preservation; and SOP 1.5, Guide to Handling, Packaging, and Shipping of Samples.

3.4.2. Documentation

A. Record any cleanup procedures and any uncompleted work (like site restoration or uncompleted sampling) in the logbook.

B. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

C. Review data collection forms for completeness.

3.4.3. Office

A. Deliver original forms and logbooks to the site manager for technical review. He/she will review, sign forms, and transmit to the document control officer (copies to the files) for eventual delivery to the Department of Energy.

B. Inventory equipment and supplies. Repair or replace all broken or damaged equipment. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

C. Contact the analytical laboratory to ensure that samples arrived safely and instructions for sample analyses are clearly understood.

4. SOURCES

Korte, Nic, and Peter Kearl. 1985. "Procedures for the Collection and Preservation of Groundwater and Surface Water Samples and for the Installation of Monitoring Wells: Second Edition." U.S. Department of Energy report GJ/TMC-08. Technical Measurements Center, Grand Junction Project Office, Grand Junction, Colorado.

EPA. 1982. "Handbook for Sampling and Sample Preservation of Water and Wastewater." U.S. Environmental Protection Agency report EPA-600/4-82-029. Washington, D.C.: U.S. Government Printing Office.

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EPA. 1983. "Manual of Groundwater Quality Sampling Procedures." U.S. Environmental Protection Agency report EPA/600/2-81-160. Washington, D.C.: U.S. Government Printing Office.

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EPA. 1985. "Practical Guide for Groundwater Sampling." U.S. Environmental Protection Agency report EPA/600/2-85/104. Washington, D.C.: U.S. Government Printing Office.

EPA. 1986. "RCRA Groundwater Monitoring Technical Enforcement Guidance Document." U.S. Environmental Protection Agency document OSWER-9950.1. Washington, D.C.: U.S. Government Printing Office.

5. APPENDIXES

5.1. Equipment and Supplies Checklist

5.2. Recommendation for Sampling and Preservation of Water Samples According to Measurement

5.3. Sampling and Preservation Procedures for RCRA Groundwater Detection Monitoring

5.4. Analysis Plan for Soil/Sediment Samples

5.5. Sample Containers for Waste

5.6. Containers for Aqueous Waste Samples

5.7. Analysis Plan for Soil/Sediment/Waste Samples

APPENDIX 5.1

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Narrow-mouth amber glass bottles with Teflon-lined caps (0.5, 1, and 2 liters)

_____ Amber glass vials with Teflon septa (40-ml)

_____ Blue Ice or equivalent

_____ 250-ml sterile bottle

_____ Cardboard boxes

_____ Insulated coolers

_____ Ballpoint pen (permanent black ink)

_____ Felt-tip marker pen (permanent black ink)

_____ Heavy-duty poly bags and ties

_____ Strapping tape

_____ Wide-mouth polyethylene bottles (0.5, 1, and 2 liters)

_____ Plastic trashcan liners

_____ 1-11 pH indicator paper

_____ Canvas bags

_____ Hach field test kit for sulfides

_____ Hach field test kit for chlorine

_____ Parafilm

_____ Ascorbic acid crystals

_____ Disposable surgical gloves (latex, PVC, other suitable plastic, or rubber)

_____ NaOH pellets

_____ Disposable wipes

_____ Crystalline $Na_2S_2O_3$

_____ Methanol and deionized water in Teflon wash bottles

APPENDIX 5.1, Continued

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Padding for packaging of samples

_____ Concentrated HNO_3, H_2SO_4, and HCl

_____ New or cleaned polyethylene narrow-mouth bottles (1.0 liter, 500 ml, 125 ml, and 60 ml)

APPENDIX 5.2

RECOMMENDATION FOR SAMPLING AND PRESERVATION OF WATER SAMPLES ACCORDING TO MEASUREMENT[1]

Measurement	Volume Requirement (m liter)	Container[2]	Preservative[3,4]	Holding Time[5]
Physical Properties				
Color	50	P,G	Ice	48 Hrs.
Conductance	100	P,G	Ice	28 Days
Hardness	100	P,G	HNO_3 to pH<2	6 Mos.
Odor	200	G only	Ice	24 Hrs.
pH	25	P,G	None Required	Analyze Immediately
Residue				
Filterable	100	P,G	Ice	7 Days
Nonfilterable	100	P,G	Ice	7 Days
Total	100	P,G	Ice	7 Days
Volatile	100	P,G	Ice	7 Days
Settleable Matter	1000	P,G	Ice	48 Hrs.
Temperature	1000	P,G	None Required	Analyze Immediately
Turbidity	100	P,G	Ice	48 Hrs.
Metals				
Dissolved	200	P,G	Filter onsite HNO_3 to pH<2	6 Mos. 6 Mos.[8]
Total	100	P,G	HNO_3 to pH<2	6 Mos.
Chromium^{+6}	200	P,G	Ice	24 Hrs.

APPENDIX 5.2, Continued

RECOMMENDATION FOR SAMPLING AND PRESERVATION OF WATER SAMPLES ACCORDING TO MEASUREMENT[1]

Measurement	Volume Requirement (m liter)	Container[2]	Preservative[3,4]	Holding Time[5]
Mercury				
Dissolved	100	P,G	Filter HNO_3 to pH<2	28 Days from date of collection
Total	100	P,G	HNO_3 to pH<2	28 Days
Inorganics, Nonmetallics				
Acidity	100	P,G	Ice	14 Days
Alkalinity	100	P,G	Ice	14 Days
Bromide	100	P,G	None Required	28 Days
Cations	1,000	P	HNO_3 to pH<2	28 Days
Chloride	50	P,G	None Required	28 Days
Chlorine	200	P,G	None Required	Analyze Immediately
Cyanides	500	P,G	NaOH to pH>12	14 Days from date of collection
Fluoride	300	P,G	Ice	28 Days
Iodide	100	P,G	Ice	24 Hrs.
Nitrogen				
Ammonia	400	P,G	Ice H_2SO_4 to pH<2	28 Days
Kjeldahl, Total	500	P,G	Ice H_2SO_4 to pH<2	28 Days
Nitrate Plus Nitrite	100	P,G	Ice H_2SO_4 to pH<2	14 Days
Nitrate[9]	100	P,G	Ice	48 Hrs.
Nitrite	50	P,G	Ice	48 Hrs.

APPENDIX 5.2, Continued

RECOMMENDATION FOR SAMPLING AND PRESERVATION OF WATER SAMPLES ACCORDING TO MEASUREMENT[1]

Measurement	Volume Requirement (m liter)	Container[2]	Preservative[3,4]	Holding Time[5]
Dissolved Oxygen				
Probe	300	G bottle and top	None Required	Analyse Immediately
Winkler	300	G bottle and top and store in dark	Fix onsite	8 Hours
Phosphorus				
Orthophosphate, Dissolved	50	P,G	Filter onsite Ice	48 Hrs.
Hydrolysable	50	P,G	Ice H_2SO_4 to pH<2	28 Days
Total, Dissolved	50	P,G	Filter on site Ice H_2SO_4 to pH<2	24 Hrs.
Silicia	50	P only	Ice	28 Days
Sulfate	50	P,G	Ice	28 Days
Sulfide	50	P,G	Ice add 2 m liter sinc acetate plus NaOH to pH >9	7 Days
Sulfite	50	P,G	None Required	Analyse Immediately
Organics				
BOD	1000	P,G	Ice	48 Hrs.
COD	50	P,G	Ice H_2SO_4 to pH<2	28 Days
Oil & Grease	1000	P,G	Ice H_2SO_4 to pH<2	28 Days

APPENDIX 5.2, Continued

RECOMMENDATION FOR SAMPLING AND PRESERVATION OF WATER SAMPLES ACCORDING TO MEASUREMENT[1]

Measurement	Volume Requirement (m liter)	Container[2]	Preservative[3,4]	Holding Time[5]
Organic Carbon	25	P,G	Ice H_2SO_4 or HCl to pH<2	28 Days
Phenolics	500	G only	Ice H_2SO_4 to pH<2	28 Days
Cyanide	1000	P,G	Ice 40% NaOH to pH>12, 0.6 g Ascorbic Acid	14 Days from date of collection
Coliform, Fecal and Total	250	P,G	Ice Sterile	6 Hrs.
Oil and Grease	1000	G	Ice H_2SO_4 to pH<2	28 Days
Phenols	1000	G, Teflon-lined Cap	Ice	7 Days until extraction; 40 Days after extraction
Total Organic Halogen and Purgeable aromatics	40	G, Teflon-lined vial septum	Ice 0.008% $Na_2S_2O_3$[6] HCl to pH<2	7 Days 14 Days
Purgeable aromatics	40	G, Teflon-lined septum	Ice 0.008% $Na_2S_2O_3$[6] HCl to pH<2	7 Days 14 Days
Acrolein and acrylonitrile	1000	G, Teflon-lined septum	Ice 0.008% $Na_2S_2O_3$[6] HCl to pH<2	7 Days 14 days
Phenols	1000	G, Teflon-lined cap	Ice 0.008% $Na_2S_2O_3$[6]	7 days until extraction; 40 days after extraction

APPENDIX 5.2, Continued

RECOMMENDATION FOR SAMPLING AND PRESERVATION OF WATER SAMPLES ACCORDING TO MEASUREMENT[1]

Measurement	Volume Requirement (m liter)	Container[2]	Preservative[3,4]	Holding Time[5]
Bensindines	1000 m liter	G, Teflon-lined cap	Ice 0.008% $Na_2S_2O_3$[6]	7 Days until extraction; 40 days after extraction
Phthalate esters	1000 m liter	G, Teflon-lined cap	Ice 0.008% $Na_2S_2O_3$[6]	7 Days until extraction; 40 days after extraction
Nitrosamines	1000 m liter	G, Teflon-lined cap	Ice store in dark 0.008% $Na_2S_2O_3$[6] extraction	7 Days until extraction; 40 days after
Nitroaromatics and isophorone	1000 m liter	G, Teflon-lined cap	Ice	7 days until extraction; 40 days after extraction
Polynuclear aromatic hydrocarbons	1000 m liter	G, Teflon-lined cap	Ice	7 days until extraction; 40 days after extraction
Haloethers	1000 m liter	G, Teflon-lined cap	Ice	7 days until extraction; 40 days after extraction
Chlorinated hydrocarbons	1000 m liter	G, Teflon-lined cap	Ice	7 days until extraction; 40 days after extraction
TCDD	1000 m liter	G, Teflon-lined cap	Ice	7 days until extraction; 40 days after extraction

APPENDIX 5.2, Continued

RECOMMENDATION FOR SAMPLING AND PRESERVATION OF WATER SAMPLES ACCORDING TO MEASUREMENT[1]

Measurement	Volume Requirement (m liter)	Container[2]	Preservative[3,4]	Holding Time[5]
Pesticides Tests				
Pesticides	1000 m liter	G, Teflon-lined cap	Ice	7 days from date of collection until extraction; 40 days after extraction
Radiological Tests				
Alpha, beta and radium	1000 m liter	P,G	HNO_3 to pH<2	6 mos.
Tritium	25 m liter	G	None	No limit
Isotopic Uranium	500 m liter	P	HNO_3 to pH<2	6 mos.
Sr-90	1000 m liter	P	HNO_3 to pH<2	6 mos.

[1]More specific instructions for preservation and sampling are found with each procedure described in this manual. A general discussion about sampling water and industrial wastewater may be found as ASTM, Part 31, p. 72-82 (1976) Method D-3370.

[2]Plastic (P) or Glass (G). For metals, polyethylene with a polypropylene cap (no liner) is preferred.

[3]Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at the time of collection. When use of an automated sample makes it impossible to preserve each aliquot, then samples may be preserved by keeping cool at 4°C until compositing and sample splitting is completed.

[4]When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 1, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following material: Hydrochloric acid (HCL) in water solutions at concentrations of 0.080% by weight or less, pH about 12.30 or less.

[5]Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permitted, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time and has received a variance from the regional administrator. Some samples may not be stable for a shorter time, if knowledge exists to show it is necessary to maintain sample stability.

[6]Should only be used in the presence of residual chlorine.

APPENDIX 5.2, Concluded

RECOMMENDATION FOR SAMPLING AND PRESERVATION OF WATER SAMPLES ACCORDING TO MEASUREMENT[1]

[7]Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before the pH adjustment in order to determine if sulfide is present. If sulfide is present, it can be removed by

the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

[8]Samples should be filtered immediately onsite before adding preservative for dissolved metals.

[9]For samples from nonchlorinated drinking water supplies, concentrated H_2SO_4 should be added to lower sample pH to less than 2. The sample should be analyzed within 14 days.

APPENDIX 5.3

SAMPLING AND PRESERVATION PROCEDURES FOR RCRA GROUNDWATER DETECTION MONITORING[a]

Parameter	Recommended Container[b]	Preservative	Maximum Holding Time	Minimum Volume Required for Analysis
		Indicators of Groundwater Contamination[c]		
pH	T,P,G	Field determined	None	25 m liter
Specific conductance	T,P,G	Field determine	None	100 m liter
TOC	G, amber, T-lined cap[e]	Ice	28 days HCl to pH<2	4 x 15 m liter
TOX	G, amber, T-lined septa or caps	Ice, add 1.1M sodium sulfite	7 days	4 x 15 m liter
		Groundwater Quality Characteristics		
Chloride	T, P, G	Ice	28 days	50 m liter
Iron Manganese Sodium	T, P	Field acidified to pH <2 with HNO_3	6 months	200 m liter
Phenols	G	Ice/H_2SO_4 to pH <2	28 days	500 m liter
Sulfate	T, P, G	Ice	28 days	50 m liter
		EPA Interim Drinking Water Characteristics		
Arsenic Barium Cadmium Chromium Lead Mercury Selenium Silver	T, P G, Amber	Field acidified to pH <2 with HNO_3 Dissolved Metals 1. Field filtration (0.45 micron) 2. Acidify to pH <2 with HNO_3	Total Metals 28 days 6 months	6 months 1000 m liter 1000 m liter

APPENDIX 5.3, Continued

SAMPLING AND PRESERVATION PROCEDURES FOR RCRA GROUNDWATER DETECTION MONITORING[a]

Parameter	Recommended Container[b]	Preservative	Maximum Holding Time	Minimum Volume Required for Analysis
Fluoride	T, P	Ice	28 days	300 m liter
Nitrate/ Nitrite	T, P, G	Ice/H_2SO_4 to pH <2	14 days	1000 m liter
Endrin Lindane Methoxychlor Toxaphene 2,4 D 2,4,5 TP Silvex	T, G	Ice	7 days	2000 m liter
Radium Gross Alpha Gross Beta	P, G	Field acidified to pH <2 with HNO_3	6 months	1 gallon
Coliform bacteria	PP, G (sterilised)	Ice	6 hours	200 m liter
		Other Groundwater Characteristics of Interest		
Cyanide	P, G	Ice, NaOH to pH >12. 0.6 g ascorbic acid[f]	14 days from date of collection	500 m liter
Oil and Grease	G only	Ice H_2SO_4 to pH <2	28 days	100 m liter
Semivolatile, nonvolatile organics	T, G	Ice	7 days from date of collection until extraction; 40 days to analysis	60 m liter
Volatiles	G, T-lined	Ice Ice and HCl to pH<2	7 days 14 days	60 m liter

[a]References:

Test Methods for Evaluation Solid Waste - Physical/Chemical Methods, SW-846 (2nd edition, 1982).
Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020.
Standard Methods for the Examination of Water and Wastewater, 16th edition (1985).

APPENDIX 5.3, Concluded

SAMPLING AND PRESERVATION PROCEDURES FOR RCRA GROUNDWATER DETECTION MONITORING[a]

[b]Container Types:

P = Plastic (polyethylene)
G = Glass
T = Fluorocarbon resins (PTFE, Teflon, FEP and PFA)
PP = Polypropylene

[c]Based on the requirements for detection monitoring (265.93), the owner/operator must collect a sufficient volume of groundwater to allow for the analysis of four separate replicates.

[d]Shipping containers (cooling chest with ice or ice pack) should be certified as to the 4°C temperature at the time of sample placement into these containers. Preservation of samples requires that the temperature of collected samples be adjusted to 4°C and maintained at 4°C upon placement of sample and during shipment. Field personnel will check the temperature in the container at the time of shipping and ice the samples to maintain a cool temperature during shipment. Maximum-minimum thermometers can be placed into the shipping chest to record temperature history. Chain-of-custody forms will include the temperature in the container at the time of shipment and delivery at the laboratory in addition to in-transit (maximum) temperature, if available.

[e]Do not allow any headspace in the container.

[f]Use ascorbic acid only in the presence of oxidising agents.

[g]Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before the pH adjustment in order to determine if sulfide is present. If sulfide is present, it can be removed by addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

APPENDIX 5.4

ANALYSIS PLAN FOR SOIL/SEDIMENT SAMPLES

Analyte	Method	Sample Container	Sample Volume (g)	Holding Preservations	Time (days)
Volatile Organics	Ref. 2	40-ml vial (2) w/Teflon-lined silicon rubber septum	5	Ice	14 from date of of collection
Base/Neutral/Acid Extractable Organics	Ref. 3	Amber G, 1 L	10-30	Ice	7/40[2]
Organochlorine Pesticide/PCB	Ref. 4	Amber G, 1 L	10-30	Ice	7/40[2]
TAL Inorganic[1]	Ref. 5	P, G, 1 L	200	Ice	180 Mercury = 28 days from date of collection
Non-TAL Metals	SW-846	P, G, 1 L	200	Ice	180
Reactivity	Ref. 6	Amber G	---	Ice	N/A
Chloride	EPA 300.0[3]	G, 1 L	20	Ice	N/A
Sulfate	EPA 300.0[3]	G, 1 L	20	Ice	N/A
Nitrate	EPA 300.0[3]	G, 1 L	20	Ice	N/A
Cyanide	Ref. 1	G, 1 L	200	Ice	12
Hexavalent Chromium	S.M. 312b[4]	G, 1 L	100	Ice	1

[1]Includes cesium, molybdenum, and strontium, which are non-TAL metals, but are analysed using the same methods.

[2]Extract within 7 days from date of collection; analysis within 40 days of extraction.

[3]Soil/sediments will be leached with Laboratory Reagent Water (20 g soil to 50 ml water) and water extract analysed using procedure in "Methods for Chemical Analysis of Water and Wastes," 1983; EPA 600/4-79-020.

[4]Soil/sediment will be leached with Laboratory Reagent Water (5 g soil and 100 ml of water) by shaking for 2 hours, and the water extract filtered and subsequently analysed. This is in accordance with method 312B in Standard Methods for Examination of Water and Wastewater, 16th Edition.

*The FSP or WP will define the actual suite of parameters to be analysed for specific samples.

APPENDIX 5.4, Continued

ANALYSIS PLAN FOR SOIL/SEDIMENT SAMPLES

Method References

Ref. 1. Method 9010 - "Test Methods for Evaluating Solid Wastes," Office of Solid Waste and Emergency Response, Washington, DC 20460, Revised September 1986.

Ref. 2. Method 8240 - "Test Methods for Evaluating Solid Wastes," Office of Solid Waste and Emergency Response, Washington, DC 20460, Revised September 1986.

Ref. 3. Method 8270 - "Test Methods for Evaluating Solid Wastes," Office of Solid Waste and Emergency Response, Washington DC 20460, Revised September 1986.

Ref. 4. Method 8080 - "Test Methods for Evaluating Solid Wastes," Office of Solid Waste and Emergency Response, Washington, DC 20460, Revised September 1986.

Ref. 5. Method 6010 or 7000 Series Methods - "Test Methods for Evaluating Solid Wastes," Office of Solid Waste and Emergency Response, Washington, DC 20460, Revised September 1986.

Ref. 6. Method 9010, 9030 - "Test Methods for Evaluating Solid Wastes," Office of Solid Waste and Emergency Response, Washington, DC 20460, Revised September 1986.

Ref. 7. Method 1310 - "Test Methods for Evaluating Solid Wastes," Office of Solid Waste and Emergency Response, Washington, DC 20460, Revised September 1986.

APPENDIX 5.5

SAMPLE CONTAINERS FOR WASTE

Waste Type	Recommended Container	Closure	Analysis
Photosensitive wastes 1000 or 2000 m liter	Amber HDPE or amber glass caps with Teflon liners for glass bottles	LPE caps for HDPE bottles; Bakelite	Waste characterization per 40 CFR-Part 261
Pesticide hydrocarbon chlorinated hydrocarbons petroleum distillates	Wide-mouth borosilicate glass bottles 1000 or 2000 m liter	Bakelite caps with Teflon liner	Waste characterization per 40 CFR-Part 261
Oil wastes	HDPE bottles wide mouth 1000 or 2000 m liter	LPE caps	Waste characterization per 40 CFR-Part 261
Strong alkali or hydrofluoric acid	HDPE bottles, 1000 m liter	LPE caps wide mouth	Waste characterization per 40 CFR-Part 261
Aqueous wastes-- characterisation of organics	Borosilicate glass bottles 1000 or 2000 m liter	Caps with Teflon liner	Waste characterization per 40 CFR-Part 261
Solids (sludge, soils, and granular)	8-oz, wide-mouth glass bottle	Bakelite caps with Teflon liners	Waste characterization per 40 CFR-Part 261

APPENDIX 5.6

CONTAINERS FOR AQUEOUS WASTE SAMPLES

Analyte	Sample Container	Sample Volume	Preservation[7]	Holding Time(days)
TCL Volatile	40 m liter vial (2)	40 m liter	HCl pH<2 with HCL	14 from date of collection
TCL Base/Neutral/Acid[1]	Amber G, 1L	1 L	Ice	7/40[5]
TCL Pesticide/PCB	Amber G, 1L	1 L	Ice	7/40[5]
TAL Inorganic[2]	P,G, 1L	1 L	pH<2,w/HNO_3[6]	180 (Mercury = 28 days from date of collection)
Non-TAL Metals	P,G, 1L	1 L	pH<2,w/HNO_3[6]	180
Cyanide	P,G, 1L	0.5L	pH>12,w/NaOH	14 from date of collection
pH[3]	P,G	N/A	None	Field Meas.
Sp. Conductivity	P,G	N/A	None	Field Meas.
Temperature[3]	P,G	N/A	None	Field Meas.
Diss. Oxygen	G	N/A	None	Field Meas.
TDS	P,G 1L	0.1 L	Ice	7
TSS	P,G 1L	0.1 L	Ice	7
Total Phosphate	P,G, 1L	1L	Ice, pH<2[6]	28
Chloride, Sulfate	P,G, 1L	1 L	Ice	28
Carbonate/Bicarbonate[4]	P,G, 1L	1 L	Ice	14
Nitrate	P,G, 1L	1L	Ice	2
Hexavalent Chromium	P,G, 1L	1 L	Ice	2

[1]The HSI Base/Neutral/Acid fractions analytical parameters are the TCL semivolatiles.
[2]Includes Cesium, Molybdenum, and Strontium, which are non-TAL metals, but are analysed using the same methods.
[3]Field Measurements.
[4]These are reported as carbonate and biocarbonate alkalinity.
[5]Seven days from date of collection to extraction; analysis within 40 days of extraction.
[6]All samples with the exception of those for total metals will be filtered within 4 hours of sample collection and preservatives added to the filtrate as specified.
[7]All samples will be kept at 4°C after arrival at the laboratory.
*The FSP or WP will define the actual suite of parameters to be analysed for specific samples.

APPENDIX 5.7

ANALYSIS PLAN FOR SOIL/SEDIMENT/WASTE SAMPLES*

Analyte	Sample Container	Sample Volume(g)	Preservations	Holding Time(days)
TCL Volatile	40 m liter vial (2)	5	Ice	14 from date of collection
TCL Base/Neutral/Acid[1]	Amber G, 1 L	10-30	Ice	7/40[3]
TCL Pesticide/PCB	Amber G, 1 L	10-30	Ice	7/40[3]
TAL Inorganic[2]	P,G, 1 L	200	Ice	180
Non-TAL Metals	P,G, 1L	200	Ice	180 (Mercury = 28 days from date of collection)
Reactivity	Amber G	100	Ice	N/A
TCLP	Amber G	100	Ice	14 days
Chloride	G, 1 L[4]	20	Ice	N/A
Sulfate	G, 1 L[4]	20	Ice	N/A
Nitrate	G, 1 L[4]	20	Ice	N/A
Cyanide	G, 1 L	200	Ice	12
Hexavalent Chromium	G, 1 L[5]	200	Ice	1

[1]The HSI Base/Neutral/Acid fractions analytical parameters are the TCL semivolatiles.
[2]Includes Cesium, Molybdenum, and Strontium, which are non-TAL metals.
[3]Extract within 7 days from date of collection; analysis within 40 days of extraction.
[4]Soil/sediments will be leached with laboratory reagent water (20 g soil to 50 m liter water) and water extract analysed using referenced procedure. Procedure reference: Methods for Chemical Analysis of Water and Wastes, 1983; EPA 600/4-79-020.
[5]Soil/sediment will be leached with laboratory reagent water (5 g soil and 100 m liter of water) by shaking for 2 hours. The water extract is filtered and subsequently analysed. This is in accordance with method 312B in Standard Methods for Examination of Water and Wastewater, 15th Edition.
*The FSP or WP Sampling Plans will define the actual suite of parameters to be analysed for specific samples.

APPENDIX 5.7, Continued

ANALYSIS PLAN FOR SOIL/SEDIMENT/WASTE SAMPLES

Method References

Ref. 1. Method 9010 - "Test Methods for Evaluating Solid Wastes," Office of Solid Waste and Emergency Response, Washington, DC 20460, Revised September 1986.

Ref. 2. Method 8240 - "Test Methods for Evaluating Solid Wastes," Office of Solid Waste and Emergency Response, Washington, DC 20460, Revised September 1986.

Ref. 3. Method 8270 - "Test Methods for Evaluating Solid Wastes," Office of Solid Waste and Emergency Response, Washington, DC 20460, Revised September 1986.

Ref. 4. Method 8080 - "Test Methods for Evaluating Solid Wastes," Office of Solid Waste and Emergency Response, Washington, DC 20460, Revised September 1986.

Ref. 5. Method 6010 or 7000 Series Methods - "Test Methods for Evaluating Solid Wastes," Office of Solid Waste and Emergency Response, Washington, DC 20460, Revised September 1986.

Ref. 6. Method 9010 or 9030 Series Methods - "Test Methods for Evaluating Solid Wastes," Office of Solid Waste and Emergency Response, Washington, DC 20460, Revised September 1986.

Ref. 7. Method 1310 - "Test Methods for Evaluating Solid Wastes," Office of Solid Waste and Emergency Response, Washington, DC 20460, Revised September 1986.

STANDARD OPERATING PROCEDURE 1.5

GUIDE TO THE HANDLING, PACKAGING, AND SHIPPING OF SAMPLES

1. PURPOSE

To provide a general guide for packaging and shipping samples of environmental and hazardous materials to the laboratory. In addition, instructions are provided to select the correct category for packaging and shipping samples of unknown contents.

2. DISCUSSION

The Field Sampling Plan (FSP) or Work Plan (WP) provides information concerning the scope and details of a specific operation. Refer to the FSP or WP for the type of samples to be collected and the destination for the samples. This SOP describes the procedures used by the ER Program technical assistance subcontractor when handling, packaging, and shipping samples. Other procedures or requirements used by installation subcontractors must conform to this SOP. The transportation of samples must protect the integrity of the sample and prevent any detrimental effects from the potentially hazardous nature of the samples.

Samples collected at a site are classified as environmental or hazardous material samples. In general, environmental samples are collected from streams, farm ponds, small lakes, wells, and offsite soils that are not expected to be contaminated with hazardous materials. Samples of onsite soils or water and materials collected from drums, bulk storage tanks, obviously contaminated ponds, impoundments, lagoons, pools, and leachates from hazardous waste sites are considered samples of hazardous materials. A distinction must be made between the two types of samples for two reasons.

1. The appropriate Department of Transportation (DOT) or International Air Transport Association (IATA) procedures for the transportation of samples must be determined. If there is any doubt, a sample should be considered hazardous and shipped accordingly.

2. The health and safety of laboratory personnel receiving samples must be protected. Special precautions are used at laboratories when samples that are not environmental are received.

Hazardous materials defined by the transportation regulations contained in 49 CFR (Subchapter C, Part 171) or the current edition of IATA regulations for dangerous goods (Sections 3 and 4) should be shipped only by the method of transportation specified in these regulations. Overnight shipments by air (Federal Express, for example) are governed by the IATA regulations. Transportation of hazardous materials exclusively by surface route is governed by the requirements of 49 CFR. This operating practice ensures compliance with the appropriate regulations and at times requires the implementation of packaging instructions that are more conservative and stringent than those required by regulation. Employees should be aware that regulatory bodies with jurisdiction have the authority to levy substantial fines and penalties to violators. Failure on the part of any employee to follow the requirements of these procedures is cause for disciplinary action, including discharge.

This SOP provides general guidance for packaging, marking, labeling, and shipping samples of environmental and hazardous materials and should not be misconstrued as the equivalent of or replacement for the DOT or IATA regulations. When shipping any potentially hazardous samples, the DOT regulations (49 CFR 171-178) and IATA regulations must be followed. This SOP should be used in conjunction with DOT and IATA regulations and advice from the freight carrier to ensure that all regulations governing transportation are being followed.

Any questions about the instructions for shipping environmental samples or hazardous materials in this SOP should be directed to the subcontractor's health and safety officer, who provides technical assistance to the ER Program.

3. PROCEDURES

3.1. Associated Procedures

Before every operation, SOPs 1.1-1.10 must be reviewed. These SOPs contain information on the performance of field activities. They should be consulted for specific information on equipment and supplies; sample collection, preservation, packaging, and shipping; decontamination procedures; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.3	Sample Control and Documentation
1.4	Sample Containers and Preservation
1.6	General Equipment Decontamination

3.2. Preparation

3.2.1. Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Obtain appropriate permission for property access.

D. Notify the analytical laboratory of sample types, the number of samples, and the approximate arrival date.

E. Contact the carrier that will transport samples to obtain information on regulations and specifications.

3.2.2. Documentation

A. Obtain a logbook from the QA officer.

B. Obtain a sufficient number of the appropriate ER Program data collection forms (see INDEX TO SOPs).

C. Consult the ER Program data administrator for a current list of information management codes, location IDs, and sample numbers used in the completion of data forms.

3.3. Operation

Procedures for shipping samples under DOT and IATA regulations are provided in Appendixes 5.1 through 5.4. The following step-by-step procedure will ensure that all applicable requirements for classifying, packing, marking, labeling, and documenting samples can be met.

A. Determine the correct technical name or composition of substances that might be in the samples. Check to see if the substance is forbidden on aircraft. Section 1 of the IATA regulations for dangerous goods contains a list of the substances that cannot be transported by air.

B. All samples must be transported by cargo aircraft or land transport. See Appendixes 5.1 through 5.4 for the appropriate DOT and IATA requirements.

C. Consult the DOT or IATA references to select the appropriate shipping container and packing material.

D. Prepare the consignment according to relevant requirements.

E. Ensure that all appropriate markings are printed on the packages and labels are attached.

F. Make any appropriate advance arrangements with the carrier and obtain current information about regulations and specifications that might affect the shipment.

G. Prepare the cargo airbill, complete, and sign the appropriate declarations for transporting dangerous goods.

H. Deliver the shipment to the local office of the freight carrier or arrange for a pickup at the site. Do not seal the container until the freight carrier is satisfied that the internal packaging meets all applicable regulations.

I. Ensure that all chain-of-custody procedures are observed. The copy of the bill of lading form will be retained as evidence of the chain-of-custody transfer.

3.4. Postoperation

3.4.1. Field

A. When transferring the samples, have the transferee sign and record the date and time on the Custody Transfer Record/Lab Work Request form (see SOP 1.3, Sample Control and Documentation). Custody transfers made to a sample custodian in the field should account for each sample, although samples may be transferred as a group. Every person who takes custody should fill in the appropriate section of the Custody Transfer Record/Lab Work Request form. Minimize the time of possession.

B. The field custodian is responsible for properly packaging and dispatching samples to the appropriate laboratory. This responsibility includes completing, dating,

and signing the appropriate portion of the Custody Transfer Record/Lab Work Request form. When samples of hazardous materials are shipped to a laboratory, provide advance notice.

C. Verify that all sample bottles have been correctly identified and labels include necessary information (for example, location, time, and date).

3.4.2. Documentation

A. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

B. As in any other activity that may be used to support litigation, regulatory agencies must be able to provide the chain of possession and custody of any samples that are offered for evidence or that form the basis of analytical test results introduced as evidence. Written procedures must be available and followed whenever samples for evidence are collected, transferred, stored, analyzed, or destroyed. The primary objective of these procedures is to create an accurate, written record that can be used to trace the possession and handling of the sample from the moment of its collection through analysis and the introduction as evidence.

A sample is in someone's custody under any of the conditions listed below.

- It is in one's actual possession.
- It is in one's view (after being in one's physical possession).
- It is one's physical possession and then locked up so that no one can tamper with it.
- It is kept in a secured area that is restricted to only authorized personnel.

C. Send all packages to the laboratory with the Custody Transfer Record/Lab Work Request form and other pertinent forms. Retain a copy of these forms at the originating office (either carbon or photocopy). Register mailed packages with a return receipt requested. For packages sent by common carrier, retain receipts as part of the permanent chain-of-custody documentation. Pack samples to eliminate the possibility of breakage during shipment. Seal or lock the package so that any tampering can be readily detected.

D. Additional guidelines for chain of custody, a sample of the form, and instructions for completing the Custody Transfer Record/Lab Work Request form are included in SOP 1.3, Sample Control and Documentation.

3.4.3. Office

A. Deliver original forms and logbooks to the site manager for technical review. He/she will review, sign forms, and transmit to the document control officer (copies to the files) for eventual delivery to the Department of Energy.

B. Contact the analytical laboratory to ensure that samples arrived safely and instructions for sample analyses are clearly understood.

4. SOURCES

International Air Transport Association. 1988. Dangerous Goods Regulations. January 1988. Montreal, Quebec, Canada.

CFR 49. 1985. Code of Federal Regulations, Title 49, U.S. Department of Transportation, Parts 100-199. November 1, 1985. Washington, D.C.: U.S. Government Printing Office.

5. APPENDIXES

5.1 Environmental Samples

5.2 Samples of Hazardous and Radioactive Materials

5.3 Transportation of Unknown Hazardous Materials by 49 CFR

5.4 Transportation of Unknown Hazardous Materials by IATA

APPENDIX 5.1

ENVIRONMENTAL SAMPLES

A. Environmental Samples

Environmental samples may be packaged and shipped according to the following procedures.

B. Packaging

Before any samples are placed in their final shipping containers, the exterior of the sample containers should be wiped clean with a damp cloth. Environmental samples must then be packaged according to the following procedures.

1. Place sample container, properly labeled and with a chain-of-custody seal on a sealed lid, into a polyethylene bag and seal the bag.

2. Place sample in a fiberboard container approved by the Department of Transportation (DOT) or picnic cooler.

3. Pack container with enough noncombustible, absorbent cushioning material to minimize the possibility of breakage and absorb any materials that may have leaked from the sample jars. Vermiculite is recommended.

4. If there are multiple samples, be sure there is sufficient cushioning material between the sample containers (each in its individual polyethylene bag) to prevent breakage from dropping or severe shock.

5. Sealed bags of ice are packed with the samples to obtain 4°C.

6. Tape a sealed plastic bag containing the completed chain-of-custody form to the inside of the shipping container lid.

7. Seal outside container with duct tape or strapping tape.

8. On each side of the shipping container, place a signed chain-of-custody seal at the junction between the shipping container and lid.

C. Marking/Labeling

Sample containers must have a completed sample identification tag (see SOP 1.3, Sample Control and Documentation), and the outside container must be marked Environmental Sample. The appropriate side of the container must be marked This End Up, and arrow labels should be used accordingly. No DOT placards or labeling are required. Assure that all sample containers are labeled identically to labels on the shipping container.

D. Shipping Papers

No DOT or International Air Transport Association (IATA) shipping papers are required for environmental samples.

APPENDIX 5.1, Continued

ENVIRONMENTAL SAMPLES

E. Transportation

There are no DOT or IATA restrictions on the mode of transportation for environmental samples. An overnight carrier is required.

F. Additional guidelines for water samples

Additional guidelines for the shipment of water samples is given in the TEGD. These include the following activities.

1. When the samples are placed in the shipping cooler, record the internal temperature of the cooler on the chain-of-custody form.

2. To determine the maximum and minimum temperature during shipment, place a thermometer that measures and records the maximum and minimum temperature range inside the shipping cooler.

3. Make sure that signed and dated chain-of-custody seals are present on the cap of each individual sample container and on the lid of the shipping cooler.

4. The internal temperature of the shipping container will be measured using a laboratory grade thermometer upon opening the cooler at the contracted laboratory.

APPENDIX 5.2

SAMPLES OF HAZARDOUS AND RADIOACTIVE MATERIALS

A. Samples of Hazardous Material

Samples that are not environmental samples or samples known or expected to contain hazardous substances must be considered samples of hazardous material and transported according to the following requirements.

If the hazardous material in the sample is known or can be accurately identified, it is packaged, marked, labeled, and shipped according to the specific instructions for that material described in the Department of Transportation Hazardous Materials Table (49 CFR 72.101) or the latest edition of the International Air Transport Association Dangerous Goods Regulations.

B. Samples of Radioactive Materials

Samples containing greater than 2,000 pCi/g of any radionuclide are considered to be radioactive materials by DOT (49 CFR 173.403y) and must be shipped following the requirements given in 49 CFR 173 for labeling and packaging. Table V.1 summarizes the shipping categories and concentration limits for several radionuclides commonly encountered during investigations. The labeling and packaging requirements for these categories are given in 49 CFR 172 subparts D and E.

TABLE V.1

RADIOACTIVE SAMPLE SHIPMENT REQUIREMENTS

Radionuclide	49 CFR Reference	Concentration	Total Quantity	DOT Class
Tritium in water				
	49 CFR 173.403(y)	<2 μCi/L	NA	Environmental
	49 CFR 173.423	2 μCi/L to 1×10^5 μCi/L	<1,000 Ci/ package	UN2910 (Limited quantity)
	49 CFR 173.403(n)	1×10^5 μCi/L to 5×10^6 μCi/L	<1,000 Ci/ package	UN2912 (LSA)
Plutonium-238 in soil or other solids				
	49 CFR 173.403(y)	0.002 μCi/g	NA	Environmental
	49 CFR 173.403 49 CFR 173.423	0.002 μCi/g to 0.1 μCi/g	<30 μCi/ package	UN2910 (Limited quantity)
	49 CFR 173.403(n) 49 CFR 173.435	0.002 μCi/g to 0.1 μCi/g	30 μCi to 3,000 μCi/ package	UN2912 (LSA)
Thorium-232 soil or other solids				
	49 CFR 173.403(y)	<0.002 μCi/g	NA	Environmental
	49 CFR 173.403 49 CFR 173.435	0.002 μCi/g to 300 μCi/g	Unlimited	UN2910 (Limited quantity)
Thorium-230 in soil or other solids				
	49 CFR 173.403(y)	<0.002 μCi/g	NA	Environmental
	49 CFR 173.403 49 CFR 173.423	0.002 μCi/g to 0.1 μCi/g	<30 μCi/ package	UN2910 (Limited quantity)
	49 CFR 173.403(n) 49 CFR 173.435	0.002 μCi/g to 0.1 μCi/g	30 μCi to 3,000 μCi/ package	UN2912 (LSA)

[a]The package refers to the shipment cooler (not the sample jars).
DOT - U.S. Department of Transportation
NA - not applicable
LSA - low-specific activity

APPENDIX 5.3

TRANSPORTATION OF UNKNOWN HAZARDOUS MATERIALS BY 49 CFR

A. Transportation of Unknown Hazardous Materials by 49 CFR

1. For samples of hazardous substances of unknown content that will be shipped by surface carrier under 49 CFR Transportation Regulations, the appropriate transportation category is selected through a process of elimination using the Department of Transportation (DOT) Hazardous Materials Classification system. While it is probable that most unknown samples of hazardous material shipped by field personnel will not contain radioactive materials or Poison A materials, it is essential for the following gradient hierarchy to be considered.

2. If radiation survey instruments demonstrate (or reasonable probability exists) that the unknown hazardous sample is radioactive, the appropriate DOT shipping regulations for radioactive material must be followed. Contact the subcontractor's health and safety officer for ER Program technical assistance for specific details.

3. If radioactive material is eliminated, the sample must then be considered to contain Poison A materials. DOT defines Poison A as an extremely dangerous, poisonous gas or a gas or liquid of the nature that a very small amount of gas (or vapor of the liquid) will be dangerous to life. Most Poison A materials are gases and would not be found in glass or drum-like containers. All samples taken from closed containers do not have to be shipped as Poison As. Based upon information available, judgment must be made as to whether a sample from a closed container is a Poison A. For specific instructions on the proper procedures for shipping Poison A, contact the subcontractor's health and safety officer for ER Program technical assistance.

4. If Poison A is eliminated as a shipment category, the next two classifications are flammable or nonflammable gases. Because an open container is not expected to contain a significant amount of gas, flammable liquid would be the next applicable category. After the categories of radioactive material, Poison A flammable gas, and nonflammable gas have been eliminated, the sample can be classified as a flammable liquid and shipped accordingly.

5. These procedures would also suffice for shipping any other samples classified below Poison A in the DOT classification table.

6. These procedures would also suffice for shipping any other samples classified below flammable liquids in the DOT classification table.

B. Shipment of Flammable Liquid by 49 CFR

The following instructions apply to the shipment of a flammable liquid by rail car, truck, or other common carrier.

1. Collect the sample in a glass or polyethylene container with a metallic, Teflon-lined screw cap. The container may be no larger than 16 fluid oz. To prevent leakage, fill the container no more than 90% full. Mark the fluid level on the outside of the sample container. If an air space in the sample container would affect sample integrity (for example, the case of a volatile organics analysis vial), place that container within a second container to meet the 90% requirement. Before any samples are placed in the final shipping container, the exterior should be wiped clean with a detergent solution.

2. Complete the sample identification tag (see SOP 1.3, Sample Control and Documentation) and attach it securely to the sample container. The sample identification tag should contain information needed to trace the sample to its point of origin and sample taker, as well as any quality assurance/quality control information.

3. Seal the container and place it in a 2-ml-thick (or thicker) polyethylene bag with one sample in each bag. Position the identification tag so that it can be read through the bag. Seal the bag.

4. Place the sealed bag inside a metal can and cushion it with enough noncombustible, absorbent material (for example, vermiculite) between the bottom and sides of the can and bag to prevent breakage and absorb leakage. Pack one bag per can. Use clips, tape, or other positive means to secure the lid onto the can.

5. Place one or more metal cans into a strong outside container (like a picnic cooler or a DOT-approved fiberboard box). Surround cans with noncombustible, absorbent cushioning material for stability during transport. Total sample volume in the picnic cooler or fiberboard box should not exceed 10 gallons. A separate air bill and shipping declaration must be processed for each container or combination of containers so that the total sample volume on any air bill will not exceed 10 gallons.

C. Shipment by Land

The following instructions apply for shipment of samples of hazardous material by car or truck (not by common carrier).

1. The above instructions for flammable liquids will apply.

2. Additionally, sample containers must be firmly secured so that they will not bounce against the sides of the vehicle during transit or in an accident.

3. Limit shipments to 1000 lbs or less. Under 1000 lbs, there are no placarding requirements under 49 CFR 172.504 (c) (1).

D. Chain of Custody

Include the Custody Transfer Record/Lab Work Request form (properly executed) in the outside container. It is also recommended to use chain-of-custody tape over each can lid.

E. Marking and Labeling Samples Classified as Flammable Liquid

1. Use abbreviations only where specified.

2. Place the information listed below on each paint can.

   - Laboratory name and address

   - Flammable Liquid, N.O.S. UN 1993. The designation N.O.S. means not otherwise specified. Use an approved DOT label.

3. Information placed on cans should also be placed on at least one side of the outside shipping containers. If labelling is placed on more than one side, it must be attached to all visible sides.

F. Shipping Papers for Samples Classified as Flammable Liquid

Shipping papers must be provided for the shipment of all samples (including those transported by rental, government, company, or personal cars).

G. Bill of Lading/Certification Statement

Complete the bill of lading and sign the certification statement. If the carrier does not provide it, use a standard industry form. Provide the information listed below in the order listed. One form may be used for more than one outside container.

- Flammable Liquid, N.O.S. UN 1993

- Limited Quantity (or Ltd. Qty.)

- Net weight or net volume (weight or volume may be abbreviated) just before or after the UN or ID number.

- Further description (like Laboratory Samples) is allowed if it does not contradict required information.

H. Transportation

1. Transport samples of unknown hazardous material that are classified as flammable liquid by rented or common carrier truck or railroad, as appropriate.

2. Do not transport by any air transport system.

APPENDIX 5.4

TRANSPORTATION OF UNKNOWN HAZARDOUS MATERIAL BY IATA

A. Transportation of Unknown Hazardous Material by International Air Transport Association (IATA)

For samples containing unknown material that will be shipped by air carrier, the most appropriate classification in the IATA regulations is the classification of other regulated substances. In order to use this designation, the categories shown below must be eliminated.

- Radioactive Materials
- Poison A Materials
- Flammable Gases
- Nonflammable Gases

B. Shipment of Other Regulated Substances

The instructions below will apply for the shipment of other regulated substances by cargo-carrying aircraft, rail car, or other common carrier.

1. Collect the sample in a glass or polyethylene container with a nonmetallic, Teflon-lined screw cap. The container may be no larger than 16 fluid oz. To prevent leakage, fill the container no more than 90% full. If an air space in the sample container would affect sample integrity (for example, the case of a volatile organics analysis vial), place that container within a second container to meet the 90% requirement. Before any samples are placed in the final shipping container, the exterior should be wiped clean with detergent solution.

2. Complete the sample identification tag (see SOP 1.3, Sample Control and Documentation) and attach it securely to the sample container. The sample identification tag should contain information needed to trace the sample to its point of origin and sample taker, as well as any quality assurance/quality control information.

3. Seal the container and place it in a 2-ml-thick (or thicker) polyethylene bag with one sample in each bag. Position the identification tag so that it can be read through the bag. Seal the bag.

4. Place the sealed bag inside a metal can and cushion it with enough noncombustible, absorbent material (for example, vermiculite) between the bottom and sides of the can and bag to prevent breakage and absorb leakage. Pack one bag per can. Use clips, tape, or other positive means to secure the lid onto the can.

5. Place one or more metal cans into a strong outside container (like a picnic cooler or a DOT-approved fiberboard box). Surround cans with noncombustible, absorbent cushioning material for stability during transport. Total sample volume in the picnic cooler or fiberboard box should not exceed 40 liters.

C. Chain of Custody

Include the Custody Transfer Record/Lab Work Request form (properly executed) in the outside container. It is also recommended to use chain-of-custody tape over each can lid.

D. Marking and Labeling Samples Classified as Other Regulated Substances

1. Use abbreviations only where specified.

2. Place the information listed below on each paint can.

   - Laboratory name and address

   - Other regulated substances, UN8027. Hazardous Class # 9

3. Information placed on cans should also be placed on at least one side of the outside shipping containers. If labelling is placed on more than one side, it must be attached to all visible sides.

4. Cargo Aircraft Only must be printed on all outside shipping containers.

5. Print Laboratory Samples and This End Up or This Side Up clearly on top of the outside shipping container. Outside containers also must contain the statement Inside Packages to Comply with Prescribed Specifications. Put upward pointing arrows on all four sides of the container.

E. Shipping Papers for Samples Classified as Other Regulated Substances

Shipping papers must be provided for the shipment of all samples (including those transported by rental, government, company, or personal cars).

F. Bill of Lading/Certification Statement

Complete the bill of lading and sign the certification statement. If the carrier does not provide it, use a standard industry form. Provide the information listed below in the order listed. One form may be used for more than one outside container.

- Other Regulated substances, UN8027

- Class or Division # 9

- Net weight or net volume (weight or volume may be abbreviated) just before or after the UN or ID number.

- Further description (like Laboratory Samples) is allowed if it does not contradict required information.

G. Transportation

1. Transport samples of unknown hazardous material that are classified as other regulated substances by rented or common carrier truck, railroad, express overnight package services, or other appropriate means.

2. Do not transport by any passenger-carrying air transport system. Ship by air carriers that transport only cargo (for example, Federal Express).

STANDARD OPERATING PROCEDURE 1.6

GENERAL EQUIPMENT DECONTAMINATION

1. PURPOSE

To describe methods for the decontamination of field equipment potentially contaminated during sample collection.

2. DISCUSSION

The Field Sampling Plan (FSP) or Work Plan (WP) provides information about the scope and details of a given operation. The FSP or WP also contains specifications for the use of decontamination agents, areas where decontamination will be performed, and quality assurance procedures to verify the effectiveness of the decontamination procedures. Decontamination is performed as a quality assurance measure and a safety precaution. It prevents cross-contamination among samples and helps maintain a clean working environment for the safety of all field personnel.

Decontamination is mainly achieved by rinsing with liquids that include soap or detergent solutions, tap water, deionized water, and methanol. Equipment is allowed to air dry after being cleaned or wiped dry with chemical-free cloths or paper towels. It can then be reused immediately. Steam cleaning should be used whenever visible contamination exists or for large machinery/vehicles.

It is the primary responsibility of the site manager to assure that proper decontamination procedures are followed and that all waste materials produced are properly stored or disposed of. It is the responsibility of all personnel involved with sample collection or decontamination to maintain a clean working environment and ensure that contaminants are not negligently introduced into the environment.

3. PROCEDURES

3.1. Associated Procedures

Before every operation, a review of SOPs 1.1-1.10 is necessary. These SOPs contain information on the performance of field activities. They should be consulted for specific information about equipment and supplies; sample collection, preservation, packaging, and shipping; decontamination procedures; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.7	Sampling for Removable Alpha Contamination
6.4	Total Alpha Surface Contamination Measurements
6.11	Beta-Gamma Radiation Measurements Using a Geiger-Mueller Detector

3.2. Preparation

3.2.1. Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Obtain appropriate permission for property access.

D. Assemble the equipment and supplies listed in Appendix 5.1.

E. Notify the analytical laboratory of the decontamination blank sample and the approximate arrival date.

F. Contact the carrier that will transport the sample to obtain information on regulations and specifications.

3.2.2. Documentation

A. Obtain a logbook from the QA officer.

B. There are no forms required to document decontamination procedures and the degree of decontamination attained.

3.2.3. Field

A. Assemble containers and equipment for decontamination.

B. Decontaminate all equipment before use.

3.3. Operation

The extent of known contamination determines the extent to which equipment must be decontaminated. If the extent of contamination cannot be readily determined, clean the equipment on the assumption that it is highly contaminated until enough data are available to allow an accurate assessment of the level of contamination.

Adequate supplies of rinsing liquids and all materials should be available. Perform decontamination in the same level of protective clothing as sampling activities unless a different level of protection is specified in the FSP or Health and Safety Plan.

The procedure for full field decontamination follows. Any deviations from this procedure for a specific project are included in the FSP or WP.

Decontamination Steps

1. The purpose of the initial step is to remove gross contamination. Remove any solid particles from the equipment or material by brushing and then rinsing with available tap water. For drilling equipment, steam cleaning is necessary.

2. Wash equipment with a nonphosphate soap or detergent solution, such as Alconox®.

3. Rinse with tap water by submerging or spraying.

4. For organic contaminants, rinse with methanol then hexane. Methanol must be of a laboratory-reagent grade and hexane a "pesticide" grade.

5. Rinse thoroughly with distilled water.

6. Air dry equipment or rinse with nanograde methanol to expedite drying.

7. If radiation screening is required by the FSP or WP, screen the equipment with a radiation detector according to SOP 1.7, Sampling for Removable Alpha Contamination; SOP 6.4, Total Alpha Surface Contamination Measurements; or SOP 6.12, Radon-222 Flux Measurements Using Charcoal Canisters. If activity above the limits for unrestricted use is detected, repeat steps 1-6.

8. Samples of drippings from the last rinse in step 5 may be collected and analyzed to verify the effectiveness of the decontamination procedure. This type of sample is called a decontamination blank. The results of these analyses are not usually available for at least one week after they arrive in the laboratory, so it is important to do a thorough decontamination from the start of the sampling activity to minimize the potential for a positive hit in the decontamination drippings.

3.4. Postoperation

3.4.1. Field

A. Decontaminate as much sampling equipment as possible and properly dispose of expendable items that cannot be decontaminated. Proper disposal may involve onsite drumming of liquids and solids in approved containers for subsequent disposal. Expensive items like machinery may require a more advanced decontamination analysis.

B. Prepare the decontamination blank sample and transport it according to SOP 1.3, Sample Control and Documentation; SOP 1.4, Sample Containers and Preservation; and SOP 1.5, Guide to Handling, Packaging, and Shipping of Samples.

C. Store containers of solutions produced during decontamination in a secure area.

D. Dispose of any soiled materials as designated in the FSP or WP.

3.4.2. Documentation

A. Record radiological measurements in the logbook before leaving the site.

B. There are no forms required to document decontamination procedures and the degree of contamination attained.

3.4.3. Office

A. Deliver original logbooks to the site manager for technical review. He/she will review and transmit to the document control officer (copies to the files) for eventual delivery to the Department of Energy.

B. Inventory equipment and supplies. Repair or replace all broken or damaged equipment. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

C. Contact the analytical laboratory to ensure that the sample arrives safely and instructions for analyses are clearly understood.

D. After receiving the results of the laboratory analyses, arrange for the disposal of wastes generated during the investigation.

4. SOURCE

NIOSH, OSHA, USCG and EPA. 1985. "Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities." prepared by the National Institute for Occupational Safety and Health (NIOSH), Occupational Safety and Health Administration (OSHA), U.S. Coast Guard (USCG), and the U.S. Environmental Protection Agency (EPA). U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control, NIOSH report, October 1985. Washington, D.C.: U.S. Government Printing Office.

5. APPENDIX

5.1. Equipment and Supplies Checklist

APPENDIX 5.1

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Decontamination solutions preselected by the laboratory

_____ Cleaning liquids: soap or detergent solutions, tap water, deionized water, and methanol

_____ Chemical-free paper towels

_____ Cleaning brushes

_____ Cleaning containers: plastic buckets and galvanized steel pans

_____ Waste storage containers: drums and plastic bags

STANDARD OPERATING PROCEDURE 1.8

PERSONNEL DECONTAMINATION--LEVEL D PROTECTION

1. PURPOSE

To describe the equipment and procedures required for the decontamination of persons who have performed field activities in Level D protective clothing.

2. DISCUSSION

The Field Sampling Plan (FSP) or Work Plan (WP) provides information about the scope and details of a specific operation. Refer to the FSP or WP Health and Safety Plan for recommendations about the level of protection worn to enter a site and the criteria for upgrading to higher levels of protection.

Level D protective clothing is primarily a work uniform. This level of protection is worn when work functions preclude splashes, immersion, inhalation, or exposure to materials above the action limits specified in the FSP or WP.

Although Level D protection is worn under these conditions, workers may be wearing disposable coveralls and gloves, safety boots/shoes, a hard hat, and safety glasses. Therefore, the site Health and Safety Plan must address the proper disposition of disposable clothing and decontamination measures that should be implemented. The disposition of disposable items must follow installation requirements and any applicable state and federal regulations.

3. PROCEDURES

3.1. Associated Procedures

Before every operation, a review of the SOPs 1.1-1.10 is necessary. These SOPs contain information on the performance of field activities. They should be consulted for specific information on equipment and supplies; sample collection, preservation, packaging, and shipping; documentation procedures; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.6	General Equipment Decontamination
1.9	Personnel Decontamination--Level C Protection
1.10	Personnel Decontamination--Level B Protection

3.2. Preparation

3.2.1. Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. The FSP or WP Health and Safety Plan will specify the procedures and equipment required for the decontamination and disposal of Level D protective clothing. All onsite personnel will be informed about the proper disposal of protective clothing and any decontamination solutions used.

C. Appendix 5.1 lists the items suggested for Level D decontamination. This list provides general guidelines and can be modified in order to meet site-specific work activities or features.

D. Obtain necessary clothing, protective gear, and equipment. Read the Health and Safety Plan and ensure that necessary decontamination materials are available.

3.2.2. Documentation

A. Obtain a logbook from the QA officer.

B. There are no forms required to document decontamination procedures and the degree of decontamination attained.

3.2.3. Field

Before initiating field activities, designate an area for decontamination activities. Although Level D areas should be minimally contaminated, always use caution to prevent the potential spread of any unknown contaminants.

3.3. Operation

The following decontamination procedures are recommended for Level D protection. These measures represent suggested guidelines and may be modified to meet site-specific conditions.

A. Remove any disposable coveralls, rubber gloves, and boot covers and place in a plastic trash sack.

B. If dusty conditions have been encountered, use water-dampened paper towels to remove the dust from hard hats and safety glasses/goggles. Place used paper towels in a trash sack.

C. If necessary, decontaminate safety boots with water and a steel brush. Do not wear muddy or dusty boots out of the exclusion zone.

D. All workers should wash hands and face before leaving the site.

E. All workers should change clothing and shower as soon as possible after the day's work activities.

3.4. Postoperation

3.4.1. Field

Collect all trash sacks containing disposable clothing. Dispose of trash sacks according to the requirements of the site Health and Safety Plan. The site manager or field team leader is responsible for the safe disposal of any items.

3.4.2. Documentation

A. Record radiological measurements in the logbook before leaving the site.

B. There are no forms required to document decontamination procedures and the degree of decontamination attained.

3.4.3. Office

Return all unused items to the equipment manager. The equipment manager should be informed of all stock items that need to be ordered to replenish the inventory.

4. SOURCES

NIOSH, OSHA, USCG and EPA. 1985. "Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities." Prepared by the National Institute for Occupational Safety and Health (NIOSH), Occupational Safety and Health Administration (OSHA), U.S. Coast Guard (USCG) and the U.S. Environmental Protection Agency (EPA). U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control, NIOSH report, October 1985. Washington, D.C.: U.S. Government Printing Office.

EPA. 1984. "Standard Operating Safety Guides." Environmental Response Branch, Hazardous Response Support Division, Office of Emergency and Remedial Response, U.S. Environmental Protection Agency, November 1984. Washington, D.C.: U.S. Government Printing Office.

5. APPENDIX

5.1. Equipment and Supplies Checklist

APPENDIX 5.1

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Plastic trash sacks
30-gallon size: (no. of boxes) _____
20-gallon size: (no. of boxes) _____

_____ Plastic wash tub: (number) _____

_____ Paper towels: (no. of rolls)_____

_____ Liquid hand soap

_____ Wet wipe towelettes

_____ Water container
(size) 1 gallon_____ 5 gallon_____

_____ Brushes (scrub or wire)

_____ Alpha scintillation detector

STANDARD OPERATING PROCEDURE 2.2

FIELD MEASUREMENTS ON GROUND AND SURFACE WATER SAMPLES

1. PURPOSE

To obtain reliable and accurate measurements of the field chemistry (pH, temperature, electrical conductivity, alkalinity, dissolved oxygen, redox potential) of water quality samples. It should be noted that dissolved oxygen is not routinely measured as part of groundwater monitoring. Refer to the Field Sampling Plan (FSP) or Work Plan (WP) for details on the measurements to be performed.

2. DISCUSSION

For several reasons, taking measurements of water chemistry in the field is preferable to taking measurements in the laboratory. If it is suspected that a particular sample is not representative or valid, resampling and reanalyzing can be immediately performed at the site. In addition, the values of pH, specific conductance, and alkalinity in water chemistry measurements in the field may differ from those in the laboratory. Disadvantages encountered in obtaining field measurements usually relate to the reliability of the particular method and equipment used for the test.

The FSP or WP contains specific details about the procedures and equipment for this SOP. Refer to the FSP or WP for the type of samples to be collected. Collection and measurement of samples and the documentation of data will be performed as described in the associated procedures.

3. PROCEDURES

3.1. Associated Procedures

Before every operation, a review of the SOPs 1.1-1.10 is necessary. These SOPs contain information on the performance of field activities. They should be consulted for specific information about equipment and supplies; sample collection, preservation, packaging, and shipping; decontamination procedures; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.6	General Equipment Decontamination
2.1	Presample Purging of Wells
2.3	Sampling Monitoring Wells with a Bladder Pump
2.4	Sampling Monitoring Wells with a Bucket-Type Bailer
2.5	Sampling Monitoring Wells with a Submersible Pump
2.6	Sampling Monitoring Wells with a Peristaltic Pump

2.8	Sampling for Volatile Organics
2.9	Surface Water Sampling
3.1	Water Level Measurement
4.3	Monitoring Well Installation

3.2. Preparation

3.2.1. Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Obtain appropriate permission for property access.

D. Assemble the equipment and supplies listed in Appendix 5.1. Ensure the proper operation of all sampling equipment. Ensure that there are sufficient quantities of reagents, solutions, and filters.

E. Notify the analytical laboratory of sample types, the number of samples, and the approximate arrival date.

F. Contact the carrier that will transport samples to obtain information on regulations and specifications.

G. Consult the site manager about purging techniques, the disposal of purged water and other sampling expendables, and the purchase of standard solutions for electrical conductivity and pH/alkalinity calibrations.

3.2.2. Documentation

A. Obtain a logbook from the QA officer.

B. Record results of the equipment check in the logbook.

C. Obtain a sufficient number of the appropriate ER Program data collection forms (see INDEX TO SOPs).

D. Consult the ER Program data administrator for a current list of information management codes, location IDs, and sample numbers used in the completion of data forms.

3.2.3. Field

A. Locate the monitoring wells to be sampled and the appropriate decontamination area. Locate the staging area and areas for managing purged water and expendable sampling materials. Check decontamination zones and barricades to public access. Plan to purge and sample the wells, moving from the least contaminated well to the well with the highest level of contamination.

B. Assemble the appropriate sampling equipment (tubing, flow-through bath, water quality meters, and other equipment). Check the condition of all supplies and the operation of the equipment. It should be noted that a flow-through bath cannot be used if a bailer is being used for the water sampling.

C. Decontaminate all sampling equipment before taking the first sample and between sampling intervals (see SOP 1.6, General Equipment Decontamination, and the FSP or WP). Rinse pH and conductivity probes with distilled water between measurements. Collect all decontamination solutions and rinsate liquids in a container or drum and dispose of them at the conclusion of the sampling event. If data exist that show that the decontamination and rinsate liquids will not contribute to contamination at the site, they may be disposed of at the site. Rinsate liquids can be disposed of at the wellsite as long as the disposal area is at least 10 ft from the nearest well.

D. Perform field calibrations for all instrumentation.

3.3. Operation

Procedures for measuring the parameters are discussed below. When practical, take final field measurements after at least three bore volumes have been pumped from the well and as close to the time of sampling as possible. Record measurements, instrument readings, calibration runs, and alkalinity determinations on the appropriate forms included in Appendixes 5.2 through 5.5. Complete the forms as described in Appendix 5.6.

3.3.1. Determining pH

A. Connect the probe to the meter. If a flow-through bath is being used, immerse the meter in the bath after pumping has begun. (A flow-through bath is an enclosed container made of inert material that houses the pH probe and calibration buffer solutions.) The bath allows the buffer solutions to reach the same temperature as the water being withdrawn, and this results in the proper calibration/standardization of the pH meter. The flow-through bath will reduce or eliminate drift. If a flow-through bath is not being used, pour an aliquot of the water into a beaker and immerse the probe in the beaker.

B. Bring the standard solutions to the temperature of the water in the flow-through bath. If the meter has a temperature adjustment, set it to the temperature of the water.

C. Rinse the probe in distilled water and pat completely dry with a clean tissue before putting it in a calibrating solution. Avoid rubbing the probe, which may cause a static charge that will disrupt measurements.

D. Standardize the meter immediately before taking the measurement. The calibrating solutions must bracket the sample--either pH 7.0 and pH 4.0 or pH 7.0 and pH 10.0. Use the procedure described below to standardize the meter.

1. Put the probe in the pH 7.0 (or in the pH 4.0 solution if the pH is less than 4.0) and adjust the standardization knob to achieve a reading of 7.0 (or 4.0 for pH less than 4.0).

2. Put the probe in the other solution and adjust the standardization knob to achieve a reading of 4.0 or 10.0, as appropriate.

3. Repeat those two steps until adjustment is no longer required.

E. Clean the probe with distilled water and clean tissues.

F. If a flow-through bath is being used, put the probe in the bath. Record the measurement within 5 min or after drift has ceased, whichever is sooner. If a bath is not being used, place the probe into a beaker containing an aliquot of water. Record the measurement as described.

G. Immediately rinse and dry the probe and put it in each calibrating solution for about 30 sec. Record the readings.

NOTE: During storage and between measurements, keep the probe immersed in the pH 4.0 solution.

H. If a flow-through bath is being used, keep hoses leading to the bath out of direct sunlight because the water can heat up quickly at low discharge rates.

3.3.2. Measuring Temperature

A. Use a Celsius thermometer to measure the temperature in a small Teflon or plastic bucket. Record measurements in the logbook periodically during pumping.

B. Place the bucket close to the wellhead. Keep the bucket and discharge hose away from direct sunlight.

3.3.3. Measuring Electrical Conductivity

A. Use three KCl solutions of known concentrations (approximately 500, 1000, and 1500 μmhos, depending on vendor availability); these concentrations must bracket the expected electrical conductivity (Ec) of water samples collected in the field. Record the electrical conductivity of the known solutions after the meter probes have been submersed and equilibrated in them. The temperature of the known solutions must be 25°C and recorded from a laboratory-grade thermometer.

B. Cool one KCl known solution to 0°C. Then record electrical conductivity measurements as the temperature of the KCl solution is warmed to 25°C. During this calibration procedure, also record the temperature scale of the Ec meter to confirm the accuracy of the Ec meter thermometer to the known accuracy of the laboratory-grade thermometer.

C. Calibrate the conductivity meter with at least three standard KCl solutions, the extremes of which bracket the anticipated conductivity of the samples. Perform this calibration at least before and after sampling. Keep a record of each calibration and record the temperature of each calibrating solution.

D. Clean the probe and cable with distilled water and clean tissues.

E. Measure conductivity in a Teflon or plastic bottle. Record conductivity periodically (after every two gallons) throughout the time of pumping. Record the position of each setting on the meter.

NOTE: Rinse the probe in distilled water and dry it completely before putting it in a calibration solution.

F. Keep the probe at least two inches away from the cell walls or bucket because most conductivity probes produce an electrical field that may be disturbed if the probe is near a solid object.

3.3.4. Alkalinity Testing

Use a digital titration kit (Hach Kit) to test for alkalinity. Field techniques can be tested by performing alkalinity tests on known solutions prepared by the quality control laboratory. When using the standard solutions, record only the value of alkalinity at a pH of 4.5 on the Standard Alkalinity Record form (Appendix 5.5). The alkalinity of three standard solutions will be determined in triplicate. Perform these tests at the start of field sampling. Report the results to the site manager for confirmation with the known values reported by the laboratory. At the discretion of the site manager, additional standard alkalinity tests may be performed periodically during or after the field sampling period.

Determine the alkalinity of each water sample at the time that the sample is collected; record the results in the appropriate sections of the Groundwater Quality Sampling Record (Appendix 5.3) or Surface Water Quality Sampling Record (Appendix 5.2). Enter alkalinity values for 13 different pH values into the table on the forms and plot the results on the graph. Perform a minimum of two tests on each sample to verify the reproducibility of the values obtained. If possible, have different field personnel run these two tests. If the calculated error is greater than 10%, perform a third test for a redetermination of the error. Three trials are usually sufficient to determine a reasonable field value. At the conclusion of the titration tests, record the pH meter readings in the pH 4.0 and 7.0 buffer solutions. The procedure used to determine alkalinity values is described below.

A. Bring the temperature of the pH 4.0 and 7.0 standard buffer solutions to the temperature of the sample in the flow-through bath. If a flow-through bath is not being used, bring the temperature of the standard solutions to the temperature of the water in the beaker. Standardize the meter with the electrode in each of these buffers according to the accepted method. Leave the electrode in the pH 4.0 buffer solution.

B. Rinse the titration flask and the volumetric flask with distilled water, followed by raw sample. Remove droplets of water by vigorous shaking.

C. Measure 100 ml of fresh, unfiltered sample in the volumetric flask and pour it into the titration flask with as little agitation as possible.

D. Rinse the pH electrode, pat it dry, and put it into the titration flask.

E. Record the titrant lot number and date on the water quality data form. Eject a few drops of titrant from the tip of the titrator and wipe the tips with a clean tissue. Reset the counter to 0000.

F. Titrate the solution with acid, gently stirring the solution to ensure mixing. Whenever feasible, use magnetic stirrers. Record pairs of pH and alkalinity readings for the pH values on the data form(s).

G. Rinse all glassware with distilled water.

3.3.5. Measuring Dissolved Oxygen

A. Connect the YSI 5739 probe to the YSI Model 50B Dissolved Oxygen meter. Place probe in calibration bottle containing a moist sponge. Allow the temperature to equilibrate (3-5 minutes).

B. Set function switch to "°C". From the solubility of oxygen chart (Table I on back of meter) find the mg/L value for the temperature indicated on the display.

C. To determine the calibration value, multiply the value in mg/L found in step B, by .97 (the value in .97 is from Table II on back of meter using an elevation of 841' for Mound Plant).

D. Set function switch to "mg/L CAL". Using the keys beneath the digit positions in the display, set the calibration value determined in step C. Set function switch to "mg/L". The display will show "CAL", then the calibration value entered will appear. Observe the reading for stability for two or three minutes.

E. Place probe in sample. Set function switch to "mg/L". Allow 3-5 minutes for the probe to come to temperature equilibrium with the sample. Begin stirring and observe the reading when the display has stabilized.

F. Record observed reading in the comment section of the field form (Appendix 5.2 or 5.3).

G. When storing probe, keep in humid environment to prevent drying out.

3.3.6. Measuring Oxidation-Reduction Potential

A Hach One Portable pH Meter may be used to measure oxidation-reduction potential with the proper electrode (Hach OPR Electrode). First the meter must be zeroed for millivolt measurements.

A. With meter turned off, insert a paper clip into the BNC connector to electrically short the circuit.

B. Press and hold the button labeled "mV", simultaneously press and release button labeled"!" then release "mV" button. The mv indicator will light, and after 10 seconds, the meter will read 0.0 mV+.2 mV. Remove paper clip and attach OPR electrode.

C. Verify the electrode response by measuring the oxidation-reduction potential of a prepared solution. No calibration can be done with this meter, if electrode is not reading correct value, replace it with a new one.

   Note: Calibration solution preparation is given in manual for Hach OPR Electrode.

D. Record observed reading in the comment section of the field form (Appendix 5.2 or 5.3).

3.4. Postoperation

3.4.1. Field

A. Ensure that all equipment is accounted for, decontaminated (see SOP 1.6, General Equipment Decontamination), and ready for shipment.

B. Restore the site to the presampling conditions and fill open sampling holes as specified in the FSP or WP.

C. Make sure all survey or sampling locations are properly staked and the location ID is readily visible on the location stake.

D. Examine electrical conductivity and temperature calibrations (red line location). Adjust data as indicated.

E. Prepare samples and transport according to SOP 1.3, Sample Control and Documentation; SOP 1.4, Sample Containers and Preservation; and SOP 1.5, Guide to Handling, Packaging, and Shipping of Samples.

3.4.2. Documentation

A. Record cleanup and hole abandonment procedures and any uncompleted work (like site restoration) in the logbook.

B. Record the sampling and operating data in the logbook.

C. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

D. Make any necessary changes on data forms after examining electrical conductivity and temperature calibrations.

E. Review data collection forms for completeness.

3.4.3. Office

A. Deliver original forms and logbooks to the site manager for technical review. He/she will review, sign forms, and transmit to the document control officer (copies to the files) for eventual delivery to the Department of Energy.

B. Inventory equipment and supplies. Repair or replace all broken or damaged equipment. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

C. Contact the analytical laboratory to ensure that samples arrived safely and instructions for sample analyses are clearly understood.

4. SOURCES

EPA. 1979. "Methods for Chemical Analysis of Water and Wastes." U.S. Environmental Protection Agency document EPA/600/4-79-020. Washington, D.C.: U.S. Government Printing Office.

Korte, N., and P. Kearl. 1984. "Procedures for the Collection and Preservation of Groundwater and Surface Water Samples and for the Installation of Monitoring Wells." U.S. Department of Energy, Grand Junction, Colorado.

5. APPENDIXES

5.1. Equipment and Supplies Checklist

5.2. Surface Water Quality Sampling Record

5.3. Groundwater Quality Sampling Record

5.4. Standard Electrical Conductivity Data Form

5.5. Standard Alkalinity Test Record

5.6. Data Form Completion

APPENDIX 5.1

EQUIPMENT AND SUPPLIES CHECKLIST

Complete all blanks before going to the field.

_____ Reagents: Alkalinity kit

- _____ Check reagent volumes
- _____ Check glass for breakage

_____ pH meter(s) (circle one)

- _____ Electrode full of fluid
- _____ Electrode glass intact
- _____ Immerse electrode in tap water
- _____ Calibrate electrode, rinse, fill, and replace cap
- _____ Temperature probe in tap water
- _____ Temperature probe in hot water

_____ Ec Meter

- _____ Battery: Ok___ Dead___
- _____ Tap water: Ok___ Faulty___
- _____ Against calibration solution
- _____ Solution temp
- _____ Conductivity of solution

_____ Hand-held thermometer

- _____ Temperature in ice water
- _____ Temperature agrees with lab thermometer

_____ KCl solutions

_____ Filters and tubing

_____ Flow-through bath

APPENDIX 5.1, Continued

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Teflon or plastic bucket

_____ Teflon or plastic bottle

_____ Titration flask

_____ Volumetric flask

_____ YSI Model 50B dissolved oxygen meter

- _____ YSI 5739 dissolved oxygen probe
- _____ YSI 5740 detachable cable
- _____ Calibration bottle and sponge
- _____ Beaker
- _____ Stirrer

_____ Hach One Portable pH meter (for redox measurements)

- _____ No. 1 steel paper clips
- _____ Hach ORP electrode
- _____ Prepared solution:

  10 mL 10N H_2SO_4 + 90 mL deionized/ distilled H_2O + 2.000g Fe $(NH_4)_2$ $(SO_4)_2$ • 6 H_2O + 0.081g $KMnO_4$

- _____ Beaker
- _____ Stirrer

APPENDIX 5.2

SURFACE WATER QUALITY SAMPLING RECORD

SURFACE WATER QUALITY SAMPLING RECORD

PAGE ____ OF ____

FACILITY CODE _____

LOCATION ID _____ LOT CONTROL NO _____

SAMPLE ID _____ SAMPLE TYPE _____

LOG DATE _____ ACCEPTANCE CODE _____

ANALYTICAL LAB CODE _____ LOGGER CODE _____

SAMPLING PERIOD: START _____ COMPLETE _____

SAMPLING METHOD _____ DATE SENT _____

COMMENTS _____

PARAMETER MEASUREMENTS:

POTENTIAL OF HYDROGEN	pH	S.U.	_____
SPECIFIC CONDUCTANCE	Ec	umhos/cm	_____
REDOX POTENTIAL	Eh	mvolts	_____
TEMPERATURE	TMP	°C	_____
ALKALINITY ($CaCO_3$)	ALK	mg/l	_____
DISSOLVED OXYGEN	DO	mg/l	_____

ACCEPTANCE CODES: A–ACCEPTABLE R–RECONNAISSANCE U–UNACCEPTABLE N–NOT DETERMINED

SAMPLE TYPES:

F – FIELD R – REPLICATE
K – KNOWN A – ACID BLANK

SAMPLING METHODS:

G – GRAB SP – SUBMERSIBLE PUMP
PP – PERISTALIC PUMP O – OTHER (SPECIFY)

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS
WQD–007 (1/88)

_____ FORM COMPLETED BY/DATE

_____ TECHNICAL REVIEWER/DATE

SURFACE WATER QUALITY SAMPLING RECORD

FACILITY CODE _____ PAGE ____ OF ____

LOCATION ID _____ **LOG DATE** _____

SAMPLING INFORMATION

WITHDRAWAL METHOD _____

FILTER SIZE _____

THERMOMETER ID _____

Ec METER ID _____

pH METER ID _____

PUMP ID _____

ALKALINITY KIT ID _____

FIELD REP _____

INSTRUMENT(S) USED _____

_____

CALIBRATION INFORMATION

DATE/TIME OF LAST Ec CALIBRATION _____

TIME OF pH CALIBRATION _____

FOR STANDARD SOLUTION pH 7, INSTRUMENT READING _____

FOR STANDARD SOLUTION pH 4, INSTRUMENT READING _____

FOR STANDARD SOLUTION pH 10, INSTRUMENT READING _____

Eh OF CALIBRATION SOLUTION _____

Eh READING IN CALIBRATION SOLUTION AFTER INSTRUMENT MEASUREMENT _____

TEMP. OF CALIBRATION SOLUTION (°C) _____

SHIPPING INFORMATION

LAB(S) SHIPPED TO: _____ _____ _____

DATE(S) SHIPPED: _____ _____ _____

METHOD OF SHIPMENT: _____ _____ _____

COMMENTS _____

_____

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS
WQD-007 (1/88)

_____ FORM COMPLETED BY/DATE

_____ TECHNICAL REVIEWER/DATE

SURFACE WATER QUALITY SAMPLING RECORD

PAGE ____ OF ____

FACILITY CODE _____

LOCATION ID _____

LOG DATE _____

ERROR AT 4.50: $\left| \frac{X1 - X2}{X5} \right| \times 100 =$ ____%

1st = ____%

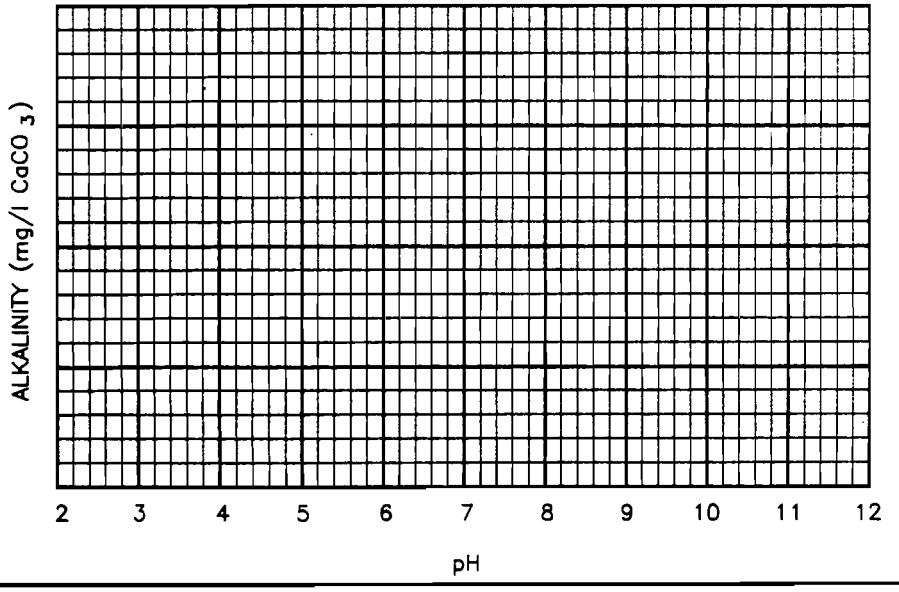
2nd = ____%

pH CHECK AFTER TITRATION

7.00 = _____

4.00 = _____

pH	ALKALINITY (mg/l Ca CO_3)		
	1st	2nd	3rd
8.90			
8.60			
8.30			
8.05			
7.80			
7.50			
6.50			
5.70			
5.10			
4.80			
4.50			
4.25			
4.00			



COMPLETE BOLDED DATA FOR ENTRY INTO TIMS
WQD-007 (3/88)

SURFACE WATER QUALITY SAMPLING RECORD

FACILITY CODE _____ PAGE ____ OF ____

LOCATION ID _____ LOG DATE _____

SAMPLE ID	TEST KIT ID	pH (S.U.)	ALKALINITY (mg/l $CaCO_3$)		UNCERTAINTY (%)
			TRUE VALUE	FIELD VALUE	

COMMENTS _____

ACCEPTANCE CODES: A–ACCEPTABLE R–RECONNAISSANCE U–UNACCEPTABLE N–NOT DETERMINED

COMPLETE BOLDED DATA FOR ENTRY INTO TEMS
WQD–007 (3/88)

APPENDIX 5.3

GROUNDWATER QUALITY SAMPLING RECORD

GROUNDWATER QUALITY SAMPLING RECORD

PAGE ___ OF ___

FACILITY CODE _____ **LOT CONTROL NO** _____

LOCATION ID _____ **SAMPLE TYPE** _____

SAMPLE ID _____ **ACCEPTANCE CODE** _____

LOG DATE _____ **LOGGER CODE** _____

ANALYTICAL LAB CODE _____

INITIAL GROUNDWATER DEPTH (FT) _____ SAMPLE DEPTH (FT) _____

SAMPLING PERIOD: START _____ COMPLETE _____

SAMPLING METHOD _____ DATE SENT _____

COMMENTS _____

FINAL PARAMETER MEASUREMENTS:

POTENTIAL OF HYDROGEN	pH	S.U.	_____
SPECIFIC CONDUCTANCE	Ec	umhos/cm	_____
REDOX POTENTIAL	Eh	mvolts	_____
TEMPERATURE	TMP	°C	_____
ALKALINITY ($CaCO_3$)	ALK	mg/l	_____
DISSOLVED OXYGEN	DO	mg/l	_____

TIME	TOTAL VOLUME WITHDRAWN		pH	Ec (umhos/cm)	TEMP (°C)	COMMENTS
	(GALS)	(Bore Volume)				
	0.0	0.0	--	--	--	START PUMPING

ACCEPTANCE CODES: A–ACCEPTABLE R–RECONNAISSANCE U–UNACCEPTABLE N–NOT DETERMINED

SAMPLE TYPES:

F – FIELD R – REPLICATE
K – KNOWN A – ACID BLANK

SAMPLING METHODS:

G – GRAB SL – SUCTION LIFT PUMP BP – BLADDER PUMP
B – BAILER SP – SUBMERSIBLE PUMP O – OTHER (SPECIFY)
PP – PERISTALIC PUMP AL – AIR–LIFT SAMPLER

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS

WQO–008 (1/88)

_____ FORM COMPLETED BY/DATE _____ TECHNICAL REVIEWER/DATE

GROUNDWATER QUALITY SAMPLING RECORD

PAGE ___ OF ___

FACILITY CODE _____

LOCATION ID _____ **LOG DATE** _____

TIME	TOTAL VOLUME WITHDRAWN		pH	Ec (umhos/cm)	TEMP (°C)	COMMENTS
	(GALS)	(Bore Volume)				

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS

WQD-006 (1/88)

GROUNDWATER QUALITY SAMPLING RECORD

PAGE ___ OF ___

FACILITY CODE _____

LOCATION ID _____ LOG DATE _____

BORE VOL CALCULATION (GAL)

$$\frac{d^2\pi}{4} (h_1 - h_2) \times 7.48$$

DEPTH OF WELL (h_2) (FT) _____

DEPTH TO WATER (h_1) (FT) _____

WELL DIAMETER (FT) _____

ONE BORE VOLUME (GAL) _____

SCREENED INTERVAL (FT) _____

FIELD REP _____

INSTRUMENT(S) USED _____

_____

SAMPLING INFORMATION

WITHDRAWAL METHOD _____

FILTER SIZE _____

THERMOMETER ID _____

Ec METER ID _____

pH METER ID _____

PUMP ID _____

ALKALINITY KIT ID _____

CALIBRATION INFORMATION

DATE/TIME OF LAST Ec CALIBRATION _____

TIME OF pH CALIBRATION _____

FOR STANDARD SOLUTION pH 7, INSTRUMENT READING _____

FOR STANDARD SOLUTION pH 4, INSTRUMENT READING _____

FOR STANDARD SOLUTION pH 10, INSTRUMENT READING _____

Eh OF CALIBRATION SOLUTION _____

Eh READING IN CALIBRATION SOLUTION AFTER MEASUREMENT _____

TEMP OF CALIBRATION SOLUTION (° C) _____

SHIPPING INFORMATION

LAB(S) SHIPPED TO: _____ _____ _____

DATE(S) SHIPPED: _____ _____ _____

METHOD OF SHIPMENT: _____ _____ _____

COMMENTS _____

_____

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS
WQD-008 (3/88)

GROUNDWATER QUALITY SAMPLING RECORD

PAGE ___ OF ___

FACILITY CODE _____

LOCATION ID _____

LOG DATE _____

ERROR AT 4.50: $\left| \frac{X1 - X2}{XS} \right| \times 100 =$ ___%

1st = _____%

2nd = _____%

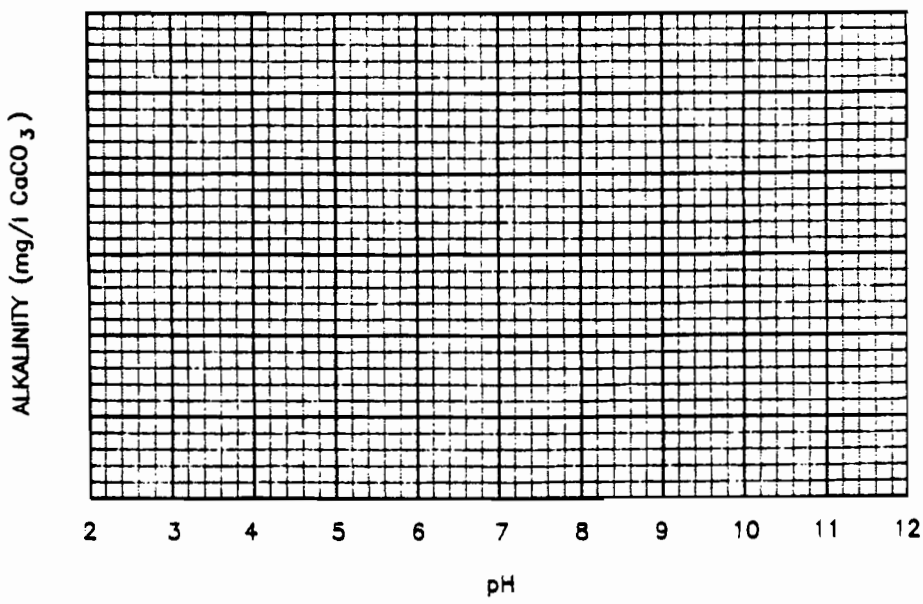
INITIAL pH _____

pH CHECK AFTER TITRATION

7.00 = _____

4.00 = _____

pH	ALKALINITY (mg/l Ca CO_3)		
	1st	2nd	3rd
8.90			
8.60			
8.30			
8.05			
7.80			
7.50			
6.50			
5.70			
5.10			
4.80			
4.50			
4.25			
4.00			



COMPLETE BOLDED DATA FOR ENTRY INTO TIMS
WQD-008 (3/88)

APPENDIX 5.4

STANDARD ELECTRICAL CONDUCTIVITY FORM

STANDARD ELECTRICAL CONDUCTIVITY DATA

FACILITY CODE _____ LOG DATE _____

LOGGER CODE _____ ACCEPTANCE CODE _____

MANUFACTURER/Ec METER ID _____

ELECTRICAL CONDUCTIVITY OF KNOWN SOLUTIONS AT 25° C:	
KNOWN (mg/l KCl)	Ec (meter)

Ec	TEMPERATURE (°C) (LAB THERMOMETER)	TEMPERATURE (°C) (Ec METER)
	0	
	1	
	2	
	3	
	4	
	5	
	6	
	7	
	8	
	9	
	10	
	11	
	12	
	13	
	14	
	15	
	16	
	17	
	18	
	19	
	20	
	21	
	22	
	23	
	24	
	25	

COMMENTS _____

ACCEPTANCE CODES: A–ACCEPTABLE R–RECONNAISSANCE U–UNACCEPTABLE N–NOT DETERMINED

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS

SEC–046 (1/88)

_____ FORM COMPLETED BY/DATE

_____ TECHNICAL REVIEWER/DATE

APPENDIX 5.5

STANDARD ALKALINITY TEST RECORD

STANDARD ALKALINITY TEST RECORD

FACILITY CODE _____

FIELD REP _____ **LOG DATE** _____

ALKALINITY TEST KIT:

MANUFACTURER _____ ALKALINITY TEST KIT ID _____

pH METER:

MANUFACTURER _____ SERIAL NO _____

LAB CODE	STANDARD SOLUTION ID	KNOWN VALUE (mg/l $CaCO_3$)	ALKALINITY TEST VALUES (at pH endpoint = 4.5) mg/l $CaCO_3$

COMMENTS _____

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS
SAT-131 (2/88)

APPENDIX 5.6

DATA FORM COMPLETION

Use a pen with black ink that is not water soluble (not a felt-tip pen). Make an entry in each blank. Where there is no data entry, enter UNK for Unknown, NA for Not Applicable, or ND for Not Done. If any procedure was not performed as prescribed, give the reason for the change or omission on the form. To change an entry, draw a single line through it, add the correct information above it, and initial the change.

SURFACE WATER QUALITY SAMPLING RECORD

1. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

2. Location ID. Four-character code assigned sequentially to each borehole, test pit, or surface location where chemical, biological, radiological, and other measurements are taken.

3. Sample ID. A four-digit number assigned to ensure that collected data retain uniqueness.

4. Log Date. The date the information recorded on the form was obtained in the format DD-MMM-YY (01-JAN-88).

5. Analytical Lab Code. A three-character code identifying the company responsible for preparing the standard solution.

6. Lot Control Number. A two-digit number denoting that a given set of samples should be analyzed as a lot or group. Normally, this number is not assigned by persons in the field collecting surface water samples.)

7. Sample Type. A one-character code distinguishing the type of sample collected. This classification permits the analysis of data for specific groups of samples. The codes used for water samples are listed below.

   D--Duplicate
   R--Replicate
   S--Spike
   K--Known
   F--Field Blank
   T--Trip Blank
   L--Lab Blank

8. Acceptance Code. A one-character code assigned by the facility manager.

9. Logger Code. Three-character or four-character code identifying the company responsible for collecting the information recorded on the form.

10. Sampling Period. Record the starting and completion times when field measurements were taken in the 24-hr format of hours:minutes (for example, 08:37 for 8:37 a.m. and 19:12 for 7:12 p.m.). See the conversion table below.

    Start--the time that collection of water sample begins.

    Complete--the time that collection of water sample ends.

Conversion Table

Conventional Time	24-Hour Time
1:00 a.m.	1:00
12:00 Noon	2:00
1:00 p.m.	13:00
2:00 p.m.	4:00
3:00 p.m.	15:00
4:00 p.m.	16:00
5:00 p.m.	17:00
6:00 p.m.	18:00
7:00 p.m.	19:00
8:00 p.m.	20:00
9:00 p.m.	21:00
10:00 p.m.	22:00
11:00 p.m.	23:00
12:00 Midnight	24:00

11. Sampling Method. A two-character code identifying the method used to collect a soil, rock, or water sample. The codes used for various water sampling methods are listed below.

B--Bailer	AL--Air Lift Sampler
C--Composite	BP--Bladder Pump
G--Grab	PP--Peristaltic Pump
R--Random	SL--Suction Lift Pump
S--Systematic	SP--Submersible Pump

12. Date Sent. The date the water sample was sent from the field collection site to the analytical laboratory in the format DD-MMM-YY (01-JAN-88).

13. Comments. Includes preservation method, acidified or nonacidified, type of acid (if acid was used to preserve water sample), and any additional information.

14. Parameter Measurements. The measurements listed below are recorded at the time the sample is collected.

    a. Potential of Hydrogen--the pH of the water sample in S.U. units at the time of collection.

b. Specific Conductance--The specific conductance of the water sample in micromhos/cm (mhos/cm) at the time of collection.

c. Redox Potential--The redox potential of the water sample in millivolts (mV) at the time of collection.

d. Temperature--The temperature of the water sample in degrees Celsius (°C) at the time of collection.

e. Alkalinity ($CaCO_3$)--The alkalinity of the water sample in milligrams per liter (mg/liter) at the time of collection.

f. Dissolved Oxygen--The dissolved oxygen content of the water sample in milligrams per liter (mg/liter) at the time of collection.

15. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

16. Location ID. Four-character code assigned sequentially to each borehole, test pit, or surface location where chemical, biological, radiological, and other measurements are taken.

17. Log Date. The date the information recorded on the form was obtained in the format DD-MMM-YY (01-JAN-88).

18. Withdrawal Method. The sampling device used to collect the surface water sample.

19. Filter Size. Size of filter in use.

20. Thermometer ID. The identification of the thermometer used.

21. Ec Meter ID. The control number and manufacturer of the meter used to measure the specific conductance of samples or calibration solutions.

22. pH Meter ID. The control number and manufacturer of the meter used to measure the pH of samples of standard solutions.

23. Pump ID. Identification of the pump in use.

24. Alkalinity Kit ID-identification and model or serial number of the alkalinity kit used.

25. Field Rep. Name of the field representative.

26. Instruments Used. The types of instruments used to obtain measurements, monitor air quality, or facilitate the collection of a sample or test performance.

27. Calibration Information. After performing the alkalinity titration test and immediately before sample collection, record the information listed below.

    a. Date/Time of Last Ec Calibration. Date and time that the specific conductivity meter was last calibrated in format of year:month:day/hour:minute.

    b. Time of pH Calibration. Time the pH meter was calibrated in hours:minutes.

    c. Standard Solution pH Readings. The pH readings are to be recorded as the probe is immersed in standard solutions of pH 7.0, 4.0 and 10.0.

    d. Eh of Calibration Solution. The Eh of the solution supplied by the laboratory to calibrate the specific conductance meter.

    e. Eh Reading in Calibration Solution after Measurement. The numerical value obtained after immersing the Eh probe in the standard solution.

    f. Temp of Calibration Solution (°C). The temperature of the Eh standard solution measured during the time that the probe is immersed.

28. Shipping Information. Information that includes the date, method, laboratory where the samples were shipped, and a comments section.

29. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

30. Location ID. Four-character code assigned sequentially to each borehole, test pit, or surface location where chemical, biological, radiological, and other measurements are taken.

31. Log Date. The date the information recorded on the form was obtained in the format DD-MMM-YY (01-JAN-88).

32. Error at 4.50. Error calculation to test the precision of the alkalinity measurement. Two tests for alkalinity are performed on the same sample, and the values are written on the pH/Alkalinity chart. The alkalinity at pH = 4.50 for test number one (x1) is subtracted from alkalinity determined in test number two (x2), and the result is divided by sx (the value of x1 or x2, whichever is smaller). The absolute value of the result is multiplied by 100 to obtain a percentage. If the calculated error is less than 10%, no further runs are necessary. If the calculated error is greater than 10%, a third test must be run. After the third test, the field sampler should use his/her judgement to recalculate the error using the value from the third test.

33. Alkalinity (mg/*l* $CaCO_3$). The alkalinity of the water sample in milligrams per liter (mg/liter) at different values of pH during titration.

34. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

35. Location ID. Four-character code assigned sequentially to each borehole, test pit, or surface location where chemical, biological, radiological, and other measurements are taken.

36. Log Date. The date the information recorded on the form was obtained in the format DD-MMM-YY (01-JAN-88).

37. Sample ID. A four-digit number assigned to ensure that collected data retain uniqueness.

38. Alkalinity Test Kit ID. The control number used to identify the kit being utilized to measure the alkalinity of a sample or standard solution.

39. Potential of Hydrogen. The pH of the water sample in S.U. units at the time of collection.

40. Alkalinity ($CaCO_3$). Enter the field alkalinity of the water sample in milligrams per liter (mg/liter) at the time of collection in the field value column.

41. True Value. Each standard solution will be prepared to a specific alkalinity as specified by the site manager. This value will not be disclosed to the field samplers until they have completed their calibration runs. After the calibration runs are complete, the samplers must contact the site manager and report the values determined in the field. If the values are within 10% of the known value, the site manager will authorize the sampling and supply the values that are to be entered in this data entry field.

42. Field Value. The alkalinity value that samplers determined in the field on the calibration run.

43. Uncertainty (%) = $(100) \bullet \left(\frac{\text{Field Value - True Value}}{\text{True Value}}\right)$

44. Comments. Any additional information.

APPENDIX 5.6, Continued

GROUNDWATER QUALITY SAMPLING RECORD

1. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

2. Location ID. Four-character code assigned sequentially to each borehole, test pit, or surface location where chemical, biological, radiological, and other measurements are taken.

3. Sample ID. A four-digit number assigned to ensure that collected data retain uniqueness.

4. Log Date. The date the information recorded on the form was obtained in the format DD-MMM-YY (01-JAN-88).

5. Analytical Lab Code. A three-character code identifying the company responsible for preparing the standard solution.

6. Lot Control Number. A two-digit number denoting that a given set of samples should be analyzed as a lot or group. Normally, this number is not assigned by persons in the field collecting surface water samples.

7. Sample Type. A one-character code distinguishing the type of sample collected. This classification permits the analysis of data for specific groups of samples. The codes used for water samples are listed below.

D--Duplicate	F--Field Blank
R--Replicate	T--Trip Blank
S--Spike	L--Lab Blank
K--Known	

8. Acceptance Code. A one-character code assigned by the facility manager.

9. Logger Code. Three-character or four-character code identifying the company responsible for collecting the information recorded on the form.

10. Initial Groundwater Depth. Distance in feet from the top of the casing to the surface of the water in the well. This length is determined by using the well sounder before measuring the depth of the well. Measure depth-to-water to the nearest tenth of a foot.

11. Sample depth. The depth at which the sample was taken.

12. Sampling Period. Record the starting and completion times when field measurements were taken in the 24-hr format of hours:minutes (for example, 08:37 for 8:37 a.m. and 19:12 for 7:12 p.m.). See the conversion table below.

Start--the time that collection of water sample begins.

Complete--the time that collection of water sample ends.

Conversion Table

Conventional Time	24-Hour Time
1:00 a.m.	1:00
12:00 Noon	2:00
1:00 p.m.	13:00
2:00 p.m.	4:00
3:00 p.m.	15:00
4:00 p.m.	16:00
5:00 p.m.	17:00
6:00 p.m.	18:00
7:00 p.m.	19:00
8:00 p.m.	20:00
9:00 p.m.	21:00
10:00 p.m.	22:00
11:00 p.m.	23:00
12:00 Midnight	24:00

13. Sampling Method. A two-character code identifying the method used to collect a soil, rock, or water sample. The codes used for various water sampling methods are listed below.

B--Bailer	AL--Air Lift Sampler
C--Composite	BP--Bladder Pump
G--Grab	PP--Peristaltic Pump
R--Random	SL--Suction Lift Pump
S--Systematic	SP--Submersible Pump

14. Date Sent. The date the water sample was sent from the field collection site to the analytical laboratory in the format DD-MMM-YY (01-JAN-88).

15. Comments. Includes preservation method, acidified or nonacidified, type of acid (if acid was used to preserve water sample), and any additional information.

16. Final Parameter Measurements. The following measurements are recorded at the time the sample is collected.

    a. Potential of Hydrogen. The pH of the water sample in S.U. units at the time of collection.

    b. Specific conductance. The specific conductance of the water sample in micromhos/cm (mhos/cm) at the time of collection.

c. Redox Potential. The redox potential of the water sample in millivolts (mV) at the time of collection.

d. Temperature. The temperature of the water sample in degrees Celsius (°C) at the time of collection.

e. Alkalinity ($CaCO_3$). The alkalinity of the water sample in milligrams per liter (mg/liter) at the time of collection.

f. Dissolved Oxygen. The dissolved oxygen content of the water sample in milligrams per liter (mg/liter) at the time of collection.

17. The Groundwater Quality Record form contains data that are collected during well purging. These are described below.

    a. Time. The time when a field measurement of purged water was performed in the format HH:MM.

    b. Total Volume Withdrawn. Using a bucket or flow meter, the field sampler will note the number of gallons of water purged from the well at the time that a field measurement is taken. This entry is cumulative and represents the total volume of water purged before sampling may begin. A second column is provided so that the field sampler can equate gallons as bore volumes purged.

    c. pH. The pH readings of water sampled during the purging process.

    d. Ec. The specific conductance readings of water sampled during the purging process in micromhos/cm (mhos/cm).

    e. Temp (°C). The temperature of the water sampled during the purging process.

    f. Comments. Includes preservation method, acidified or nonacidified, type of acid (if acid was used to preserve water sample), and any additional information.

18. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

19. Location ID. Four-character code assigned sequentially to each borehole, test pit, or surface location where chemical, biological, radiological, and other measurements are taken.

20. Log Date. The date the information recorded on the form was obtained in the format DD-MMM-YY (01-JAN-88).

21. Depth of Well (h_2). Distance in feet from the top of the casing to the bottom of the well. This length can be determined by bottoming out a water level sounder against the bottom cap of the well. Measure the depth of the well to the nearest tenth of a foot.

22. Depth-to-Water (h_1). Distance in feet from the top of the casing to the surface of the water in the well. This length is determined by using the well sounder before measuring the depth of the well. Measure depth-to-water to the nearest tenth of a foot.

23. Well Diameter. The inside diameter of the well casing measured to the nearest tenth of a foot.

24. One Bore Volume. The volume of water contained in the well before purging is started. One bore volume is determined by the equation included on the form.

25. Screened Interval (Ft). Depth from the top of the casing to the midpoint of the screen or well section open to the aquifer. Well completion information must be consulted to determine the length and position of the screen.

26. Field Rep. Name of the field representative.

27. Instruments Used. Well sounding instruments and the type of system used to purge the well.

28. Withdrawal Method. The method used to sample the well.

29. Filter Size. The filter size being used.

30. Thermometer ID. The identification of the thermometer being used.

31. Ec Meter ID. The control number and manufacturer of the meter used to measure the specific conductance of samples or calibration solutions.

32. pH Meter ID. The control number and manufacturer of the meter used to measure the pH of samples of standard solutions.

33. Pump ID. Identification and model or serial number of the alkalinity kit used.

34. Alkalinity Kit ID. Identification and model or serial number of the alkalinity kit used.

35. Calibration Information. After performing the alkalinity titration test and immediately before sample collection, the information listed below will be recorded.

a. Date/Time of Last Ec Calibration. Date and time that the specific conductivity meter was last calibrated in format of year:month:day/hour:minute.

b. Time of pH Calibration. Time the pH meter was calibrated in hours:minutes.

c. Standard Solution pH readings. The pH readings are to be recorded as the probe is immersed in standard solutions of pH 7.0, 4.0, and 10.0.

d. Eh of Calibration Solution. The Eh of the solution supplied by the laboratory to calibrate the specific conductance meter.

e. Eh Reading in Calibration Solution after Measurement. The numerical value obtained after immersing the Eh probe in the standard solution.

f. Temp of Calibration Solution (°C). The temperature of the Eh standard solution measured during the time that the probe is immersed.

36. Shipping Information. Information that includes the date, method, laboratory where the samples were shipped, and a comments section.

37. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

38. Location ID. Four-character code assigned sequentially to each borehole, test pit, or surface location where chemical, biological, radiological, and other measurements are taken.

39. Log Date. The date the information recorded on the form was obtained in the format DD-MMM-YY (01-JAN-88).

40. Error at 4.50. Error calculation to test the precision of the alkalinity measurement. Two tests for alkalinity are performed on the same sample, and the values are written on the pH/Alkalinity chart. The alkalinity at pH = 4.50 for test number one (x1) is subtracted from alkalinity determined in test number two (x2), and the result is divided by sx (the value of x1 or x2, whichever is smaller). The absolute value of the result is multiplied by 100 to obtain a percentage. If the calculated error is less than 10%, no further runs are necessary. If the calculated error is greater than 10%, a third test must be run. After the third test, the field sampler should use his/her judgement to recalculate the error using the value from the third test.

41. Alkalinity (mg/*l* $CaCO_3$). The alkalinity of the water sample in milligrams per liter (mg/liter) at different pH values during titration.

APPENDIX 5.6, Continued

STANDARD ELECTRICAL CONDUCTIVITY FORM

1. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

2. Logger Code. Three-character or four-character code identifying the company responsible for collecting the information recorded on the form.

3. Ec Meter ID. The control number and manufacturer of the meter used to measure the specific conductance of samples or calibration solutions.

4. Log Date. The date the information recorded on the form was obtained in the format DD-MMM-YY (01-JAN-88).

5. Acceptance Code. A one-character code assigned by the facility manager.

6. Known Value. Each standard solution will be prepared to a specific alkalinity as specified by the site manager. This value will not be disclosed to the field samplers until they have completed their calibration runs. After the calibration runs are complete, the samplers must contact the site manager and report the values determined in the field. If the values are within 10% of the known value, the site manager will authorize the sampling and supply the values that are to be entered in this data entry field.

7. Ec. The specific conductance of the water sample in micromhos/cm (mhos/cm) at the time of collection.

8. Temperature (°C). The temperature of the water sample in degrees Celsius (°C) at the time of collection.

9. Comments. Any other information.

APPENDIX 5.6, Concluded

STANDARD ALKALINITY TEST RECORD

1. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

2. Field Rep. Name of the field representative.

3. Log Date. The date the information recorded on the form was obtained in the format DD-MMM-YY (01-JAN-88).

4. Manufacturer. The company that manufactures the alkalinity test kit.

5. Alkalinity Test Kit ID. The control number used to identify the kit being utilized to measure the alkalinity of a sample or standard solution.

6. pH Meter ID. The control number and manufacturer of the meter used to measure the pH of samples of standard solutions.

7. Lab Code. A three-character code identifying the company responsible for preparing the standard solution.

8. Standard Solution ID. The control number used to identify the standard solution prepared by the quality control laboratory. The control number is assigned by the laboratory and conforms to the internal numbering system.

9. Known Value. Each standard solution will be prepared to a specific alkalinity as specified by the site manager. This value will not be disclosed to the field samplers until they have completed their calibration runs. After the calibration runs are complete, the samplers must contact the site manager and report the values determined in the field. If the values are within 10% of the known value, the site manager will authorize the sampling and supply the values that are to be entered in this data entry field.

10. Alkalinity Test Values ($CaCO_3$). The alkalinity of the water sample in milligrams per liter (mg/liter) at the endpoint of titration (pH = 4.50).

11. Comments. Includes preservation method, acidified or nonacidified, type of acid (if acid was used to preserve water sample), and any additional information.

STANDARD OPERATING PROCEDURE 2.8

SAMPLING FOR VOLATILE ORGANICS

1. PURPOSE

To outline procedures for collecting a representative groundwater sample and transporting it from its original environment to the laboratory for analysis of trace volatile organics.

2. DISCUSSION

The growing concern over the past several years about low levels of volatile organic compounds in water supplies has led to the development of highly sophisticated analytical methods that can provide detection limits at parts-per-trillion levels. Although laboratory methods are extremely sensitive, well controlled, and quality assured, they cannot compensate for a poorly collected sample. The collection of a sample should be as sensitive, highly developed, and quality assured as the analytical procedures.

A sample to be analyzed for dissolved volatile organics must be collected with minimal disturbance of the sample to limit aeration, which would cause a loss of volatiles from the sample.

Sample retrieval systems suitable for the valid collection of volatile organic samples are positive-displacement bladder pumps; reciprocating piston-type, submersible pumps; gear-driven, submersible pumps; syringe samplers; and bailers (Barcelona 1984; Bennett 1988; Nielsen 1985). Field conditions and other considerations will limit the choice of system. The focus of concern must be to provide a valid sample for analysis, one that has been subjected to the least amount of turbulence possible.

Materials of construction for bladders, pumps, bailers, and tubing are limited to stainless steel, Teflon, and glass. The tendency of organics to leach into and out of many materials makes the selection of materials critical for these trace analyses. The use of plastics, for example, Tygon, must be avoided. There are numerous ways of introducing foreign contaminants into a sample; these must be avoided by following strict sampling procedures and using only trained personnel.

Treatment of the sample with sodium thiosulfate preservative is required only when there is residual chlorine in the water. Residual chlorine could cause free, radical chlorination and change the identity of the original contaminants.

Holding time for the analysis of volatiles is seven days. It is imperative that the samples are shipped or delivered to the analytical laboratory daily. The bottles must be shipped on their sides to aid in maintaining the airtight seal during shipment, with adequate packing and cooling to ensure that they arrive intact. The sensitivity of the analysis and the fragility of the samples require that all volatile samples are collected in duplicate.

The Field Sampling Plan (FSP) or Work Plan (WP) contains site-specific details about the procedures and equipment for this SOP. Refer to the FSP or WP for the analyses that are to be performed. Collection and measurement of samples and the documentation of data will be performed as described in the associated procedures.

3. PROCEDURES

3.1. Associated Procedures

Before every operation, a review of the SOPs 1.1-1.10 is necessary. These SOPs contain information on the performance of field activities. They should be consulted for specific information about equipment and supplies; sample collection, preservation, packaging, and shipping; decontamination procedures; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.3	Sample Control and Documentation
1.4	Sample Containers and Preservation
1.5	Guide to the Handling, Packaging, and Shipping of Samples
1.6	General Equipment Decontamination
2.1	Presample Purging of Wells
2.2	Field Measurement on Ground and Surface Water Samples
2.3	Sampling Monitoring Wells with a Bladder Pump
2.4	Sampling Monitoring Wells with a Bucket-Type Bailer

3.2. Preparation

3.2.1. Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Obtain appropriate permission for property access.

D. Assemble the equipment and supplies listed in Appendix 5.1. Ensure the proper operation of all sampling equipment.

E. Notify the analytical laboratory of sample types, the number of samples, and the approximate arrival date.

F. Contact the carrier that will transport samples to obtain information on regulations and specifications.

G. Obtain appropriate discharge permits or containment vessels for purged water.

3.2.2. Documentation

A. Obtain a logbook from the QA officer.

B. Record results of the equipment check in the logbook.

C. Obtain a sufficient number of the appropriate ER Program data collection forms (see INDEX TO SOPs).

D. Consult the ER Program data administrator for a current list of information management codes, location IDs, and sample numbers used in the completion of data forms.

3.2.3. Field

A. Locate monitoring wells to be sampled and the appropriate decontamination area. Locate the staging area and areas for managing purged water and expendable sampling materials. Check decontamination zones and barricades to public access.

B. Decontaminate all sampling equipment before taking the first sample and between sampling intervals (see SOP 1.6, General Equipment Decontamination, and the FSP or WP). Perform other sampling tasks before collecting the volatile samples (see SOP 2.1, Presample Purging of Wells; SOP 2.2, Field Measurements on Ground and Surface Water Samples; and SOP 2.8, Sampling for Volatile Organics).

C. Sample monitoring wells from least to most contaminated to reduce the possibility for cross-contamination.

3.3. Operation

3.3.1. Preliminary Determinations

A. Floating Organics. If floating organics are of concern (as determined in the FSP or WP and by field measurement for floating organics), obtain the sample with a bucket-type or bottom-valve bailer (see SOP 2.4, Sampling Monitoring Wells with a Bucket-Type Bailer).

B. Purging. Purge the well before sampling, as specified in the FSP or WP and according to SOP 2.1, Presample Purging of Wells. Ensure that the well was not pumped dry and that high flow rates were not employed to cause turbulence in the formation.

C. Residual Chlorine. Using the Hach Field Test Kit for Chlorine, determine if there is residual chlorine in the water to be sampled. If there is, treat the sample vial with a crystal of sodium thiosulfate before sample collection (see SOP 1.4, Sample Containers and Preservation).

3.3.2. Sample Retrieval

A. Bladder Pumps. Using a nongas, contact positive-displacement bladder pump is often mandated when dedicated pumps have been installed on wells. These bladder pumps are also suitable for shallow wells (less than 100 ft). They are somewhat difficult to clean, but can be used with well-dedicated sample tubing to avoid some cleaning. These pumps require a power supply and a compressed gas

supply (or compressor). They may be operated at variable flow and pressure rates, which makes them ideal for both purging and sampling.

1. Increase the cycle time and reduce the pressure to the minimum that will allow the sample to come to the surface.

2. Open both vials, set caps in a clean place, and collect the sample during the middle of the cycle. Collecting both samples at the same time provides more similar samples than those collected at different times, but may be difficult. At a minimum, collect two samples from consecutive discharge cycles.

3. Holding the edge of the sample line at the top edge of the sample vial, allow the water to run down the side into the vial. Do not allow the water to drop or fall into the vial; avoid splashing. The discharge rate cannot exceed 100 mL/minute.

4. Fill the vials just to overflowing. Do not rinse the vials or excessively overflow them. There should be a convex meniscus on the top of the vials.

5. Be sure the caps have not been contaminated (splashed) and carefully cap the vials. Place the caps directly over the top and screw down firmly. Do not overtighten and break the cap.

6. Invert the vials and tap gently. Observe vials for at least 10 sec. If an air bubble appears, discard the sample and begin again. It is imperative that no entrapped air is in the sample vial.

7. Immediately place the vials in the protective foam sleeve and place into the cooler. They should be on their sides, rather than straight up.

8. Ship or deliver samples to the laboratory daily. Ensure that the samples are iced, but do not allow them to freeze.

NOTE: Whenever a sample is collected, a custody record must be initiated on the Custody Transfer Record/Lab Work Request form and a Water Sample Identification Label affixed to the sample container. SOP 1.3, Sample Control and Documentation, contains copies of the form and label and instructions for completing the form and label.

B. Reciprocating Piston-Type, Submersible Pumps. These systems are portable, self-contained, and capable of delivery flow rates of 30 gallons per hour at lifts up to 500 ft. The pump fits into 2-inch wells, which is the most common monitoring well diameter. The flow rate of the pump is varied by increasing or decreasing the driving pressure supplied to the pump from a compressed air container. The gas driving the pump does not contact the sample being purged, making this type of pump the preferred tool to collect samples for volatile organic analyses in deep wells. The samples are collected as described in steps 2-8 of Section 3.3.2.A.

C. Gear-Driven, Submersible Pumps. These pumps are not as complicated or expensive as the bladder pumps. They provide comparable samples and are often easier to handle and clean than are other pumps. However, they do not have good control of flow rate, and more care must be exercised when sampling with them.

Collect the sample as described in steps 2-8 of Section 3.3.2.A. Use more care with this kind of a pump because the flow rate is not controllable and there is a greater potential for splashing and aeration of the sample.

D. Syringe Samplers. Only a limited number of commercial, syringe-type samplers are available (two are manufactured by IEA and TIMCO). Although some homemade devices apparently provide good, quality samples for volatile analysis, these devices are severely limited in sample volume and are specific to sampling for volatiles. Essentially, they operate with an evacuated chamber that is lowered down the well and allowed to fill from the pressure of the water. The entire mechanism is then brought to the surface with the sample. The sample can then be transferred to a sample vial, or the entire unit may be sent as the sample container.

1. If necessary, evacuate the syringe and lower the sampling device to just below the well screen.

2. Remove the constriction from the device and allow the syringe to fill with sample, applying slight suction.

3. Bring unit to the surface. If necessary, transfer the sample to vials (see steps 2-8 of Section 3.3.2.A).

E. Bailers. The positive-displacement, volatile-sampling bailer by GPI is the most appropriate bailer to collect water samples for volatile analysis. Other bailer types (messenger and bottom fill) are less desirable, but may be mandated by cost and site considerations. Generally, bailers can provide an acceptable sample, provided that the sampling personnel use extra care in the collection process.

1. If using the GPI bailer, insert a volatile organic analysis (VOA) vial into the clamp and assemble the unit.

2. Securely attach the bailer to the cable that is on a reel.

3. Lower the bailer slowly and gently into the well, taking care not to shake the casing sides or splash the bailer into the water.

4. Check the reel meter to note the location of the bailer in the well. Stop when the bailer reaches the screened interval.

5. Retrieve the bailer from the well slowly and gently, avoiding bumping and splashing.

6. Once the bailer reaches the surface, detach the bailer from the cable. Repeat steps 1-6 for the second vial.

7. Carefully open the GPI bailer unit and remove the vial. If using a conventional bailer, skip the next step. If using a GPI bailer, repeat steps 1-7 to collect the duplicate sample.

8. Begin slowly pouring from the bailer; collect the duplicate samples from the midstream sample.

9. Proceed as described in steps 2-8 of Section 3.3.2.A.

3.4. Postoperation

3.4.1. Field

A. Ensure that all equipment is accounted for, decontaminated (see SOP 1.6, General Equipment Decontamination), and ready for shipment.

B. Restore the site to presampling conditions as specified in the FSP or WP.

C. Make sure all wells are properly labeled and the location ID is readily visible on the protective casing.

D. Prepare samples and transport according to SOP 1.3, Sample Control and Documentation; SOP 1.4, Sample Containers and Preservation; and SOP 1.5, Guide to Handling, Packaging, and Shipping of Samples.

3.4.2. Documentation

A. Record cleanup and hole abandonment procedures and any uncompleted work (like site restoration or long-term monitoring) in the logbook.

B. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

C. Review data collection forms for completeness.

3.4.3. Office

A. Deliver original forms and logbooks to the site manager for technical review. He/she will review, sign forms, and transmit to the document control officer (copies to the files) for eventual delivery to the Department of Energy.

B. Inventory equipment and supplies. Repair or replace all broken or damaged equipment. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

C. Contact the analytical laboratory to ensure that samples arrived safely and instructions for sample analyses are clearly understood.

4. SOURCES

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5. APPENDIX

5.1. Equipment and Supplies Checklist

APPENDIX 5.1

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Teflon stainless steel bladder pump

_____ Teflon stainless steel gear-driven submersible pump

_____ Syringe sampler; stainless steel, Teflon, or glass

_____ Teflon stainless steel bailer (positive-displacement or point-retrieval)

_____ Teflon or other chemically inert tubing

_____ Fittings for pump

_____ 40-ml amber glass vials; Teflon-lined septa

_____ Hach field kit for chlorine

_____ $Na_2S_2O_4$ crystals

_____ Foam sleeves, coolers, and Blue Ice (or equivalent)

_____ Cable, reel, and tripod

_____ Air compressor or bottled nitrogen

_____ Plastic sheet

_____ Any additional supplies listed in associated procedures, as needed

STANDARD OPERATING PROCEDURE 2.9

SURFACE WATER SAMPLING

1. PURPOSE

To define guidelines followed by field personnel in sampling surface water bodies and documenting all aspects of surface water sample collection.

2. DISCUSSION

The sampling of streams, rivers, ponds, and lakes requires care to avoid contamination and collect representative samples.

The preferred method for collecting surface water samples uses a peristaltic pump (Korte and Kearl 1984). The pump system allows the union of the filtration assembly with the pump and the sample container. In this method, surface samples are filtered and collected directly with minimal elapsed time. With a peristaltic pump, only inert materials contact the sample. The inert pump tubing is relatively inexpensive and easily replaced.

An alternate method for this sampling is to collect surface water as grab samples. This method involves dipping a beaker, dipper, or other transfer device into the surface water to retrieve samples. The water sample can also be collected directly by dipping the collection bottle into the water and filling, removing, and capping it. This method has several drawbacks, including problems associated with sampling shallow waters like seeps, springs, or shallow streams. The likelihood of extensive air contact during the filtering of a sample and the time lapse before preservatives are added to samples are also problems. The only advantage of the grab-sample method is the low cost.

The Field Sampling Plan (FSP) or Work Plan (WP) includes guidelines for selecting sampling equipment and analytical methods to be used at each sampling location. The FSP or WP also has specific details about the procedures for this SOP. Collection and measurement of samples and the documentation of data will be performed as described in the associated procedures.

3. PROCEDURES

3.1. Associated Procedures

Before every operation, a review of the SOPs 1.1-1.10 is necessary. These SOPs contain information on the performance of field activities. They should be consulted for specific information about equipment and supplies; sample collection, preservation, packaging, and shipping; decontamination procedures; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.3	Sample Control and Documentation

1.4	Sample Containers and Preservation
1.5	Guide to the Handling, Packaging, and Shipping of Samples
1.6	General Equipment Decontamination
2.1	Presample Purging of Wells
2.2	Field Measurement on Ground and Surface Water Samples
2.6	Sampling Monitoring Wells with a Peristaltic Pump
2.8	Sampling for Volatile Organics

3.2. Preparation

3.2.1. Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Obtain appropriate permission for property access.

D. Assemble the equipment and supplies listed in Appendix 5.1. Ensure the proper operation of all sampling equipment.

E. Notify the analytical laboratory of sample types, the number of samples, and the approximate arrival date.

F. Contact the carrier that will transport samples to obtain information on regulations and specifications.

3.2.2. Documentation

A. Obtain a logbook from the QA officer.

B. Record results of the equipment check in the logbook.

C. Obtain a sufficient number of the appropriate ER Program data collection forms (see INDEX TO SOPs).

D. Consult the ER Program data administrator for a current list of information management codes, location IDs, and sample numbers used in the completion of data forms.

3.2.3. Field

A. Locate sampling sites along the surface water body and the appropriate decontamination area. Check decontamination zones and barricades to public access.

B. Locate a docking area for the boat, when appropriate.

C. Decontaminate all sampling equipment before taking the first sample and between sampling intervals (see SOP 1.6, General Equipment Decontamination, and the FSP or WP).

3.3. Operation

3.3.1. Surface Water Sampling with a Peristaltic Pump

A. Whenever a sample is collected, record all field measurements and chemistry determinations on the Surface Water Quality Sampling Record form. A copy of the form and instructions for completing the form are provided in SOP 2.2, Field Measurements on Ground and Surface Water Samples.

B. Refer to the general discussion about the operation of peristaltic pumps in SOP 2.1, Presample Purging of Wells, and SOP 2.6, Sampling Monitoring Wells with a Peristaltic Pump. This method will not be used to sample for volatile organic compounds in surface water.

C. To collect a sample, use the procedure outlined below.

1. Attach tubing to peristaltic pump.

2. Lower intake into surface water and begin water removal. Collect or dispose of water in an acceptable container, as specified in the FSP or WP.

3. Record amount of water removed and discharge rates. A calibrated bucket and stopwatch are commonly used for this step.

4. If possible, remove enough surface water to ensure that field chemistry parameters are stabilized. Refer to SOP 2.2, Field Measurements on Ground and Surface Water Samples.

5. Perform final field measurements (see SOP 2.2, Field Measurements on Ground and Surface Water Samples).

6. Sampling surface water tends to clog filters rapidly. Attach clean filter (if applicable) and prepare to collect raw water samples into empty bottles.

   NOTE: If filtering is required, run a few hundred milliliters (ml) of raw water through the filter before filling the sample bottles.

7. Fill the sample bottle, allowing the sample stream to flow gently down the side of the bottle with minimal entry turbulence. Do not allow dirt or dust to blow into the bottle or bottle cap. Shield bottles or bottle cap, as necessary, to eliminate entry of windblown material.

8. Rinse the inside of the bottle cap with raw water (using a Teflon-lined cap, if applicable). Screw the cap onto bottle tightly and shake the bottle if a preservative (for example, HNO_3, H_2SO_4, or HCl) has been added.

9. Immediately put the samples requiring refrigeration into an insulated cooler with Blue Ice or the equivalent. Put all other samples into a box and keep away from direct sunlight. For additional instructions, refer to SOP 5.4, Sample Containers and Preservation.

NOTE: Whenever a sample is collected, a custody record must be initiated on the Custody Transfer Record/Lab Work Request form and a Water Sample Identification Label affixed to the sample container. SOP 1.3, Sample Control and Documentation, contains copies of the form and label and instructions for completing the form and label.

3.3.2. Surface Water Sampling with a Transfer Device

A. Collect grab samples with a carboy container constructed of inert material like Teflon or stainless steel. Use the container to transfer liquid and liquid wastes from surface waters to a sample bottle. The purpose of this method is to obtain a representative sample of moving surface water for all parameters to be analyzed. This method prevents unnecessary contamination that would result if the outer surface of the sample bottle were directly immersed in the liquid. In general, field personnel must avoid using metal transfer devices for trace-metal analysis or plastic devices for sampling trace organics.

B. Direct sampling of surface water will be required for volatile organic compounds, rather than use of a carboy container.

C. Base the exact sampling location and number of grab samples obtained on the known tendency of waste to mix slowly in surface water (Korte and Kearl 1984). The site manager or field personnel should designate the number of samples to be gathered and the sampling locations.

D. To collect a sample, follow the procedure outlined below.

1. Ensure the carboy is cleaned according to EPA specifications.

2. To fill the carboy, make sure the sample stream flows gently down the sidewall.

3. Record the amount of water removed.

4. Perform field chemistry on raw water in the carboy (see SOP 2.2, Field Measurements on Ground and Surface Water Samples).

5. Perform steps 5-9 in Section 3.3.1.C (Surface Water Sampling with a Peristaltic Pump).

6. When collecting a sample from a frozen body of water, a carboy may not be effective because of its narrow mouth. In that event, use an appropriate receptacle and document its use in the logbook. The procedure for this kind of sampling is described below.

   a. Clean the receptacle according to EPA specifications.

   b. Grab or cut pieces of ice and fill the carboy or receptacle. If necessary, crush ice to fit.

c. Prepare the sample as described in the procedures for surface sampling. ~~with a transfer device~~.

7. If the raw water is collected directly into the collection bottle, add preservatives after the sample is collected. Rinse the bottle thoroughly and shake it if a preservative (for example, HNO_3, HCL, or H_2SO_4) has been added.

8. Consult the FSP or WP for specific instructions for filtering or preserving the sample in the field. Store the sample immediately according to the proper procedures (see SOP 1.4, Sample Containers and Preservation).

NOTE: Whenever a sample is collected, a custody record must be initiated on the Custody Transfer Record/Lab Work Request form and a Water Sample Identification Label affixed to the sample container. SOP 1.3, Sample Control and Documentation, contains copies of the form and label and instructions for completing the form and label.

3.4. Postoperation

3.4.1. Field

A. Ensure that all equipment is accounted for, decontaminated (see SOP 1.6, General Equipment Decontamination), and ready for shipment.

B. Place a permanent reference (sampling point) marker (for example, a wooden or metal stake with flagging that includes the location and site code) as close to the sampling location as possible.

3.4.2. Documentation

A. Record cleanup procedures and any uncompleted work (like site restoration or long-term monitoring) in the logbook.

B. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

C. Review data collection forms for completeness.

3.4.3. Office

A. Deliver original forms and logbooks to the site manager for technical review. He/she will review, sign forms, and transmit to the document control officer (copies to the files) for eventual delivery to the Department of Energy.

B. Inventory equipment and supplies. Repair or replace all broken or damaged equipment. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

C. Contact the analytical laboratory to ensure that samples arrived safely and instructions for sample analyses are clearly understood.

4. SOURCES

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5. APPENDIX

5.1. Equipment and Supplies Checklist

APPENDIX 5.1

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Peristaltic pump

_____ Filtration unit

_____ Teflon bore and fittings

_____ Clean filters and prefilters

_____ Transfer device for grab samples

_____ 2 or 5-gallon carboy container

_____ Wooden stakes

_____ Survey flagging

_____ Plastic or Teflon bucket

_____ Stopwatch

_____ Sample containers and preservatives

_____ Blue Ice or equivalent

_____ Any additional supplies listed in associated procedures, as needed

STANDARD OPERATING PROCEDURE 4.1

SOIL BORING

1. PURPOSE

To ensure acceptable, consistent soil-boring procedures for all pertinent aspects of hazardous waste investigations.

2. DISCUSSION

The Field Sampling Plan (FSP) or Work Plan (WP) provides information about the scope and details of the soil-boring operation, including specifications for drilling techniques. Refer to the FSP or WP for the type, number, and depth intervals at which samples will be collected. Collection and measurement of samples and the documentation of data will be performed as specified in the associated procedures.

To the extent possible, the boring process should not alter the medium that is being investigated. Various methods can be used for soil boring. These include, but are not limited to, hollow-stem augering, cable tool, mud rotary, and air rotary. For most investigations, dry hollow-stem augering or cable tool are the preferred drilling methods. Additional information concerning boring techniques is contained in the SOP 4.2, Rock Boring.

3. PROCEDURES

3.1. Associated Procedures

Before every operation, a review of the SOPs 1.1-1.10 is necessary. These SOPs contain information on the performance of field activities. They should be consulted for specific information about equipment and supplies; sample collection, preservation, packaging, and shipping; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.3	Sample Control and Documentation
1.4	Sample Containers and Preservation
1.5	Guide to Handling, Packaging, and Shipping of Samples
1.6	General Equipment Decontamination
4.2	Rock Boring
4.3	Monitoring Well Installation

5.1	Soil and Rock Borehole Logging and Sampling
6.1	Health and Safety Monitoring of Combustible Gas Levels
6.2	Health and Safety Monitoring of Organic Vapors with a Photoionization Detector
6.3	Health and Safety Monitoring of Organic Vapors with a Flame Ionization Detector

3.2. Preparation

3.2.1. Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Obtain appropriate permission for property access.

D. Assemble the equipment and supplies listed in Appendix 5.1. Ensure the proper operation of all sampling equipment.

E. If samples are to be collected for analyses, notify the laboratory of sample types, the number of samples, and the approximate arrival date. In addition, contact the carrier that will transport samples to obtain information on regulations and specifications.

F. Ensure that boring or well-drilling permits required by state or local authorities have been obtained, as well as procedures for compliance with state or local regulations regarding the submission of well logs and samples.

G. Research the site hydrogeology to estimate the key parameters (for example, anticipate the aquifer depth and thickness and types of contaminants).

3.2.2. Documentation

A. Obtain a logbook from the QA officer.

B. Record results of the equipment check in the logbook.

C. Obtain a sufficient number of the appropriate ER Program data collection forms (see INDEX TO SOPs).

D. Consult the ER Program data administrator for a current list of information management codes, location IDs, and sample numbers used in the completion of data forms.

3.2.3. Field

A. Before drilling, decontaminate all downhole drilling and sampling equipment, as well as the back of the drilling rig (as described in SOP 1.6, General Equipment Decontamination).

B. Clear the work site of all brush and minor obstructions.

C. Stake the location of utilities and the proposed boring areas.

NOTE: Ensure that proposed boring areas are not traversed by utility transmission ways.

D. If drilling fluid or grout is required, the source(s) of any water to be used in grouting and well installation must be approved by the site manager before field operations.

3.3. Operation

A. Decontaminate sampling equipment between sampling events (as described in SOP 1.6, General Equipment Decontamination).

B. Inventory all samples as specified in SOP 1.3, Sample Control and Documentation.

C. Handle all samples as specified in SOP 1.5, Guide to Handling, Packaging, and Shipping of Samples.

D. Soil sampling and borehole logging must conform to SOP 5.1, Soil and Rock Borehole Logging and Sampling.

E. If field screening of samples for organic vapors is required, conduct the survey as described in SOP 6.2, Health and Safety Monitoring of Organic Vapors with a Photoionization Detector, and SOP 6.3, Health and Safety Monitoring of Organic Vapors with a Flame Ionization Detector.

F. If the boring is to be completed as a monitoring well, review SOP 4.3, Monitoring Well Installation, for pertinent information.

G. Ensure that the back of the drilling rig is free of any leaking hydraulic lines. Do not grease surfaces to the point that excess grease could be dislodged during drilling.

H. Conduct work in compliance with all Occupational Safety and Health Administration (OSHA) regulations regarding drilling safety and the detection of underground utilities. If required by safety considerations, staked boring should be moved.

I. For each operating drill rig, designate an individual to be responsible for logging the samples, preparing the boring logs and well sketches, and the well installation of that rig. More than one rig at a time may be supervised by a qualified individual.

J. Log the samples, prepare the boring logs and well sketches, and supervise the well installation.

NOTE: Whenever a sample is collected, a custody record must be initiated on the Custody Transfer Record/Lab Work Request form and a Soil Sample Identification Label affixed to the sample container. SOP 1.3, Sample Control and Documentation, contains copies of the form and label and instructions for completing the form and label.

K. Do not use dyes, tracers, or other substances or introduce them into borings, wells, lysimeters, grout, backfill, groundwater, or surface water unless specifically required by the contract.

L. If drilling fluids are required, maintain portable recirculation tanks for accurate records of fluid loss.

M. If specified, air systems include an air line oil filter that requires frequent replacement to remove all oil residue from the air compressor. Describe the air system manufacturer's name, model number, air pressures used, the frequency of changing the oil filter, and the evaluation of air line filtering in the logbook.

N. Follow the sampling interval and type of sampling equipment specified in the FSP or WP unless directed by the site manager.

O. Record all field measurements and comments on the Borehole Log (Soil) form (see SOP 5.1). Complete all lines on the forms. Use the letter designation NA for not applicable, ND for not done, or UNK for unknown when applicable. If some steps or procedures are not performed as described, state the reason (as is practicable) on the Borehole Log (Soil) form or submit it as an attachment.

P. Maintain a daily detailed driller's report during drilling. Resolve all disputes concerning drilling time, standby time, and work progress at the end of each day. The driller's representative should sign and initial the daily report to indicate concurrence. The report should provide a complete description of all formations encountered, the number of feet drilled, the number of hours on the job, any shutdowns because of breakdown, the feet of casing set, and other pertinent data.

Q. Collect, containerize, and store all excess soil cuttings, waste materials, and decontamination solutions for proper disposal as specified in the FSP or WP.

R. If temporary casing is specified in the FSP or WP, advance the casing to the specified depth. Remove all loose material within the casing before sampling. Advance the casing according to project requirements. Use the type of casing-advance technique that is specified in the FSP or WP. The casing will be of the flush-joint or flush-couple type and of sufficient size to allow for soil sampling, coring, or well installation. All casing sections should be straight and free of any obstructions. If hollow-stem augers are used, equip the bit with a plug device that can be removed at the required sampling depth.

S. The abandonment of any boring should follow any appropriate state regulations and be approved before any casing removal or sealing/backfilling. Seal borings by grouting from the bottom of the boring or well to the ground surface. This can be accomplished by placing a tremie pipe at the bottom of the boring and pumping grout through this pipe until undiluted grout flows from the boring at ground surface. The grout or tremie pipe may be gradually withdrawn, as long as the end of the pipe is at least 10 ft below the grout surface. The grout should consist of a neat cement with 4 lbs of commercial bentonite and approximately 7.5 gallons of water added for every 94-lb bag of cement. After the grout has set (about 72 hrs), the contractor should check the abandoned site for grout settlement. Fill any depression in the grout with the grout mix described above. Methods other than those described here may be implemented as dictated by appropriate state or local agencies. Do not use any grout additives except the 4% bentonite.

T. Safety equipment should be specified by the site health and safety officer. In all cases, the minimum physical protection worn by drilling personnel should include a hard hat, safety glasses, gloves, steel-toed leather boots, and hearing protection.

3.4. Postoperation

3.4.1. Field

A. Ensure that all equipment is accounted for, decontaminated (see SOP 1.6, General Equipment Decontamination), and ready for shipment.

B. Restore the site to presampling conditions and fill open sampling holes as specified in the FSP or WP.

C. Make sure all borehole locations are properly staked and the location ID is readily visible on the location stake.

D. Have the driller approve and initial the report of progress at the end of each operating day.

3.4.2. Documentation

A. Record cleanup and hole abandonment procedures and any uncompleted work (like site restoration or long-term monitoring) in the logbook.

B. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

C. Review data collection forms for completeness.

D. If required by local or state law, file well-installation reports.

E. After a land survey, verify that the drilling permit describes the site location accurately. If necessary, modify and resubmit the permit.

3.4.3. Office

A. Deliver original forms and logbooks to the site manager for technical review. He/she will review, sign forms, and transmit to the document control officer (copies to the files) for eventual delivery to the Department of Energy.

B. Inventory equipment and supplies. Repair or replace all broken or damaged equipment. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

C. If samples have been collected for analysis, contact the laboratory to ensure that samples arrived safely and instructions for sample analyses are clearly understood.

D. If drilling wastes were stored, determine the appropriate disposal (based on laboratory analysis) of the soils from the borings.

4. SOURCES

Barcelona, M. J., J. P. Gibb, J. A. Helfrich, and E. E. Garske. 1985. "Practical Guide to Groundwater Sampling." U.S. Environmental Protection Agency report EPA/600/2-85/104. Washington, D.C.: U.S. Government Printing Office.

DOE. 1985. "Field Technical Representaive Manual." 2d ed. U.S. Department of Energy, Uranium Mill Tailings Remedial Action Project Office, Albuquerque Operations Office document, June 1985. Albuquerque, New Mexico.

5. APPENDIX

5.1. Equipment and Supplies Checklist

APPENDIX 5.1

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Sample containers

_____ Appropriate clothing

_____ Sprayer with clean water for dust control

_____ Any applicable licenses and permits

_____ Camera and film

_____ Measuring tape

_____ Plastic sheets

_____ Any additional supplies listed in associated procedures, as needed

STANDARD OPERATING PROCEDURE 4.2

ROCK BORING

1. PURPOSE

To ensure acceptable, consistent rock boring procedures for all pertinent aspects of hazardous waste investigations.

2. DISCUSSION

The Field Sampling Plan (FSP) or Work Plan (WP) provides information about the scope and details of the rock-boring operation, including specifications on drilling techniques to be used at a site. Refer to the FSP or WP for the type, number, and depth intervals at which samples will be collected. Collection and measurement of samples and the documentation of data will be performed as specified in the associated procedures.

Rock boring provides samples for logging, geotechnical analysis, and monitoring well installation. The boring process should not (to the extent practicable) alter the medium that is being investigated. Rock-boring techniques include, but are not limited to, cable tool, rotary, reverse-circulation rotary, and coring.

3. PROCEDURES

3.1. Associated Procedures

Before every operation, a review of the SOPs 1.1-1.10 is necessary. These SOPs contain information on the performance of field activities. They should be consulted for specific information about equipment and supplies; sample collection, preservation, packaging, and shipping; decontamination procedures; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.3	Sample Control and Documentation
1.4	Sample Containers and Preservation
1.5	Guide to Handling, Packaging, and Shipping of Samples
1.6	General Equipment Decontamination
4.3	Monitoring Well Installation
5.1	Soil and Rock Borehole Logging and Sampling
6.1	Health and Safety Monitoring of Combustible Gas Levels

6.2 Health and Safety Monitoring of Organic Vapors with a Photoionization Detector

6.3 Health and Safety Monitoring of Organic Vapors with a Flame Ionization Detector

3.2. Preparation

3.2.1. Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Obtain appropriate permission for property access.

D. Assemble the equipment and supplies listed in Appendix 5.1. Ensure the proper operation of all sampling equipment.

E. If samples are to be collected for analyses, notify the laboratory of sample types, the number of samples, and the approximate arrival date. In addition, contact the carrier that will transport samples to obtain information on regulations and specifications.

F. Obtain all boring or well-drilling permits required by state or local authorities and informtion required for compliance with state or local regulations regarding the submission of well logs and samples. Obtain utility maps for the site and coordinate boring locations with utility companies.

G. Research the site hydrogeology to estimate the key parameters (for example, anticipated aquifer depth and thickness, types of contaminants, and grain-size distribution).

3.2.2. Documentation

A. Obtain a logbook from the QA officer.

B. Record results of the equipment check in the logbook.

C. Obtain a sufficient number of the appropriate ER Program data collection forms (see INDEX TO SOPs).

D. Consult the ER Program data administrator for a current list of information management codes, location IDs, and sample numbers used in the completion of data forms.

3.2.3. Field

A. Before drilling, decontaminate all downhole drilling and sampling equipment, as well as the back of the drilling rig (as described in SOP 1.6, General Equipment Decontamination).

B. Clear the working areas of brush and minor obstructions.

C. Stake the location of utilities and locations for proposed borings.

NOTE: Ensure that proposed boring areas are not traversed by utility transmission ways.

D. If drilling fluid or grout is required, the source(s) of any water to be used in grouting and well installation must be approved by the site manager before field operations.

3.3. Operation

A. Decontaminate sampling equipment between sampling events as described in the SOP 1.6, General Equipment Decontamination.

B. Handle all samples as described in the SOP 1.5, Guide to Handling, Packaging, and Shipping of Samples.

C. Rock sampling and borehole logging must conform to the specifications defined in the SOP 5.1, Soil and Rock Borehole Logging and Sampling.

D. If field screening of samples for organic vapors is required, conduct it as described in the SOP 6.2, Health and Safety Monitoring of Organic Vapors with a Photoionization Detector, and SOP 6.3, Health and Safety Monitoring of Organic Vapors with a Flame Ionization Detector.

E. If the boring is to be completed as a monitoring well, review the SOP 4.3, Monitoring Well Installation, for pertinent information.

F. The back of the drilling rig must be free of any leaking hydraulic lines. Do not grease surfaces to the point that excess grease could be dislodged during drilling.

G. Conduct work in compliance with Occupational Safety and Health Administration (OSHA) regulations regarding drilling safety and the detection of underground utilities. If required by safety considerations, relocate the borings.

H. For each operating drill rig, designate an individual to be responsible for logging samples, preparing boring logs and well sketches, and the well installation of that rig. More than one rig at a time may be supervised by a qualified individual.

I. Do not use dyes, tracers, or other substances or introduce them into borings, wells, soil moisture (water) samplers, grout, backfill, groundwater, or surface water unless specifically required by the contract.

J. If drilling fluids are required, do not use dug (lined) sumps; portable recirculation tanks are required.

K. Log the samples, prepare the boring logs and well sketches, and supervise the well installation.

NOTE: Whenever a sample is collected, a custody record must be initiated on the Custody Transfer Record/Lab Work Request form and a Soil Sample Identification Label affixed to the sample container. SOP 1.3, Sample Control and Documentation, contains copies of the form and label and instructions for completing the form and label.

L. If specified, air systems should include an air line oil filter that requires frequent replacement to remove all oil residue from the air compressor. The use of any air system should be fully described in the driller's log and include an equipment description, the manufacturer's name, model name, air pressures used, the frequency of changing the oil filter, and an evaluation of the air line filtering.

M. Follow the sampling interval and type of sampling equipment specified in the FSP or WP unless directed by the site manager.

N. Record all field measurements and comments on the Borehole Log (Rock) form (see SOP 5.1). Complete all lines on the forms. Use the letter designation NA for not applicable, ND for not done, or UNK for unknown when applicable. If some steps or procedures were not performed as described, state the reason (as practicable) on the Borehole Log (Rock) or submit it as an attachment.

O. During drilling, maintain a daily detailed driller's report and have a qualified person submit it if requested by the site manager. The report will provide a complete description of all formations encountered, the number of feet drilled, the number of hours on the job, any shutdowns because of breakdown, the feet of casing set, and other pertinent data.

P. If rock coring is specified, follow the procedure described below.

1. Casing is required for the full depth of the overburden in borings when rock is cored.

2. Advance the casing according to specifications in the FSP or WP. The casing should be of the flush-joint or flush-couple type and of sufficient size to allow for soil sampling, coring, and well installation. All casing sections should be straight and free of any obstructions. If hollow-stem augers are to be used, equip the bit with a plug device that can be removed at the required sampling depth.

3. Use drill rods for drilling rock that are NW in size to minimize vibration and chattering. Rock core size should be NX, NQ (wire line), or a size specified in the FSP or WP.

4. Use core barrels of the improved double-tube varieties (like the Christensen Series C or D models or the equivalent) that are equipped with a split inner tube.

5. Use five-ft barrels at the discretion of the site manager.

6. Make every effort to use clear water as a drilling fluid. In the event that this is impractical, recirculated water may be used at the discretion of the site manager, as long as a settling tank and filtering system are provided.

7. To minimize core losses in soft, erodable rock, the measures listed below should be required by the supervising field technician.

   a. Restrict drilling to short runs of 2 to 3 ft.

   b. Keep drilling water pressure low (under 150 psi).

c. Keep feed pressure under 100 psi.

8. The supervising geologist should not permit a full coring run to be drilled if he/she suspects that core was left in the hole on the previous run. If this is believed to have occurred, shorten the next coring run by the length of core believed to have been left in the hole. This is necessary to prevent blocking the core barrel and grinding the core.

9. Upon removal of the core barrel from the drill hole, the driller should remove and open the split inner tube and deliver it to the field technician. If necessary to facilitate accurate logging, wipe off the core while it rests in the inner half.

10. Store rock cores in cardboard or wood core boxes so that their relative position by depth is preserved. Note intervals of lost core in the core sequence. Clearly mark the top of the core sequence. Mark boxes with the boring number, cored interval, and box number in cases of multiple boxes. The weight of each fully loaded box should not exceed 75 lbs. No data should appear on or within the box that is not specified on the Boring Log (Rock).

11. Each box should contain core from only one borehole. If spacers are required to separate intervals of core runs, use wooden blocks that have been clearly marked with the missing interval of core.

Q. If tri-cone rotary drilling is used, record the items below.

1. Rate of drilling

2. Percent of drilling fluid recovery

3. Changes in drilling fluid and water color

4. Lithologic description

Determine lithologies by using a kitchen screen to separate rock particles from the return waters. Describe the cuttings according to SOP 5.1, Soil and Rock Borehole Logging and Sampling. Place the cuttings on plastic sheets for easier tracking of depth and examination.

R. In the abandonment of any boring, follow any appropriate state regulations and obtain approval before any casing removal or sealing/backfilling. Seal borings by grouting from the bottom of the boring or well to the ground surface. This will be accomplished by placing a tremie pipe at the bottom of the boring and pumping grout through this pipe until undiluted grout flows from the boring at the ground surface. The grout or tremie pipe may be gradually withdrawn, as long as the end of the pipe is at least 10 ft below the grout surface. The grout should consist of a neat cement with 4 lbs of commercial bentonite and approximately 7.5 gallons of water added for every 94-lb bag of cement. After the grout has set (about 72 hrs), the contractor should check the abandoned site for grout settlement. Fill any depression in the grout with the grout mix described above. Methods other than those described here may be implemented as dictated by appropriate state or local agencies. Do not use any grout additives except the 4% bentonite.

S. Safety equipment should be specified by the site and safety officer. In all cases, the minimum physical protection worn by drilling personnel should include a hard hat, safety glasses, gloves, steel-toed boots, and hearing protection.

3.4 . Postoperation

3.4.1. Field

A. Ensure that all equipment is accounted for, decontaminated (see SOP 1.6, General Equipment Decontamination), and ready for shipment.

B. Restore the site to presampling conditions as specified in the FSP or WP.

C. Make sure all borehole locations are properly staked and the location ID is readily visible on the location stake or protective casing.

D. Have the driller approve and initial the report of progress at the end of each operating day.

E. Prepare samples and transport according to SOP 1.3, Sample Control and Documentation; SOP 1.4, Sample Containers and Preservation; and SOP 1.5, Guide to Handling, Packaging, and Shipping of Samples.

3.4.2. Documentation

A. Record cleanup and hole abandonment procedures and any uncompleted work (like site restoration or long-term monitoring) in the logbook.

B. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

C. Review data collection forms for completeness.

D. If required by local or state law, file well-installation reports.

E. After a land survey, verify that the drilling permit describes the site location accurately. If necessary, modify and resubmit the permit.

3.4.3. Office

A. Deliver original forms and logbooks to the site manager for technical review. He/she will review, sign forms, and transmit to the document control officer (copies to the files) for eventual delivery to the Department of Energy.

B. Inventory equipment and supplies. Repair or replace all broken or damaged equipment. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

C. If samples have been collected for analysis, contact the laboratory to ensure that samples arrived safely and instructions for sample analyses are clearly understood.

D. If drilling wastes were stored, determine the appropriate disposal (based on laboratory analysis) of the soils from the borings.

4. SOURCES

Barcelona, M. J., J. P. Gibb, J. A. Helfrich, and E. E. Garske. 1985. "Practical Guide to Groundwater Sampling." U.S. Environmental Protection Agency report EPA/600/2-85/104. Washington, D.C.: U.S. Government Printing Office.

DOE. 1985. "Field Technical Representaive Manual." 2d ed. U.S. Department of Energy, Uranium Mill Tailings Remedial Action Project Office, Albuquerque Operations Office document, June 1985. Albuquerque, New Mexico.

5. APPENDIX

5.1. Equipment and Supplies Checklist

APPENDIX 5.1.

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Sprayer with clean (potable) water for dust control

_____ Core boxes

_____ Wood block or lath

_____ Measuring tape (tenths)

_____ Large, black permanent marker

_____ Strapping tape

_____ Appropriate clothing

_____ Sprayer with clean water for dust control

_____ Any applicable licenses and permits

_____ Camera and film

_____ Sample containers

_____ Plastic sheets

_____ Any additional supplies listed in associated procedures, as needed

STANDARD OPERATING PROCEDURE 4.6

TEST PIT LOGGING AND SAMPLING

1. PURPOSE

To describe the physical nature of consolidated or unconsolidated subsurface earthen materials encountered during the excavation of a pit or trench.

2. DISCUSSION

The Field Sampling Plan (FSP) or Work Plan (WP) includes specific details about the location and procedures, equipment, and other details related to the trenching task. Refer to the FSP or WP for the type and number of samples to be collected in each trench. Collection and measurement of samples and the documentation of data will be performed as described in the associated procedures.

Before beginning the field test pit excavation program, office and field management personnel should obtain information about geologic conditions expected at the site. The three basic methods of sampling a test pit are described below.

1. Strip sampling a pit involves the vertical cutting of a representative sample from each distinctive lithologic unit of the test pit wall.

2. Undisturbed block sampling can be performed within a test pit. Normally, the block sample size is approximately 1 ft^3. Microcrystalline wax sealing and proper storage and shipment are essential to avoid drying and breaking block samples.

3. When the test pit is inaccessible or groundwater is infiltrating, the excavation consists of selectively digging in certain lithologic units with a backhoe.

The investigation team should be composed of technical specialists with backgrounds in the fields of geology, engineering geology, or geotechnical engineering. The locations and dimensions of the trench should be identified in the FSP or WP. Mapping the trench walls and sampling should be conducted by the members of the investigation team. The construction subcontractor should be responsible for supplying excavation equipment, trained operators, materials for shoring the trench walls, and the preparation of the walls for mapping.

3. PROCEDURES

3.1. Associated Procedures

Before every operation, a review of the SOPs 1.1-1.10 is necessary. These SOPs contain information on the performance of field activities. They should be consulted for specific information on equipment and supplies; sample collection, preservation, packaging, and shipping; decontamination procedures; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.3	Sample Control and Documentation
1.4	Sample Containers and Preservation
1.5	Guide to Handling, Packaging, and Shipping of Samples
1.6	General Equipment Decontamination
5.2	Soil Sampling with a Spade and Scoop

3.2. Preparation

3.2.1. Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Obtain appropriate permission for property access.

D. Assemble the equipment and supplies listed in Appendix 5.1. Ensure the proper operation of all sampling equipment.

E. Notify the analytical laboratory of sample types, the number of samples, and the approximate arrival date.

F. Contact the carrier that will transport samples to obtain information on regulations and specifications.

3.2.2. Documentation

A. Obtain a logbook from the QA officer.

B. Record results of the equipment check in the logbook.

C. Obtain a sufficient number of the appropriate ER Program data collection forms (see INDEX TO SOPs).

D. Consult the ER Program data administrator for a current list of information management codes, location IDs, and sample numbers used in the completion of data forms.

3.2.3. Field

Decontaminate all sampling equipment before taking the first sample and between sampling intervals.

3.3. Operation

A. Excavate trenches with a backhoe that uses a 2- to 3-ft-wide bucket capable of efficient excavation to a depth of approximately 15 ft.

B. At those sites requiring restoration or containing contaminated materials, remove and stockpile the topsoil or contaminated material before trench excavation. Make shallow cuts of 1- to 2-ft depths and stockpile the material on the downwind or downslope side of the trench. Maintain ample space (a minimum of 2 ft) between the stockpiled material and the trench to maneuver excavation equipment.

C. Maintenance of stable walls, particularly in deep trenches, is best accomplished with straight-line sections. Because shoring in corners or curve areas is potentially ineffective, avoid the excavation of nonlinear trenches.

D. Make trench walls vertical, uniform in width, and as smooth as practical to facilitate the efficient use of portable shoring braces. If caving of the trench walls occurs during excavation, widen the trench and terrace it at the top to allow placement of the shoring in the lower section of the trench. Mitigate severe caving of the trench walls by using multiple terraces that incrementally increase the trench width toward the ground surface. Instances may occur in which the only way to examine vertical walls in problem areas is to map the walls in small increments of depths and lengths, stopping the trenching as required and using the backhoe bucket as safety equipment. After each trench section is adequately excavated, the backhoe operator should scrape the trench bottom to remove loose debris and obstructions. Trench investigators should be allowed unrestricted movement to the degree practically achievable, particularly with respect to ladder access.

F. Place excavated subsoil as a windbreak on the upwind or upgradient side of the trench. Maintain sufficient space (a minimum of 2 ft) between the material and the trench edge to ensure that it will not fall back into the trench or impede the advancement of the backhoe.

G. Depending on the strength of the surficial deposits being trenched, mapping may occur concurrently with the advancement of the trench. The site geologist and an assistant should conduct the mapping of the trench wall at a safe distance behind the backhoe, lessening the risk of exposure to caving induced by backhoe vibrations. One team member should remain at the surface to direct the excavation activity and assist the trench wall mapping team. The topside member can ensure that the backhoe operator excavates the trench as specified by the site geologist, who should advise the operator of trench conditions requiring immediate attention and direct the operator to trim and terrace trench walls when appropriate. The safety of the trench mappers is the paramount responsibility of the topside team member, who should be constantly alert for indications of potential wall caving (like tension cracks). All persons entering any portion of the trench should wear personal equipment for protection of the head and eyes.

H. For trenches in excess of 5 ft deep or shallower trenches that have been examined and require structural reinforcement, the most efficient way to strengthen a trench wall is with hydraulically operated portable shoring. Shore up only the section of trench being mapped. As safety is the primary consideration, the

placement of shoring relative to the mapping activity in the trench is of critical importance. No person may enter any portion of the trench that has not been shored. Alternately, a specially constructed trench box can be used to shore the walls to facilitate sampling.

I. Project managers should document the required safety protocol related to Occupational Safety and Health Administration (OSHA) requirements according to the OSHA publication 2226 (Excavating and Trenching Operations, U.S. Department of Labor) and 29 CFR 1926, Construction and Industry Standards. Trenches deeper than 5 ft and longer than 8 ft must be shored. For trenches approximately 10 ft deep or less, use a group of at least 3 portable shoring braces. Space them roughly 8 ft apart to provide the field personnel at the site with a working space. For depths greater than 10 ft, use at least 4 shoring braces. When 2 or more people will be in the trench, use additional shoring to expand the working space. The shoring braces should not be separated by more than 8 ft. Shoring should be placed in true horizontal position, spaced vertically, and secured to prevent sliding, falling, or kickouts.

J. Provide access to the trench with ladder(s) placed next to the shoring. The ladder(s) should be aluminum and capable of extending from the trench bottom to at least 3 ft above the ground surface. Provide a ladder for each person working in the trench.

K. The topside team member should install shoring by placing a 2-inch by 2-inch board across the width of the trench for support. The topside person should relocate the portable shoring and ladder(s) as mapping operations advance (or as warranted by unstable trench wall conditions).

L. Inclement weather accompanied by intense or steady precipitation is sufficient cause to temporarily suspend fault-trenching operations. Careful examination of the trench wall, bottom, and vicinity conditions should precede the resumption of the trenching operations.

M. When appropriate, restrict access to the trench area(s). Use barricades with flashing lights to preclude vehicle and pedestrian traffic and delineate a perimeter that contains the trench, windbreak, and topsoil/contaminated material stockpiles. If the trench area is accessible to livestock, install a single-wire electric fence or other type of temporary fencing to prevent entry into the site area.

N. Each trench or trench segment is usually maintained in an accessible condition until all trenches have been mapped and data collected to the satisfaction of the site geologist. No portion of any trench should be reentered unless that segment has been inspected for stability and adequately reinforced with portable shoring.

O. Plot physical attributes of units that are distinct because of lithology, texture, or color as feet and inches, plus or minus the baseline elevation and station position. In addition to bedding planes and lithologic interfaces, geologic features (like cobble strings) may aid in following stratum continuity, particularly if individual units are difficult to discern. Assessment of stratigraphy in a deposit lacking distinct strata or containing similar geologic units may be aided by the use of a 5-ft-square aluminum frame marked with string in a 1-ft grid pattern. To demonstrate the continuity of surficial deposits, detailing the geologic features (like cobble strings or lenses) with the grid frame across the entire length of the

trench may be necessary. Other features like gouge or breccia zones, zones of alteration, and fracture systems should be mapped, as well as distinct reference features like large boulders or organic debris.

P. Visual Description. Description of the soil will be according to the ASTM Designation D 2488-84, Standard Recommended Practice for Description of Soils (Visual-Manual Procedure).

Q. The technical specialist assisting the mapper in the trench should prepare the trench wall ahead of the mapper using a trowel and large paint brushes. Physical support of the grid frame when mapping the middle and upper reaches of a trench wall will be the responsibility of this person.

R. When there appears to be evidence of structural deformation or where there is doubt of the lateral continuity of a deposit or stratum, take at least two photographs of the trench wall. The first should record the unaltered appearance of the wall. The second should record the same location after it has been divided into a grid reference using nails and string line and highlighted with orange, white, or black spray paint. Take flash photos if ambient light is insufficient for a clear, bright exposure.

S. In the event that faulting is indicated by a bedding disturbance, attempt to date the youngest offset bed or the oldest bed that is not offset by acceptable dating methods. After delineating the trend of the fault trace, confirm the orientation of the fault by excavating a second trench across the same fault.

T. Collect samples from the trench wall or floor according to SOP 5.2, Soil Sampling with a Spade and Scoop.

NOTE: Whenever a sample is collected, a custody record must be initiated on the Custody Transfer Record/Lab Work Request form and a Soil Sample Identification Label affixed to the sample container. SOP 1.3, Sample Control and Documentation, contains copies of the form and label and instructions for completing the form and label.

U. Backfill the trenches with the backhoe used to perform the excavation. Alternatively, a rubber tire front-end loader or backhoe with a front-end loader bucket may be more efficient. Use material excavated and functioning as a windbreak for backfill. It is important at most sites that the restored site is as close to the original surface contour as practical. Compact the backfill by wheel rolling with the backhoe or loader to accomplish this objective.

V. In pasture land or any place where restoration is required and vegetation cover is important, replace the topsoil that was excavated and stockpiled downgradient from the trench at the end of the restoration process. After the surface is wheel rolled and dressed down, restore the area as specified by the access agreement.

3.4. Postoperation

3.4.1. Field

A. Fill the test pit to its original level.

B. Ensure that all equipment is accounted for, decontaminated (see SOP 1.6, General Equipment Decontamination), and ready for shipment.

C. Restore the site to presampling conditions as specified in the FSP or WP.

D. Make sure the test pits are properly staked and the location ID is readily visible on the location stake.

E. Prepare samples for transport according to SOP 1.3, Sample Control and Documentation; SOP 1.4, Sample Containers and Preservation; and SOP 1.5, Guide to Handling, Packaging, and Shipping of Samples.

3.4.2. Documentation

A. Record cleanup procedures and any uncompleted work (like site restoration or long-term monitoring) in the logbook.

B. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

C. Review data collection forms for completeness.

3.4.3. Office

A. Deliver original forms and logbooks to the site manager for technical review. He/she will review, sign forms, and transmit to the document control officer (copies to the files) for eventual delivery to the Department of Energy.

B. Inventory equipment and supplies. Repair or replace all broken or damaged equipment. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

C. Contact the analytical laboratory to ensure that samples arrived safely and instructions for sample analyses are clearly understood.

4. SOURCE

ASTM. 1986. Standard Recommended Practice for Description of Soils (Visual-Manual Procedure)," 411-25, ASTM D:2488-84. American Society of Testing Methods, Philadelphia, Pennsylvania.

5. APPENDIXES

5.1. Equipment and Supplies Checklist

5.2. Test Pit Log Form

5.3. Data Form Completion

5.4. Checklist to Describe Fine-Grained Soils

5.5. Checklist to Describe Coarse-Grained Soils

APPENDIX 5.1

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Flat-nosed shovel and scoops

_____ Sample containers and preservatives

_____ Hand lens

_____ Ladders

_____ Portable shoring braces (minimum of 3)

_____ Spray paint

_____ Trowel

_____ Large paint brushes

_____ Barricades or temporary fencing

_____ Camera and flash attachment

APPENDIX 5.2

TEST PIT LOG

TEST PIT LOG

PAGE 1 OF _____

FACILITY CODE _____ **EXCAVATOR CODE** _____

LOCATION ID _____ **EXCAVATION DATE** _____

COORDINATES (FT): **DEPTH (FTFD)** _____

NORTH _____ **EAST** _____ CONSTRUCTION METHOD _____

GROUND ELEVATION (FT MSL) _____ **ACCEPTANCE CODE** _____

LOCATION TYPE TP

GROUNDWATER LEVELS		
DATE	TIME	DEPTH (FT)

LOCATION DESCRIPTION

LITHOLOGIC LOG

LOGGER CODE _____

DEPTH (FT)	SAMPLE INTERVAL	SAMPLE METHOD	SAMPLE ID	**USCS**	**VISUAL DESCRIPTION**

ACCEPTANCE CODES: A–ACCEPTABLE R–RECONNAISSANCE U–UNACCEPTABLE N–NOT DETERMINED

CONSTRUCTION METHODS:

D – DUG O – OTHER (SPECIFY)

T – TRENCHING

SAMPLE METHODS:

B – UNDISTURBED BLOCK

D – DISTURBED BULK

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS
TPO-004 (3/88)

FORM COMPLETED BY/DATE

TECHNICAL REVIEWER/DATE

TEST PIT LOG

PAGE ____ OF ____

FACILITY CODE ____________________

LOCATION ID ____________________ COMPLETION DATE ____________

LITHOLOGIC LOG

DEPTH (FT)	SAMPLE INTERVAL	SAMPLE METHOD	SAMPLE ID	USCS	VISUAL DESCRIPTION

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS
TPD-004 (3/88)

FORM COMPLETED BY/DATE ____________ TECHNICAL REVIEWER/DATE ____________

APPENDIX 5.3

DATA FORM COMPLETION INSTRUCTIONS

Use a pen with black ink that is not water soluble (not a felt-tip pen). Make an entry in each blank. Where there is no data entry, enter UNK for Unknown, NA for Not Applicable, or ND for Not Done. If any procedure was not performed as prescribed, give the reason for the change or omission on the form. To change an entry, draw a single line through it, add the correct information above it, and initial the change.

TEST PIT LOG

1. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

2. Location ID. Four-character code assigned sequentially to each test pit location where physical, chemical, biological, radiological, and other measurements are taken.

3. Coordinates (Ft): North/East. The coordinates refer to the horizontal location of the test pit. At the time of the field investigation, the exact coordinate position of the borehole will not be known. In this case, UNK must be within the two spaces on the form. This information will be provided when the survey data comes in after the test pit program has been completed.

4. Ground Elevation (Ft MSL). At the time of the field investigation, the exact ground elevation of the test pit will not be known. In this case, the exact ground elevation will be determined when the survey data comes in after the program has been completed.

5. Location Type. This line is for data processing personnel only, and no additional information needs to be given on this line.

6. Excavator Code. Three-character code identifying the company responsible for excavating a test pit.

7. Excavation Date. The date when the test pit was excavated in the format DD-MMM-YY (01-JAN-88).

8. Depth (FTFD). The total depth of the test pit in feet and tenths of feet.

9. Construction Method. The construction or excavation method used in the advancement of the test pit. A table of various construction methods is included at the bottom of each Test Pit Log form.

10. Acceptance Code. One-character code assigned by the installation manager.

11. Groundwater Levels. The date, time, and depth (in feet) of any water encountered during the excavation of a test pit should be recorded by the distance from the ground surface to the location where it is seeping from the sides of the excavated trench.

12. Location Description. A written description of the approximate test pit location in respect to some nearby permanent topographic or geographic location.

13. Logger Code. Three-character code identifying the company that employs the person filling out the Test Pit Log form.

14. Lithologic Log

    a. Depth (Ft). A numerical designation that generally depicts lithologic soil boundaries. Each space is usually designated as equal to 1.0 ft of depth below the ground surface. Depths will be recorded on the Test Pit Log in feet and tenths of feet.

CONVERSION TABLE

(INCHES TO TENTHS OF FEET)

Inches	Tenths of Feet
1	.08
2	.17
3	.25
4	.33
5	.42
6	.50
7	.58
8	.67
9	.75
10	.83
11	.92
12	1.00

    b. Sample Interval. A graphical representation depicting the interval from which the sample was collected.

    c. Sample Method. The method by which the samples will be obtained. A list of test pit sampling methods is included at the bottom of each Test Pit Log form.

    d. Sample ID. Four-digit number assigned to ensure that data collected retains uniqueness from other data collected at the same location ID.

e. USCS (The Unified Soil Classification System). A method of grouping unconsolidated earth materials according to their engineering properties. It is based on soil behavior, which is a reflection of the physical properties of the soil and its constituents. The system established 15 distinct soil groups with different engineering properties. Boundary classifications are provided for soils having characteristics of 2 groups. The 15 soil groups are divided into the categories of fine-grained and coarse-grained materials and are described in Appendixes 5.4 and 5.5, respectively.

f. Visual Description. The visual description of material being excavated.

APPENDIX 5.4

CHECKLIST TO DESCRIBE FINE-GRAINED SOILS

1. Typical Name
   Sandy Silt, Silt, Clayey Silt, Sandy Clay, Silty Clay, Clay, Organic Silt, and Organic Clay

2. Size Distribution
   Approximate percent of gravel, sand, and fines in fractions finer than 3 inches

3. Color
   Note presence of mottling and banding, as well as color of the soil.

4. Moisture Content
   Dry, Moist, Wet, and Saturated

5. Consistency
   Soft, Firm (medium), Stiff, Very Stiff, or Hard

6. Structure
   Stratified, Laminated (Varved), Fissured, Blocky, Lensed, and Homogeneous (nonstratified)

7. Cementation
   Weak or Strong

8. Local or Geologic Name

9. Group Symbol

<u>Soil Classification</u>

Group Symbol	Group Name
CL	Lean clay (low plasticity)
ML	Silt
OL	Organic clay or silt (lean)
CH	Fat clay (high plasticity)
MH	Elastic silt
OH	Organic clay or silt (Fat)
PT	Peat

APPENDIX 5.5

CHECKLIST TO DESCRIBE COARSE-GRAINED SOILS

1. Typical Name
   Sand, Clayey Sand, Silty Sand, Gravel, Clayey Gravel, Silty Gravel, Cobbles, and Boulders

2. Gradation
   Well Graded (uniformly graded) or Poorly Graded (gap graded)

3. Size Distribution.
   Approximate percent of gravel, sand, and fines in fractions finer than 3 inches

4. Grain Shape
   Angular, Subangular, Subrounded, and Rounded

5. Color

6. Moisture Content
   Dry, Moist, Wet, and Saturated

7. Structure
   Stratified, Lensed, and Nonstratified

8. Cementation
   Weak and Strong

9. Local or Geologic Name

10. Group Symbol

<u>Soil Classification</u>

Group Symbol	Group Name
GW	Well-graded gravel
GP	Poorly graded gravel
GM	Silty gravel
GC	Clayey gravel
SW	Well-graded gravel
SP	Poorly graded gravel
SM	Silty sand
SC	Clayey sand

STANDARD OPERATING PROCEDURE 5.1

SOIL AND ROCK BOREHOLE LOGGING AND SAMPLING

1. PURPOSE

To describe the physical nature of consolidated or unconsolidated subsurface earthen materials encountered during auger, rotary, or other drilling activities and collect samples of the earthen materials for further evaluation.

2. DISCUSSION

The Field Sampling Plan (FSP) or Work Plan (WP) contains specific details about the procedures and equipment for this SOP. Refer to the FSP or WP for the type of samples to be collected. Collection and measurement of samples and the documentation of data will be performed as described in the associated procedures.

Before field drilling begins, all management personnel should obtain information about expected geologic and hydrogeologic conditions at the site. The FSP or WP includes directions for sampling methods, location, and details for the site where the field personnel will work.

Most sites where soil and rock borehole logging and sampling are conducted include unconsolidated deposits of varying thickness over consolidated bedrock at depth. Two forms are used for describing the soil and rock encountered during drilling and sampling. The Borehole Log (Soil) form (Appendix 5.2) is used when describing unconsolidated alluvium, colluvium, and regolith. The Borehole Log (Rock) form (Appendix 5.3) is used when drilling or coring rock to describe the lithology and other physical characteristics of sedimentary, igneous, and metamorphic rocks.

3. PROCEDURES

3.1. Associated Procedures

Before every operation, a review of the SOPs 1.1-1.10 is necessary. These SOPs contain information on the performance of field activities. They should be consulted for specific information on equipment and supplies; sample collection, preservation, packaging, and shipping; decontamination procedures; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.3	Sample Control and Documentation
1.4	Sample Containers and Preservation
1.5	Guide to Handling, Packaging, and Shipping of Samples

1.6	General Equipment Decontamination
4.1	Soil Boring
4.2	Rock Boring

3.2. Preparation

3.2.1. Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Obtain appropriate permission for property access.

D. Assemble the equipment and supplies listed in Appendix 5.1. Ensure the proper operation of all sampling equipment.

E. Notify the analytical laboratory of sample types, the number of samples, and the approximate arrival date.

F. Contact the carrier that will transport samples to obtain information on regulations and specifications.

G. Contact the members of the survey crew and inform them of the approximate date that ground surveys will begin.

3.2.2. Documentation

A. Obtain a logbook from the QA officer.

B. Record results of the equipment check in the logbook.

C. Obtain a sufficient number of appropriate ER Program data collection forms (see INDEX TO SOPs).

D. Consult the ER Program data administrator for a current list of information management codes, location IDs, and sample numbers used in the completion of data forms.

3.2.3. Field

A. Decontaminate all sampling equipment before taking the first sample and between sampling intervals (see SOP 1.6, General Equipment Decontamination, and the FSP or WP).

B. Record all pertinent information (date, site, ID number, and location) in the logbook or the appropriate data form. Note field conditions, unusual circumstances, and weather conditions.

C. Permanently attach a soil sample identification label to each sample container.

3.3. Operation

3.3.1. Logging Samples

A. Whenever a sample is collected, complete a description of the sample using the Borehole Log (Soil) form (Appendix 5.2) or Borehole Log (Rock) form (Appendix 5.3). Fill out these forms according to the information described in Appendix 5.4. Include all scale drawings that further describe textural variations in the logbook. If cameras are permitted on the site, use photographs instead of drawings.

NOTE: Whenever a sample is collected, a custody record must be initiated on the Custody Transfer Record/Lab Work Request form and a Soil Sample Identification Label affixed to the sample container. SOP 1.3, Sample Control and Documentation, contains copies of the form and label and instructions for completing the form and label.

B. Gross physical properties of the sample can be described by unaided visual observation or inspection under a hand lens. Whenever feasible, prepare sample descriptions by observing fresh surfaces or cuttings.

C. Wash cuttings from mud rotary boring in clear water to remove drilling mud. Use a strainer with a large capacity during the washing process.

D. Split the core lengthwise, so that the interior can be inspected. Place core in cardboard box after inspection. Label top, bottom, and core interval on box. The core box will be lined with plastic to minimize cross-contamination. Mark unrecovered core intervals with blocks of wood. Use two permanent markers of different colors to draw parallel lines on the core so that its proper position in the core box can be maintained during and after future inspections.

E. Record all linear measurements in feet or tenths of feet, not inches.

F. Blow counts are to be counted for each 0.5-ft penetration of the Standard Penetration Test.

G. Use the standard Unified Soil Classification System (USCS) designations for identifying soil strata. Detailed descriptions of the different soil classifications are included in the U.S. Corps of Engineers Technical Memorandum, 1953.

H. Visual descriptions will be according to ASTM Designation D 2488-69 "Standard Recommended Practice for Description of Soils (Visual-Manual Procedure)."

I. Percent core recovery is a measure of coring efficiency and rock quality. If coring progresses gradually, a hard sandstone to a strongly fissile shale, the percent recovery will be affected. Once the core is shown to the field representative (preferably while still in the core barrel if it is a split-core barrel), measure the total length recovered. Sometimes it is possible to determine where core loss occurred in the run. The following clues are sometimes present: plugging off during drilling, intense fracturing in certain sections of core (possibly correlated with rough, high vibrations during drilling), and rolled and recut pieces of core.

J. The rock quality designation (RQD) is based on a modified core recovery procedure and indirectly on the number of fractures and the amount of softening

or alteration in the rock mass. Obtain the measure by summing up the total length of core recovered and counting only those relatively hard, sound pieces of core that are four inches in length or longer. See Stags and Zienkiewitz, 1968 for an example of an RQD computation. It is necessary to distinguish between natural fractures and those caused by drilling or recovery operations. Depending on the engineering or hydrogeologic requirements of the project, breaks induced along high anisotropic planes (like foliation or bedding) may be counted as natural fractures. Lengths that contain strong and recemented fractures should be measured in total.

RQD	QUALITY OF ROCK MASS
90-100	Excellent
75-90	Good
50-75	Fair
25-50	Poor
0-25	Very Poor

K. Percent Drilling Fluid Recovery--the volume of fluid losses and the interval over which they occur. For example, no fluid loss means that no fluid was lost except through spillage and filling the hole. Partial fluid loss means that a return was achieved, but the amount of return was significantly less than the amount being pumped. Complete water loss means that no fluid returned to the surface during the pumping operation. A combination of opinions from the field personnel and the driller on this matter will result in the best estimate. Make a crude, relatively effective estimate by placing a calibrated stick in the recirculation pit. Estimate the return flow and volume of the pit at intervals of one-half ft to one ft on the stick. Obtain the pumping rate by measuring elapsed time to withdraw a specified volume from the pit without return. After estimating the return flow, a percentage can be obtained. The return flow can sometimes be crudely measured with a five-gallon bucket.

Record a change in the fluid recovery rates. If the recovery is essentially the same for an entire core run, only one call per change is necessary. Record the change in fluid recovery rate to the nearest 10%.

L. Spacing. Describe the spacing of discontinuities as close or wide according to the following tables.

SPACING	FOLIATION, BANDING, OR BEDDING	SYMBOL
(1) More than 6 ft	Very thickly (bedded or banded)	VT
(2) 2 to 6 ft	Thickly	T
(3) 8 to 24 inches	Medium	M
(4) 2 1/2 to 8 inches	Thinly	TN
(5) 3/4 to 2 1/2 inches	Very thinly	VTN

In describing structural features, describe the rock mass as thickly bedded or thinly bedded according to the above criteria. Depending on the project requirements, identify the form of joint (stepped, smooth, undulating, or planar), its dip (in degrees), its surface (rough, smooth, or slickensided), its opening (giving width), and its filling (none, sand, clay, or breccia).

M. Orientation. The orientation is the angle of bedding, joints, or fractures measured with a clear plastic protractor to the nearest 10°. Reference all measurements with 0° at horizontal. If fractures have a persistent orientation, record at the beginning and repeat every 5 ft in depth.

N. Condition. The texture of the fractures. Examples of condition are smooth, clean, rough, and stained.

O. Weathering. Describe the degree of weathering according to the following table.

GRADE	SYMBOL	DIAGNOSTIC FEATURES
Fresh	F	No visible sign of decomposition or discoloration. Rings under hammer impact present.
Slightly	SLW	Slight discoloration inward from open fractures; otherwise, similar to fresh.
Moderately Weathered	MW	Discoloration throughout. Weaker minerals (like feldspar) have decomposed. Strength somewhat less than fresh rock, but cores cannot be broken by hand or scraped by knife. Texture has been preserved.
Highly Weathered	HW	Most minerals are somewhat decomposed. Specimens can be broken by hand with effort or shaved with a knife. Core stones present in rock mass. Texture becoming indistinct, but fabric has been preserved.
Completely	CW	Minerals decomposed to soil, but fabric and structure have been preserved (saprolite). Easily crumbled or penetrated. Advanced state of decomposition resulting in plastic soils. Rock fabric and structure completely destroyed. Large volume change.

CAUTION: Soft rock is not necessarily weathered. Look for alteration minerals that indicate true weathering has occurred.

P. Hardness. Describes the degree of hardness according to the following table.

CLASS	HARDNESS	SYMBOL	FIELD TEST
I	Extremely hard	EH	Many blows with geologic hammer required to break intact specimen.
II	Very hard	VH	Hand-held specimen breaks with hammer end of pick under more than one blow.
III	Hard	H	Cannot be scraped or peeled with knife; hand-held specimen can be broken with single moderate blow with pick.
IV	Soft	SO	Can just be scraped or peeled with knife. Indentations 1 mm to 3 mm show in specimen with moderate blow with pick.
V	Very soft	VSO	Material crumbles under moderate blow with sharp end of pick and can be peeled with a knife, but specimen is too hard to hand trim to a size usable in a triaxial test apparatus.

Q. Lithologic. The type of strata encountered during various subsurface exploratory investigations. The geologic lithology codes that are to be used are listed in Appendix 5.7.

R. Visual Description. Other petrologic descriptors that aid in the classification of the rock or soil. Information that should be included is listed below.

1. Typical Name
   Sandstone, schist, granite, or any of the appropriate names/descriptions included in Appendix 5.6

2. Grain Size
   Fine, medium, or coarse

3. Structure
   Stratified, laminated (varved), fissured, slickensided, blocky, lensed, or homogeneous (nonstratified)

4. Color
   Use A.S.A. rock color chart for rocks; use Mansell color chart for soils. This should include grey colors for reduced soils.

5. Cementation
   Weak or strong

6. Local or Geologic Name

7. Group Symbol from Appendix 5.5 or 5.6

S. Place samples for analyses in appropriate containers (see SOP 1.5, Guide to Handling, Packaging, and Shipping of Samples). Designate sample intervals removed from core by wooden blocks that have been properly labeled according to the interval sampled.

3.4. Postoperation

3.4.1. Field

A. Ensure that all equipment is accounted for, decontaminated (see SOP 1.6, General Equipment Decontamination), and ready for shipment.

B. Restore the site to presampling conditions and plug open sampling holes, as specified in the FSP or WP.

C. Make sure all borehole locations are properly staked and the location ID is readily visible on the location stake.

D. Prepare samples and transport according to SOP 1.3, Sample control and Documentation; SOP 1.4, Sample Containers and Preservation; and SOP 1.5, Guide to Handling, Packaging, and Shipping of Samples.

3.4.2. Documentation

A. Record cleanup and hole abandonment procedures and any uncompleted work (like site restoration or long-term monitoring) in the logbook.

B. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

C. Review data collection forms for completeness.

3.4.3. Office

A. Deliver original forms and logbooks to the site manager for technical review. He/she will review, sign forms, and transmit to the document control officer (copies to the files) for eventual delivery to the Department of Energy.

B. Inventory equipment and supplies. Repair or replace all broken or damaged equipment. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

C. Contact the analytical laboratory to ensure that samples arrived safely and instructions for sample analyses are understood.

4. SOURCES

ASTM. 1986. "Standard Method for Penetration Test and Split-Barrel Sampling of Soils," 298-303, ASTM D: 1586-84. American Society of Testing Methods, Philadelphia, Pennsylvania.

ASTM. 1986. "Standard Practice for Thin-Walled Tube Sampling of Soils," 304-307, ASTM D: 1587-83. American Society of Testing Methods, Philadelphia, Pennsylvania.

ASTM. 1986. "Standard Practice for Diamond Core Drilling For Site Investigation," 333-337, ASTM D: 2113-83. American Society of Testing Methods, Philadelphia, Pennsylvania.

ASTM. 1986. "Standard Test Method for Classification Of Soils For Engineering Purposes," 397-410, ASTM D: 2487-85. American Society of Testing Methods, Philadelphia, Pennsylvania.

ASTM. 1986. "Standard Recommended Practice for Description of Soils (Visual-Manual Procedure)," 411-25, ASTM D:2488-84. American Society of Testing Methods, Philadelphia, Pennsylvania.

ASTM. 1986. "Standard Practice for Ring-Lined Barrel Sampling of Soils," 560-563, ASTM D: 3550-84. American Society of Testing Methods, Philadelphia, Pennsylvania.

U.S. Corps of Engineers. 1953. "The Unified Soil Classification System." Technical Memorandum No. 3-357. Washington, D.C.: U.S. Government Printing Office.

5. APPENDIXES

5.1. Equipment and Supplies Checklist

5.2. Borehole Log (Soil) Form

5.3. Borehole Log (Rock) Form

5.4. Data Form Completion

5.5. Checklist for Description of Fine-Grained Soils

5.6. Checklist for Description of Coarse-Grained Soils

5.7. Geologic Lithology Codes

APPENDIX 5.1

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Plastic sheet

_____ Hand lens

_____ Tape measure (divided by feet and tenths of feet)

_____ Wooden blocks (for core)

_____ Core boxes

_____ Grain-size chart

_____ Color chart

_____ Dilute HCl

_____ Red and blue permanent markers

_____ Strainer

_____ Sample labels

APPENDIX 5.2

BOREHOLE LOG (SOIL) FORM

BOREHOLE LOG (SOIL)

PAGE 1 OF ____

FACILITY CODE _____ DRILLER CODE _____
LOCATION ID _____ COMPLETION DATE _____
COORDINATES (FT): DIAMETER (IN) _____
NORTH _____ EAST _____ DEPTH (FTFD) _____
GROUND ELEVATION (FT MSL) _____ CONSTRUCTION METHOD _____
LOCATION TYPE BH ACCEPTANCE CODE _____
COMMENTS _____

GROUNDWATER LEVELS

DATE	TIME	DEPTH (FT)

LOCATION DESCRIPTION

_____

LITHOLOGIC LOG

LOGGER CODE _____

DEPTH (FT)	SAMPLE INTERVAL	SAMPLE RECOVERY	SAMPLE RETAINED	SAMPLE METHOD	SAMPLE ID	BLOW COUNT (PER 6 IN)	N VALUE	USCS	VISUAL DESCRIPTION

ACCEPTANCE CODES: A–ACCEPTABLE R–RECONNAISSANCE U–UNACCEPTABLE N–NOT DETERMINED

CONSTRUCTION METHODS:

A – AIR ROTARY
B – BORED OR AUGERED
C – CABLE–TOOL
D – DUG
H – HYDRAULIC–ROTARY
J – JETTED
P – AIR–PERCUSSION
R – REVERSE ROTARY
T – TRENCHING
V – DRIVEN
W – DRIVE AND WASH
Z – OTHER (SPECIFY)

SAMPLE METHODS:

A – AUGER CUTTINGS
S – 2" O.D. 1.38" I.D. DRIVE SAMPLE
U – 3" O.D. 2.42" I.D. TUBE SAMPLE
T – 3" O.D. THIN–WALLED SHELBY TUBE
O – OTHER (SPECIFY)

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS
BHD–002 (3/88)

FORM COMPLETED BY/DATE _____ TECHNICAL REVIEWER/DATE _____

BOREHOLE LOG (SOIL)

PAGE ____ OF ____

FACILITY CODE _____

LOCATION ID _____ **COMPLETION DATE** _____

LITHOLOGIC LOG

DEPTH (FT)	SAMPLE INTERVAL	SAMPLE RECOVERY	SAMPLE RETAINED	SAMPLE METHOD	SAMPLE ID	BLOW COUNT (PER 6 IN)	N VALUE	USCS	VISUAL DESCRIPTION

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS

SHD—002 (3/88)

FORM COMPLETED BY/DATE _____

TECHNICAL REVIEWER/DATE _____

APPENDIX 5.3

BOREHOLE LOG (ROCK) FORM

BOREHOLE LOG (ROCK)

PAGE 1 OF ____

FACILITY CODE _____ **DRILLER CODE** _____
LOCATION ID _____ **COMPLETION DATE** _____
COORDINATES (FT): **DIAMETER (IN)** _____
NORTH _____ **EAST** _____ **DEPTH (FTFD)** _____
GROUND ELEVATION (FT MSL) _____ CONSTRUCTION METHOD _____
LOCATION TYPE BH _____ **ACCEPTANCE CODE** _____

GROUNDWATER LEVELS

DATE	TIME	DEPTH (FT)

LOCATION DESCRIPTION

LITHOLOGIC LOG

LOGGER CODE _____

DEPTH (FT)	PACKER TEST INTERVAL	DRILLING METHOD	SAMPLE ID	% CORE RECOVERY	% ROCK QUALITY DESIGNATION (RQD)	% DRILLING FLUID RECOVERY	SPACING	ORIENTATION	CONDITION	WEATHERING	HARDNESS	LITHOLOGIC	VISUAL DESCRIPTION

ACCEPTANCE CODES: A–ACCEPTABLE R–RECONNAISSANCE U–UNACCEPTABLE N–NOT DETERMINED

CONSTRUCTION METHODS:

A – AIR ROTARY
B – BORED OR AUGERED
C – CABLE–TOOL
D – DUG
H – HYDRAULIC–ROTARY
J – JETTED
P – AIR–PERCUSSION
R – REVERSE ROTARY
T – TRENCHING
V – DRIVEN
W – DRIVE AND WASH
O – OTHER (SPECIFY)

DRILLING METHODS:

H – 8 1/2" HOLLOW STEM AUGER
C – 4 1/2" CONTINUOUS FLIGHT AUGER
GB – GEAR BIT
NX – NX ROCK CORING
NQ – NQ WIRELINE ROCK CORING

SPACING:

BEDDED, FOLIATION, ETC:
VT – VERY THICKLY
T – THICKLY
M – MEDIUM
TN – THINLY
VTN – VERY THINLY

JOINTS/FAULTS:
VW – VERY WIDELY
W – WIDELY
M – MEDIUM
C – CLOSELY
VC – VERY CLOSELY

CONDITION:

ND – NODULAR
MT – MOTTLED
V V – VARVED
BN – BANDED
ST – STINGERS
SM – SMOOTH
CL – CLEAN
RH – ROUGH
SL – SLIGHT

WEATHERING:

F – FRESH
SL – SLIGHTLY WEATHERED
MW – MODERATLY WEATHERED
HW – HIGHLY WEATHERED
CW – COMPLETELY WEATHERED
RS – RESIDUAL SOIL

HARDNESS:

EH – EXTREMELY HARD
VH – VERY HARD
H – HARD
S – SOFT
VS – VERY SOFT

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS
BHD–003 (3/88)

FORM COMPLETED BY/DATE _____ TECHNICAL REVIEWER/DATE _____

BOREHOLE LOG (ROCK)

PAGE ____ OF ____

FACILITY CODE _____

LOCATION ID _____ COMPLETION DATE _____

LITHOLOGIC LOG

DEPTH (FT)	PACKER TEST INTERVAL	DRILLING METHOD	SAMPLE ID	% CORE RECOVERY	% ROCK QUALITY DESIGNATION (RQD)	% DRILLING FLUID RECOVERY	SPACING	ORIENTATION	CONDITION	WEATHERING	HARDNESS	LITHOLOGIC	VISUAL DESCRIPTION

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS

BHD-003 (1/88)

FORM COMPLETED BY/DATE _____ TECHNICAL REVIEWER/DATE _____

APPENDIX 5.4

DATA FORM COMPLETION INSTRUCTIONS

Use a pen with black ink that is not water soluble (not a felt-tip pen). Make an entry in each blank. Where there is no data entry, enter UNK for Unknown, NA for Not Applicable, or ND for Not Done. If any procedure was not performed as prescribed, give the reason for the change or omission on the form. To change an entry, draw a single line through it, add the correct information above it, and initial the change.

BOREHOLE LOG (SOIL) FORM AND BOREHOLE LOG (ROCK) FORM

The following information explains the completion of the upper portion of both forms.

1. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

2. Location ID. Four-character code assigned sequentially to each borehole, test pit, or surface location where chemical, biological, radiological, and other measurements are taken.

3. Coordinates (Ft): North/East. The horizontal location of the borehole. At the time of the field investigation, the exact coordinate position of the borehole will not be known. In this case, write NA in the two spaces on the form. This information will be provided on an addendum form when the survey data comes in after the drilling program has been completed.

4. Ground Elevation. At the time of the field investigation, the exact ground elevation of the borehole will not be known. In this case, write NA in the space on the form. This information will be provided on an addendum to the original data form when the survey data comes in after the drilling program has been completed.

5. Location Type BH is a standard location type for a borehole log or well.

6. Driller Code. A maximum of four characters noting the company responsible for drilling.

7. Completion Date. The date when the borehole reached total depth in the format DD-MMM-YY (01-JAN-88).

8. Diameter. Diameter of borehole in inches and tenths of inches.

9. Depth. Total depth of borehole in feet and tenths of feet.

10. Construction Method. The construction or drilling method used in the advancement of the borehole. A table of various construction methods is included at the bottom of each Borehole Log form.

11. Acceptance Code. A one-character code assigned by the facility manager.

12. Groundwater Levels. Record depth-to-water when encountered during the drilling. Take at least one water level measurement after the completion of drilling and the borehole has been left open or a piezometer or monitoring well has been installed. Before taking water level measurements, see SOP 3.1, Water Level Measurement.

13. Location Description. Description of the approximate borehole location in respect to some recognizable, permanent topographic or geographic location that is nearby.

14. Logger Code. Four-character code identifying the company responsible for collecting the information recorded on the form.

15. Depth in Feet That Depicts Lithologic or Soil Boundaries. Record depths on the Borehole Logs in feet and fractions (tenths of feet).

Conversion Table

Inches to Tenths of Feet

Inches	Tenths of Feet
1	.08
2	.17
3	.25
4	.33
5	.42
6	.50
7	.58
8	.67
9	.75
10	.83
11	.92
12	1.00

16. Sample ID. Four-digit number assigned to ensure that data collected retains uniqueness from other data collected at the same location ID.

A. The following describes the nonrepetitive information required for completing the Borehole Log (Soil) form.

1. Comments. Includes preservation method, acidified or nonacidified type of acid (if acid was used to preserve water sample), and any additional information.

2. Sample Interval. The designated starting and ending depth of the appropriate sampling technique. The following table summarizes common intervals for sample lengths.

Sampling Device	Sample Interval (Feet)	Example Depth Internal
SPT	1.5 or 2.0	14.8 - 16.3
Large-diameter ring sample	1.0, 1.5, or 2.0	14.8 - 15.8
Shelby tube	2.0	14.8 - 16.8
Continuous sampler	5.0	14.8 - 19.8

If the designated sample is 1.5 ft standard penetration test (SPT), but in actuality the SPT only penetrates 0.5 ft (or some length less than the sample interval) in 50 blows per 6 inches or less (refusal blow count), the graphical representation for the sample interval remains at 1.5 ft.

3. Sample Recovery. The numerical representation of the actual sample recovered by the respective sampling method.

4. Sample Retained. The numerical representation of the actual sample retained for future laboratory testing. This number cannot be larger than the sample recovery number. If no sample is recovered, write NR for no recovery in the space provided.

5. Sample Method. One-character code to identify the sampling method used to retain a subsurface soil sample. Sample types are listed below. The box in the lower right-hand corner of the Borehole Log (Soil) form lists the types of samples.

   Sample Types:

   A Auger cuttings
   S 2-inch O.D., 1.38-inch I.D. drive sample
   U 3-inch O.D., 2.42-inch I.D. tube sample
   T 3-inch O.D., thin-walled Shelby tube
   O Other (specify)

6. Blow Count. The number of blows (blow counts) required for every 6 inches of the 18-inch or 24-inch SPT.

7. N Value. The sum of the number of blows in the last two 6-inch intervals of the SPT.

8. Unified Soil Classification System (USCS). Two-character code describing soil strata encountered during drilling. Use the standard Unified Soil Classification (USCS) designations except when a combination of two soils is encountered. In that case, use two different two-character codes (separated by a dash, like CL--ML). Codes may also be derived from the agricultural classifications of soils. Agricultural classifications are not standard and vary from region to region for the same basic soil type.

For the purpose of classifications, the USCS system recognizes 15 soil groups, each having distinctive engineering properties. A listing of these 15 USCS classifications is shown below.

CH	Fat Clays
CL	Lean Clays, Sandy Clays, or Gravelly Clays
GC	Clayey Gravel or Clayey Sandy Gravel
GM	Silty Gravel or Silty Gravelly Sand
GP	Gravel or Sandy Gravel--Poorly Graded
GW	Gravel or Sandy Gravel--Well Graded
MH	Micaceous Silts or Diatomaceous Soils
OH	Fat Organic Clays
OL	Organic Silts or Lean Organic Clays
PT	Peat, Humus, and Other Organic Swamp Materials
SC	Clayey Sand or Clayey Gravelly Sand
SM	Silty Sand or Silty Gravelly Sand
SP	Sand or Gravelly Sand--Poorly Graded
SW	Sand or Gravelly Sand--Well Graded

9. Visual Description. The soil description will be according to the ASTM Designation, D 2488-69 Standard Recommended Practice for Description of Soils (Visual-Manual Procedure). Appendixes 5.5 and 5.6 include checklists outlining minimum descriptions of fine-grained and coarse-grained soils.

B. The following describes the nonrepetitive information required to complete the Borehole Log (Rock) form.

1. Packer Test Interval. A graphical representation that denotes the bottom of the top packer and the top of the bottom packer by a solid line drawn in the column.

2. Drilling Method. The drilling method could be, but is not limited to, gear bit, NX rock coring, or NQ wireline rock coring. The box in the lower right-hand corner of the Borehole Log (Rock) form lists the accepted abbreviations for drilling methods shown below.

Drilling Methods

GB	Gearbit (specify diameter)
NX	NX Rock Coring
NQ	NQ Wireline Rock Coring
AR	Air Rotary
MR	Mud Rotary
WR	Water Rotary
CT	Cable Tool

3. Percent Core Recovery. The total length of the recovered core multiplied by 100 and divided by the length of the core run results in the percentage of recovery. Record the recovery of core to the nearest 5%.

4. Rock Quality Designation (RQD). The Rock Quality Designation (RQD) is computed by summing the lengths of all pieces of core equal to or longer than 4 inches and dividing by the percent recovery. The result is multiplied by 10 to yield the RQD in a percentage form that can be recorded to the nearest 5%.

$$RQD = \frac{\text{Sum of Length} > 4 \text{ inches}}{\text{Total Length of Core}}$$

5. Percent Drilling Fluid Recovery. After estimating the return flow, a percentage can be obtained. Record a change in the fluid recovery rates. If the recovery is essentially the same for the entire core run, only one call per change is necessary. Record it to the nearest 10%.

6. Spacing. Describe the spacing of discontinuities with the symbols or verbiage described in section 3.3.1.M.

7. Orientation. Record this call to the nearest 10°.

8. Condition. Examples of conditions are smooth, clean, rough, or stained.

9. Weathering. Describes as fresh the degree of weathering (slight, moderate, or high), complete, or residual soil.

10. Hardness. Describes sample as extremely hard, very hard, hard, soft, or very soft.

11. Lithologic. Two-character code describing the lithology encountered during various subsurface exploratory investigations. Use the geologic lithology codes listed in Appendix 5.7.

12. Visual Description. Other petrologic descriptors to assist in the classification of the rock.

APPENDIX 5.5

CHECKLIST FOR DESCRIPTION OF FINE-GRAINED SOILS

1. Typical Name

   Sandy silt, silt, clayey silt, sandy clay, silty clay, clay, organic silt, organic clay, or fill

2. Size Distribution

   Approximate percent gravel, sand, and fines in fractions finer than three inches

3. Color

   Note presence of mottling and banding, as well as soil color

4. Moisture Content

   Dry, moist, wet, or saturated

5. Consistency

   Soft, firm (medium), stiff, very stiff, or hard

6. Structure

   Stratified, laminated (varved), fissured, blocky, lensed, or homogeneous (nonstratified)

7. Cementation

   Weak, strong, or absent

8. Local or Geologic Name

9. Group Symbol

   U.S.C.S. Soil Classification

Group Symbol	Group Name
CL	Lean clay (low to medium plasticity)
ML	Silt
OL	Organic clay or silt (lean)
CH	Fat clay (high plasticity)
MH	Elastic silt
OH	Organic clay or silt (fat)
PT	Peat

APPENDIX 5.6

CHECKLIST FOR DESCRIPTION OF COARSE-GRAINED SOILS

1. Typical Name

   Sand, clayey sand, silty sand, gravel, clayey gravel, silty gravel, cobbles, or boulders

2. Gradation

   Well graded (uniformly graded) or poorly graded (gap-graded)

3. Size Distribution

   Approximate percent gravel, sand, and fines in fractions finer than three inches.

4. Grain Shape

   Angular, subangular, subrounded, or rounded

5. Color

6. Moisture Content

   Dry, moist, wet, or saturated

7. Structure

   Stratified, lensed, or nonstratified

8. Cementation

   Weak or strong

9. Local or Geologic Name

10. Group Symbol

    U.S.C.S. Soil Classification

Group Symbol	Group Name
GW	Well-graded gravel
GP	Poorly graded gravel
GM	Silty gravel
GC	Clayey gravel
SW	Well-graded sands
SP	Poorly graded sands
SM	Silty sand
SC	Clayey sand

APPENDIX 5.7

GEOLOGIC LITHOLOGY CODES

LITHOLOGIC	NAME/DESCRIPTION
AL	Alluvium
BA	Basalt
BR	Undifferentiated Bedrock
BT	Bandelier Tuff
CG	Conglomerate
CH	Fat Clays
CL	Lean Clays, Sandy or Silty Clays, or Gravelly Clays
CA	Calcrete
CR	Chert
CS	Claystone
DI	Diorite
DO	Dolomite
FL	Fill Material
GC	Clayey Gravel or Clayey Sandy Gravel Soils
GM	Silty Gravel or Silty Soils
GN	Gneiss
GP	Gravel or Sandy Gravel, Poorly Graded
GR	Granite
GW	Gravel or Sandy Gravel, Well Graded
GY	Gypsum
LG	Lignite
LS	Limestone
MH	Micaceous Silts or Diatomaceous Soils
ML	Silts, Sands, or Clayey Fine Sands
NR	No Recovery of Data For Classifying
OH	Organic Clays of High Plasticity
OL	Organic Silts or Organic Clays of Low Plasticity
OT	Other General Materials
PT	Peat, Humus, and Other Organic Swamp Soils
QT	Quartzite
RY	Rhyolite
SA	Salt
SC	Clayey Sand or Sand-Clay Mixture
SH	Shale
SI	Silcrete, Poorly Graded
SM	Silty Sand or Sand-Silt Mixtures
SP	Sand or Gravelly Sand, Poorly Graded
SS	Sandstone
ST	Siltstone
SW	Sand or Gravelly Sand, Well Graded
TF	Tuff
TI	Till
TP	Unknown
TS	Topsoil, Undifferentiated
VO	Volcanics

STANDARD OPERATING PROCEDURE 5.2

SOIL SAMPLING WITH A SPADE AND SCOOP

1. PURPOSE

To describe a method for collecting a soil sample less than 4 ft below the land surface.

2. DISCUSSION

The Field Sampling Plan (FSP) or Work Plan (WP) contains specific details about the procedures and equipment for this SOP. Refer to the FSP or WP for the type of samples to be collected. Collection and measurement of samples and documentation of data will be performed as described in the associated procedures.

Sampling of the soil horizons above the groundwater table can detect contaminants before they migrate into the water table. Soil sample analysis can assist in determining the extent of the contaminant source term and establish the amount of contamination absorbed into aquifer solids that have the potential to contaminate groundwater.

Accurate, representative samples can be collected with this procedure, if care and precision are demonstrated by the technician. The spade-and-scoop method can be used in most soil types, but is somewhat limited to sampling near the soil surface. Sample collection from depths greater than 50 inches can become extremely labor-intensive. Collection of samples from near the soil surface can be accomplished with tools like spades, shovels, and scoops. Spades and shovels will be used to remove surficial material to the required depth. Then a stainless steel or Teflon scoop is used to collect the sample. Devices plated with chrome or other materials are not acceptable for sample collection. The use of a flat, pointed mason trowel often aids in collecting undisturbed soil profile samples. To the extent possible, the sampling process should not alter the medium being investigated. Samples should be kept at 4°C, protected from direct light, sealed tightly in glass bottles, and analyzed as soon as possible.

3. PROCEDURES

3.1. Associated Procedures

Before every operation, a review of the SOPs 1.1-1.10 is necessary. These SOPs contain information on the performance of field activities. They should be consulted for specific information about equipment and supplies; sample collection, preservation, packaging, and shipping; decontamination procedures; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.3	Sample Control and Documentation
1.4	Sample Containers and Preservation

1.5	Guide to Handling, Packaging, and Shipping of Samples
1.6	General Equipment Decontamination
5.1	Soil and Rock Borehole Logging and Sampling

3.2. Preparation

3.2.1. Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Obtain appropriate permission for property access.

D. Assemble the equipment and supplies listed in Appendix 5.1. Ensure the proper operation of all sampling equipment.

E. Notify the analytical laboratory of sample types, the number of samples, and the approximate arrival date.

F. Contact the carrier that will transport samples to obtain information on regulations and specifications.

3.2.2. Documentation

A. Obtain a logbook from the QA officer.

B. Record results of the equipment check in the logbook.

C. Obtain a sufficient number of appropriate ER Program data collection forms (see INDEX TO SOPs).

D. Consult the ER Program data administrator for a current list of information management codes, location IDs, and sample numbers used in the completion of data forms.

3.2.3. Field

Decontaminate all sampling equipment before taking the first sample and between sampling intervals (see SOP 1.6, General Equipment Decontamination, and FSP or WP).

3.3. Operation

A. Whenever a sample is collected, complete a description of the sample using the Borehole Log (Soil) form. Copies of this form and instructions for completing the form are supplied in SOP 5.1, Soil and Rock Borehole Logging and Sampling.

B. Procedure for Soil Sampling

1. Carefully remove the top layer of soil to the desired sample depth with a spade which has been appropriately decontaminated.

2. Using an appropriate decontaminated stainless steel or Teflon scoop or trowel, remove and discard a thin layer of soil from the area that comes in contact with the shovel.

3. Transfer the sample into an appropriate sample bottle with a stainless steel or Teflon scoop, lab spoon, or equivalent.

4. If required, be sure the Teflon liner is present in the cap. Secure the cap tightly. Preservation with chemical additives is not necessary; it is achieved by cooling the sample with ice. If feasible, minimize holding time and transport the sample to the laboratory no later than two days after collection.

5. Label the sample bottle with the appropriate sample tag. Be sure to label the tag carefully and clearly, addressing all the categories of parameters.

   NOTE: Whenever a sample is collected, a custody record must be initiated on the Custody Transfer Record/Lab Work Request form and a Soil Sample Identification Label affixed to the sample container. SOP 1.3, Sample Control and Documentation, contains copies of the form and label and instructions for completing the form and label.

6. Decontaminate equipment between sample locations according to SOP 1.6, General Equipment Decontamination.

7. Fill the hole and replace any grass turf.

3.4. Postoperation

3.4.1. Field

A. Ensure that all equipment is accounted for, decontaminated (see SOP 1.6, General Equipment Decontamination), and ready for shipment.

B. Restore the site to presampling conditions and fill open sampling holes as specified in the FSP or WP.

C. Make sure all sampling locations are properly staked and the location ID is readily visible on the location stake.

D. Prepare samples and transport according to SOP 1.3, Sample Control and Documentation; SOP 1.4, Sample Containers and Preservation; and SOP 1.5, Guide to Handling, Packaging, and Shipping of Samples.

3.4.2. Documentation

A. Record pertinent sample location information in the logbook.

B. Record cleanup and hole abandonment procedures and any uncompleted work (like site restoration or long-term monitoring) in the logbook.

C. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

D. Review data collection forms for completeness.

3.4.3. Office

A. Deliver original forms and logbooks to the site manager for technical review. He/she will review, sign forms, and transmit to the document control officer (copies to the files) for eventual delivery to the Department of Energy.

B. Inventory equipment and supplies. Repair or replace all broken or damaged equipment. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

C. Contact the analytical laboratory to ensure that the samples arrived safely and the instructions for sample analyses are clearly understood.

4. SOURCE

Ford, Patrick J., Paul J. Turina, and Douglas E. Seely. 1984. Available Sampling Methods, 2d ed. Vol. 2, Characterization of Hazardous Waste Sites - A Methods Manual. U.S. Environmental Protection Agency document EPA/600/4-84/076. Washington, D.C.: U.S. Government Printing Office.

5. APPENDIX

5.1. Equipment and Supplies Checklist

APPENDIX 5.1

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Stainless steel scoop or lab spoon (scoopulas)

_____ Stainless steel shovel or fat-pointed mason trowel

_____ Stainless steel spade

_____ Tape measure (tenths)

_____ Teflon sheets or stainless steel sampling trays

_____ Plastic Sheet

_____ Alconox

_____ Brushes (long handle, scrub, and wire)

_____ Galvanized tub

_____ Trash bags

_____ Buckets (galvanized, stainless steel, and plastic)

_____ Garden pressure sprayer

_____ Cleaning wipes

_____ Kim wipes

_____ Storage containers for waste decontamination solutions

_____ Blue Ice or equivalent

_____ Disposable laboratory gloves

_____ Camera and film

_____ Sample containers and preservatives

_____ Any additional supplies listed in associated procedures, as needed

STANDARD OPERATING PROCEDURE 5.3

SUBSURFACE SOLID SAMPLING WITH HAND AUGER AND THIN-WALL SAMPLER

1. PURPOSE

To define a method of collecting subsurface solid samples with a hand auger and thin-wall tube sampler.

2. DISCUSSION

The Field Sampling Plan (FSP) or Work Plan (WP) contains specific details about the procedures and equipment for this SOP. Refer to the FSP or WP for the type of samples to be collected. Collection and measurement of samples and the documentation of data will be performed as described in the associated procedures.

Solid samples can be recovered directly with a hand auger or with a thin-wall tube sampler. The thin-wall tube sampler provides a less disturbed sample than that obtained with a hand auger. However, it may not be possible to force the thin-wall tube sampler through some soil, and sampling with the hand auger may be the most viable alternative. It is usually not practical to use the hand auger or thin-wall sampler at depths beyond 20 ft.

3. PROCEDURES

3.1. Associated Procedures

Before every operation, a review of the SOPs 1.1-1.10 is necessary. These SOPs contain information on the performance of field activities. They should be consulted for specific information about equipment and supplies; sample collection, preservation, packaging, and shipping; decontamination procedures; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.3	Sample Control and Documentation
1.4	Sample Containers and Preservation
1.5	Guide to Handling, Packaging, and Shipping of Samples
1.6	Equipment Decontamination

3.2. Preparation

3.2.1. Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Obtain appropriate permission for property access.

D. Assemble the equipment and supplies listed in Appendix 5.1. Ensure the proper operation of all sampling equipment.

E. Notify the analytical laboratory of sample types, the number of samples, and the approximate arrival date.

F. Contact the carrier that will transport samples to obtain information on regulations and specifications.

3.2.2. Documentation

A. Obtain a lobgook from the QA officer.

B. Record results of the equipment check in the logbook.

C. Obtain a sufficient number of the appropriate ER Program data collection forms (see INDEX TO SOPs).

D. Consult the ER Program data administrator for a current list of information management codes, location IDs, and sample numbers used in the completion of data forms.

E. Document field conditions, unusual circumstances, and weather in the logbook.

3.2.3. Field

Decontaminate all sampling equipment before taking the first sample and between sampling intervals (see SOP 1.6, General Equipment Decontamination, and the FSP or WP).

3.3. Operation

This method uses an auger, a series of rods, a T handle, and a thin-wall tube or split-spoon sampler (Appendix 5.2). Bore a hole to a desired sampling depth (up to 20 ft) with the auger and then withdraw it. Replace the auger tip with a tube core (sampler), lower it down the borehole, and drive the auger into the soil at the completion depth. Withdraw the core and collect the sample.

Three types of augers are available for this method of sampling. They are bucket-type, continuous flight (screw), and posthole augers. The bucket-type auger is more appropriate for direct sample recovery. The bucket-type auger is fast and provides a large volume of sample. When a continuous flight auger is used, the sample can be collected directly off the flights that are usually in 5-ft intervals. The continuous flight auger is satisfactory for use when a composite of the complete soil column is desired. Posthole augers have limited utility for sample collection because they are designed to cut through fibrous,

rooted swampy areas. In soil where the borehole will not remain open when the tool is removed, use a temporary casing until the desired sampling depth is reached.

3.3.1 Collection of Samples

A. Clear any surface debris (twigs, rocks, and litter) from the area to be sampled. It may be advisable to remove the first three to six inches of surface soil for an area approximately six inches in radius around the drilling location.

B. Begin augering, periodically removing accumulated soils. This prevents accidentally brushing loose material back down the borehole when removing the auger or adding drill rods.

C. After reaching the desired depth, slowly and carefully remove the auger from boring.

NOTE: When sampling directly from the auger, collect the sample after the auger is removed from boring and proceed to step I.

D. Remove auger tip from drill rods and replace with a precleaned, thin-wall tube sampler. Install the proper cutting tip.

E. Lower the tube sampler down the borehole, being careful not to scrape soil from the sides of the borehole that will fall into the bottom of the borehole. Gradually force the tube sampler into the soil. Care should be taken to avoid scraping the borehole sides. Avoid hammering the drill rods to facilitate coring because the vibrations may cause the boring walls to collapse.

F. Remove the tube sampler and unscrew the drill rods.

G. Remove the cutting tip and remove the core from the device.

H. Discard the top of the core (approximately one inch, which represents any material collected by the tube sampler before penetration of the layer in question).

I. Place sample into an appropriate container.

NOTE: Whenever a sample is collected, a custody record must be initiated on the Custody Transfer Record/Lab Work Request form and a Soil Sample Identification Label affixed to the sample container. SOP 1.3, Sample Control and Documentation, contains copies of the form and label and instructions for completing the form and label.

J. If required, be sure a Teflon liner is present in the cap. Secure the cap tightly onto the sample container. Preserve the sample container with ice or in a refrigerator. Freezing may be required (for instance, Hg or tritium determinations). Consult SOP 1.5, Guide To Handling, Packaging, and Shipping of Samples, and SOP 1.4, Sample Containers and Preservation.

K. If another sample is to be collected at a greater depth in the same hole, reattach the auger bit to the drill and assembly and follow steps 3-11.

L. Refill the hole and restore the sampling area according to instructions provided in the FSP or WP.

3.4. Postoperation

3.4.1. Field

A. Ensure that all equipment is accounted for, decontaminated (see SOP 1.6, General Equipment Decontamination), and ready for shipment.

B. Restore the site to presampling conditions and fill open sampling holes as specified in the FSP or WP.

C. Make sure all sampling locations are properly staked and the location ID is readily visible on the location stake.

D. Prepare samples and transport according to SOP 1.3, Sample Control and Documentation; SOP 1.4, Sample Containers and Preservation; and SOP 1.5, Guide to Handling, Packaging, and Shipping of Samples.

3.4.2. Documentation

A. Record cleanup and hole abandonment procedures and any uncompleted work (like site restoration or long-term monitoring) in the logbook.

B. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

C. Review data collection forms for completeness.

3.4.3. Office

A. Deliver original forms and logbooks to the site manager for technical review. He/she will review, sign forms, and transmit to the document control officer (copies to the files) for eventual delivery to the Department of Energy.

B. Inventory equipment and supplies. Repair or replace all broken or damaged equipment. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

C. Contact the analytical laboratory to ensure that samples arrived safely and instructions for sample analyses are clearly understood.

4. SOURCE

deVera, E. R., B. P. Simmons, R. D. Stephens, and D. L. Storm. 1980. "Samplers and Sampling Procedures for Hazardous Waste Streams." U.S. Environmental Protection Agency report EPA 600/2-80-018, January 1980. Washington, D.C.: U.S. Government Printing Office.

5. APPENDIXES

5.1. Equipment and Supplies Checklist

5.2. Augers and Thin-Wall Tube Sampler

APPENDIX 5.1

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Thin-wall tube sampler

_____ Drill rods

_____ T handle

_____ Auger

- _____ Bucket type
- _____ Continuous flight
- _____ Posthole

_____ Sample container(s)

_____ Decontamination equipment

- _____ Sprayer
- _____ Distilled water
- _____ Scrub brush
- _____ Methanol
- _____ Disposable laboratory gloves

_____ Combustible gas indicator

_____ Portable photoionization detector (PID) or flame ionization detector (FID)

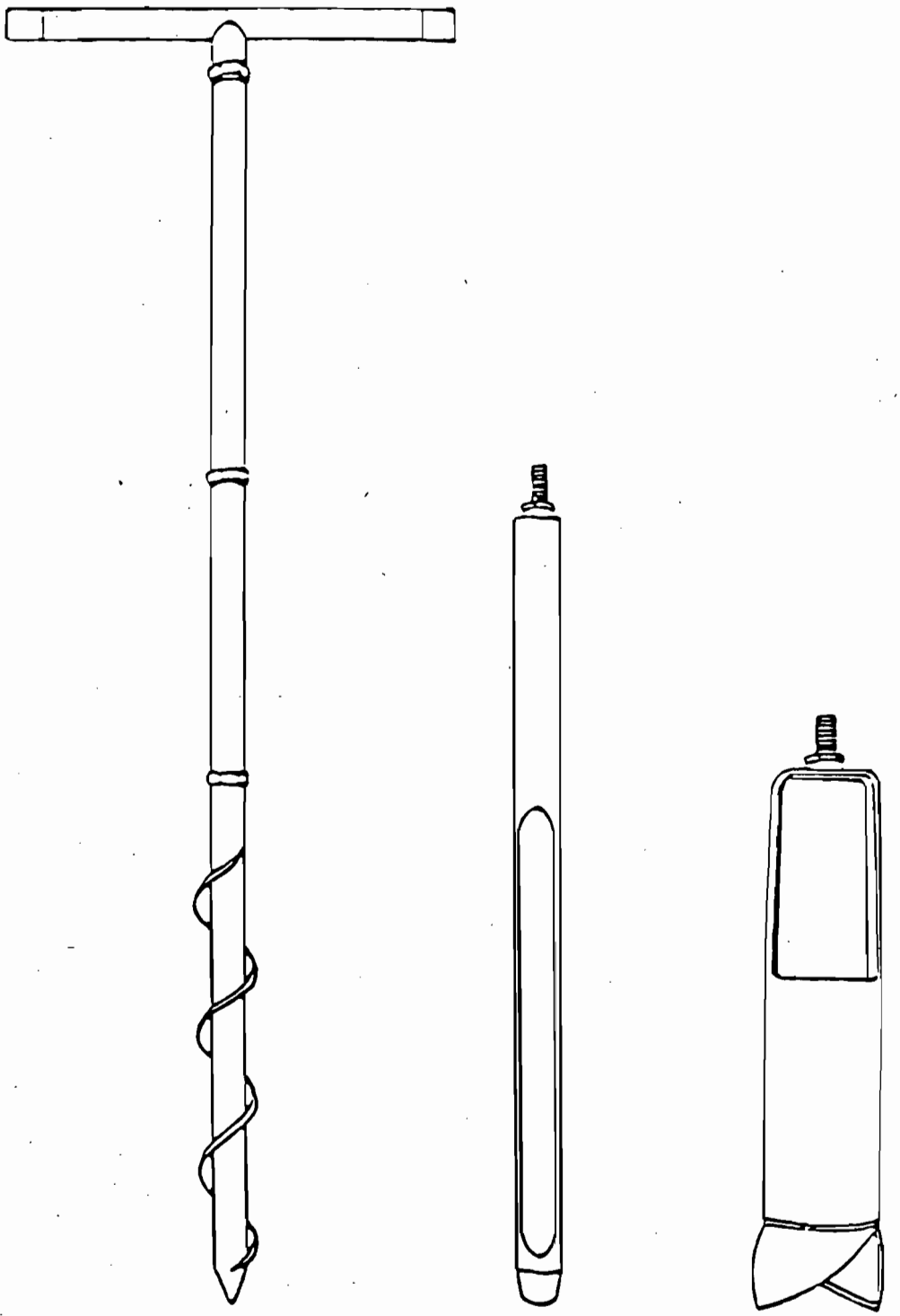
_____ Blue Ice or equivalent

_____ Plastic sheet

_____ Any additional supplies listed in associated procedures, as needed

APPENDIX 5.2

AUGERS AND THIN-WALL TUBE SAMPLER



STANDARD OPERATING PROCEDURE 6.1

HEALTH AND SAFETY MONITORING OF COMBUSTIBLE GAS LEVELS

1. PURPOSE

To describe the equipment and proper method for monitoring combustible gas levels in order to determine when an explosion hazard exists in the work environment.

2. DISCUSSION

The Field Sampling Plan (FSP) or Work Plan (WP) provides information on the scope of specific operations, related health and safety requirements, and the applicability of this procedure. Combustible gas indicators (or explosimeters) are used to determine the potential for the combustion or explosion of unknown atmospheres. A typical combustible gas indicator (CGI) determines the level of organic vapors and gases present in an atmosphere as a percentage of the lower explosive limit (LEL) or lower flammability limit (LFL) by measuring the change in electrical resistance in a Wheatstone bridge circuit.

CGIs provide readouts in units of percent LEL, in parts per million (ppm) combustible gases by volume, or both. The types of combustible gases to be encountered are often unknown. In those instances, the more explosive the calibration gas (the lower the LEL), the more sensitive the indication of explosivity, and a greater margin of safety results. The operator should be familiar with the LEL concentrations for specific gases to effectively use instruments that provide data only in ppm combustible gas (by volume).

Although instruments can be purchased that are factory-calibrated for gases like butane, pentane, natural gas, or petroleum vapors, methane calibration is the most common. The LEL of methane is 5% by volume in air; therefore, an air mixture containing 5% methane will be read as 100% LEL and is explosive. When combustible gases other than methane are sampled, the relative response of the detector must be considered. Recalibration to other gases may be possible (see the manufacturer's recommendations), and National Bureau of Standards (NBS) traceable calibration gases should be used. The relative sensitivity of the detector and the differences in LEL for different gases will produce varying meter-responses. Correlation equations that will convert the percent LEL (based on methane) indicated by the instrument to a percent LEL for another combustible gas can usually be found in the CGI operating manual. Many units have alarm systems that can be adjusted for various LELs, and several incorporate oxygen analyzers.

2.1. Definitions

A. Lower Explosive Limit (LEL)

The LEL (also LFL, lower flammability limit) is defined as the lowest concentration of gas or vapor in air by volume that can be ignited and cause an explosion or flame propagation.

B. Upper Explosive Limit (UEL)

The UEL (also UFL, upper flammability limit) is the concentration of gas in air above which there is insufficient oxygen available to support combustion and an explosion is unlikely. A flame, however, may burn at the gas/air interface. Should additional air enter the mixture, a very explosive atmosphere may develop.

2.2. Instrument Limitations

A. Of the many instruments commercially available for detecting combustible or explosive gas, some are not certified safe for operation in the atmospheres they can detect. It is important to use only those instruments that are certified safe for use in atmospheres greater than 25% of the LEL. The instrument manufacturer's operating manual should be consulted to determine safety certification in specific atmospheres.

B. Combustible gas measurement instruments do not indicate if a given atmosphere contains hazardous or toxic compounds.

C. The CGI cannot be used in atmospheres containing silanes, silicones, silicates, or other compounds containing silicon because these substances seriously impair the instrument response.

D. If the detector has a platinum filament, its sensitivity may be reduced by exposure to gases like leaded gasoline vapors (tetraethyl lead), sulfur compounds (mercaptans and hydrogen sulfide), and sulfide compounds. An inhibitor filament that will nullify the effect of leaded gasoline vapors is available on some commercial units (Mine Safety Appliances Company, Model 260 Portable Combustible Gas and Oxygen Alarm). The instrument manufacturer's operating manual should be consulted to determine the instrument's ability to function in leaded gasoline atmospheres.

E. An oxygen detector should be used in conjunction with a CGI. Select a unit with this feature and follow the operating manual when the oxygen detector is calibrated and used. This is especially important when atmospheres are monitored within enclosed spaces or where oxygen deficient atmospheres (<19.5%) may exist.

F. Unusually high concentrations of sulfur dioxide, fluorine, chlorine, bromine, iodine, and oxides of nitrogen cause measurement interference.

G. Combustible gas indicator instruments must be calibrated frequently. Using an NBS traceable calibration gas, consult the manufacturer's operating manual for calibration frequency. Also, frequent calibration will be necessary if several known organic species are present. Maximum accuracy requires a recalibration for each gas.

3. PROCEDURE

3.1. Associated Procedures

Information that applies to most field activities is provided in SOPs 1.1-1.10. In addition to the FSP or WP, those SOPs provide guidance that may supplement the information in

this procedure. They should be consulted as necessary to obtain specific information about equipment and supplies; sample collection, preservation, packaging, and shipping; decontamination procedures; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.6	General Equipment Decontamination

3.2. Preparation

3.2.1. <u>Office</u>

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Obtain appropriate permission for property access.

D. Assemble the equipment and supplies listed in Appendix 5.1. Perform a minimal check of the CGI in the office to ensure that it is functioning properly. Obtain the CGI, its operating manual, and a supply of NBS traceable gas. Methane is the factory calibration gas, but other gaes may be used for specific requirements. Perform the equipment checks described below.

1. Make sure the instrument is clean and serviceable, especially sample lines and detector surfaces.

2. Check the battery charge level. If in doubt, charge the battery as described in the operating manual. Some units have charge level meters, while others have only low charge alarms.

3. Turn the unit to the on position and allow the instrument sufficient warmup time.

4. Verify that the sample pump is operable when the analyzer is on. The pump can usually be heard when operating.

5. With the intake assembly in combustible gas-free ambient air, zero the meter by rotating the zero control until the meter reads 0% LEL. For instruments with an additional oxygen meter, adjust the dial to 21% oxygen in nonhazardous locations.

6. Calibrate the unit against a known concentration of a calibration gas like hexane by rotating the calibration control (span or gain) until the meter reads the same concentration as the known standard.

7. Some instruments, like the Gas Tech Model 1314, require internal calibrating with a small screwdriver. Consult the operating manual before calibration. With this model, it is also necessary to maintain the proper flow rate during calibration. Connect a flow meter between the CGI and the calibration gas cylinder to monitor the flow rate.

8. The Gas Tech Model 1314 and others are equipped with three meters that read in percent O_2, percent LEL, and ppm. A correctly calibrated instrument for determining percent LEL is critical for monitoring many work environments. The percent oxygen is usually factory calibrated and should not be adjusted in the field. The ppm dial is often not used in the field unless a Photoionization Detector (PID) or Flame Ionization Detector (FID) is not available, as these instruments are considered to be more accurate.

3.2.2. <u>Documentation</u>

A. Obtain a logbook from the QA officer.

B. Record results of the equipment check in the logbook.

C. Obtain a sufficient number of the appropriate ER Program data collection forms (see INDEX TO SOPs).

D. Consult the ER Program data administrator for a current list of information management codes and location IDs used in the completion of data forms.

3.2.3. <u>Field</u>

A. Instrument Check

Before using the CGI in the field, follow the procedures in Section 3.2.1.D. Additional adjustments may be made. If necessary, adjust the alarm setting to the appropriate combustibility limit. The action level or the point at which activities are halted and personnel removed from the immediate vicinity is usually less than 25% of the LEL for the gases that are present.

B. Record necessary calibration data in the logbook and include the information listed below.

1. Date and time of arrival at the site

2. Site identification

3. Instrument, model number, and serial number

4. Date/time calibrated

5. Calibration gas used

6. Calibration location

7. Operator's signature

3.3. **Operation**

3.3.1. <u>Field Measurements</u>

A. Calibrate the CGI daily before use in the field. The calibration procedure for the Gas Tech Model 1314 is outlined in Appendix 5.4. Also, consult the manufacturer's manual.

B. Complete the Combustible Gas Indicator Monitoring Data form (Appendix 5.2) as described in Appendix 5.3, Data Form Completion.

C. Position the intake assembly close to the area in question to get an accurate reading. For readings taken downhole during drilling, there will be a slight delay between positioning the intake tubing downhole and registering accurate meter readings because of the time required for the sample to travel the length of the tube.

D. In general, combustible gas indicator instruments respond in the manner described below.

1. The meter indicates 0.5 LEL (50%). This means that 50% of the concentration of combustible gas needed to reach an unstable combustible situation is present. If the LEL of the gas is 5% in air, then the instrument indicates the presence of a 2.5% mixture.

2. The meter needle stays above 1.0 LEL (100%). This means that the concentration of combustible gas is greater than the LEL and less than the UEL. Therefore, the concentration is immediately combustible and explosive.

3. The meter needle rises above the 1.0 (100%) mark and then returns to zero. This response indicates that the ambient atmosphere has a combustible gas concentration greater than the UEL.

E. Personnel should evacuate the area if any of the events listed below occur.

1. Sounding of the alarm

2. Readings that reach the action levels designated in the Health and Safety Plan

3. Malfunctioning of the CGI

4. Condition encountered or suspected that indicates oxygen enrichment or depletion of the atmosphere (specially designed units are available for operation in those atmospheres)

F. Some important factors to keep in mind during use are listed below.

1. Slow, sweeping motions of intake or cell assembly will help ensure that problem atmospheres are not bypassed. Cover an area from floor (ground) to ceiling, the breathing zone, and areas where maximum concentrations may be expected (for example, downhole during drilling).

2. Operation of the unit in temperatures outside the recommended operating range may compromise the accuracy of readings or damage the instrument. Check the operating manual for the temperature limitations of a particular model.

3. Many combustible gas indicators are not designed for use in oxygen-enriched or depleted atmospheres. If this condition is encountered or suspected, personnel should evacuate the area. Specially designed units are available for operation in those atmospheres.

4. Use an oxygen detector in conjunction with a CGI. Select a unit and follow the operating manual for calibration and use of the oxygen detector.

5. Calibrate the equipment regularly and charge the battery after each field use. See the operating manual for details.

6. The operator should fully understand the operating principles and procedures for the specific CGI in use.

3.4 Postoperation

3.4.1 Field

A. When the activity is completed or at the end of the day, carefully clean the outside of the CGI with a damp disposable towel to remove any visible dirt. Return the CGI to a secure area and place on charge.

B. Ensure that all equipment is accounted for, decontaminated (see SOP 1.6, General Equipment Decontamination), and ready for shipment.

C. Make sure all survey or sampling locations are properly staked and the location ID is readily visible on the location stake.

3.4.2. Documentation

A. Record any uncompleted work (like additional monitoring) in the logbook.

B. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

C. Review data collection forms for completeness.

3.4.3. Office

A. Deliver original forms and logbooks to the document control officer (with copies to the site manager and files) for eventual delivery to the Department of Energy.

B. Inventory equipment and supplies. Repair or replace all broken or damaged equipment. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

4. SOURCE

EPA. 1984. "Characterization of Hazardous Waste Sites - A Methods Manual: Volume II, Available Sampling Methods, Second Edition," U.S. Environmental Protection Agency report EPA-600/4-84-076. Environmental Monitoring Systems Laboratory, Office of Research and Development, Las Vegas, Nevada.

5.. APPENDIXES

5.1. Equipment and Supplies Checklist

5.2. Combustible Gas Indicator Monitoring Data Form

5.3. Data Form Completion

5.4. Calibration Procedure for Gas Tech Model 1314

APPENDIX 5.1

EQUIPMENT AND SUPPLIES CHECKLIST

_____ CGI

_____ Battery charger for CGI

_____ Oxygen sensor

_____ Battery charger for oxygen sensor

_____ Spare gas detector filaments

_____ Spare batteries for CGI

_____ Jeweler's screwdrivers for internal adjustment

_____ Calibration kit

A) Spare gas cylinder (NBS traceable calibration gas)
B) Valve attachment
C) Flexible tubing (tygon)
D) Cylinder to encapsulate sensor probe

_____ Probe extensions

APPENDIX 5.2

COMBUSTIBLE GAS INDICATOR MONITORING DATA FORM

COMBUSTIBLE GAS INDICATOR (CGI) MONITORING DATA

FACILITY CODE _____ **LOG DATE** _____

LOGGER CODE _____ FIELD REP _____

CGI MANUFACTURER _____ CGI MODEL NO _____

SERIAL NO _____ CALIBRATION DATE/TIME _____

ACCEPTANCE CODE _____ BATTERY CONDITION _____

CALIBRATION GAS (%LEL):
TYPE _____ CONCENTRATION _____ % LEL CYLINDER SERIAL NO _____

CALIBRATION GAS (PPM):
TYPE _____ CONCENTRATION _____ PPM CYLINDER SERIAL NO _____

COMMENTS _____

LOCATION ID OR DESCRIPTION	COORDINATES (FT)		MONITORING TIME (HH:MM)	LOCATION TYPE	% LOWER EXPLOSIVE LIMIT	PPM	% OXYGEN
	NORTH	EAST					

ACCEPTANCE CODES: A–ACCEPTABLE R–RECONNAISSANCE U–UNACCEPTABLE N–NOT DETERMINED

LOCATION TYPES: BH – BOREHOLE SL – SURFACE LOCATION TP – TEST PIT WL – WELL

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS

CGI–101 (1/88)

FORM COMPLETED BY/DATE _____ TECHNICAL REVIEWER/DATE _____

APPENDIX 5.3

DATA FORM COMPLETION

Use a pen with black ink that is not water soluble (not a felt-tip pen). Make an entry in each blank. Where there is no data entry, enter UNK for Unknown, NA for Not Applicable, or ND for Not Done. If any procedure was not performed as prescribed, give the reason for the change or omission on the form. To change an entry, draw a single line through it, add the correct information above it, and initial the change.

COMBUSTIBLE GAS INDICATOR MONITORING DATA FORM

1. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

2. Log Date. The date the information recorded on the form was obtained, in the format DD-MMM-YY (01-JAN-88).

3. Logger Code. Three-character or four-character code identifying the company responsible for collecting the information recorded on the form.

4. Field Rep. The name of the field representative.

5. CGI Manufacturer. The company that manufactured the CGI.

6. CGI Model No. The model number of the CGI.

7. CGI Serial No. The serial number of the CGI.

8. CGI Calibration Date/Time. The date and time when the CGI was last calibrated.

9. Acceptance Code. One-character code assigned by the site manager.

10. Battery Condition. The battery charge reading at the beginning of the measurement.

11. Calibration Gas (% LEL). This information consists of three data fields: the chemical name of the calibration gas (type), concentration of the calibration gas (% LEL), and the serial number of the gas cylinder.

12. Calibration Gas (ppm). This information consists of three data fields: the chemical name of the calibration gas (type), concentration of the calibration gas (ppm), and the serial number of the gas cylinder.

13. Comments. Any additional information.

14. Location ID or Description. Four-character code assigned sequentially to each borehole, test pit, or surface location where physical, chemical, biological, radiological, and other measurements are taken.

15. Coordinates (Ft). The location of the measurement of the survey grid in units of feet. The two coordinate fields are in the format north, east.

16. Monitoring Time (HH:MM). The time when a field measurement was taken in the format hours:minutes using a 24-hr clock. Example: 08:37 for 8:37 a.m. and 19:12 for 7:12 p.m. (See conversion table below.)

Conversion Table

Conventional Time	24-Hr Time
1:00 a.m.	1:00
12:00 Noon	12:00
1:00 p.m.	13:00
2:00 p.m.	14:00
3:00 p.m.	15:00
4:00 p.m.	16:00
5:00 p.m.	17:00
6:00 p.m.	18:00
7:00 p.m.	19:00
8:00 p.m.	20:00
9:00 p.m.	21:00
10:00 p.m.	22:00
11:00 p.m.	23:00
12:00 Midnight	24:00

17. Location Type. Code describing the location of the CGI reading. The location type codes are: BH--borehole, TP--test pit, SL--surface location, and WL--well.

18. Percent Lower Explosive Limit. The reading obtained with the meter set to the LEL Scale.

19. PPM. The reading with the meter set to the PPM scale.

20. Percent Oxygen. Record the percent oxygen reading in this data field.

GASTECH MODEL 1314 CALIBRATION

CALIBRATION PROCEDURE

1. PPM RANGE
   - 1.01 Turn on instrument, allow to warm up and adjust zero in normal way.
   - 1.02 Add a few drops of water to glass wool packing inside humidifier. Glass wool should be moist but not dripping.
   - 1.03 Couple flowmeter to Cliplok fitting, and humidifier to flowmeter inlet, as shown.
   - 1.04 Couple Cliplok fitting to instrument inlet.
   - 1.05 Readjust zero as required after instrument stabilizes.
   - 1.06 Note flowmeter reading.
   - 1.07 Connect calibration valve to ppm–range cylinder. Open valve slightly to produce a small flow.
   - 1.08 Couple valve outlet to humidifier inlet.
   - 1.09 Adjust valve to give same flow on flowmeter as observed in 1.06.
   - 1.10 Watch meter as gas enters instrument. Observe highest reading.
   - 1.11 Compare reading with marked gas concentration on cylinder.
   - 1.12 If not correct, adjust calibration as shown in Section V of Instruction Manual.
   - 1.13 Turn off valve and disconnect calibration components.
2. LEL Range

   Use same procedure as above, but in LEL range. However, omit humidifier as it is not necessary in LEL range. Use LEL range cylinder.

GMC-130 (1/88)

STANDARD OPERATING PROCEDURE 6.2

HEALTH AND SAFETY MONITORING OF ORGANIC VAPORS WITH A PHOTOIONIZATION DETECTOR

1. PURPOSE

To describe the equipment and proper method for environmental monitoring of toxic gases and vapors using a portable photoionization detector (PID).

2. DISCUSSION

The Field Sampling Plan (FSP) or Work Plan (WP) provides information on the scope of the given operation and the applicability of this procedure to the work activities.

The PID is useful as a general survey instrument at hazardous waste sites. A PID is capable of detecting and measuring real-time concentrations of many organic and inorganic vapors in the air. A PID is similar to a flame ionization detector (FID) in application. The PID has somewhat broader capabilities because it can detect certain inorganic vapors. Conversely, the PID is unable to respond to certain low molecular weight hydrocarbons (like methane and ethane) that are readily detected by FID instruments. Appendix 5.1 describes the application comparisons between an FID organic vapor analyzer and a PID.

A PID will respond to most vapors that have an ionization potential less than or equal to that supplied by the ionizing source in the detector, which is an ultraviolet (UV) lamp. Several probes are available for the PID, each having a different source and a different ionization potential. For this reason, the selection of the appropriate probe is essential in obtaining useful field results. Though it can be calibrated to a particular compound, the instrument cannot distinguish between detectable compounds in a mixture of gases. Therefore, it indicates an integrated response to the mixture.

2.1. PID Instrument Limitations

A. The PID is a nonspecific total vapor detector. It cannot be used to identify unknown substances; it can only quantify them.

B. The PID must be calibrated to a specific compound.

C. The PID does not respond to certain low molecular weight hydrocarbons like methane and ethane.

D. Certain toxic gases and vapors like carbon tetrachloride and hydrogen cyanide have high ionization potentials and cannot be detected with a PID.

E. Certain models of PID instruments are not intrinsically safe. Refer to the manufacturer's operating manual for use in potentially flammable or combustible atmospheres. A PID should be used in conjunction with a combustible gas indicator (see SOP 6.1, Health and Safety Monitoring of Combustible Gas Levels).

F. Electrical power lines or power transformers close to the PID instrument may cause measurement errors. Under this circumstance, refer to the operating manual for proper procedures.

G. High winds and high humidity will affect measurement readings. Certain models of PID instruments become unusable under foggy conditions. An indication of this is the needle dropping below 0.

H. The lamp window must be periodically cleaned to ensure ionization of the air contaminants.

I. One PID instrument, the HNu, measures concentrations from about 1 to 2000 ppm, although the response is not linear over this entire range. For example, the response to benzene is linear from about 0 to 600 ppm. This means the HNu reads a true concentration of benzene only between 0 and 600. Greater concentrations are read at a lower level than the true value. Consult the manufacturer's operating manual to determine the instrument's response to various chemicals.

2.2. Regulatory Limitations

A. Transport of calibration gas cylinders by passenger and cargo aircraft follow the U.S. Code of Federal Regulations, 49 CFR Parts 100-177. Benzene is a typical calibration gas included with a PID. Benzene is classified as a nonflammable gas, UN 1556, and the proper shipping name is compressed gas. It must be shipped in cargo aircraft only.

3. PROCEDURE

3.1. Associated Procedures

Information that applies to most field activities is provided in SOPs 1.1-1.10. In addition to the FSP or WP, those SOPs provide guidance that may supplement the information in this procedure. They should be consulted as necessary to obtain specific information about equipment and supplies; sample collection, preservation, packaging, and shipping; decontamination procedures; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.6	General Equipment Decontamination
6.1	Health and Safety Monitoring of Combustible Gas Levels

3.2. Preparation

3.2.1 Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Obtain appropriate permission for property access.

D. Assemble the equipment and supplies listed in Appendix 5.2. Perform the procedures described below.

1. Start-Up Procedure

   a. Before attaching the probe, check the function switch on the control panel to ensure that it is in the off position. Attach the probe by plugging it into the interface on the top of the readout module. Use care in aligning the prongs in the probe cord with the plug interface. Do not use excessive force.

   b. Turn the function switch to the battery check position. The needle on the meter should be within or above the green battery arc on the scale; if not, recharge the battery. If the red indicator light comes on, the battery needs recharging.

   c. Turn the function switch to any range setting. Look into the end of the probe to see if the lamp is on. If it is on, it will emit a purple glow. Do not stare into the probe any longer than 3 sec. Long-term exposure to UV light will damage the eyes. Also, listen for the hum of the fan motor.

   d. To zero the instrument, turn the function switch to the standby position and rotate the zero adjustment until the meter reads zero. A calibration gas is not needed because this is an electronic zero adjustment. If the span adjustment setting is changed after the zero is set, the zero should be rechecked and adjusted (if necessary). Wait 15 to 20 sec to ensure that the zero reading is stable. If necessary, readjust the zero.

2. Operational Check

   a. Follow the start-up procedure.

   b. With the instrument set on the 0 to 20 range, hold a solvent-based marker pen near the probe tip. If the meter deflects upscale, the instrument is working.

3. Calibration Procedure

   a. Follow the start-up procedure and the operational check.

   b. Set the function switch to the range setting for the concentration of the calibration gas.

   c. Remove the detector from the outer casing by loosening the screw on the bottom of the casing.

   d. Attach a regulator to a disposable cylinder of calibration gas. Connect the regulator to the probe of the PID with a piece of clean tygon tubing. Open the valve on the regulator.

e. After 15 sec, adjust the internal calibration screw until the meter reading equals the concentration of the calibration gas used. Consult the operating manual for the location of this screw.

f. If the PID does not start up, check out or calibrate properly and notify the equipment manager immediately. Under no circumstances should work requiring monitoring with a PID be performed without a properly functioning instrument.

g. Replace the detector in the outer casing.

h. Contact the carrier that will transport equipment and hazardous materials to obtain information on regulations and specifications.

3.2.2. Documentation

A. Obtain a logbook from the QA officer.

B. Record results of the equipment check in the logbook.

C. Obtain a sufficient number of the appropriate ER Program data collection forms (see INDEX TO SOPs).

D. Consult the ER Program data administrator for a current list of management codes, location IDs, and sample numbers used in the completion of data forms.

E. Record the calibration data on the Photoionization Detector Field Data form (Appendix 5.3). See Appendix 5.4 (Data Form Completion) for instructions.

3.2.3. Field

A. Follow the start-up procedure, operational check, and calibration check described in Section 3.2.1.D.

B. Set the function switch to the appropriate range. If the concentration of gases or vapors is unknown, set the function switch to the 0 to 20 ppm range; adjust the range if necessary.

C. With the exception of the probe's inlet and exhaust, wrap the PID in clear plastic to prevent it from becoming contaminated and to prevent water from getting inside the instrument in the event of precipitation.

3.3. Operation

3.3.1 Measuring organic vapor levels using the PID

A. As with any field instrument, accurate results depend on the operator's knowledge of the operator's manual. Follow the instructions in the operating manual explicitly in order to obtain accurate results.

B. Position the intake assembly close to the monitoring area because the low sampling rate allows for only very localized readings. Do not immerse the intake assembly in fluid under any circumstances.

C. While taking care not to permit the PID to be exposed to excessive moisture, dirt, or contamination, monitor the work activity as specified in the site Health and Safety Plan. Conduct the PID survey at a slow to moderate rate of speed and slowly sweep the intake assembly (the probe) from side to side.

D. During drilling activities, perform PID monitoring at every 5-ft interval downhole, at the headspace, and in the breathing zone. In addition, monitoring may be performed in the breathing zone during actual drilling when elevated organic vapor levels are encountered. When the activity being monitored does not involve drilling (like surface sampling), readings may only be recorded in the breathing zone. Refer to the site Health and Safety Plan for specific monitoring instructions.

E. Be prepared to evacuate the area if the preset alarm sounds. Operators using supplied air systems may not need to evacuate the work area, but they should frequently observe the levels indicated by the instrument.

F. Static voltage sources like power lines, radio transmissions, or transformers may interfere with measurements. See the operator's manual for a discussion of necessary considerations.

3.4. Postoperation

3.4.1. Field

A. When the activity is completed or at the end of the day, carefully clean the outside of the PID with a damp disposable towel to remove any visible dirt. Return the PID to a secure area and place on charge.

B. Ensure that all equipment is accounted for, decontaminated (see SOP 1.6, General Equipment Decontamination), and ready for shipment.

C. Make sure all survey or sampling locations are properly staked and the location ID is readily visible on the location stake.

3.4.2. Documentation

A. Record any uncompleted work (like additional monitoring) in the logbook.

B. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

C. Review data collection forms for completeness.

3.4.3. Office

A. Deliver original forms and logbooks to the document control officer (with copies to the site manager and files) for eventual delivery to the Department of Energy.

B. Inventory equipment and supplies. Repair or replace all broken or damaged equipment and charge the batteries. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

4. SOURCES

HNU Systems, Inc. 1986. "Instruction Manual for the Trace Gas Analyzer Model PI 101." Newton, Massachusetts.

CFR 49. 1985. Code of Federal Regulations, Title 49, U.S. Department of Transporatation, Parts 100-177. November 1, 1985. Washington, D.C.: U.S. Government Printing Office.

EPA. 1984. "Characterization of Hazardous Waste Sites--A Methods Manual: Volume II, Available Sampling Methods, Second Edition" U.S. Environmental Protection Agency report EPA-600/4-84-076. Environmental Monitoring Systems Laboratory, Office of Research and Development, Las Vegas, Nevada.

5. APPENDIXES

5.1. Comparison of the FID and PID

5.2. Equipment and Supplies Checklist

5.3. Photoionization Detector Field Data Form

5.4. Data Form Completion

APPENDIX 5.1

COMPARISON OF THE FID AND PID

	FID	PID
Response	Responds to many organic gases and vapors, especially low molecular weight hydrocarbons.	Responds to many organic and some inorganic gases and vapors, especially heavy hydrocarbons.
Application	In survey mode, detects total concentrations of gases and vapors. In GC mode, identifies and measures specific compounds.	In survey mode, detects total concentrations of gases and vapors. Some identification of compounds possible if GC column and standards are used.
Limitations	Does not respond to inorganic gases and vapors with a higher ionization potential than the flame detector. No temperature control.	Does not respond to methane or inorganic aliphatic chlorinated solvents. Does not respond properly in presence of water vapor (high humidity). Does not detect a compound if probe (lamp) has a lower energy than compound's ionization potential.
Calibration gas	Methane and others	Benzene (1,3-butadiene) and others
Ease of operation	Requires experience to interpret correctly, especially in GC mode.	Fairly easy to use and interpret. More difficult in the GC mode.
Detection limits	0.1 ppm (methane)	0.1 ppm (benzene), depends on lamp voltage.
Response time	2-3 sec (survey mode)	3 sec for 90% of total concentration

APPENDIX 5.1, Continued

	FID	PID
Maintenance	Periodically clean and inspect particle filters, valve rings, and burner chamber. Check calibration and pumping system for leaks. Recharge battery after each use.	Clean UV lamp frequently. Check calibration regularly. Recharge battery after each use.
Useful range	0-1000 ppm	0-2000 ppm
Service life	8 hrs; 3 hrs with strip chart recorder	10 hrs; 5 hrs with strip chart recorder

APPENDIX 5.2

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Photoionization detector (PID)

_____ Operating manual

_____ Probes: 9.5eV_____, 10.2eV_____, and 11.7eV_____

_____ Battery charger for PID

_____ Spare batteries

_____ Jeweler's screwdriver for adjustments

_____ Tygon tubing

_____ NBS traceable calibration gas (type)_____

_____ "T" valve for calibration

_____ Intake assembly extension

_____ Strap for carrying PID

_____ Teflon tubing for downhole measurements

_____ Plastic bags for protecting the PID from moisture and dirt

APPENDIX 5.3

PHOTOIONIZATION DETECTOR FIELD DATA FORM

PHOTOIONIZATION DETECTOR FIELD DATA

FACILITY CODE _____ **LOG DATE** _____

LOCATION ID _____ LOCATION TYPE _____

LOGGER CODE _____ FIELD REP _____

PHOTOIONZATION DETECTOR INSTRUMENT: MODEL _____

MANUFACTURER _____ DATE/TIME CALIBRATED _____

SERIAL NO _____ **ACCEPTANCE CODE** _____

CALIBRATION GASES:	
TYPE/CYLINDER ID NO	CONCENTRATION (PPM)/SPAN
1	1
2	2

COMMENTS _____

TIME (HH:MM)	SAMPLE ID	OBSERVED READING (ppm)					DRILLING DEPTH (FT)	COMMENTS
		DH	HS	BZ	D	OT		

ACCEPTANCE CODES: A–ACCEPTABLE R–RECONNAISSANCE U–UNACCEPTABLE N–NOT DETERMINED

LOCATION TYPES:
BH – BOREHOLE
SS – SOIL SAMPLE
SL – SURFACE LOCATION
SB – SAMPLE BOTTLE
TP – TEST PIT
WL – WELL
OT – OTHER

OBSERVED READING: D – DURING DRILLING (BZ)
DH – DOWNHOLE BZ – BREATHING ZONE
HS – HEADSPACE OT – OTHER

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS
PHO–040 (1/88)

FORM COMPLETED BY/DATE _____ TECHNICAL REVIEWER/DATE _____

APPENDIX 5.4

DATA FORM COMPLETION

Use a pen with black ink that is not water soluble (not a felt-tip pen). Make an entry in each blank. Where there is no data entry, enter UNK for Unknown, NA for Not Applicable, or ND for Not Done. If any procedure was not performed as prescribed, give the reason for the change or omission on the form. To change an entry, draw a single line through it, add the correct information above it, and initial the change.

PHOTOIONIZATION DETECTOR FIELD DATA FORM

1. Facility Code. Five-character code abbreviating the facility name where the program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

2. Log Date. The date the information recorded on the form was obtained in the format DD-MMM-YY (01-JAN-88).

3. Location ID. Four-character code assigned sequentially to each borehole, test pit, or surface location where physical, chemical, biological, radiological, and other measurements are taken.

4. Location Type. Two-character code identifying where the sample was taken. There is one location type for each location ID. Location types include those listed below.

   BH--Borehole

   TP--Test Pit

   SL--Surface Location

   WL--Well

   SB--Sample Bottle

   SS--Soil Sample

   OT--Other (explain)

5. Logger Code. Three-character or four-character code identifying the company responsible for collecting the information on the form.

6. Field Rep. The name of the field representative.

7. PID Model. Model of photoionization detector (PID) instrument.

8. PID Manufacturer. Manufacturer's name on the PID instrument used.

9. Date/Time Calibrated. Last day and time when the PID instrument was calibrated. Calibration should be performed daily.

10. Serial No. Serial No. of PID instrument.

11. Acceptance Code. One-character code assigned by the site manager.

12. Calibration Gases

    a) Type/Cylinder ID No. Name of the calibration gas and the identification number of the cylinder.

    b) Concentration (ppm)/span. Concentration of calibration gas in parts per million (ppm) and the span setting for calibration.

13. Comments. Any additional information.

14. Time (HH:MM). The time when a field measurement was taken in the 24-hr clock format of hours:minutes (for example, 08:37 for 8:37 a.m. and 19:12 for 7:12 p.m.). See the conversion table below.

Conversion Table

Conventional Time	24-Hr Time
1:00 a.m.	1:00
12:00 Noon	2:00
1:00 p.m.	13:00
2:00 p.m.	14:00
3:00 p.m.	15:00
4:00 p.m.	16:00
5:00 p.m.	17:00
6:00 p.m.	18:00
7:00 p.m.	19:00
8:00 p.m.	20:00
9:00 p.m.	21:00
10:00 p.m.	22:00
11:00 p.m.	23:00
12:00 Midnight	24:00

15. Sample ID. When samples are being taken during a PID monitoring, the identification number or code assigned to a particular sample (like 01) is correlated with the observed readings and appropriate drilling depth (if drilling is being performed). This is useful in selecting samples for analyses and in the correlation of laboratory data with PID measurements.

16. Observed Reading (ppm). PID reading at the respective location ID in the units indicated on the meter. When the calibration gas and the gas being measured for the environment are the same, the meter reads in parts per million (ppm) during drilling. Readings may be taken downhole, at the headspace, and in the breathing zone, and data should be recorded in the appropriately marked column.

17. Drilling Depth (Ft). PID monitoring is performed every 5 ft during drilling. The depth of the drilling is listed in feet and can be given as the most recent interval (like 5-10) or as the ending depth (like 10).

18. Comments. Any additional information, including the type of gas being measured if this determination can be made (for example, by labels on drums).

STANDARD OPERATING PROCEDURE 6.4

TOTAL ALPHA SURFACE CONTAMINATION MEASUREMENTS

1. PURPOSE

To provide guidance for determining levels of total surface alpha contamination on equipment, vehicles, and personnel that have been in contact with material that was potentially contaminated with alpha-emitting radionuclides.

2. DISCUSSION

The Field Sampling Plan (FSP) or Work Plan (WP) provides information on the scope of a given operation, related health and safety requirements, and the applicability of this procedure to the activities.

Equipment and vehicles must be monitored for contamination before release from radiologically controlled areas for unrestricted use. Levels of alpha contamination on equipment will be determined and compared to release criteria in draft DOE Order 5480.11 that are taken from the U.S. Nuclear Regulatory Commission Regulatory Guide 1.86. The limit for alpha activity applies to natural uranium, U-235, U-238, and associated decay products and is 5000 disintegrations per minute (dpm)/100 cm^2 averaged over 1 m^2 or 15,000 dpm/100 cm^2, not to exceed an area of 100 cm^2. Limits for other radionuclides apply to alpha, beta, and gamma radiations, and some are set below the detection limits for portable instruments. As a result, the applicable release criterion for alpha contamination is the minimum detectable activity above background as measured by a direct-reading alpha scintillation detector with a minimum $2\upsilon\tau\pi$ efficiency of 20%. This measurement typically equals about 350 dpm/100 cm^2. Equipment must be decontaminated to levels that are as low as reasonably achievable and below the applicable release criterion in all cases. Personnel must be monitored for contamination before leaving a controlled area and decontaminated to the lowest reasonably achievable levels.

High-voltage plateau curves and National Bureau of Standards (NBS) traceable source calibrations must be performed on the detector semiannually to ensure proper operation. Alpha detector counting efficiencies must be determined daily before using the instrument for contamination monitoring. The counting efficiency also must be determined following any adjustments or repairs on the instrument. The counting efficiency is used to convert instrument readings to a measure of activity in units of dpm per 100 cm^2.

3. PROCEDURE

3.1. Associated Procedures

Information that applies to most field activities is provided in SOPs 1.1-1.10. In addition to the FSP or WP, those SOPs provide guidance that may supplement the information in this procedure. They should be consulted as necessary to obtain specific information about equipment and supplies; sample collection, preservation, packaging, and shipping; decontamination procedures; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.6	General Equipment Decontamination
1.7	Sampling for Removable Alpha Contamination
6.11	Beta-Gamma Radiation Measurements Using a Geiger-Mueller Detector

3.2. **Preparation**

3.2.1. Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Obtain appropriate permission for property access.

D. Assemble the equipment and supplies listed in Appendix 5.1. Ensure the proper operation of all field equipment. Ensure that the alpha scintillator and the ratemeter/scaler have current calibrations.

3.2.2. Documentation

A. Obtain a logbook from the QA officer.

B. Record results of the equipment check in the logbook.

C. Obtain a sufficient number of the appropriate ER Program data collection forms (see INDEX TO SOPs).

D. Consult the ER Program data administrator for a current list of codes used in the completion of data forms.

3.2.3. Field

A. Complete the Daily Alpha Efficiency Check form (Appendix 5.2) by following instructions in Appendix 5.4, Data Form Completion.

B. Perform a daily 10-min background count and a 1-min alpha source count during use and record the results on the Daily Alpha Efficiency Check form. To perform a background count, place the probe on a clean, uncontaminated surface and record the number of counts accumulated over a period of 10 min. To perform a check source count, place the alpha source in the detector tray or against the detector surface and record the number of counts accumulated per minute (cpm).

C. Calculate the counting efficiency (E) using the formula shown below.

$$E = \frac{\text{(source cpm) - (background cpm)}}{\text{(source dpm)}}$$

D. While counting samples or performing surveys, the alpha probe may be contaminated, causing the background count rate to increase. If this is suspected, repeat the 10-min background count. If the background count rate is more than 50% above the average value, the detector should be cleaned.

3.3 Operation

3.3.1 Total Alpha Survey

A. Complete the Total Alpha Contamination Survey Data form by following instructions in Appendix 5.4, Data Form Completion.

B. List the items to be surveyed in the first column on the form. Items must be identified as specifically as possible with serial numbers, model numbers, license numbers, or other forms of unique descriptions. If the items to be surveyed need to be labeled with the assigned identification number, use an indelible marker, spray paint, or some type of permanent marker. Use a separate line of the form to list each area surveyed on the items.

C. List the surveyor's name, date of survey, and identification number of the monitoring instrument/detector.

D. Switch the instrument on, check the batteries for adequate power, and check the instrument for damage. Record the instrument daily background, efficiency, and calibration factor in the appropriate spaces. The instrument background and efficiency should be determined at least once during each operational day.

E. Monitor potentially contaminated surfaces by passing the probe face along each surface at a rate of 5 cm/sec or less. Without touching it, hold the probe face as close as possible to the surface being monitored and not more than 0.5 cm away. Be careful not to damage the Mylar face of the probe. Hold the probe steady at any area that appears to indicate an elevated reading. Record the highest reading for each separate area of the item monitored, listing a description of each area in the space provided under the first column.

F. When monitoring potentially contaminated skin and clothing surfaces, hold the probe face as close as possible to the surface being monitored, no more than 0.5 cm away. Move the probe along the surface at a rate of 5 cm/sec or less. At a minimum, monitor the areas listed below.

   1. Both sides of each hand

   2. Tops, sides, and bottoms of shoes or boots

   3. The torso of the body, both front and back

   4. All loose equipment (for example, papers, clipboards, and hand-carried tools)

G. Instrument readings will fluctuate during monitoring. Investigate any significant elevation of the meter reading by holding the meter in the suspected area. A noticeable elevation in the meter reading identifies contamination that may need to be removed.

H. Multiply each instrument reading (cpm) by the calibration factor to obtain the contamination level in dpm/100 cm^2.

I. If natural uranium, U-235, U-238, and associated decay products are the only contaminants present and the results of the survey for total alpha contamination are below the applicable release criterion for removable contamination (1000 dpm/100 cm^2), the item may be released without a survey for removable alpha contamination. If the total alpha activity is above the release criterion for removable contamination, perform the swipe or smear survey procedure. See SOP 1.7, Sampling for Removable Alpha Contamination.

J. Wash contaminated skin and equipment with water and soap. Contaminated clothing may be removed and laundered at an appropriate facility.

K. Give the survey results to the personnel responsible for releasing equipment. Equipment that fails to meet the release limits must undergo additional decontamination according to SOP 1.6, General Equipment Decontamination, and must be resurveyed.

3.4. Postoperation

3.4.1. Field

A. Turn the power off.

B. Ensure that all equipment is accounted for, decontaminated (see SOP 1.6, General Equipment Decontamination), and ready for shipment.

3.4.2. Documentation

A. Record any uncompleted work (like additional monitoring) in the logbook.

B. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

C. Review data collection forms for completeness.

3.4.3. Office

A. Deliver original forms and logbooks to the document control officer (with copies to the site manager and files) for eventual delivery to the Department of Energy.

B. Inventory equipment and supplies. Repair or replace all broken or damaged equipment. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

4. SOURCE

NRC. 1974. Regulatory Guide 1.86, "Termination of Operating Licenses for Nuclear Reactors." U. S. Regulatory Commission, Washington, D.C.: U.S. Government Printing Office.

5. APPENDIXES

5.1. Equipment and Supplies Checklist

5.2. Daily Alpha Efficiency Check Form

5.3. Total Alpha Contamination Survey Data Form

5.4. Data Form Completion

APPENDIX 5.1

EQUIPMENT AND SUPPLIES CHECKLIST

_____ Alpha scintillation probe (Ludlum 43-5 or the equivalent)

_____ Ratemeter/scaler (Ludlum Model 3 or the equivalent)

_____ Alpha check source (Am-241 or the equivalent)

_____ Data forms

_____ Voltage meter

_____ Hand-held calculator

_____ Tape measure

APPENDIX 5.2

DAILY ALPHA EFFICIENCY CHECK FORM

DAILY ALPHA EFFICIENCY CHECK

FACILITY CODE _____ FIELD REP _____

LOGGER CODE _____ ACCEPTANCE CODE _____

RATEMETER/SCALER:

MODEL NO _____ SERIAL NO _____ CALIBRATION DATE _____

WINDOW OUT THRESHOLD _____ HIGH VOLTAGE _____ BATTERY _____

ALPHA SCINTILLATION PROBE:

MODEL NO _____ SERIAL NO _____ CALIBRATION DATE _____

SOURCE:

SERIAL NO _____ ISOTOPE _____ ACTIVITY _____ DPM

LOG DATE	TIME (HH:MM)	COUNTING TIME (MIN)	BACKGROUND CPM	GROSS COUNTS	GROSS CPM	NET CPM	EFFICIENCY (NET CPM/DPM)

ACCEPTANCE CODES: A—ACCEPTABLE R—RECONNAISSANCE U—UNACCEPTABLE N—NOT DETERMINED

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS
DAE-103 (3/88)

_____ FORM COMPLETED BY/DATE

_____ TECHNICAL REVIEWER/DATE

APPENDIX 5.3

TOTAL ALPHA CONTAMINATION SURVEY DATA FORM

TOTAL ALPHA CONTAMINATION SURVEY DATA

FACILITY CODE _____ **LOG DATE** _____

LOGGER CODE _____ FIELD REP _____

RATEMETER/SCALER: **ACCEPTANCE CODE** _____

MODEL NO _____ SERIAL NO _____ CALIBRATION DATE _____

WINDOW OUT THRESHOLD _____ HIGH VOLTAGE _____ BATTERY _____

ALPHA PROBE:

MODEL NO _____ SERIAL NO _____ EFFICIENCY _____ CPM/DPM

PROBE FACE AREA[1] _____ CALIBRATION FACTOR[2] _____ $\frac{(DPM/100cm^2)}{CPM}$

ITEM SURVEYED (SPECIFY)	GROSS COUNTS	COUNT TIME	NET CPM	CONTAMINATION[3] LEVEL (IN DPM/100cm²)	MEETS RELEASE LIMIT (YES/NO)	SWIPE NECESSARY (YES/NO)

1 LUDLUM MODEL 43-1 PROBE FACE AREA = 83.0cm² LUDLUM MODEL 43-5 PROBE FACE AREA = 78.5 cm²

2 CALIBRATION FACTOR = (100/PROBEFACE AREA (cm²))/EFFECIENCY(CPM/DPM)

3 CONTAMINATION LEVEL = (NET CPM)(CALIBRATION FACTOR)

ACCEPTANCE CODES: A-ACCEPTABLE R-RECONNAISSANCE U-UNACCEPTABLE N-NOT DETERMINED

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS
TAC-128 (3/88)

_____ FORM COMPLETED BY/DATE

_____ TECHNICAL REVIEWER/DATE

APPENDIX 5.4

DATA FORM COMPLETION

Use a pen with black ink that is not water soluble (not a felt-tip pen). Make an entry in each blank. Where there is no data entry, enter UNK for Unknown, NA for Not Applicable, or ND for Not Done. If any procedure was not performed as prescribed, give the reason for the change or omission on the form. To change an entry, draw a single line through it, add the correct information above it, and initial the change.

DAILY ALPHA EFFICIENCY CHECK

1. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

2. Field Rep. The name of the field representative.

3. Logger Code. Three-character or four-character code identifying the company responsible for collecting the information recorded on the form.

4. Acceptance Code. One-character code assigned by the site manager.

5. Ratemeter/Scaler Model No. The model number of the ratemeter/scaler.

6. Ratemeter/Scaler Serial No. The serial number of the ratemeter/scaler.

7. Ratemeter/Scaler Calibration Date. The date when the ratemeter/scaler was last calibrated.

8. Window. The window is in the out position unless otherwise specified.

9. Threshold. The adjustment for the lower energy level of the discriminator shown on the calibration sticker.

10. High Voltage. The voltage that is applied to the alpha scintillation probe shown on the calibration sticker. This voltage is determined semiannually using a voltage plateau.

11. Battery. The battery voltage reading at the beginning of the measurement.

12. Alpha Scintillation Probe Model No. The model number of the alpha detector probe.

13. Alpha Scintillation Probe Serial No. The serial number of the alpha probe.

14. Alpha Scintillation Probe Calibration Date. The date the probe was last calibrated.

15. Source Serial No. The serial number of the radiation source.

16. Source Isotope. The identity of the radioactive isotope contained in the source given as element and mass number, like Am-241.

17. Source Activity. The activity of the radioactive source in disintegrations per minute (dpm). If the check source activity is given in microcuries (μCi), it can be converted to dpm using 1 μCi = 2.22×10^6 dpm.

18. Log Date. The date the information recorded on the form was obtained in the format DD-MMM-YY (01-JAN-88).

19. Time (HH:MM). The time the efficiency was determined using the 24-hr clock in the format hours:minutes.

20. Counting Time (Min). The time in minutes over which the scaler counts. Enter N/A if using a ratemeter.

21. Background cpm. The count rate with no source present.

22. Gross Counts. The number of pulses recorded by the scaler during the counting time. Enter N/A if using a ratemeter.

23. Gross cpm. The count rate with the source present given in pulses per minute.

24. Net cpm. Net counts per minute (cpm) equals gross cpm minus background cpm.

25. Efficiency (Net cpm/dpm). The ratio of the observed count rate to the true disintegration rate.

$$\text{Efficiency} = \frac{\text{Net cpm}}{\text{Source dpm}}$$

APPENDIX 5.4, Continued

TOTAL ALPHA CONTAMINATION SURVEY DATA FORM

1. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

2. Log Date. The date the information recorded on the form was obtained in the format DD-MMM-YY (01-JAN-88).

3. Logger Code. Three-character or four-character code identifying the company responsible for collecting the information recorded on the form.

4. Field Rep. The name of the field representative.

5. Acceptance Code. One-character code assigned by the site manager.

6. Ratemeter/Scaler Model No. The model number of the ratemeter/scaler.

7. Ratemeter/Scaler Serial No. The serial number of the ratemeter/scaler.

8. Ratemeter/Scaler Calibration Date. The date when the ratemeter/scaler was last calibrated.

9. Window. The window will be in the out position unless otherwise specified.

10. Threshold. The adjustment for the lower energy level of the discriminator shown on the calibration sticker.

11. High Voltage. The voltage applied to the alpha detector shown on the calibration sticker.

12. Battery. The battery voltage reading at the beginning of the measurement.

13. Alpha Probe Model No. The model number of the alpha detector probe.

14. Alpha Probe Serial No. The serial number of the alpha detector probe.

15. Alpha Probe Efficiency. The ratio of observed net count rate to the known disintegration rate of the check source from the Daily Alpha Efficiency Check form (Appendix 5.2).

16. Probe Face Area. The surface area of the Mylar window on the alpha scintillation detector in square cm. Values for Ludlum Models 43-1 and 43-5 are listed at the bottom of the form.

17. Calibration Factor. Factor that takes the detector efficiency and surface area into account to convert from cpm to dpm per 100 cm^2. The calibration factor in (dpm/100cm^2)/cpm equals (100/Probe Face Area in cm^2)/efficiency in cpm/dpm.

18. Item Surveyed (Specify). A description or identification number of the article surveyed. A separate line on the form is used to list and describe each area to be surveyed on the article.

19. Gross Counts. The total counts collected during the counting period.

20. Count Time. The time (in minutes) during which the counts were collected.

21. Net cpm. Gross count cpm minus background cpm.

22. Contamination Level (in dpm/100cm^2). This is calculated by multiplying the net cpm by the calibration factor.

    Contamination level = (Net cpm) (Calibration Factor)

23. Meets Release Limit (Yes/No). If the contamination level is greater than the applicable release limit, a no is written here. If the contamination level is less than the release limit, a yes is written here.

24. Swipe Necessary (Yes/No). If the total alpha contamination level exceeds the applicable removable contamination criteria, a swipe must be performed to determined the activity contribution of fixed and loose contamination.

STANDARD OPERATING PROCEDURE 6.7

NEAR SURFACE AND SOIL SAMPLE SCREENING FOR LOW-ENERGY GAMMA RADIATION USING THE FIDLER

1. PURPOSE

To describe the procedure in which a field instrument for the detection of low-energy radiation (FIDLER) is used to monitor surfaces and soil samples for the presence of low-energy gamma radiations that accompany some alpha emissions.

2. DISCUSSION

The Field Sampling Plan (FSP) or Work Plan (WP) provides information on the scope of specific operations, related health and safety requirements, and the applicability of this procedure.

The FIDLER uses a thin, 5-inch-diameter sodium iodide (NaI) crystal to detect low-energy radiation. The NaI crystal is optically coupled to a quartz light pipe and installed in a standard 5-inch probe housing that has an entrance window of beryllium. The principal use of this detector is for photons with energies less than 75 kilo-electron volts (keV).

The FIDLER probe can be used to scan individual samples for low-energy photons that normally accompany alpha emissions. Uranium is principally an alpha emitter. However, the radiation from its daughter products includes low-energy photons, principally L-orbital x rays from thorium. In the case of U-238, for example, two low-energy photons from Th-234 can be detected by the FIDLER. During most investigations, the instrument will be adjusted for maximum response for the 63 keV photon from Th-234 or the 60 keV photon of Am-241 formed by the beta decay of Pu-241.

Data from these measurements are presented as gross counts in the 60 keV energy range. The combination of this information with gross alpha counts of the same sample conducted according to SOP 6.5, Screening Soil Samples for Alpha Emitters, can be used to determine the presence or absence of radionuclides.

Included in this procedure are instructions for 1) initial instrument setup (voltage plateau), 2) daily response standardization using a sealed check source, 3) determination of the instrument's response to terrestrial background radiation, and 4) use of the instrument to scan ground surface areas and samples.

3. PROCEDURE

3.1. Associated Procedures

Information that applies to most field activities is provided in SOPs 1.1-1.10. In addition to the FSP or WP, those SOPs provide guidance that may supplement the information in this procedure. They should be consulted as necessary to obtain specific information about equipment and supplies; sample collection, preservation, packaging, and shipping; decontamination procedures; and documentation requirements. Procedures directly associated with this SOP are listed below.

SOP No.	SOP Title
1.1	General Instructions for Field Personnel
1.6	General Equipment Decontamination
6.5	Screening Soil Samples for Alpha Emitters

3.2. Preparation

3.2.1. Office

A. Review the FSP or WP and SOPs listed in Section 3.1.

B. Coordinate schedules/actions with the installation staff.

C. Assemble the equipment and supplies listed in Appendix 5.1. Ensure the proper operation of all equipment.

D. Obtain appropriate permission for property access.

E. Before the FIDLER can be used in the field, it is necessary to determine the correct operating voltage. This is accomplished by determining the plateau for background radiation and for radiation from a source of Am-241 (60 keV) in which the intensity is several times greater than background radiation levels. Determine the operating voltage once each week.

1. Inspect the FIDLER, the ratemeter/scaler, and interconnecting cable for obvious damage.

2. If no damage is observed, adjust the high voltage to 0 volts before connecting the FIDLER and the ratemeter/scaler.

3. Connect the FIDLER to the ratemeter/scaler in an area that has exhibited background radiation in previous measurements.

4. Turn the ratemeter/scaler selector switch to the high voltage setting and slowly adjust the high voltage to 900 volts (V).

5. Place the FIDLER in a position so that a series of 0.5-min counts can be made at various high-voltage settings.

6. Make three 0.5-min readings and record the voltage, count time, counts, and counts per minute in the columns on the Plateau Curve Record. When these three readings have been completed, adjust the high voltage upward 40 to 50 V. Repeat the three readings. Continue this procedure until three recordings have been made at a high-voltage setting of 1400 V. Do not exceed 1400 V.

7. Place the Am-241 source approximately 1 inch from the beryllium entrance window near the center of the probe and repeat the series of 0.5-min readings for high-voltage settings between 900 and 1400 V.

8. On the Plateau Curve Record, plot the FIDLER response as a function of high voltage. Between 1100 and 1300 V, there is little change in the

detector's response. This represents the operating plateau. Set the ratemeter/scaler high voltage at the midpoint of the plateau (typically 1200 V) for field operation. Minor fluctuations in the high voltage caused by environmental conditions or battery drain will have little effect on the count rate. Record this ratemeter/scaler voltage on the Plateau Curve Record form.

3.2.2. **Documentation**

A. Obtain a logbook from the QA officer.

B. Record results of the equipment check and information concerning the initial setup of the FIDLER in the logbook.

C. Obtain a sufficient number of the appropriate ER Program data collection forms (see INDEX TO SOPs).

D. Consult the ER Program data administrator for a current list of information management codes, location IDs, and sample numbers used in the completion of data forms.

E. Record information concerning the initial setup of the FIDLER on the Plateau Curve Record form (Appendix 5.2). Instructions for completing the form are in Appendix 5.5 (Data Form Completion).

3.2.3. **Field**

A. Daily Source Check

1. Establish a fixed geometry between the detector and the source so that their relative position is a matter of record and reproducible from one work period to the next.

2. Make five 1-min counts with the Am-241 source in its check position. Record each count in the logbook. Repeat this step twice daily when the instrument is in use or at any new locations.

3. Determine the average and standard deviation of the five 1-min counts. Record these values and three times the standard deviation in the logbook.

The average source count (X) = $\frac{x_1+x_2+x_3+x_4+x_5}{5}$

The standard deviation of the average source count (SDX) = $(X/5)^{1/2}$

B. Monitoring the performance of the FIDLER

1. Prepare a control chart at the beginning of the site characterization and use it each day to record the FIDLER's response to the Am-241 source.

2. On a piece of linear graph paper, place consecutive calendar dates on the x-axis. Define a range along the y-axis that includes the average FIDLER source count $\pm$ 3 standard deviations. Draw three horizontal lines that intersect the y-axis at these three points.

3. Determine the average count rate and plot this value on the control chart for that day. If the average count rate falls within the ± 3 standard deviations as defined above, the FIDLER may be used for field measurements. If the average count rate is not within this range, corrective action must be taken (see Section 3.2.3.C).

C. Corrective Action

1. Check the ratemeter/scaler calibration due date.

2. Check to see that the ratemeter/scaler high voltage is set at the plateau midpoint.

3. Turn off the ratemeter/scaler and disconnect the cable to the FIDLER. Clean the cable and chassis connectors with ethyl alcohol and let dry.

4. Reconnect the cable, turn on the ratemeter/scaler, and check the voltage setting.

5. Check the source-to-detector distance and make any necessary corrections.

6. Repeat the daily source check procedure. If the FIDLER response is within the range of the average count rate $\pm$ 3 standard deviations, the unit may be used. If the average count rate is still outside the control boundary, turn off the instrument and use a substitute instrument. Repeated failures will require attention by the manufacturer. The FIDLER is temperature sensitive and will not function correctly at temperatures below 32°F.

D. Determination of Background

1. In order to provide a statistical basis to determine if samples or locations are contaminated, calculate the mean background and standard deviation.

   a. In a location designated as background (not in the contaminated area) or using five soil samples collected from a background area, perform a series of five 1-minute counts with the FIDLER probe in the same position as it will be for screening samples in locations (see Sections 3.3.1 and 3.3.2).

   b. Determine the mean and standard deviation of the five background counts.

$$\text{Mean} = X = \frac{x_1+x_2+x_3+x_4+x_5}{5}$$

where

x_1, x_2, x_3, x_4, x_5 = the background counts

standard deviation = $SDX = (X/5)^{1/2}$

2. The contamination criterion used for further samples is the mean background plus 3 standard deviations (X + 3SDX). Samples with 1-min counts greater than this criterion should be considered contaminated.

3. Record this number or criterion on the FIDLER Soil Sample Screening Log (Appendix 5.3) or the FIDLER Measurement Data form (Appendix 5.4), depending on the type of screening to be performed.

3.3. Operation

3.3.1 Screening Soil Samples

A. Record the soil sample screening data on the FIDLER Soil Sample Screening Log form (Appendix 5.3) following the instructions in Appendix 5.5, Data Form Completion.

B. Place the soil samples in the petri dishes (fill to the top).

C. The steps for screening samples are described below.

1. Place the soil sample container (petri dish) in the counting shield.

2. Place the FIDLER probe in a counting jig inside the lead shield so that it is positioned above the center of a sample container holder. Adjust the height so that the FIDLER is one inch above the sample container. Close the shield door.

3. Turn the ratemeter/scaler selector switch to preset time and set the time for one min. Push the reset button to start and count.

4. Record the counts and the counting time on the FIDLER Soil Sample Screening Log form.

6. Remove the sample container, store it in sample archive, and repeat with additional samples.

3.3.2. Near-Surface Screening

A. The FIDLER Measurement Data form (Appendix 5.4) is completed as described in Appendix 5.5, Data Form Completion.

B. Determine that the FIDLER system has been checked and is ready for field measurements.

C. Refer to the FSP or WP for the areas to be scanned, the number of people required, time requirements, and special instructions.

D. Subdivide the grid block squares into square (or regular) grid blocks, the total area of which can be scanned in 0.5 or 1-min intervals. Use a calibrated measurement tape.

E. Drainage paths can only be scanned with a FIDLER if the area is free of standing or flowing water. If the area is dry, place grid stakes at regular intervals between the preexisting stakes.

F. Record scan measurements as integral counts over the area to be scanned. Pulses from the FIDLER will be summed for 0.5 min or 1 min. Turn the main selector switch of the ratemeter/scaler to either 0.5 or 1 min (as specified in the FSP or WP).

G. Begin scans at one corner of a grid block and progress in a serpentine pattern over the entire block, ending at the diagonally opposite corner of the block. Push the ratemeter/scaler reset button as the scan begins. With practice, the timed count should end upon reaching the opposite corner. During the scan, the field representative must listen to the audible signal from the meter. Although scanning speed should not be slowed when increased audible signals are heard, note the presence of apparent hot spots when recording the integrated count for the scan.

H. After completing the scan for an individual grid block, record the integrated count in its respective position on the FIDLER Measurement Data form (Appendix 5.4). Place a check mark above the recorded count to signify that anomalous or hot spot areas may exist on the basis of audible signals for that grid block.

I. Compare the location count rate to the contamination criterion (average background and 3 standard deviations). If the count rate is greater, the location is marked Y for further study or characterization.

3.4. Postoperation

3.4.1. Field

A. Ensure that all equipment is accounted for, decontaminated (see SOP 1.6, General Equipment Decontamination), and ready for shipment.

B. If necessary, make sure all survey or sampling locations are properly staked and the location ID is readily visible on the location stake.

C. Ensure that all radiological sources and standards have been stored in a locked area.

3.4.2. Documentation

A. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages.

B. Review data collection forms for completeness.

3.4.3. Office

A. Deliver original forms and logbooks to the document control officer (with copies to the site manager and files) for eventual delivery to the Department of Energy.

B. Inventory Equipment and supplies. Repair or replace all broken or damaged equipment. Replace expendable items. Return equipment to the equipment manager and report incidents of malfunction or damage.

4. SOURCE

Becron. 1985. "Technical Manual, Model: Labtech Scaler/Ratemeter/Analyzer with 2-Channel Option." Bicron Corporation, Newbury, Ohio.

5. APPENDIXES

5.1. Equipment and Supplies Checklist

5.2. Plateau Curve Record

5.3. FIDLER Soil Sample Screening Log Form

5.4. FIDLER Measurement Data Form

5.5. Data Form Completion

APPENDIX 5.1

EQUIPMENT AND SUPPLIES CHECKLIST

_____ FIDLER probe

_____ Ratemeter/scaler with voltage indicator and optional headphones

_____ Connector cable

_____ Lead shield and counting jig (optional)

_____ Plan view (site map) of the area to be surveyed that includes grid system coordinates

_____ Calibrated measurement tape or chain

_____ Am-241 source or the equivalent

_____ Hand-held calculator

_____ FIDLER heat shield, if available

_____ Linear graph paper for the performance control chart

_____ Petri dishes for soil samples

APPENDIX 5.2

PLATEAU CURVE RECORD

PLATEAU CURVE RECORD

PAGE 1 OF ____

FACILITY CODE _____ **LOG DATE** _____

LOGGER CODE _____ FIELD REP _____

RATEMETER/SCALER: **ACCEPTANCE CODE** _____

MODEL NO _____ SERIAL NO _____ CALIBRATION DATE _____

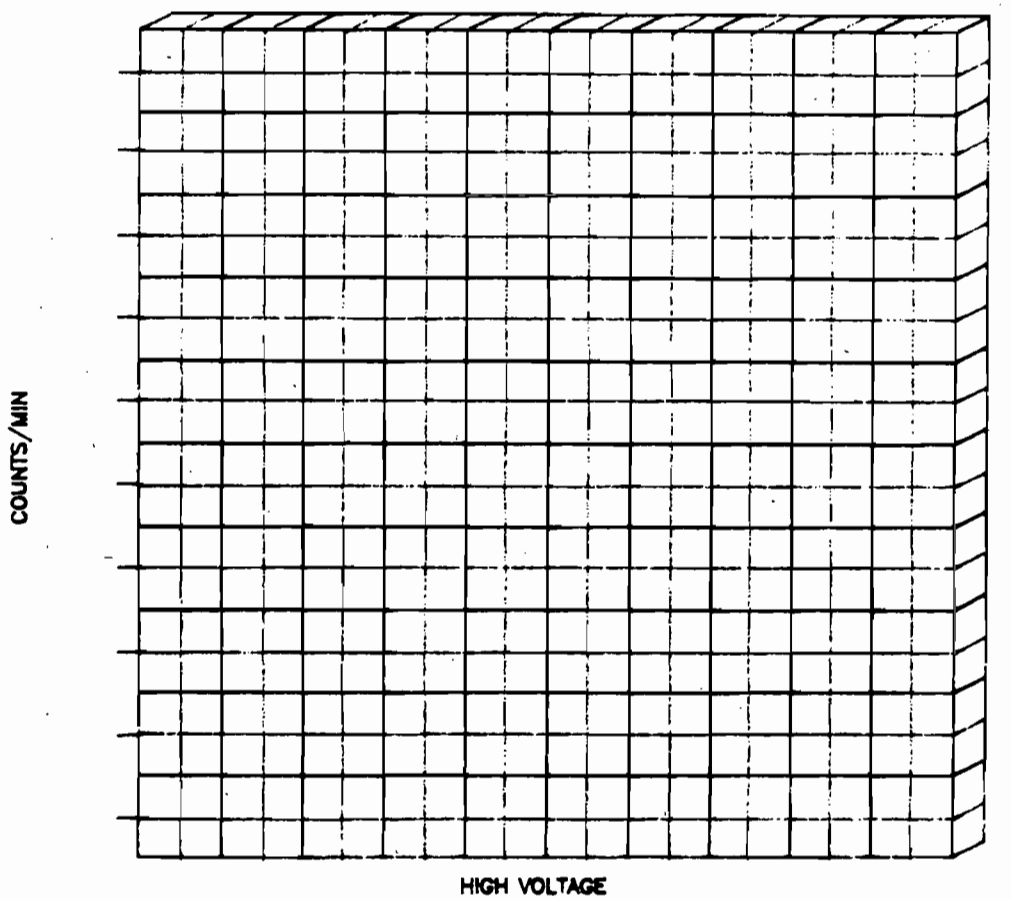
WINDOW _____ THRESHOLD _____ BATTERY _____

PROBE:

MODEL NO _____ SERIAL NO _____ HIGH VOLTAGE _____

CHECK SOURCE:

SERIAL NO _____ ISOTOPE _____ ACTIVITY _____



ACCEPTANCE CODES: A—ACCEPTABLE R—RECONNAISSANCE U—UNACCEPTABLE N—NOT DETERMINED

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS

PCR-102 (3/88)

FORM COMPLETED BY/DATE _____ TECHNICAL REVIEWER/DATE _____

APPENDIX 5.3

FIDLER SOIL SAMPLE SCREENING LOG

FIDLER SOIL SAMPLE SCREENING LOG PAGE 1 OF ____

FACILITY CODE _____ **LOG DATE** _____

LOGGER CODE _____ FIELD REP _____

RATEMETER/SCALER: **ACCEPTANCE CODE** _____

MODEL NO _____ SERIAL NO _____ CALIBRATION DATE _____

WINDOW _____ THRESHOLD _____ HIGH VOLTAGE _____ BATTERY _____

NaI PROBE :

MODEL NO _____ SERIAL NO _____ CALIBRATION DATE _____

CHECK SOURCE:

ISOTOPE _____ ACTIVITY _____ DPM SERIAL NO _____

SOURCE CHECK

METER READING _____ COUNTS/_____ MIN = _____ CPM

(WITHIN ± 3 STANDARD DEVIATIONS?[1] _____ (Y/N))

AVERAGE BACKGROUND + 3 STANDARD DEVIATIONS = _____ CPM

COMMENTS _____

LOCATION ID	**COORDINATES (FT)**		SAMPLE ID	COUNTS (=CPM)	CONTAMINATED YES/NO	ESTIMATED[2] pCi/gram
	NORTH	**EAST**				

1 SOURCE CHECK WITHIN ± 3 STANDARD DEVIATIONS (YES/NO), FROM THE CONTROL CHART

2 THE pCi/gram RADIONUCLIDE CONCENTRATION CAN BE ESTIMATED ONLY IF FIELD CALIBRATION FACTORS ARE AVAILABLE

ACCEPTANCE CODES: A–ACCEPTABLE R–RECONNAISSANCE U–UNACCEPTABLE N–NOT DETERMINED

COMPLETE BOLDED DATA FOR ENTRY IN TMS
FSS–125 (3/88)

_____ FORM COMPLETED BY/DATE

_____ TECHNICAL REVIEWER/DATE

APPENDIX 5.4

FIDLER MEASUREMENT DATA FORM

FIDLER MEASUREMENT DATA

PAGE 1 OF ___

FACILITY CODE _____ **LOG DATE** _____

LOGGER CODE _____ FIELD REP _____

RATEMETER/SCALER: **ACCEPTANCE CODE** _____

MODEL NO _____ SERIAL NO _____ CALIBRATION DATE _____

WINDOW _____ THRESHOLD _____ VOLTAGE _____ BATTERY _____

NaI PROBE:

MODEL NO _____ SERIAL NO _____ CALIBRATION DATE _____

CHECK SOURCE:

ISOTOPE _____ ACTIVITY _____ DPM SERIAL NO _____

SOURCE CHECK

METER READING _____ COUNTS/_____ MIN = _____ CPM

(WITHIN ± 3 STANDARD DEVIATIONS?[1] _____ (Y/N)

AVERAGE BACKGROUND + 3 STANDARD DEVIATIONS = _____

COMMENTS _____

LOCATION ID	**GRID BLOCK SCANNED (FROM N.E. CORNER)**		INTEGRATED COUNT (CPM)	AUDIBLE (Y)es or (N)o	CONTAMINATED (Y)es or (N)o
	NORTH	**EAST**			

1 SOURCE CHECK WITH ± 3 STANDARD DEVIATIONS, (YES/NO) FROM THE CONTROL CHART

ACCEPTANCE CODES: A—ACCEPTABLE R—RECONNAISSANCE U—UNACCEPTABLE N—NOT DETERMINED

COMPLETE BOLDED DATA FOR ENTRY INTO TIMS
FMD-120 (3/88)

_____ FORM COMPLETED BY/DATE

_____ TECHNICAL REVIEWER/DATE

APPENDIX 5.5

DATA FORM COMPLETION

Use a pen with black ink that is not water soluble (not a felt-tip pen). Make an entry in each blank. Where there is no data entry, enter UNK for Unknown, NA for Not Applicable, or ND for Not Done. If any procedure was not performed as prescribed, give the reason for the change or omission on the form. To change an entry, draw a single line through it, add the correct information above it, and initial the change.

PLATEAU CURVE RECORD

1. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

2. Log Date. The date the information recorded on the form was obtained in the format DD-MMM-YY (01-JAN-88).

3. Logger Code. Three-character or four-character code identifying the company responsible for collecting the information recorded on the form.

4. Field Rep. The name of the field representative.

5. Acceptance Code. One-character code assigned by the site manager.

6. Ratemeter/Scaler Model No. The model number of the ratemeter/scaler.

7. Ratemeter/Scaler Serial No. The serial number of the ratemeter/scaler.

8. Ratemeter/Scaler Calibration Date. The date when the ratemeter/scaler was last calibrated.

9. Window. The window will be in the out position unless otherwise specified.

10. Threshold. The adjustment for the lower energy level of the discriminator shown on the calibration sticker.

11. Battery. The battery voltage reading at the beginning of the measurement.

12. Probe Model Number. The model number of the FIDLER probe.

13. Probe Serial Number. The serial number of the FIDLER probe.

14. Probe High Voltage. The final voltage setting that will be applied to the detector, as determined by the Plateau Curve procedure. For the FIDLER, the operating voltage should be about 1200V.

15. Check Source Serial No. The serial number of the radiation check source.

16. Check Source Isotope. The radioactive isotope contained in the check source given as element and mass number, like Am-241.

17. Check Source Activity. The activity of the radioactive check source in disintegrations per minute (dpm). An activity given in microcuries (μCi) can be converted to dpm using 2.22×10^6 dpm = 1 μCi.

18. Counts/Min. The count rate given in counts per minute (cpm).

19. High Voltage. The voltage applied to the detector during the collection of the associated counts.

APPENDIX 5.5, Continued

FIDLER SOIL SAMPLE SCREENING LOG

1. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

2. Log Date. The date the information recorded on the form was obtained in the format DD-MMM-YY (01-JAN-88).

3. Logger Code. Three-character or four-character code identifying the company responsible for collecting the information recorded on the form.

4. Field Rep. The name of the field representative.

5. Acceptance Code. One-character code assigned by the site manager.

6. Ratemeter/Scaler Model No. The model number of the ratemeter/scaler.

7. Ratemeter/Scaler Serial No. The serial number of the ratemeter/scaler.

8. Ratemeter/Scaler Calibration Date. The date when the ratemeter/scaler was last calibrated.

9. Window. The window will be in the out position unless otherwise specified.

10. Threshold. The adjustment for the lower energy level of the discriminator shown on the calibration sticker.

11. High Voltage. The voltage setting that is applied to the probe as determined by the Plateau Curve procedure.

12. Battery. The battery voltage reading at the beginning of the measurement.

13. NaI Probe Model No. The model number of the FIDLER probe.

14. NaI Probe Serial No. The serial number of the FIDLER probe.

15. NaI Probe Calibration Date. The date when the FIDLER probe was last calibrated.

16. Check Source Isotope. The radioactive isotope that the source contains, given as element and mass number, like Am-241.

17. Check Source Activity. The activity of the check source, measured in disintegrations per minute (dpm). An activity given in microcuries can be converted to dpm using 2.22×10^6 dpm = 1 μCi.

18. Check Source Serial No. The serial number of the check source.

19. Source Check Meter Reading. The results of a count on a check source. The check source data consists of three fields: total counts, counting time in minutes, and count rate in counts per minute (cpm).

20. Within ± 3 Standard Deviations (Y/N). This field describes the performance of the FIDLER, from the control chart in 3.2.3.B., Field Preparation.

21. Average Background + 3 Standard Deviations (cpm). This field gives the average background count rate + 3 standard deviations of the average count rate. It is used as a contamination criterion. Count rates greater than this are considered contaminated. Count time equals 1 min.

    X = Average or Mean Background = $\frac{x_1+x_2+x_3 \ldots x_n}{N}$

    where

    x_1, x_2, x_3 etc = individual background counts

    N = the number of counts taken

    SDX = standard deviation of the average background = $(X/N)^{1/2}$

22. Comments. Any additional information.

23. Location ID. Four-character code assigned sequentially to each borehole, test pit, or surface location where physical, chemical, biological, radiological, and other measurements are taken.

24. Coordinates (Ft). The location of the FIDLER measurement on the survey grid in units of feet. The two coordinate fields are in the format north and east.

25. Sample ID. The identifying code or number given to the sample.

26. Counts (=cpm). The number of counts registering on the FIDLER meter during the 1-min counting period.

27. Contaminated (Yes/No). If the counts per 1 min obtained are greater than the average background plus 3 standard deviations, the sample is considered contaminated. Enter Yes or Y if contaminated and No or N if not contaminated.

28. Estimated pCi/gram. If calibration factors are available for the specific site under investigation, the FIDLER cpm can be converted to a pCi/gram concentration. Enter N/A if these factors are not available.

APPENDIX 5.5, Continued

FIDLER MEASUREMENT DATA FORM

1. Facility Code. Five-character code abbreviating the facility name where program activity is being conducted. The first three characters indicate the facility, and the remaining two numbers designate the specific site within the facility.

2. Log Date. The date the information recorded on the form was obtained in the format DD-MMM-YY (01-JAN-88).

3. Logger Code. Three-character or four-character code identifying the company responsible for collecting the information recorded on the form.

4. Field Rep. The name of the field representative.

5. Acceptance Code. One-character code assigned by the site manager.

6. Ratemeter/Scaler Model No. The model number of the ratemeter/scaler.

7. Ratemeter/Scaler Serial No. The serial number of the ratemeter/scaler.

8. Ratemeter/Scaler Calibration Date. The date when the ratemeter/scaler was last calibrated.

9. Window. The window will be in the out position unless otherwise specified.

10. Threshold. The adjustment for the lower energy level of the discriminator shown on the calibration sticker.

11. Voltage. The voltage setting that is applied to the probe, as determined by the Plateau Curve procedure.

12. Battery. The battery voltage reading at the beginning of the measurement.

13. NaI Probe Model No. The model number of the FIDLER probe.

14. NaI Probe Serial No. The serial number of the FIDLER probe.

15. NaI Probe Calibration Date. The date when the FIDLER probe was last calibrated.

16. Check Source Isotope. The radioactive isotope that the source contains, given as element and mass number, like Am-241.

17. Check Source Activity. The activity of the check source, measured in disintegrations per minute (dpm). An activity given in microcuries (μCi) can be converted to dpm using 2.22×10^6 dpm = 1 μCi.

18. Check Source Serial No. The serial number of the check source.

19. Source Check Meter Reading. The results of a count on a check source. Check source data consists of three fields: total counts, count time in minutes, and count rate in cpm.

20. Within ± 3 Standard Deviations. This field describes the performance of the FIDLER, from the control chart in Section 3.2.3.B, Field Preparation. Enter Y (yes) or N (no).

21. Average Background + 3 Standard Deviations. This field gives the average background count rate (cpm) + 3 standard deviations of the average. It is used as a contamination criterion; count rates greater than this number indicate contaminated areas.

$$X = \text{Average or Mean Background} = \frac{x_1+x_2+x_3 \ldots x_n}{N}$$

where

x_1, x_2, x_3, etc. = individual background counts

N = the number of counts taken

SDX = standard deviation of the average background = $(X/N)^{1/2}$

22. Comments. Any additional information.

23. Location ID. Four-character code assigned sequentially to each borehole, test pit, or surface location where physical, chemical, biological, radiological, and other measurements are taken.

24. Grid Block Scanned (From N.E. Corner). The location of the grid block that was scanned, identified by the survey coordinates of the northeast corner relative to the survey grid in units of feet. There are two fields in the coordinate description: north and east.

25. Integrated Count (cpm). The count rate in counts per minute obtained from the walkover scan of the grid.

26. Audible. If an audible anomaly was noted, enter YES; if no audible anomaly was detected, enter NO.

27. Contaminated. If the count rate recorded is greater than the average background plus 3 standard deviations, the location is considered contaminated (Y). If it is less, it is not considered contaminated (N).

APPENDIX F

FIDLER SCREENING OF SOIL SAMPLES

PROGRAM	MANUAL NUMBER	SHEET	OPERATION	ISSUE
ENVIRONMENTAL ANALYTICAL PROCEDURES	PD-80030	1 of 10	1355	1

OPERATION TITLE	EFFECTIVITY DATE
Plutonium and Thorium in Soil Using the Bicron Detector (Soil Screening)	1-1-88

SUBMITTED BY	ECN(S) INCORPORATED
C. T. Bishop, D. G. Draper, J. Y. Jarvis, T. J. Pearson	890905MD-C

CLASSIFICATION	CLASSIFIER	SUPERVISOR	SECTION MANAGER
Unclassified		Carl T. Bishop	

1. SCOPE

This procedure is a rapid screening method for the measurement of plutonium and thorium in soil. It involves the measurement of x-rays or gamma rays from these two radioactive elements with a thin sodium iodide detector. Plutonium can be measured down to concentrations of about 25 pCi/g and thorium can be measured to about 5 pCi/g.

2. SUMMARY

Plutonium-238 (or -239) is measured by detecting the 17 keV plutonium x-ray with a 1/16-inch thick, 5-inch diameter NaI (Tl) detector manufactured by the Bicron Corporation. Thorium-232 is measured by counting lead x-rays at an energy of 88 keV. Soil samples are counted in disposable polyethylene containers 4 inches in diameter and 2-5/8 inches high.

Standards were prepared by adding known amounts of plutonium-238 or thorium-232 standard solutions to a soil that had been found to be free of these two isotopes. The counting system is calibrated on a monthly basis, but a standard is counted daily to evaluate the accuracy of the counting system, and to give assurance that the system is not out of calibration.

A multichannel analyzer is used to accumulate counts in the region of interest for both plutonium-238 or thorium-232. A sample is normally counted for 400 seconds, but can be counted for a longer or shorter period of time depending on the activity present in the sample being measured.

2. Continued

A computer program has been prepared to calculate the plutonium and thorium concentrations in picocuries per gram based on the sample counts and the data on the standards.

If a high plutonium concentration is observed, reliable thorium concentrations can also be observed. However, if the thorium concentration is high, the plutonium concentration computed will not be reliable since the x-ray detected for thorium is higher in energy than the plutonium x-ray and its Compton distribution will interfere with the plutonium measurement. In general any high energy (> 17 keV) x-ray or gamma ray will interfere with the plutonium peak because of the Compton distribution.

3. SPECIAL APPARATUS

- Three Davidson multichannel analyzers, Model 4106
- Three Bicron scintillation detectors
- Three high voltage power supplies
- Three Bicron pre-amplifiers
- Three amplifiers
- One IBM/PC with printer
- Assorted soil standards in varing concentration of plutonium and thorium along with background soils.

4. REAGENTS

Does not apply.

5. SOIL SCREENING PROCEDURE

5.1. Gamma Pulse Height Analysis

5.1.1. If the sample container is less than half full, weigh it.

5.1.2. If the weight is less than 250 grams, record the weight.

5.1.3. Place the sample over the center of the detector and place the lid on top of the shielded well.

5.1.4. Check to see that the multichannel analyzer (MCA) parameters are properly set. (See Section 5.3 in this procedure.)

5.1.5. Press the display button.

5.1.6. Move RESET DATA/TIME switch up and down (this resets the time and date).

5.1.7. Start the count by pressing the accumulate button (ACCUM.). Normally the count time is set at 400 seconds.

5.1.8. After counts have been accumulated, display the data by pressing the DISPLAY button.

5.1.9. Adjust the marker location knob to give the channels corresponding to the region of interest for plutonium (example: channels 71 - 170). This region of interest has been determined from calibration with the plutonium standard.

5.1.10. Record the number of counts as displayed on the screen corresponding to the plutonium activity.

5.1.11. Continue to turn the Marker Location Knob counter-clockwise to give the channels corresponding to the region of interest for thorium (example: channels 279 - 338). This region of interest has been determined from calibration with a thorium standard.

5.1.12. Record the number of the counts as displayed on the screen corresponding to the thorium activity.

5.2. Using PC for Calculating and Logging System (C&L).

5.2.1. Hardware configuration:

IBM or compatible PC
640K of memory
IBM compatible printer
10M or higher hard drive

5.2.2. Software requirements:

dBase III Plus
DOS 2.1 or Higher
Soil menu program to run in dBase environment

5.2.3. Move switch on right side of PC up. (This will boot up PC.)

5.2.4. When menu appears on screen, select key associated with Soil Data Management System.

5.2.5. The Soil program (Soil menu) will appear. Select [D] Input Well (A,B, & C) Data.

5.2.6. The next screen asks you to [enter cal date you are looking for]. The date that you see is the last known calibration date. Press return.

5.2.7. The next screen that appears is [Soil data].

Enter sample type:

CONT....: constructions
SM.....: SM building
PP.....: PP building
HS3.....: hot spot No. 3
Misc....: miscellaneous

Enter sample condition:

This is always wet unless you process it through Bldg. 30 microwave.

Sampler initials:

This information is obtained from sample label.

Enter collection date:

Enter the date the sample was collected.

Enter date screen:

This is always the date on which you put the sample result into the computer.

5.2.8. Input gross data from MCA:

Enter gross cts Pu-238:

Enter the gross count from the (MCA) (ROI) for plutonium

Enter gross cts Th-232:

Enter the gross count from the (MCA) (ROI) for thorium

5.2.8. Continued

Enter sample weight in grams:
(Use 300 g when sample is not weighed.)
Enter the sample weight

Enter sample grid & location:
Enter the remarks from the container

Was the sample counted in A, B, or C:
Enter the well where sample was counted

NOTE: At the bottom of the screen will appear the calculated results for this sample. You may continue from here if the following conditions are the same: sample type, condition, sampler, and collection date.

5.2.9. After this you will see the sample renumbered after returning to the Soil Menu program's main menu.

5.2.10. To print the report, press [E]. Select the report best suited for your needs.

5.3. Calibration of Multichannel Analyzer (MCA).

5.3.1. Move the memory selector knob to position 1 (at 12:00 on the dial). This displays all 4096 channels.

5.3.2. Move ROI/ALL switch to ALL.

5.3.3. Clear all data from the channels by pressing Reset DATA/TIME up and down.

5.3.4. Press ROI ENTER/RESET switch down to reset

5.3.5 Set the analyzer to the following:

HV.....	+ 1100 volts
Conversion gain....	4
Mode.....	T
Offset.....	off
ULD.....	10
LDL.....	0
Gain.....	variable
Time.....	(400) variable
Memory location....	1-8 (512 channels)
Data.....	add
Time.....	add
ROI.....	variable
PMT-AMP.....	AMP
CNC-F-ANC.....	F

5.3.6. Move ROI/ALL switch to ROI (Region of Interest).

5.3.7. Place standard No. 1 into a detector well.

5.3.8. Move the memory selector knob to position 1 of 8 (the memory selector knob must correspond with the standard ID).

5.3.9. Press the acquire button and accumulate spectrum for a preset time. (Repeat step 5.3.7 - 5.3.9 until all the standards have been counted.)

5.3.10. Move the memory location knob to 5 of 8.

5.3.11. Set the ROI/ALL switch to ALL.

5.3.12. Move the marker location knob right or left, to find the peak, and place the CRT highlight over the entire peak area of interest.

5.3.13. Press the ROI ENTER/RESET switch to enter.

5.3.14. Move the ROI/ALL switch to ROI.

5.3.15. Move the marker location knob until the ROI channel and gross counts appear on the top left corner of the screen.

5.3.16. The number to the right of the "=" is the gross count of the standard.

5.3.17. Move the memory location knob to 4 of 8.

5.3.18. Set ROI/ALL switch to ALL.

5.3.19. Position the marker location knob on the entire peak of interest.

5.3.20. Move ROI ENTER/RESET/ switch to ENTER.

5.3.21. Move ROI/ALL switch to ROI.

5.3.22. Move the marker location knob until the ROI channel and gross counts appear on the top left corner of the screen.

5.3.23. This is the ROI for the plutonium standards.

5.3.24. Move the memory location knob to the next location(s).

5.3.25. Repeat steps 5.3.18 - 5.3.22.

5.3.26. Move the memory location knob to 1 of 8.

5.3.27. Use the ROI channel previous setting (highlighted area) to set up 1 of 8, unknown sample region.

6. CALCULATIONS

6.1 Calculate the plutonium and thorium efficiencies and thorium cross-over from the following equations:

$$EFF(Pu) = \frac{C(PuROI) - B(PuROI)}{S(Pu) \times CT}$$

$$EFF(Th) = \frac{C(ThROI) - B(ThROI)}{S(Th) \times CT}$$

$$Th(CO) = \frac{C(PuROI) - B(PuROI)}{C(ThROI) - B(ThROI)}$$

Where: EFF(Pu) and EFF(Th) are plutonium and thorium efficiencies, respectively.

C(PuROI) and C(ThROI) are counts in plutonium and thorium regions of interest, respectively.

B(PuROI) and B(ThROI) are counts in a background soil at the plutonium and thorium regions of interest, respectively.

S(Pu) and S(Th) are the concentrations of plutonium and thorium in pCi/g of each standard, respectively.

CT is the counting time.

Th(CO) - The thorium crossover is the amount of counts observed in the plutonium region of interest from the thorium standard and varies with sample weight (self-absorption).

6.2 Calculate the plutonium and thorium concentrations in the unknown sample:

$$Pu(pCi/g) = \frac{[C(PuROI) - B(PuROI)] - [C(ThROI \times Th(Co)]}{EFF(Pu) \times CT}$$

$$Th(pCi/g) = \frac{C(ThROI) - B(ThROI)}{EFF(Th) \times CT \times EC}$$

Where: Pu(pCi/g) and Th(pCi/g) are the concentrations of plutonium and thorium in pCi/g in the unknown sample. Other symbols are given in Step 6.1.

EC is the correction of the thorium efficiency due to different sample weights.

7. REFERENCES

7.1 Operating Manual, Bicron Scintillation Detectors (1984). Bicron Corporation Newbury, Ohio.

7.2 Casella, V. R., Environmental Science Report, MLM-MU-85-63-0001 (Fourth Quarter, 1984) pp 14-15.

7.3 Stought, R. L., D. A. Edling, and D. G. Draper, The Mound Site Survey Project for the Characterization of Radioactive Materials in Site Soils, MLM-3517 (May 16, 1988) Appendix A-3, Soil Screening of Samples, pp 82-96.

7.4 Umbarger, C. J., "Soil Monitoring Instrumentation", CONF-791234, Environmental Decontamination Workshop, Oak Ridge, TN, 1979, pp 41-45.

7.5 Umbarger, C. J. and M. A. Wolf, "A Battery Operated Portable Phoswich Detector for Field Monitoring of Low Levels of Transuranic Contaminants," Nucl. Inst. and Meth. 155, p. 453 (1978).

APPENDIX G

USATHAMA PROCEDURES FOR EXPLOSIVES IN SOILS

INTERNATIONAL TECHNOLOGY CORPORATION

November 11, 1987

Mr. Les Brown
USATHAMA
AMXPH/A5/Building 4435
Aberdeen Proving Ground, MD 21010-5401

Ref. No.: IT/COE-308002-L-337

Dear Mr. Brown:

Enclosed are four copies of the certification packages for ten explosives by HPLC at one-half the original levels.

If you have any questions, please call Doug Peery or myself at (615) 690-3211.

Sincerely,

R. Alan Doughty

R. Alan Doughty, Ph.D.
Director, Field Analytical and Sampling

RAD/jch

Enclosures

cc: Project Files (308003)
COE File (B-1.12)

Regional Office
312 Directors Drive • Knoxville Tennessee 37923 • 615-690-3211

INTERNATIONAL
TECHNOLOGY
CORPORATION

CERTIFICATION PERFORMANCE

DATA PACKAGE

TEN EXPLOSIVES

BY

HPLC

Regional Office
312 Directors Drive • Knoxville, Tennessee 37923 • 615-690-3211

CERTIFICATION PERFORMANCE DATA PACKAGE CHECKLIST
(ONE FOR EACH METHOD)

Contract/Task No. DACA 45-87-C-0103 303002.12.01.04 Installation Cornhusker Army Ammunition Plant

The following items are included in this Certification Performance Data Package for HMX;RDX;NB;1,3-DNB;1,3,5-TNB; TNT; 2,4-DNT;2,6-DNT; 2,A,4,6-DNT; Tetryl (Analyte(s)) in Soil/Ash (Matrix).

Yes Method written up in USATHAMA format.

Calibration:

Yes Calibration curves from days of certification (plot of raw data).

Yes Documentation for Lack of Fit and Zero Intercept Tests.

Certification:

Yes Data summary - target versus found.

Yes Reporting limit, precision and accuracy calculations.

Yes Reporting limit plot.

Yes Data summary - statistics.

Yes Lack of Fit and Zero Intercept Tests.

Yes Chromatograms from each day of certification analytes for the highest tested concentration and for the tested concentration closest to calculated reporting limit.

N/A Spectra for all target analytes (if applicable).

N/A Identity and purity determinations for off-the-shelf reference materials, either purchased by the Contractor or supplied by the Central QA Laboratory.

Paul [illegible]
Contractor QAC

11/10/87
Date

Ten Explosives by High Performance Liquid Chromatography

I. SUMMARY

A. Analytes

The following nitro-explosives are quantified:

HMX ..... Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine Cyclotetramethylenetetranitramine
RDX ..... Hexahydro-1,3,5-trinitro-s-triazine Cyclotetramethylenetetranitramine
NB ..... Nitrobenzene
1,3-DNB 1,3-dinitrobenzene
1,3,5-TNB .. 1,3,5-trinitrobenzene
2,4-DNT 2,4-dinitrotoluene
2,6-DNT 2,6-dinitrotoluene
TNT 2,4,6-trinitrotoluene
2A,4,6-DNT . 2-amino-4,6-dinitrotoluene
Tetryl A-methyl-N,2,4,6-tetranitroanaline

B. Matrix

This procedure is applicable to the analysis of nitro-explosive compounds in soil and ash.

C. General Method

A fixed amount of acetonitrile is pipetted into a clean vial containing an accurately weighed aliquot of representative soil. The mixture is thoroughly mixed and filtered. The resulting filtrate is analyzed by high performance liquid chromatography (HPLC) and the concentration, in µg/g of soil, for each nitro-explosive is calculated.

II. APPLICATION

A. Calibration Range

HMX	0.5 to 10 µg/ml	Tetryl	0.25 to 5 µg/ml
RDX	0.5 to 10 µg/ml	TNT	0.25 to 5 µg/ml
1,3,5-TNB ..	0.25 to 5 µg/ml	2A,4,6-DNT ..	0.25 to 5 µg/ml
1,3-DNB	0.25 to 5 µg/ml	2,6-DNT	0.25 to 5 µg/ml
NB	0.25 to 5 µg/ml	2,4-DNT	0.05 to 1 µg/ml

Test Concentration Range

HMX	2.43 to 48.7 µg/g	Tetryl	1.20 to 24.0 µg/g
RDX	2.46 to 49.3 µg/g	TNT	1.23 to 24.6 µg/g
1,3,5-TNB ..	1.25 to 24.9 µg/g	2A,4,6-DNT ..	1.24 to 24.8 µg/g
1,3-DNB	1.19 to 23.8 µg/g	2,6-DNT	1.26 to 25.2 µg/g
NB	1.25 to 25.0 µg/g	2,4-DNT	0.235 to 4.71 µg/g

B. Sensitivity

HMX ..... 0.0735 ng/kilo count
RDX ..... 0.0611 ng/kilo count
1,3,5-TNB ... 0.0275 ng/kilo count
1,3-DNB ..... 0.0207 ng/kilo count
NB ..... 0.0286 ng/kilo count
Tetryl ..... 0.0517 ng/kilo count
2A,4,6-DNT .. 0.0286 ng/kilo count
2,6-DNT ..... 0.0233 ng/kilo count
2,4-DNT ..... 0.0396 ng/kilo count
TNT ..... 0.0178 ng/kilo count

C. Reporting Limits

Compound in (µg/gram)

HMX 4.08
RDX 3.25
1,3,5-TNB ... 1.51
1,3-DNB 1.36
NB 1.38
Tetryl 10.85
TNT 1.47
2A,4,6-DNT .. 2.23
2,6-DNT 1.44
2,4-DNT 0.329

D. Interferences

1. Any compound that is extracted from the soil with similar retention time and absorbance will cause a high value.

2. Tetryl will break down in the presence of water.

E. Analysis Rate

Approximately eighteen samples could be analyzed in an 8-hour time period. This does not include QA/QC samples.

F. Safety Information

All compounds are potentially explosive. Caution must be exercised in the following areas:

1. Keep all materials cooled and properly stored.

2. Some materials in their pure form need to be stored in water.

3. Do not allow solvents to evaporate.

III. APPARATUS AND CHEMICALS

A. Glassware/Hardware

100 5-ml glass serum vials + teflon septa + aluminum crimp tops
100 12-ml glass vials with teflon lined screw caps
20 10-ul, 20-ul, 100-ul and 500-ul glass syringes
6 1.0-ml, 2.0-ml, 5.0-ml volumetric pipets
100 1.8-ml autosampler vials with screw caps and teflon septa
100 Lidex LID-X filter syringes with 0.45 μ glass microfiber filter
1 Sonicator
20 Class A 5-ml and 1-ml volumetric flasks

B. Equipment and Operation

1. Analytical System

Perkin Elmer Series 4 HPLC metering and pumping system
Perkin Elmer ISS 100 auto-injector
Perkin Elmer LC95 spectrophotometric detector
Perkin Elmer LCI 100 integrator

2. Chromatographic System

LC 18 guard column 2 cm x 4.6 mm 5-μ
LC 18 column (25 cm long by 4.6 mm inside diameter)

3. Analytical Conditions

a. Auto-injector

15 μl injection
50 μl injection loop

b. Metering and Pumping

	Time (min)	Flow (ml/min)	% Acetonitrile	% Methanol	% Water
Equilibrium	0.5	1.0	15	40	45
1.0	13.0	1.0	15	40	45

c. Detector

Wave length - 254 nm
Response time - 5000 milliseconds
Range - 0.5

d. Integrator

Start time - 0.00 min
End time - 20.0 min
Chart speed - 5 mm/min
Attenuation - 8
Offset - 5
Auto zero on
Tic marks on
Start time 4.8 min

e. Retention Times (±0.15 min)

HMX	5.63 min	Tetryl	12.67 min
RDX	7.29 min	TNT	14.03 min
1,3,5-TNB ...	9.34 min	2A,4,6-DNT ..	14.67 min
1,3-DNB	11.17 min	2,6-DNT.....	15.70 min
NB	12.07 min	2,4-DNT	16.29 min

C. Analytes

	CAS #	Melting Point (°C)	g/cm^3 Density
HMX	2691-41-0	---	---
RDX	121-82-4	205	1.82 @ 20°C
1,3,5-TNB ...	99-35-04	122.5	1.76 @ 20°C
1,3-DNB	99-65-01	118	1.57 @ 20°C
NB	98-95-3	6	1.205 @ 15°C
Tetryl	---	131	1.57 @ 20°C
2,A,4,6-DNT .	35572-78-2	---	---
2,6-DNT	606-20-2	66	1.28 @ 111°C
2,4-DNT	121-14-2	71	1.32 @ 71°C
TNT	118-96-7	80.1	1.65 @ 20°C

D. Reagents and SARMS

1. Acetonitrile, distilled in glass, HPLC grade.

2. Methanol, distilled in glass, HPLC grade.

3. ASTM Type I water.

4. SARMS, provided by USATHAMA Central QA Lab, as follows:

Compound	Lot Number	% Purity
HMX	1217	99.12
RDX	1130	99.95
1,3,5-TNB ...	1154	99.99
1,3-DNB	2250	99.99
NB	2177	99.76
Tetryl	1149	99.95
2,A,4,6-DNT .	1279	99.99
2,6-DNT	1148	99.99
2,4-DNT	1147	99.98
TNT	1129	99.99

IV. CALIBRATION

A. Initial Calibration

1. Standards

a. Stock Standard

Compound	Lot #	% Purity	Actual Weight (mg)	Final Volume (ml)	Conc µg/ml
HMX	1217	99.12	12.41	5.0	2482
RDX	1130	99.95	11.73	5.0	2346
1,3,5-TNB	1154	99.99	9.895	5.0	1979
1,3-DNB	2250	99.99	9.89	5.0	1978
NB	2177	99.76	10.13	5.0	2026
Tetryl	1149	99.95	9.11	5.0	1822
TNT	1129	99.99	10.06	5.0	2012
2A,4,6-DNT	1279	99.99	9.36	5.0	1872
2,6-DNT	1148	99.99	11.23	5.0	2246
2,4-DNT	1147	99.98	9.34	5.0	1868

All SARMs were accurately weighed to four places directly into a clean, tared 5-ml serum vial. 5 ml of solvent was added to each using a 5-ml volumetric pipet. Those compounds which are stored in water were allowed to air dry prior to weighing.

The stock standard is stored at 0-4°C in a 5-ml serum vial with a teflon septum and crimped seal. The standard can be kept up to three months.

b. Intermediate Standard (ID # 90987-2)

Compound	Conc Stock (µg/ml)	Volume (ml)	Dilution Volume (ml)	Conc µg/ml
HMX	2482	0.400	5	198.6
RDX	2346	0.400	5	187.7
1,3,5-TNB	1979	0.250	5	98.95
1,3-DNB	1978	0.250	5	98.90
NB	2026	0.250	5	101.3
Tetryl	1822	0.270	5	98.39
TNT	2012	0.250	5	100.6
2A,4,6-DNT	1872	0.260	5	97.34
2,6-DNT	2246	0.230	5	103.3
2,4-DNT	1868	0.050	5	18.68

Using glass syringes with an accuracy of ±0.1% aliquots of the stock solutions are transferred to a 5-ml Class A volumetric flask and brought to volume with acetonitrile.

The intermediate standard is stored at 0-4°C in a serum vial with a septum and crimped seal. The intermediate standard can be stored up to 30 days.

c. Calibration Standard

1. ID # 927-05 (0.05 x TRL)

Compound	Parent ID #	Conc Parent (µg/ml)	Aliquot Volume (ml)	Final Volume (ml)	Final Conc (µg/l)
HMX	90987-2	198.6	0.012	5.0	0.4766
RDX	90987-2	187.7	0.012	5.0	0.4505
1,3,5-TNB	90987-2	98.95	0.012	5.0	0.2374
1,3-DNB	90987-2	98.90	0.012	5.0	0.2374
NB	90987-2	101.3	0.012	5.0	0.2431
Tetryl	90987-2	98.39	0.012	5.0	0.2361
TNT	90987-2	100.6	0.012	5.0	0.2414
2A,4,6-DNT	90987-2	97.34	0.012	5.0	0.2336
2,6-DNT	90987-2	103.3	0.012	5.0	0.2479
2,4-DNT	90987-2	18.68	0.012	5.0	0.0448

The aliquot volume was obtained using a microliter syringe and was diluted to volume in a Class A volumetric flask with a 15% acetonitrile, 40% methanol, and 45% water solution.

2. ID # 927-1 (1.0 x TRL)

Compound	Parent ID #	Conc Parent (µg/ml)	Aliquot Volume (ml)	Final Volume (ml)	Final Conc (µg/l)
HMX	90987-2	198.6	0.025	5.0	0.9930
RDX	90987-2	187.7	0.025	5.0	0.9385
1,3,5-TNB	90987-2	98.95	0.025	5.0	0.4945
1,3-DNB	90987-2	98.90	0.025	5.0	0.4945
NB	90987-2	101.3	0.025	5.0	0.5065
Tetryl	90987-2	98.39	0.025	5.0	0.4920
TNT	90987-2	100.6	0.025	5.0	0.5030
2A,4,6-DNT	90987-2	97.34	0.025	5.0	0.4867
2,6-DNT	90987-2	103.3	0.025	5.0	0.5165
2,4-DNT	90987-2	18.68	0.025	5.0	0.0934

The aliquot volume was obtained using a microliter syringe and was diluted to volume in a Class A volumetric flask with a 15% acetonitrile, 40% methanol, and 45% water solution.

3. ID # 927-2 (2 x TRL)

Compound	Parent ID #	Conc Parent (µg/ml)	Aliquot Volume (ml)	Final Volume (ml)	Final Conc (µg/l)
HMX	90987-2	198.6	0.050	5.0	1.986
RDX	90987-2	187.7	0.050	5.0	1.877
1,3,5-TNB	90987-2	98.95	0.050	5.0	0.9895
1,3-DNB	90987-2	98.90	0.050	5.0	0.9890
NB	90987-2	101.3	0.050	5.0	1.013
Tetryl	90987-2	98.39	0.050	5.0	0.9839

Compound	Parent ID #	Conc Parent (µg/ml)	Aliquot Volume (ml)	Final Volume (ml)	Final Conc (µg/ml)
TNT	90987-2	100.6	0.050	5.0	1.006
2A,4,6-DNT	90987-2	97.34	0.050	5.0	0.9734
2,6-DNT	90987-2	103.3	0.050	5.0	1.033
2,4-DNT	90987-2	18.68	0.050	5.0	0.1868

The aliquot volume was obtained using a microliter syringe and was diluted to volume in a Class A volumetric flask with a 15% acetonitrile, 40% methanol, and 45% water solution.

4. ID # 927-5 (5 x TRL)

Compound	Parent ID #	Conc Parent (µg/ml)	Aliquot Volume (ml)	Final Volume (ml)	Final Conc (µg/ml)
HMX	90987-2	198.6	0.120	5.0	4.766
RDX	90987-2	187.7	0.120	5.0	4.505
1,3,5-TNB	90987-2	98.95	0.120	5.0	2.374
1,3-DNB	90987-2	98.90	0.120	5.0	2.374
NB	90987-2	101.3	0.120	5.0	2.431
Tetryl	90987-2	98.39	0.120	5.0	2.361
TNT	90987-2	100.6	0.120	5.0	2.414
2A,4,6-DNT	90987-2	97.34	0.120	5.0	2.336
2,6-DNT	90987-2	103.3	0.120	5.0	2.479
2,4-DNT	90987-2	18.68	0.120	5.0	0.4483

The aliquot volume was obtained using a microliter syringe and was diluted to volume in a Class A volumetric flask with a 15% acetonitrile, 40% methanol, and 45% water solution.

5. ID # 927-10 (10 x TRL)

Compound	Parent ID #	Conc Parent (µg/ml)	Aliquot Volume (ml)	Final Volume (ml)	Final Conc (µg/ml)
HMX	90987-2	198.6	0.250	5.0	9.930
RDX	90987-2	187.7	0.250	5.0	9.385
1,3,5-TNB	90987-2	98.95	0.250	5.0	4.948
1,3-DNB	90987-2	98.90	0.250	5.0	4.945
NB	90987-2	101.3	0.250	5.0	5.065
Tetryl	90987-2	98.39	0.250	5.0	4.919
TNT	90987-2	100.6	0.250	5.0	5.030
2A,4,6-DNT	90987-2	97.34	0.250	5.0	4.867
2,6-DNT	90987-2	103.3	0.250	5.0	5.165
2,4-DNT	90987-2	18.68	0.250	5.0	0.9340

The aliquot volume was obtained using a microliter syringe and was diluted to volume in a Class A volumetric flask with a 15% acetonitrile, 40% methanol, and 45% water solution.

These standards can be stored up to 5 days. They should be kept in a crimp topped 5-ml serum vial with a teflon septum, at 0-5°C.

2. Instrument Calibration

Flow rate should be checked upon initial start-up of the HPLC system (by visually examining the flow rate and checking for any pressure drop), along with other operating parameters i.e. wavelength, mobile phase concentration, pressure drop over the system, to ensure stable conditions.

An initial equilibration time of approximately 15 minutes is recommended.

At the end of the day the HPLC system should be flushed with 100% acetonitrile until a stable pressure drop is observed and then allowed to equilibrate for 15 minutes prior to shut down.

The first analysis of the day is a mobile phase blank to ensure that there are no system interferences.

3. Analysis of Calibration Data

The criteria for acceptability is 1) 10 x TRL ±10% and 2) good recovery of a standard prepared by an independent party from SARM.

a. Standard Criteria

Compound	Lower Limit (µg/l)	Upper Limit (µg/l)
HMX	8.77	10.71
RDX	8.87	10.85
1,3,5-TNB	4.48	5.48
1,3-DNB	4.28	5.24
NB	4.50	5.50
Tetryl	4.32	5.28
TNT	4.43	5.41
2A,4,6-DNT	4.46	5.46
2,6-DNT	4.59	5.61
2,4-DNT	0.85	1.04

b. Check Standard

Compound	Lot #	Conc (µg/l)	Average Pre-Certification Concentration	Upper Limit	Lower Limit
NB	2117	1.100	N/A	1.298	0.902

3. Daily Calibration

1. Standards

a. Stock Standard

Compound	Lot #	% Purity	Actual Weight (mg)	Final Volume (ml)	Conc (µg/ml)
HMX	1217	99.12	12.41	5.0	2482
RDX	1130	99.95	11.73	5.0	2346
1,3,5-TNB	1154	99.99	9.895	5.0	1979
1,3-DNB	2250	99.99	9.89	5.0	1978
NB	2177	99.76	10.13	5.0	2026
Tetryl	1149	99.95	9.11	5.0	1822
TNT	1129	99.99	10.06	5.0	2012
2A,4,6-DNT	1279	99.99	9.36	5.0	1872
2,6-DNT	1148	99.99	11.23	5.0	2246
2,4-DNT	1147	99.98	9.34	5.0	1868

All SARMs were accurately weighed to four places directly into a clean, tared 5-ml serum vial. 5 ml of solvent was added to each using a 5-ml volumetric pipet. Those compounds which are stored in water were allowed to air dry prior to weighing.

The stock standard is stored at 0-4°C in a 5-ml serum vial with a teflon septum and crimped seal. The standard can be kept up to three months.

b. Intermediate Standard (ID # 90987-2)

Compound	Conc Stock (µg/ml)	Volume (ml)	Dilution Volume (ml)	Conc µg/ml
HMX	2482	0.400	5	198.6
RDX	2346	0.400	5	187.7
1,3,5-TNB	1979	0.250	5	98.95
1,3-DNB	1978	0.250	5	98.90
NB	2026	0.250	5	101.3
Tetryl	1822	0.270	5	98.39
TNT	2012	0.250	5	100.6
2A,4,6-DNT	1872	0.260	5	97.34
2,6-DNT	2246	0.230	5	103.3
2,4-DNT	1868	0.050	5	18.68

Using glass syringes with an accuracy of ±0.1% aliquots of the stock solutions are transferred to a 5-ml Class A volumetric flask and brought to volume with acetonitrile.

The intermediate standard is stored at 0-4°C in a serum vial with a septum and crimped seal. The intermediate standard can be stored up to 30 days.

C. Calibration Standard

1. ID # 927-10 (10 x TRL)

Compound	Parent ID #	Conc Parent (µg/ml)	Aliquot Volume (ml)	Final Volume (ml)	Final Conc (µg/l)
HMX	90987-2	198.6	0.250	5.0	9.930
RDX	90987-2	187.7	0.250	5.0	9.385
1,3,5-TNB	90987-2	98.95	0.250	5.0	4.948
1,3-DNB	90987-2	98.90	0.250	5.0	4.945
NB	90987-2	101.3	0.250	5.0	5.065
Tetryl	90987-2	98.39	0.250	5.0	4.919
TNT	90987-2	100.6	0.250	5.0	5.030
2A,4,6-DNT	90987-2	97.34	0.250	5.0	4.867
2,6-DNT	90987-2	103.3	0.250	5.0	5.165
2,4-DNT	90987-2	18.68	0.250	5.0	0.9340

The aliquot volume was obtained using a microliter syringe and was diluted to volume in a Class A volumetric flask with a 15% acetonitrile, 40% methanol, and 45% water solution.

Shelf life is 5 days if stored at 0-4°C.

2. Instrument Calibration

Flow rate should be checked upon initial start-up of the HPLC system (by visually examining the flow rate and checking for any pressure drop), along with other operating parameters i.e. wavelength, mobile phase concentration, pressure drop over the system, to ensure stable conditions.

An initial equilibration time of approximately 15 minutes is recommended.

At the end of the day the HPLC system should be flushed with 100% acetonitrile until a stable pressure drop is observed and then allowed to equilibrate for 15 minutes prior to shut down.

The first analysis of the day is a mobile phase blank to ensure that there are no system interferences.

1. Blank
2. 10 x TRL #927-10
3. Blank
4. Check STD
5. Blank
6. Sample
7. Sample

End of day

8. 10 x TRL #927-10
9. Blank
10. Check Std

3. Analysis of Calibration Data

The criteria for acceptability of data is ±10% of the peak area found in the initial calibration of the 10 x TRL standard.

4. Calibration Checks

a. Standard Check

	% Return on 10 TRL			
	Day 1	Day 2	Day 3	Day 4
HMX				
Initial	98.28	104.26	104.51	106.34
Final	104.26	105.48	106.84	105.63
RDX				
Initial	98.87	98.87	101.50	101.52
Final	98.78	104.28	101.52	105.04
1,3,5-TNB				
Initial	98.94	99.56	99.02	100.99
Final	99.56	99.38	100.99	101.83
1,3-DNB				
Initial	99.17	98.36	99.01	101.70
Final	98.36	102.22	101.70	100.87
NB				
Initial	99.09	98.57	99.94	101.95
Final	98.57	99.94	101.95	101.14
Tetryl				
Initial	95.57	41.15	0	0
Final	41.15	27.20	0	53.6*
2A,4,6-DNT				
Initial	99.60	94.45	101.14	102.69
Final	94.45	101.52	102.69	96.12
2,6-DNT				
Initial	99.20	97.73	99.27	101.42
Final	87.73	100.12	101.42	101.33
2,4-DNT				
Initial	99.26	95.10	98.46	99.49
Final	95.10	97.21	99.49	97.76

*Fresh solution prepared.

b. Check Standard

	% Return on 10 TRL			
	Day 1	Day 2	Day 3	Day 4
NB				
Initial	99.80	93.87	90.52	84.93
Final	93.87	89.06	84.93	89.60

NOTE: Check standard was made by diluting a stock standard prepared by a qualified independent party from a USATHAMA SARM.

V. CERTIFICATION TESTING

A. Soil Spiking

1. 36 aliquots of ~2 grams of USATHAMA standard soil were weighed out in clean, tared 5-ml serum vials and the weight recorded to the nearest 0.001 gram.

2. The vials were labelled as follows:

Group I	Group II	Group III	Group IV	Group V	Group VI
D1-HP-00	D1-HP-0.5	D1-HP-1	D1-HP-2	D1-HP-5	D1-HP-10.0
D2-HP-00	D2-HP-0.5	D2-HP-1	D2-HP-2	D2-HP-5	D2-HP-10.0
D3-HP-00	D3-HP-0.5	D3-HP-1	D3-HP-2	D3-HP-5	D3-HP-10.0
D4-HP-00	D4-HP-0.5	D4-HP-1	D4-HP-2	D4-HP-5	D4-HP-10.0
D5-HP-00	D5-HP-0.5	D5-HP-1	D5-HP-2	D5-HP-5	D5-HP-10.0
D6-HP-00	D6-HP-0.5	D6-HP-1	D6-HP-2	D6-HP-5	D6-HP-10.0

3. The following solution #092787-1 was used for spiking the soils.

Compound Name	Conc. of Parent Sol'n. ng/µl	Aliq. Vol. (ml)	Final Vol. (ml)	Final Conc. ng/µl
HMX	2482	0.7	5.0	347.5
RDX	2346	0.75	5.0	351.9
1,3,5-TNB	1979	0.45	5.0	178.1
1,3-DNB	2020	0.42	5.0	169.7
NB	2882	0.31	5.0	178.7
Tetryl	1822	0.47	5.0	171.3
TNT	2012	0.44	5.0	177.1
2A,4,6-DNT	1872	0.47	5.0	176.0
2,6-DNT	2246	0.40	5.0	179.7
2,4-DNT	1868	0.09	5.0	33.62

4. Spike the samples as follows:

	Group I 00 ul Spike	Group II 14 ul Spike	Group III 28 ul Spike	Group IV 56 ul Spike	Group V 140 ul Spike	Group VI 280 ul Spike
HMX	0.0	4.87	9.73	19.46	48.65	97.30
RDX	0.0	4.93	9.85	19.71	49.27	98.53
1,3-DNB	0.0	2.49	4.99	9.87	24.93	49.87
1,3,5-TNB	0.0	2.38	4.75	9.50	23.76	47.52
NB	0.0	2.50	5.00	10.01	25.02	50.04
Tetryl	0.0	2.40	4.80	9.59	23.98	47.96
TNT	0.0	2.48	4.96	9.92	24.79	49.59
2A,4,6-DNT	0.0	2.46	4.93	9.86	24.64	48.28
2,6-DNT	0.0	2.52	5.03	10.06	25.16	50.32
2,4-DNT	0.0	0.471	0.941	1.88	4.71	9.41

a. These values are the total ug of each compound spiked onto the 2-gram aliquots of USATHAMA standard soil.

b. The soil was spiked by injecting the appropriate volume indicated in each group of the intermediate standard 092787-1 directly on to the weighed soil in the 10-ml vial with a microsyringe.

c. The solvent was evaporated from the soil using a stream of helium to gently blow the solvent off. The soil was allowed to set 1 hour before extraction solvent was added.

B. Soil Extraction

1. To each vial with the weighed and spiked soil, 5 ml of acetonitrile was accurately pipetted and capped securely with a crimped top and teflon septum.

2. The solution was vigorously shaken by hand for 5 seconds to assure that all the soil surface areas were wetted.

3. The vials containing the soil and extract were placed so that they did not touch and were suspended approximately 0.25 inches above the sonication head in the cup horn attachment for the sonicator. The solutions were sonicated for 10 minutes at a power control knob setting of 7 after tuning. The sonicating bath was maintained at a temperature of less than 120°F.

4. The soil and solvent were shaken thoroughly and then separated by centrifuging for 5 minutes at 200 rpm.

5. An aliquot was taken from the vial and filtered through a 0.45-micron glass fiber using a syringe filtration system.

5. 400 µl of filtrate was transferred to an autosampler vial to which 400 µl of MeOH and water solution (20% MeOH - 80% H_2O) was added.

C. Analysis by HPLC

Day I	Day II	Day III	Day IV
Blank	Blank	Blank	Blank
Check STD	10 x TRL	10 x TRL	10 x TRL
Blank	Blank	Blank	Blank
0.50 x TRL	Check STD	Check STD	Check STD
1 x TRL	Blank	Blank	Blank
2 x TRL	D2-HP-00	D3-HP-00	D4-HP-00
5 x TRL	D2-HP-0.5	D3-HP-0.5	D4-HP-0.5
10 x TRL	D2-HP-1	D3-HP-1	D4-HP-1
Blank	D2-HP-2	D3-HP-2	D4-HP-2
D1-HP-00	D2-HP-5	D3-HP-5	D4-HP-5
D1-HP-0.5	D2-HP-10	D3-HP-10	D4-HP-10
D1-HP-1	Blank	Blank	Blank
D1-HP-2	10 x TRL	10 x TRL	10 x TRL
D1-HP-5	Blank	Blank	Blank
D1-HP-10	Check STD	Check STD	Check STD
Blank			
10 x TRL			
Blank			
Check STD			

VI. SAMPLE HANDLING & STORAGE

A. Sampling Procedure

1. Field

Field sampling will be performed by inserting stainless steel shelby tube into the processed soil in the bucket of the front end loader at regular pre-determined intervals. The processed soil core collected will be sealed in the tube by covering both ends with aluminum caps and stored in a secure repository.

The shelby tubes will be collected from the respository on a routine basis which will not exceed 24 hours and taken to the laboratory.

2. Laboratory

In the laboratory, equal weights of soil, free of rocks and extraneous material, will be taken from the end of each tube for the 24-hour sampling period and will be composited together. The weight from each tube will vary depending on the number of tubes collected, but it will be enough to give a final composite weight of 200 grams.

a. Dry Brittle Soils

If the samples are dry and brittle, then each weighed aliquot will be broken up and placed in a clean glass container. The glass container will contain a volume in excess of the amount of soil to be added and sealed with a teflon lined screw cap. The jar will be shaken and/or stirred until its contents are homogenous.

An initial sub-sample will be taken for analysis. A second sub-sample will be used to fill a labelled 40-ml glass vial with a teflon lined screw cap. The glass vial will be archived in a refrigerator at 4°C.

b. Moist Cohesive Soils

If the samples are moist and cohesive then each weighed sample will be placed in a clean stainless steel round-bottom bowl. The material will be thoroughly mixed and sub-divided by hand until it is mixed uniformly.

An initial sub-sample will be taken for analysis. A second sub-sample will be used to fill a labelled 40-ml glass vial with a teflon lined screw cap. The glass vial will be archived in a refrigerator at 4°C.

B. Container

Stainless steel shelby tubes with aluminum caps
500-ml, amber, wide-mouth jars with teflon lined screw caps
40-ml, amber glass vials

C. Storage Conditions

1. Field

A secure and tightly sealed repository box with an opening that allows the tubes to be dropped in as they are collected. The box will be at ambient temperature.

2. Laboratory

Sample storage after compositing will be in a refrigerator at 4°C.

D. Holding Times

Samples are to be extracted within seven days of sampling and analyzed within 30 days of extraction.

E. Solution Verification

A control reference standard will be made in a separate lab by qualified individuals who are not involved in the project from USATHAMA supplied SARMs.

VII. PROCEDURE

A. Soil Extraction

1. Soil (0.25 grams for feed pile, 2 grams for processed soil) is weighed to the third decimal and placed in a glass serum vial.

2. To each vial with the weighed and spiked soil, 5 ml of acetonitrile was accurately pipetted and capped securely with a crimped top and teflon septum.

3. The solution was vigorously shaken by hand in order to assure that all soils were wetted.

4. The vials containing the solutions were placed in a water bath so that they did not touch, and were suspended approximately 0.25 inches above the sonication head in the cup horn attachment. The solutions were sonicated for 10 minutes at a power control setting of 7 after proper tuning. The sonicating bath was maintained at a temperature of less than 120°F.

5. The sonicated solution was thoroughly shaken and then separated by centrifuging for 5 minutes at 2000 rpm.

6. An aliquot was taken from the vial and filtered through a 0.45-micron glass fiber using a syringe filtration system.

7. 400 µl of filtrate was transferred to an autosampler vial to which 400 µl of MeOH and water solution (20% MeOH + 80% H_2O) was added.

B. Chemical Reactions

There are no compound modifications or derivatizations in this method.

C. Instrumental Analysis

1. Analytical System

Perkin Elmer Series 4 HPLC metering and pumping system
Perkin Elmer ISS 100 auto-injector
Perkin Elmer LC95 spectrophotometric detector
Perkin Elmer LCI 100 integrator

2. Chromatographic System

LC 18 guard column Supelco 2 cm x 4.6 mm 5-µ
LC 18 column (25 cm long by 4.6 mm inside diameter) 5-µ

3. Analytical Conditions

a. Auto-injector

15 µl
50 µl injection loop

b. Metering and Pumping

	Time (min)	Flow (ml/min)	% Acetonitrile	% Methanol	% Water
Equilibrium	0.5	1.0	15	40	45
Analysis Run	13.0	1.0	15	40	45

Pressure drop = ~14.3 mpa.

c. Detector

Wave length - 254 nm
Response time - 5000 milliseconds
Range - 0.5

d. Integrator

Start time - 0.00 min
End time - 20.0 min
Chart speed - 5 mm/min
Attenuation - 8
Offset - 5
Auto zero on
Tic marks on
Start time 4.8 min

VIII. CALCULATION

$$\text{Conc } \mu g/g = \frac{10B}{W \text{ wet soil extracted}}$$

Where B = concentration in µg/ml as obtained from the calibration curve using peak areas
W = weight of soil in grams
10 = 2 (dilution factor) x 5 ml (acetonitrile)

Concentration is expressed in µg/g wet weight.

IX. DAILY QUALITY CONTROL

A. Control Samples

1. Stock Standard

Compound	Lot #	% Purity	Actual Weight (mg)	Final Volume (ml)	Conc µg/ml
HMX	1217	99.12	12.41	5.0	2482
RDX	1130	99.95	11.73	5.0	2346
1,3,5-TNB	1154	99.99	9.895	5.0	1979
1,3-DNB	2250	99.99	9.89	5.0	1978
NB	2177	99.76	10.13	5.0	2026

Compound	Lot #	% Purity	Actual Weight (mg)	Final Volume (ml)	Conc µg/ml
Tetryl	1149	99.95	9.11	5.0	1822
TNT	1129	99.99	10.06	5.0	2012
2A,4,6-DNT	1279	99.99	9.36	5.0	1872
2,6-DNT	1148	99.99	11.23	5.0	2246
2,4-DNT	1147	99.98	9.34	5.0	1868

All SARMs were accurately weighed to four places directly into a clean, tared 5-ml serum vial. 5 ml of solvent was added to each using a 5-ml volumetric pipet. Those compounds which are stored in water were allowed to air dry prior to weighing.

The stock standard is stored at 0-4°C in a 5-ml serum vial with a teflon septum and crimped seal. The standard can be kept up to three months.

2. Intermediate Standard

Compound	Conc Stock (µg/ml)	Volume (ml)	Dilution Volume (ml)	Conc µg/ml
HMX	2482	0.700	5.0	347.5
RDX	2346	0.750	5.0	351.9
1,3,5-TNB	1979	0.450	5.0	178.1
1,3-DNB	1978	0.450	5.0	178.0
NB	2026	0.450	5.0	182.3
Tetryl	1822	0.500	5.0	182.2
TNT	2012	0.450	5.0	181.1
2A,4,6-DNT	1872	0.500	5.0	187.2
2,6-DNT	2246	0.400	5.0	179.7
2,4-DNT	1868	0.090	5.0	33.62

Using glass syringes with an accuracy of ±0.1% aliquots of the stock solutions are transferred to a 5-ml Class A volumetric flask and brought to volume with acetonitrile.

The intermediate standard is stored at 0-4°C in a serum vial with a septum and crimped seal. The intermediate standard can be stored up to 30 days.

3. 10 CRL

Compound	Conc Parent (µg/ml)	Aliquot Volume (ml)	Final Volume (ml)	Final Conc (µg/l)
HMX	347.5	0.35	5.0	24.3
RDX	351.9	0.35	5.0	24.6
1,3,5-TNB	178.1	0.35	5.0	12.5
1,3-DNB	170.0	0.35	5.0	11.9

Compound	Conc Parent (µg/ml)	Aliquot Volume (ml)	Final Volume (ml)	Final Conc (µg/l)
NB	178.7	0.35	5.0	12.5
Tetryl	171.4	0.35	5.0	12.0
TNT	177.1	0.35	5.0	12.4
2A,4,6-DNT	175.7	0.35	5.0	12.3
2,6-DNT	179.7	0.35	5.0	12.6
2,4-DNT	33.62	0.35	5.0	2.35

The aliquot volume was obtained using a microliter syringe and was diluted to volume in a Class A volumetric flask with a 21% acetonitrile, 35% methanol, and 44% water solution.

4. 2 CRL

Compound	Conc Parent (µg/ml)	Aliquot Volume (ml)	Final Volume (ml)	Final Conc (µg/l)
HMX	347.5	0.075	5.0	5.21
RDX	351.9	0.075	5.0	5.28
1,3,5-TNB	178.1	0.075	5.0	2.67
1,3-DNB	170.0	0.075	5.0	2.55
NB	178.7	0.075	5.0	2.68
Tetryl	171.4	0.075	5.0	2.57
TNT	177.1	0.075	5.0	2.66
2A,4,6-DNT	175.7	0.075	5.0	2.64
2,6-DNT	179.7	0.075	5.0	2.70
2,4-DNT	33.62	0.075	5.0	0.504

B. Control Charts

1. Description of Charts to be Maintained

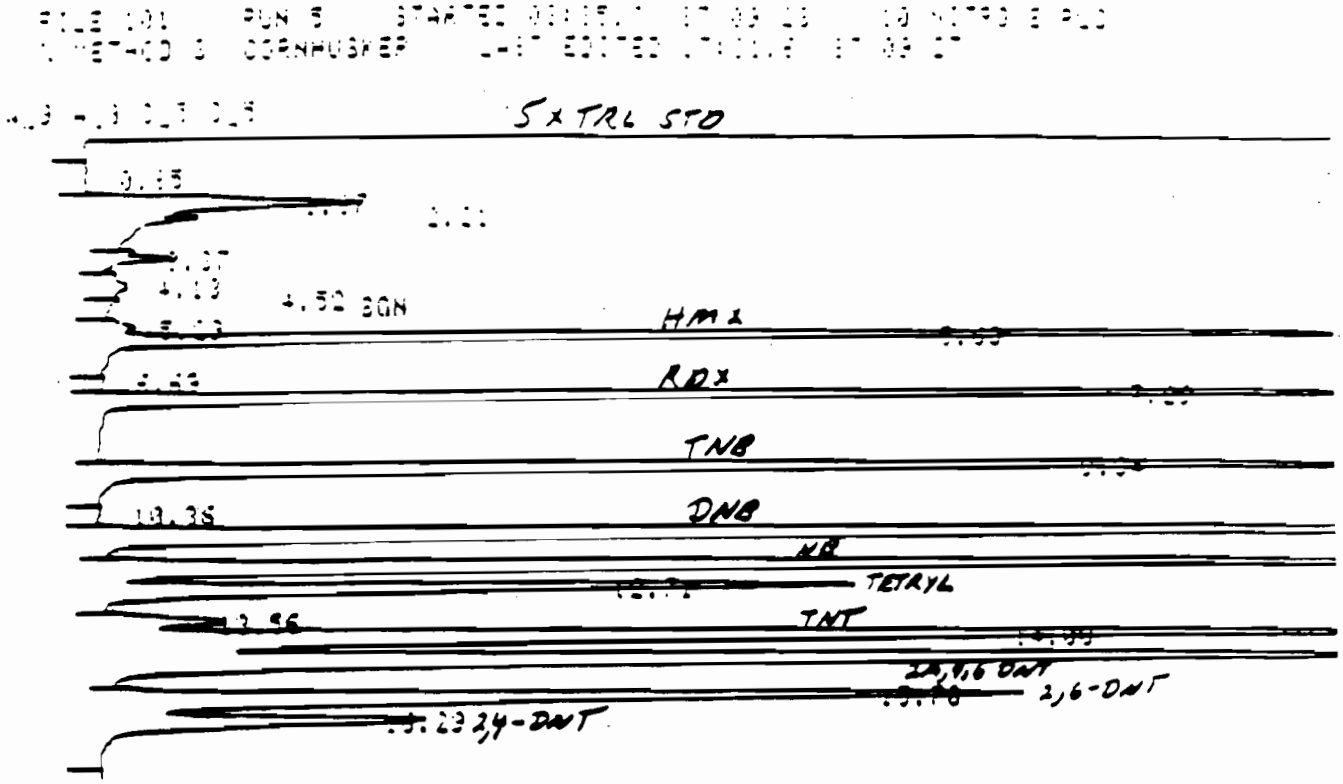
Single day $\bar{X}$-R control charts are prepared for each control analyte using data from the duplicate spiked QC samples in each lot to determine percent recovery:

$$\frac{\text{Found Concentration}}{\text{Spiked Concentration}} \times 100$$

Use of percent recovery allows for minor variations in spiking solution concentrations.

To prepare control charts, the analyst should have access to the following data:

- Percent recovey of each analyte in the two high concentration spiked QC samples (Class 1).
- Average ($\bar{X}$) percent recovery for the two spiked QC samples (Class 1) in each lot.

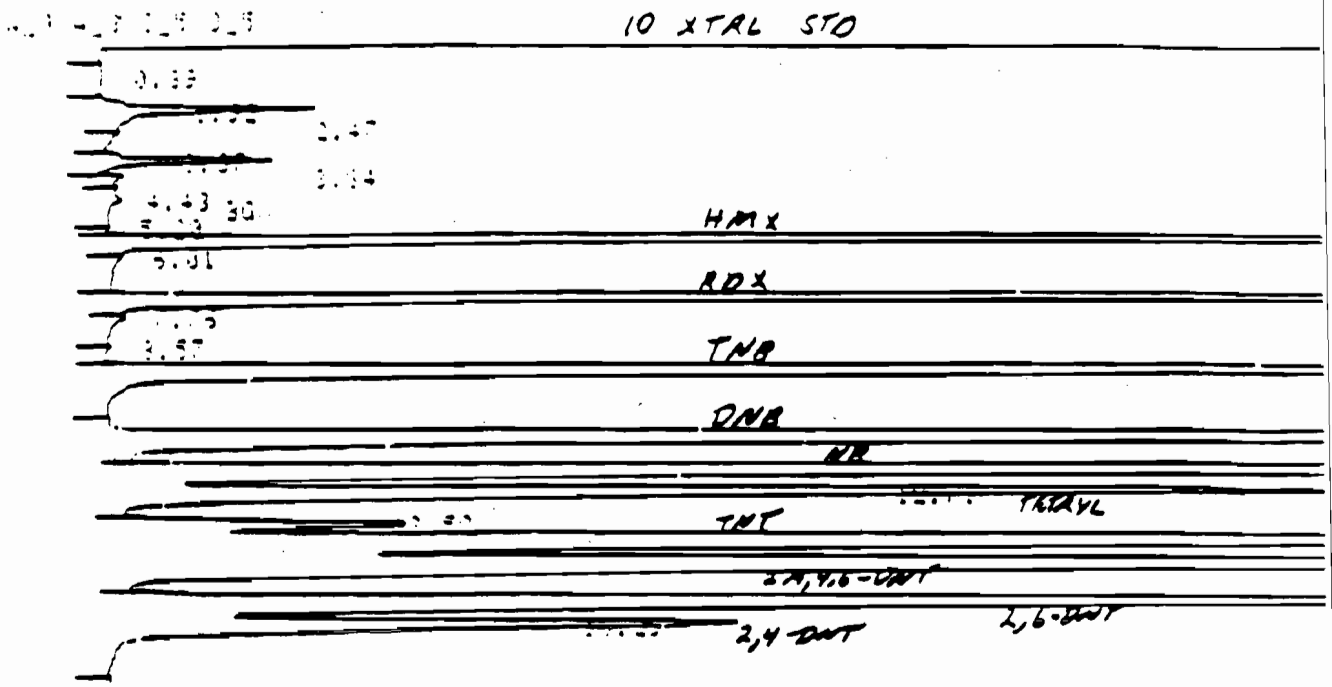


FILE 101 RUN 5 STARTED 03:05.7 87/09/28 10 NITRO EXPLO
% METHOD 3 CORNHUSKER LAST EDITED 17:31.6 87/09/27

RT	AREA	HEIGHT	BC	AREA PERCENT	HEIGHT PERCENT
5.22	53063	2.4515	T	0.4744	0.3303
5.[illegible]	[illegible]	[illegible]	T	[illegible].2604	[illegible]
6.69	1824	0.1402	TS	0.0163	0.0189
7.29	1003872	79.1812	T	8.9677	10.6637
9.34	1265755	101.2544	T	11.3161	13.6423
10.36	2957	0.1420	TS	0.0264	0.0191
11.17	1740386	123.3943	T	15.5537	16.6258
12.07	1267229	85.6904	T	11.3293	11.5457
12.71	678668	41.9994	T	6.0674	5.6539
13.56	118183	6.3940	T	1.0566	0.9289
14.08	1348729	76.4990	T	11.9864	10.3073
14.67	1505873	79.6117	T	13.4628	10.7267
15.70	916654	50.2399	T	8.1951	6.7692
16.29	367139	17.9644	U	3.2923	2.4205

14 PEAKS > AREA REJECT 11195417 TOTAL AREA
14 PEAKS > HEIGHT REJECT 742.1347 TOTAL HEIGHT

FILE 102 RUN 6 STARTED 03:55.0 87 09/28 10 NITRO EXPLO
METHOD 3 CORNHUSKER LAST EDITED 17:31.6 87/09/27



FILE 102 RUN 6 STARTED 03:55.0 87 09/28 10 NITRO EXPLO
METHOD 3 CORNHUSKER LAST EDITED 17:31.6 87/09/27

RT	AREA	HEIGHT	BC	AREA PERCENT	HEIGHT PERCENT
5.20	11579	0.5113	T	0.0537	0.0352
5.63	1680685	150.5060	T	7.7932	10.3553
6.01	16518	0.6623	TS	0.0767	0.0456
7.29	1897493	156.3223	T	8.8053	10.7555
7.76	9658	0.6375	TS	0.0448	0.0439
8.57	2168	0.2073	TS	0.0101	0.0143
9.34	2452682	199.6763	T	11.3917	13.7383
11.17	3430196	244.2933	T	15.9179	16.8082
12.07	2482082	169.5225	T	11.5178	11.6637
12.71	1152454	71.1143	T	5.3480	4.8929
13.52	239767	16.2833	T	1.1448	1.1203
14.03	2706513	151.5256	T	12.5596	10.4254
14.67	2919652	157.8634	T	13.5487	10.8615
15.70	1739906	99.2026	T	8.3061	6.8254
16.29	708052	35.0936	U	3.2857	2.4145

15 PEAKS > AREA REJECT 21549345 TOTAL AREA
15 PEAKS > HEIGHT REJECT 1453.4232 TOTAL HEIGHT

CERTIFICATION PERFORMANCE

DATA PACKAGE AND METHOD:

NITRO-EXPLOSIVES IN WATER BY HPLC

(CERTIFICATION PERFORMED IN KNOXVILLE, TN)

USATHAMA METHOD NUMBER UW11

IT Analytical Services

Knoxville, TN

ORIGINAL SUBMISSION: June 16, 1988

REVISED SUBMISSION: September 30, 1988

CERTIFICATION PERFORMANCE DATA PACKAGE CHECKLIST
(ONE FOR EACH METHOD)

Contract/Task No. DACA45-87-C0103 Installation Louisiana Army Ammunition Plant

The following items are included in this Certification Performance Data Package for 10 Nitroexplosives (Analyte(s)) in water (Matrix).

X Method written up in USATHAMA format.

Calibration:

X Calibration curves from days of certification (plot of raw data).

X Documentation for Lack of Fit and Zero Intercept Tests.

Certification:

X Data summary - target versus found.

X Reporting limit, precision and accuracy calculations.

X Reporting limit plot.

X Data summary - statistics.

X Lack of Fit and Zero Intercept Tests.

X Chromatograms from each day of certification analyses for the highest tested concentration and for the tested concentration closest to calculated reporting limit.

X Spectra for all target analytes (if applicable). Not applicable

X Identity and purity determinations for off-the-shelf reference materials, either purchased by the Contractor or supplied by the Central QA Laboratory. Included

Paul Mills 6/14/88
Contractor QAC Date
Paul Mills

NITROAROMATICS IN WATER SAMPLES
UW11

I. SUMMARY

This method is applicable to the determination of the following compounds in ground water and surface water samples:

Acronym		Compound
HMX	:	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine Cyclotetramethylenetetranitramine
RDX	:	Hexahydro-1,3,5-trinitro-s-triazine Cyclotetramethylenetetranitramine
1,3,5-TNB	:	1,3,5-trinitrobenzene
1,3-DNB	:	1,3-dinitrobenzene
NB	:	Nitrobenzene
Tetryl	:	A-methyl-N,2,4,6-tetranitroanaline
2A,4,6-DNT	:	2-amino-4,6-dinitrotoluene
2,4,6-TNT	:	2,4,6-trinitrotoluene
2,6-DNT	:	2,6-dinitrotoluene
2,4-DNT	:	2,4-dinitrotoluene

After concentration samples are analyzed by direct injection HPLC with UV detection.

II. APPLICATION

A. Calibration Range

The concentration range of calibration standards tested is as follows:

Compound	Concentration (ng/ul = ug/l)		
HMX	1,320	to	26,300
RDX	1,340	to	26,760
1,3,5-TNB	653	to	13,050
1,3-DNB	675	to	13,490
NB	655	to	13,100
Tetryl	653	to	13,100
2A,4,6-DNT	673	to	13,450
2,4,6-TNT	669	to	13,450
2,6-DNT	66	to	1,320
2,4-DNT	67	to	1,342

B. Tested Concentration Range

Based upon the procedure, the tested concentration range in water is as follows:

Compound	Concentration (ug/l)		
HMX	5.06	to	101.2
RDX	5.14	to	102.9
1,3,5-TNB	2.51	to	50.2
1,3-DNB	2.59	to	51.9
NB	2.52	to	50.4
Tetryl	2.51	to	50.20
2A,4,6-DNT	2.59	to	51.80
2,4,6-TNT	2.57	to	51.40
2,6-DNT	0.255	to	5.10
2,4-DNT	0.258	to	5.16

C. Sensitivity

The instrumental response observed for each analyte at the calculated reporting limit is as follows: (taken from 0.5X TRL, Day #1 Certification Standard Run)

Compound	Peak Height	Concentration (ug/l)
HMX	16.5249	1320.
RDX	19.5417	1340.
1,3,5-TNB	21.7569	653.
1,3-DNB	25.9442	675.
NB	17.6209	655.
Tetryl	11.4344	653.
2A,4,6-DNT	15.3310	673.
2,4,6-TNT	16.3188	669.
2,6-DNT	1.4371	66.
2,4-DNT	2.4314	67.

D. Certified Reporting Limits

Each analyte in ug/liter:

Compound	CRL	UPPER LIMITS
HMX	19.8	50.6
RDX	5.84	51.45
1,3,5-TNB	14.2	50.2
1,3-DNB	12.0	51.9
NB	11.2	50.4
Tetryl	2.51	50.2
2A,4,6-DNT	2.74	51.8
2,4,6-TNT	2.94	51.4
2,6-DNT	0.47	5.1
2,4-DNT	0.38	5.16

E. Analysis Rate

After calibration, up to twelve (12) samples could be analyzed in a typical 8-hour workday.

F. Interferences

Solvents, reagents, glassware and other sample processing equipment may yield artifacts that can interfere with HPLC analysis. Reagent blanks should be prepared and analyzed prior to sample extraction to verify the purity of the analytical system.

Interferences coextracted from the samples will vary considerably from source to source. Compounds which coelute with the compounds of interest and absorb at 210-260 nm will interfere.

G. Safety Information

Use caution in handling. Compounds are explosives. Toxic inhalation and skin absorption hazards exist.

RDX and HMX can undergo alkaline hydrolysis and both degrade at temperatures greater than 80°C in an organic solvent. TNT and others may photodecompose and standards and samples should, therefore, be kept in amber containers and in the dark as much as possible. When handling small quantities of explosives, safety precautions should be expanded. Caution must be exercised in the following areas:

1. Keep all materials cooled and properly stored.
2. Some materials in their pure form need to be stored in water.
3. Do not allow solvents to evaporate.
4. Protect from violent shock.

III. APPARATUS AND CHEMICALS

A. Equipment and Operation

1. Analytical System

Perkin Elmer Series 4 HPLC metering and pumping system
Perkin Elmer ISS 100 Auto-injector
erkin Elmer LC95 spectrophotometric detector
Perkin Elmer LCI 100 integrator

2. Chromatographic System

LC 18 guard column
LC 18 column (25 cm long by 4.6 mm inside diameter)

3. Analytical Conditions

a. Auto-injector

10 ul

b. Metering and Pumping

	Time (min)	Flow (ml/min)	% Aceto-nitrile	% Methanol	% Water
*Equilibrium	0.1	1.0	15	40	45
Analysis Run	15	1.0	15	40	45

c. Detector

Wave Length - 254 nm
Response time - 2000 milliseconds
Range - 0.5

*This equilibrium is entered to satisfy the instrument's running requirements. To run isocratically no equilibration time is necessary between runs.

d. Integrator

Start time - 0.00 min
End time - 15.0 min
Chart speed - 10mm/min
Attenuation - 8
Offset - 5
Auto zero on at 0.01 sec

e. Retention Times

Compound		Rention Time Window (Minutes)		
HMX	:	5.50	±	0.2025
RDX	:	6.875	±	0.2085
1,3,5-TNB	:	8.9475	±	0.289
1,3-DNB	:	9.952	±	0.402
NB	:	10.7275	±	0.412
Tetryl	:	11.455	±	0.649
2A,4,6-DNT	:	12.715	±	0.813
2,4,6-TNT	:	13.150	±	0.655
2,6-DNT	:	13.795	±	0.7256
2,4-DNT	:	14.2025	±	0.776

Retention time windows are determined by taking four (4) days of 10XIRL runs, finding the mean retention time and ± three times the standard deviation of the mean. If these retention times shift due to significant system changes (e.g. new column, new pump) and no other system problem is apparent, then new retention time windows will be established.

B. Analytes

		CAS #	Melting Point (°C)	Density (g/ml)
HMX	:	2691-41-0	—	—
RDX	:	121-82-4	205	1.82 @ 20°C
1,3,5-TNB	:	99-35-04	122.5	1.76 @ 20°C
1,3-DNB	:	99-65-01	118	1.57 @ 20°C
NB	:	98-95-3	6	1.205 @ 15°C
Tetryl	:	479-45-8	131	1.57 @ 20°C
2A,4,6-DNT	:	35572-78-2	—	—
2,4,6-TNT	:	118-96-7	80.1	1.65 @ 20°C
2,6-DNT	:	606-20-2	66	1.28 @ 111°C
2,4-DNT	:	121-14-2	71	1.32 @ 71°C

C. Reagents and SARMS

1. Acetonitrile, distilled in glass, HPLC grade.

2. Methanol, distilled in glass, HPLC grade.

3. ASTM Type I and II water, with 100 mg/L sulfate and chloride added to the Type II water.

4. SARMS, provided by USATHAMA Central QA Lab, as follows:

COMPOUND	LOT NUMBER	% PURITY
HMX	1217	99.12
RDX	1130	99.95
1,3,5-TNB	1154	99.99
1,3-DNB	2250	99.99
NB	2177	99.76
Tetryl	1149	99.95
2A,4,6-DNT	1279	99.99
2,4,6-TNT	1129	99.99
2,6-DNT	1148	99.99
2,4-DNT	1147	99.98

IV. CERTIFICATION CALIBRATION

A. Preparation of Standards

1. Stock Standard

Compound	Lot #	Purity %	Weight (mg)	Dilution Volume (ml)	Conc mg/l
HMX	1217	99.12	23.00	5.0	4600
RDX	1130	99.95	20.58	5.0	4116
1,3,5-TNB	1154	99.99	12.55	5.0	2510
1,3-DNB	2250	99.99	21.63	5.0	4326
NB	2177	99.76	21.01	5.0	4202
Tetryl	1149	99.95	10.04	5.0	2008
2A,4,6-DNT	1279	99.99	10.78	5.0	2156
2,4,6-TNT	1129	99.99	14.69	5.0	2939
2,6-DNT	1148	99.99	9.80	5.0	1960
2,4-DNT	1147	99.98	17.20	5.0	3440
EPA 2,6-DNT	D002	99.26	50.82	N/A	

All reagents were accurately weighed and diluted to volume with methanol in individual Class "A" volumetric flasks. Those compounds which are stored in water are allowed to air dry prior to weighing.

The stock standards are stored at 0-4°C in an amber serum vial with a septum and crimped seal. The stock standards can be kept up to 90 days.

2. Intermediate Standard (ID #L-I-03)

Compound	Conc Stock (mg/l)	Aliquot Volume (ml)	Dilution Volume (ml)	Final Conc. ug/ml
HMX	4600	0.22	5	202
RDX	4116	0.25	5	205.8
1,3,5-TNB	2510	0.20	5	100.
1,3-DNB	4326	0.12	5	104.
NB	4202	0.12	5	100.8
Tetryl	2008	0.25	5	100.
2A,4,6-DNT	2156	0.24	5	104.
2,4,6-TNT	2939	0.175	5	103.
2,6-DNT	1960	0.026	5	10.2
2,4-DNT	3440	0.015	5	10.3

Each was diluted into one Class "A" volumetric flask and brought up to volume with methanol.

The intermediate standard is stored at 0-4°C in an amber serum vial with a septum and crimped seal. The intermediate standard can be stored up to 30 days.

3. Calibration Standard

a. ID #L-W-07 (0.500 x TRL)

Compound	Parent I.D. Number	Conc. Parent ng/ul	Aliq. Vol. (ml)	Dilution Vol. (ml)	Final Conc. ng/ml
HMX	L-I-03	202.	0.0325	5	1320.
RDX	L-I-03	205.8	0.0325	5	1340.
1,3,5-TNB	L-I-03	100.	0.0325	5	653.
1,3-DNB	L-I-03	104.	0.0325	5	675.
NB	L-I-03	100.8	0.0325	5	655.
Tetryl	L-I-03	100.	0.0325	5	653.
2A,4,6-DNT	L-I-03	104.	0.0325	5	673.
2,4,6-TNT	L-I-03	103.	0.0325	5	669.
2,6-DNT	L-I-03	10.2	0.0325	5	66.
2,4-DNT	L-I-03	10.3	0.0325	5	67.

The aliquot volume was obtained using a 50 ul microliter syringe and was diluted to volume in a Class "A" volumetric flask with methanol.

b. ID #L-W-08 (1.0 x TRL)

Compound	Parent I.D. Number	Conc. Parent ng/ul	Aliq. Vol. (ml)	Dilution Vol. (ml)	Final Conc. ng/ml
HMX	L-I-03	202.	0.065	5	2631.
RDX	L-I-03	205.8	0.065	5	2680.
1,3,5-TNB	L-I-03	100.	0.065	5	1305.
1,3-DNB	L-I-03	104.	0.065	5	1349.
NB	L-I-03	100.8	0.065	5	1310.
Tetryl	L-I-03	100.	0.065	5	1305.
2A,4,6-DNT	L-I-03	104.	0.065	5	1346.
2,4,6-TNT	L-I-03	103.	0.065	5	1338.
2,6-DNT	L-I-03	10.2	0.065	5	132.5
2,4-DNT	L-I-03	10.3	0.065	5	134.2

The aliquot volume was obtained using a 100 ul microliter syringe and was diluted to volume in a Class "A" volumetric flask with methanol.

c. ID #L-W-9 (2 x TRL)

Compound	Parent I.D. Number	Conc. Parent ng/ul	Aliq. Vol. (ml)	Dilution Vol. (ml)	Final Conc. ng/ml
HMX	L-I-03	202.	0.130	5	5262.
RDX	L-I-03	205.8	0.130	5	5350.
1,3,5-TNB	L-I-03	100.	0.130	5	2610.
1,3-DNB	L-I-03	104.	0.130	5	2699.
NB	L-I-03	100.8	0.130	5	2620.
Tetryl	L-I-03	100.	0.130	5	2610.
2A,4,6-DNT	L-I-03	104.	0.130	5	2691.
2,4,6-TNT	L-I-03	103.	0.130	5	2675.
2,6-DNT	L-I-03	10.2	0.130	5	264.9
2,4-DNT	L-I-03	10.3	0.130	5	268.3

The aliquot volume was obtained using a 100 ul microliter syringe and was diluted to volume in a Class "A" volumetric flask with methanol.

d. ID #L-W-10 (5 x TRL)

Compound	Parent I.D. Number	Conc. Parent ng/ul	Aliq. Vol. (ml)	Dilution Vol. (ml)	Final Conc. ng/ml
HMX	L-I-03	202.	0.325	5	13160.
RDX	L-I-03	205.8	0.325	5	13380.
1,3,5-TNB	L-I-03	100.	0.325	5	6526.
1,3-DNB	L-I-03	104.	0.325	5	6747.
NB	L-I-03	100.8	0.325	5	6550.
Tetryl	L-I-03	100.	0.325	5	6526.
2A,4,6-DNT	L-I-03	104.	0.325	5	6728.
2,4,6-TNT	L-I-03	103.	0.325	5	6689.
2,6-DNT	L-I-03	10.2	0.325	5	662.4
2,4-DNT	L-I-03	10.3	0.325	5	670.8

The aliquot volume was obtained using a 500 ul microliter syringe and was diluted to volume in a Class "A" volumetric flask with methanol.

e. ID #L-W-11 (10.0 x TRL)

Compound	Parent I.D. Number	Conc. Parent ng/ul	Aliq. Vol. (ml)	Dilution Vol. (ml)	Final Conc. ng/ml
HMX	L-I-03	202.	0.650	5	26300.
RDX	L-I-03	205.8	0.650	5	26760.
1,3,5-TNB	L-I-03	100.	0.650	5	13050.
1,3-DNB	L-I-03	104.	0.650	5	13490.
NB	L-I-03	100.8	0.650	5	13100.
Tetryl	L-I-03	100.	0.650	5	13100.
2A,4,6-DNT	L-I-03	104.	0.650	5	13450.
2,4,6-TNT	L-I-03	103.	0.650	5	13450.
2,6-DNT	L-I-03	10.2	0.650	5	1320.
2,4-DNT	L-I-03	10.3	0.650	5	1342.

The aliquot volume was obtained using a 500 ul microliter syringe and was diluted to volume in a Class "A" volumetric flask with methanol.

B. Instrument Calibration

Flow rate should be checked upon initial start-up of the HPLC system (by visually examining the flow rate of 1-2 ml through the system over time and checking for any pressure drop; 1 ml/min is the desired flow rate) along with other operating parameters to ensure stable conditions.

An initial equilibration time of approximately 15 minutes is recommended.

At the end of the day the HPLC system should be flushed with 100% acetonitrile until a stable pressure drop is observed and then allowed to equilibrate for 15 minutes prior to shut down.

The first analysis of the day is a mobile phase blank to ensure that there are no system interferences.

Inject 10 ul of the 10XTRL working calibration standard into the chromatographic system. Check the chromatogram to ensure separation of the nitrated toluenes and separation of the nitrobenzene and tetryl. If necessary, adjust the ratio of the mobile phase until separate peaks are distinguished. As the column ages, less methanol is required. Generally, the column ages rapidly the first 24 hours, after which it is fairly stable.

Once good peak separation is obtained, introduce 10 ul of each working calibration standard and an acetonitrile blank into the chromatographic system using the manufacturer's recommended procedure. Both sets of calibration standards are analyzed sequentially, preceded and followed by a 1.01 ug/ml check standard which is prepared from an independent, certified standard (2,6-DNT from the USEPA).

C. Analysis of Calibration Data

The criteria for acceptability of daily calibration standard data is ± 25% of the peak height found in the initial calibration of the 10X TRL standard.

More specifically, for the first seven calibrations, the response must agree within 25% of the mean response for the same concentration, as determined from precertification and the certification Initial Calibration. After seven calibrations, responses must agree within two standard deviations rather than a percentage. If the response fails the test, the daily standard shall be reanalyzed. If the response from the second analysis is not within the required percentage or two standard deviations of the mean response from precertification and the certification Initial Calibration, the system is considered to have failed calibration. All data generated since the last satisfactory calibration are considered questionable and must be reanalyzed after repeating Initial Calibration.

D. Calibration Checks

1. Standard Check, 10X TRL

	Peak Heights/% Differences*							
	DAY 1		DAY 2		DAY 3		DAY 4	
	Peak Height	% Diff	Peak Height	% Diff	Peak Height	% Diff	Peak Height	% Diff
HMX								
Initial	360.6	—	355.5	1.4	353.6	2.0	401.4	10.2
Final	350.0	2.9	348.8	3.3	345.1	4.5	366.0	1.5
RDX								
Initial	406.4	—	400.1	1.6	398.8	1.9	451.7	10.0
Final	392.9	3.3	391.3	3.9	388.4	4.6	412.6	1.5
1,3,5-TNB								
Initial	448.6	—	440.5	1.8	441.2	1.7	498.1	9.9
Final	434.1	3.2	430.8	4.1	429.9	4.3	453.6	1.1
1,3-DNB								
Initial	552.2	—	540.7	2.1	543.1	1.7	612.5	9.8
Final	533.9	3.3	528.4	4.5	527.6	4.6	556.6	0.79
NB								
Initial	376.5	—	369.5	1.9	372.1	1.2	419.1	10.2
Final	365.0	3.1	359.7	4.7	364.0	3.4	383.3	1.8
Tetryl								
Initial	266.2	—	242.8	9.6	260.4	2.2	305.6	12.9
Final	249.4	6.3	226.2	17.8	248.6	7.1	269.2	1.11
2A,4,6-DNT								
Initial	320.2	—	311.1	2.8	310.5	3.1	359.6	11.0
Final	304.3	5.0	302.6	5.8	300.6	6.5	321.9	0.52
2,4,6-TNT								
Initial	334.9	—	332.4	0.75	336.3	0.41	374.6	10.6
Final	329.1	1.8	323.8	3.4	330.2	1.4	342.7	3.9
2,6-DNT								
Initial	27.6	—	26.7	3.4	27.2	1.6	30.4	9.3
Final	25.3	8.9	25.9	6.5	25.9	6.2	26.9	2.6
2,4-DNT								
Initial	44.7	—	43.2	3.4	43.5	2.8	49.9	10.5
Final	42.2	5.5	41.8	6.8	41.6	6.9	45.0	0.67

*% differences are for each individual standard run compared to the initial response on Day 1.

V. CERTIFICATION TESTING

A. Water Spiking

1. Each day of testing six (6) 500 ml volumetrics were spiked with aliquots of the intermediate standard (L-I-03) and brought to volume with ASTM Type II water (with 100 mg/L sulfate and chloride added) to achieve the following concentrations:

Spiked Concentration: ug/L

	Blank	0.5X TRL	1X TRL	2X TRL	5X TRL	10X TRL
HMX	0	5.06	10.12	20.24	50.6	101.2
RDX	0	5.14	10.29	20.58	51.45	102.9
1,3,5-TNB	0	2.51	5.02	10.04	25.10	50.20
1,3-DNB	0	2.59	5.19	10.38	25.95	51.90
NB	0	2.52	5.09	10.08	25.20	50.40
Tetryl	0	2.51	5.02	10.04	25.10	50.20
2A,4,6-DNT	0	2.59	5.18	10.36	25.90	51.80
2,4,6-TNT	0	2.57	5.14	10.28	25.70	51.4
2,6-DNT	0	0.255	0.510	1.020	2.55	5.10
2,4-DNT	0	0.258	0.516	1.032	2.58	5.16
Aliquot(ml)	0	0.0125	0.025	0.050	0.125	0.25

VI. SAMPLE HANDLING AND STORAGE

A. Containers

Grab and/or composite samples will be collected and stored in 1-liter amber glass bottles with teflon-lined caps. Bottles should be filled to at least 3/4 total capacity.

B. Storage Conditions

Samples should be stored in appropriate containers in a refrigerator at 4° C prior to analysis.

C. Holding Times

Samples will be extracted within seven (7) days of collection and analyzed within 40 days of extraction.

VII. PROCEDURE

A. Separations

1. COLUMN PREPARATION

a. Insert divinyl benzene column into vacuum manifold, plugging any unused holes.

b. Attach vacuum gauge/controller and hoses to an aspirator nozzle on a water faucet.

c. Add 3 ml of HPLC grade methanol to each divinyl benzene column. Pull methanol through columns by use of vacuum system. Release vacuum.

d. Attach 75 ml column reservoir to top of divinyl benzene column.

e. Add 15 ml of reagent water to each reservoir. Pull water through divinyl benzene column to displace methanol.

2. SAMPLE SORPTION

a. Add a 500 ml aliquot of sample (prefiltered through a 0.45 micron filter) to an Erlenmeyer flask.

b. Check pH with wide range pH paper and adjust to 7 (± 0.5) with 1N NaOH or 1:1 H_2SO4.

c. Place approximately 40 ml of the 500 ml aliquot into the 75 ml reservoir.

d. Place stopper with glass tube in hole onto the reservoir. Attach tygon tubing to glass tube and insert disposable pipet into opposite end of tubing.

e. Pull sample through divinyl benzene column by use of vacuum system. Keep vacuum between 5" and 15" Hg at a rate of approximately 10 ml/min.

f. After all of the sample is filtered through the divinyl benzene column, pull air through the column for a minimum of 5 minutes.

g. Water rinse with approximately 10 milliliters of ASTM Class I water.

3. SAMPLE ELUTION

a. Remove the 75 ml reservoir and dry the inside of the vacuum manifold, being careful not to disturb the divinyl benzene columns.

b. Install the collection rack with 2 ml volumetric flasks under each divinyl benzene column.

c. Add 2 ml of HPLC grade methanol in 1 ml aliquots and pull it through the divinyl benzene column at approximately 4" to 5" Hg. Release vacuum slowly at gauge.

d. Remove the collection rack and bring the volume in each flask up to the mark with methanol, as required. Transfer the 2 ml of methanol to sample vials with teflon-lined caps.

e. The samples can now be directly injected for analysis.

B. CHEMICAL REACTIONS

No chemical reactions are involved in this analytical method. However, RDX and HMX undergo alkaline hydrolysis and both degrade at temperatures greater than 80°C in an organic solvent. Solutions of calibration standards, spiking mixtures, and sample extracts are refrigerated at 4°C and protected from the photodecomposition effect of light when not in use.

C. INSTRUMENTAL ANALYSIS

After proper calibration of the instrument introduce 10 ul of each sample into the system.

II. CALCULATIONS

Concentration (ug/l) =

$$\frac{\text{(peak height of sample) (vol. of extract) (1000) (dilution)}}{\text{(response factor) (vol. extracted)}}$$

where:

$$\text{response factor} = \frac{\text{peak height of standard}}{\text{concentration injected}}$$

vol. of extract = total volume of extract, in ml

vol. extracted = total volume of sample extracted, in ml

1000 = 1000 ml/l conversion factor

NOTE: For non-linear parameters, the response factor of the standard closest in concentration to the concentration in the sample will be used for this calculation.

IX. DAILY QUALITY CONTROL

A. Control Samples

See Section V.A. for preparation of the 2X CRL and 10X CRL spiking solutions.

B. Control Charts

1. Description of Charts to be Maintained

Single day X-R control charts are prepared for each control analyte using data from the duplicate spiked QC samples in each lot to determine percent recovery:

$$\frac{\text{Found Concentration}}{\text{Spiked Concentration}} \times 100$$

Use of percent recovery allows for minor variations in spiking solution concentrations.

To prepare the single day control charts, the analyst should have access to the following data:

- Percent recovery of each analyte in the two high concentration spiked QC samples.
- Average ($\overline{X}$) percent recovery for the two high level QC samples in each lot.
- Difference (R) between the percent recoveries for the two high level QC samples in each lot.

The initial control chart shall be prepared using the four days of certification data closest to the spiking concentration used during analyses. The average $\overline{X}$ ($\overline{\overline{X}}$), average range ($\overline{R}$), and control limits for $\overline{X}$ and R shall be updated after each in-control lot for the first 20 lots. Limits established after lot 20 shall be used for the next 20 lots. Control charts shall be updated after each 20 lots, thereafter, using the most recent 40 points.

Three-point moving average control charts shall be maintained for each control analyte spiked in the single low concentration spiked QC sample. The X-R three-point moving average control charts shall be constructed for each control analyte as follows:

- Use percent recovery to allow for minor variations in spiking concentration.
- The first plotted point is the average of the first three recoveries (from certification, at concentrations nearest the spiking level).
- Subsequent points are obtained by averaging the three most recent individual recovery values (outliers excluded from calculation, but not from plot).
- The range for each point is the difference between the highest and lowest value for each group of three values.

2a. Initial Control Limits - Single Day $\bar{X}$ and R

The control limits establihsed as of day 4 of the certification runs are as follows for the high level spike:

COMPOUND	$UCL_{\bar{X}}$	$LCL_{\bar{X}}$	$\bar{\bar{X}}$	UCL_R	$\bar{R}$
HMX	105.0	39.2	72.1	57.2	17.6
RDX	124.6	46.0	85.4	68.3	20.9
1,3,5-TNB	142.1	86.9	114.5	48.0	14.8
1,3-DNB	106.2	87.0	96.6	16.7	5.1
NB	102.0	84.4	93.2	15.4	4.8
Tetryl	146.8	94.6	120.7	45.4	13.9
2A,4,6-DNT	133.2	89.6	111.4	37.9	11.6
2,4,6-TNT	128.8	79.2	104.0	43.1	13.2
2,6-DNT	84.0	62.6	73.4	18.6	5.8
2,4-DNT	100.7	66.5	83.6	29.7	9.2

2b. Initial Control Limits - Three Day $\bar{X}$ and R

The control limits established as of day 4 of the certification runs are as follows for the low level spike:

COMPOUND	$UCL_{\bar{X}}$	$LCL_{\bar{X}}$	$\bar{\bar{X}}$	UCL_R	$\bar{R}$
HMX	94.8	55.0	74.9	50.2	19.6
RDX	127.0	36.6	78.5	113.8	44.2
1,3,5-TNB	138.4	62.6	100.6	95.3	37.0
1,3-DNB	128.2	93.6	110.9	43.5	16.9
NB	120.6	89.8	105.2	38.9	15.2
Tetryl	129.8	74.6	102.2	69.5	27.0
2A,4,6-DNT	119.1	64.7	91.9	68.5	26.6
2,4,6-TNT	115.0	49.8	82.4	82.1	31.9
2,6-DNT	95.8	70.8	83.4	31.4	12.2
2,4-DNT	107.9	40.5	74.2	84.7	32.9

X. REFERENCES

"Explosives in Soil by HPLC (Acetonitrile Extraction)" Method #8H; Prepared by Roy F. Weston, Inc., for USATHAMA; Revision Date 7/27/83.

"Nitroaromatics in Water Samples"; Prepared by Metatrace for USATHAMA.

XI. DATA

A. EPA Reference Solution Analysis

EPA reference material was 5000 ± 50 ug/ml of 2,6-DNT

Run	Concentration Found (ppb)	Concentration Expected (ppb)	% Return
Day 1	0.695	1.016	68.4%
Day 2	0.695	1.016	68.4%
Day 3	0.715	1.016	70.4%
Day 4	0.71	1.016	70. %

Low recovery was observed using the EPA standard. The labeled purity of this standard was 99.7% and no breakdown products were observed. Integration differences (single peak versus multiple peak; i.e.,

merging peaks, TNT, 2,6-DNT, 2,4-DNT) may account for some of the difference.

B. Raw data and statistical evaluations are appended as follows:

Appendix I - Response Versus Concentration Data
Response Versus Concentration Graphs
Lack of Fit (LOF) Tests
Zero Intercept (ZI) Tests

Appendix II - Raw Data

ENVIRONMENTAL RESTORATION PROGRAM

LIMITED FIELD INVESTIGATION
ADDENDUM TO THE QUALITY ASSURANCE PROJECT PLAN

MOUND PLANT
MISCELLANEOUS SITES, OPERABLE UNIT 3

October 1991

DEPARTMENT OF ENERGY
ALBUQUERQUE OPERATIONS OFFICE

ENVIRONMENTAL RESTORATION PROGRAM
TECHNICAL SUPPORT OFFICE
LOS ALAMOS NATIONAL LABORATORY

DRAFT

(REVISION 0)

OPERABLE UNIT 3 ADDENDUM
FOR DIOXIN/FURAN ANALYSES

LIMITED FIELD INVESTIGATION

QUALITY ASSURANCE PROJECT PLAN

DEPARTMENT OF ENERGY, MOUND PLANT

MIAMISBURG, OHIO

Approved by Diana Malley Date 1/23/92
U. S. EPA Region V Remedial Project Manager

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DISTRIBUTION LIST

This section of the Operable Unit 3 QAAP applies to this addendum.

ACRONYMS

This section of the Operable Unit 3 QAAP applies to this addendum, except for the following additions:

2,3,7,8-TCDD	-	tetrachlorodibenzo-p-dioxin
2,3,7,8-TCDF	-	tetrachlorodibenzofuran
1,2,3,7,8-PeCDD	-	pentachlorodibenzo-p-dioxin
1,2,3,7,8-PeCDF	-	pentachlorodibenzofuran
1,2,3,6,7,8-HxCDD	-	hexachlorodibenzo-p-dioxin
1,2,3,6,7,8-HxCDF	-	hexachlorodibenzofuran
1,2,3,4,6,7,8-HpCDD	-	heptachlorodibenzo-p-dioxin
1,2,3,4,6,7,8-HpCDF	-	heptachlorodibenzofuran
1,2,3,4,5,6,7,8-OCDD	-	octachlorodibenzo-p-dioxin
1,2,3,4,5,6,7,8-OCDF	-	octachlorodibenzofuran
HpCDD	-	hexachlorinated dibenzo-p-dioxin
HpCDF	-	hexachlorinated dibenzofuran
HxCDD	-	heptachlorinated dibenzo-p-dioxin
HxCDF	-	heptachlorinated dibenzofuran
MDL	-	method detection limit
MS	-	matrix spike
OCDD	-	octachlorinated dibenzo-p-dioxin
OCDF	-	octachlorinated dibenzofuran
OU	-	operable unit
PCDD	-	polychlorinated dibenzo-p-dioxin
PCDF	-	polychlorinated dibenzofuran
PeCDD	-	pentachlorinated dibenzo-p-dioxin
PeCDF	-	pentachlorinated dibenzofuran
TCDD	-	tetrachlorinated dibenzo-p-dioxin
TCDF	-	tetrachlorinated dibenzofuran

1. INTRODUCTION

This addendum to the Operable Unit 3 Quality Assurance Project Plan (QAPP) addresses quality assurance and quality control for the sampling and analysis of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs). The technical content for most of the QAPP elements for this addendum is contained within the Operable Unit 3 QAPP. Additional information and/ or procedures are provided in this document where necessary.

Table I.1 addresses analytical levels for PCDDs, PCDFs, 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD and 2,3,7,8-tetrachlorodibenzofuran (2,3,7,8,-TCDF).

Table I.1. Analytical Levels for Addendum to Operable Unit 3 Limited Field Investigation

Site	Purpose	Media	Field Parameters	Laboratory Parameters	Analytical Level
Glass melter room sump	Per OU 3 QAPP Table I.1	Sediment	Per O.U 3 QAPP Table I.1	PCDDs/PCDFs 2,3,7,8-TCDD 2,3,7,8-TCDF	IV IV IV
Building 34 sites:					
Oil burn structure	Per OU 3 QAPP Table I.1	Subsurface soil and sediment	Per O.U 3 QAPP Table I.1	PCDDs/PCDFs 2,3,7,8-TCDD 2,3,7,8-TCDF	IV IV IV
Fire fighting training facility		Subsurface soil and sediment	Per O.U 3 QAPP Table I.1	PCDDs/PCDFs 2,3,7,8-TCDD 2,3,7,8-TCDF	IV IV IV

PCDD - polychlorinated dibenzo-p-dioxin
PCDF - polychlorinated dibenzofuran
TCDD - tetrachlorodibenzo-p-dioxin
TCDF - tetrachlorodibenzofuran
QAPP - Quality Assurance Project Plan
OU - operable unit

2. PROJECT ORGANIZATION AND RESPONSIBILITY

This section of the Operable Unit 3 QAPP applies to this addendum, with the following amendment:

- Laboratory analyses of dioxins and furans will be performed by IT Technology Development Laboratory, Knoxville, TN, with the possible exception of samples taken from the glass melter room.

3. QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA IN TERMS OF ACCURACY, COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY

Quality control procedures for the dioxin and furan analyses are listed in Table III.1. Laboratory advisory limits for matrix spikes are listed in Table III.2.

Table III.1. Summary of Quality Control Procedures for Field Activities and Laboratory Measurements: Water and Soil/Sediment Samples

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
		Field quality control			
SW8280[a]	PCDD PCDF 2,3,7,8-TCDD 2,3,7,8-TCDF	Duplicate	1 every 10 or fewer field samples	Not applicable	Evaluate variability.
		Equipment (rinsate) blank[b]	1 every 20 or fewer field samples	≤ 10 x level in associated samples	Evaluate potential sources. Evaluate associated data for usability.
		Laboratory quality control			
		Method blank	1 per 20 samples or fewer of a given matrix	≤ quantitation limit	Investigate source; reextract and reanalyze associated samples with positive results for the same analyte(s).
		Matrix spike	1 per 20 samples or fewer of a given matrix	See Table III.2	Evaluate data for usability.
		Matrix spike duplicate	1 per 20 samples or fewer of a given matrix	See Table III.2	Evaluate data for usability.
		Tuning criteria	Daily or each 12-hour period, whichever is more frequent	Per SW8280	Correct problem; reanalyze required standards; recalibrate if necessary.
		Calibration	Initial 5 point	Per SW8280	Recalibrate before sample analysis.
		Internal standard	Every sample	25% - 200% recovery of internal standard	Evaluate data for usability.

Table III.1. (Continued)

Analytical Method	Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SW8280[a] (continued)	PCDD PCDF 2,3,7,8-TCDD 2,3,7,8-TCDF	System performance check compound-2,3,7,8-TCDD only	Beginning and end of each 12-hour period and every calibration	≤ 25% resolution for TCDD	Identify source and correct.
		Continuing calibration check compounds	Once at beginning of 12-hour period	≤ 30% difference from mean RRF	Identify source and correct. Recalibrate if source not found and corrected.
		PCDD/PCDF GC window definition	Daily and with every calibration	Windows within MS descriptor switch-points	Adjust MS switch-points.

[a] "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, EPA, November 1986 or most recent version.
[b] All field quality control samples associated with a batch of samples will be evaluated as a unit. This criterion is designed for evaluating an isolated quality control sample and does not take into account the interdependencies of quality control results. Corrective actions will be taken at all levels of detection in the blank samples associated with field sampling. The criterion applies only if there is a positive detection of the same compound in associated samples. All data will be evaluated on a case by case basis; therefore, this criterion may not be applicable at times, (e.g., reported levels near detection limit).

PCDD - polychlorinated dibenzo-p-dioxin
PCDF - polychlorinated dibenzofuran
2,3,7,8-TCDD - tetrachlorodibenzo-p-dioxin
2,3,7,8-TCDF - tetrachlorodibenzofuran
GC - gas chromatography
MDL - method detection limit
MS - matrix spikes

Table III.2. Laboratory Advisory Limits for Matrix Spikes and Matrix Spike Duplicates: Water and Soil/Sediment Samples IT Technology Development Laboratory, Knoxville, TN

Analytical Method	Spiking Compounds	Spike Concentration		Advisory Limits	
		Soil (ug/kg)	Water (ng/L)	Percent Recovery (%)	Relative Percent Difference (%)
SW8280[a]	2,3,7,8-TCDD	1.25	12.5	60 - 140	± 50%
	2,3,7,8-TCDF	1.25	12.5	60 - 140	± 50%
	1,2,3,7,8-PeCDD	1.25	12.5	60 - 140	± 50%
	1,2,3,7,8-PeCDF	1.25	12.5	60 - 140	± 50%
	1,2,3,6,7,8-HxCDD	3.13	31.3	60 - 140	± 50%
	1,2,3,4,7,8-HxCDF	3.13	31.3	60 - 140	± 50%
	1,2,3,4,6,7,8-HpCDD	3.13	31.3	60 - 140	± 50%
	1,2,3,4,6,7,8-HpCDF	3.13	31.3	60 - 140	± 50%
	1,2,3,4,5,6,7,8-OCDD	6.25	62.5	60 - 140	± 50%
	1,2,3,4,5,6,7,8-OCDF	6.25	62.5	60 - 140	± 50%

[a] "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, EPA, November 1986 or most recent version.

2,3,7,8-TCDD - tetrachlorodibenzo-p-dioxin
2,3,7,8-TCDF - tetrachlorodibenzofuran
1,2,3,7,8-PeCDD - pentachlorodibenzo-p-dioxin
1,2,3,7,8-PeCDF - pentachlorodibenzofuran
1,2,3,6,7,8-HxCDD - hexachlorodibenzo-p-dioxin
1,2,3,6,7,8-HxCDF - hexachlorodibenzofuran
1,2,3,4,6,7,8-HpCDD - heptachlorodibenzo-p-dioxin
1,2,3,4,6,7,8-HpCDF - heptachlorodibenzofuran
1,2,3,4,5,6,7,8-OCDD - octachlorodibenzo-p-dioxin
1,2,3,4,5,6,7,8-OCDF - octachlorodibenzofuran

4. SAMPLING PROCEDURES

Table IV.1 lists the container, preservation, and holding time requirements for dioxin and furan samples. Table IV.2 summarizes the sampling and analysis program for dioxins and furans.

Table IV.1. Sample Containers, Volumes, Preservation, and Holding Times: Soil/Sediment Samples and Water Samples

Parameters	Analytical Method	Container[a]	Minimum Volume/Weight	Preservation	Holding Time[b]
PCDD/PCDF 2,3,7,8-TCDD 2,3,7,8-TCDF	SW8280[c]	Amber glass jar with Teflon-lined lid	50 grams (soil) 1 liter (water)	Cool 4 deg. C	30 days extraction/ 45 days analysis[b]

[a]Sample containers will be certified clean by the manufacturer according to EPA standards.
[b]From date of collection.
[d]"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, EPA, November 1986 or most recent version.

PCDD - polychlorinated dibenzo-p-dioxin
PCDF - polychlorinated dibenzofuran
2,3,7,8-TCDD - tetrachlorodibenzo-p-dioxin
2,3,7,8-TCDF - tetrachlorodibenzofuran

Table IV.2. Summary of Sampling and Analysis Program

Sample Matrix	Laboratory Parameters	Investigative Samples			Field Quality Control Samples						MS/MSD			Matrix Total
					Duplicates			Equipment Blanks						
		No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	
Subsurface Soil	PCDD PCDF 2,3,7,8-TCDD 2,3,7,8-TCDF	20	1	20	2	1	2	1	1	1	1	1	1	23
Sediment	PCDD PCDF 2,3,7,8-TCDD 2,3,7,8-TCDF	3	1	3	1	1	1	1	1	1	1	1	1	5

PCDD - polychlorinated dibenzo-p-dioxin
PCDF - polychlorinated dibenzofuran
2,3,7,8-TCDD - tetrachlorodibenzo-p-dioxin
2,3,7,8-TCDF - tetrachlorodibenzofuran
MS/MSD - matrix spike/matrix spike duplicate

5. SAMPLE CUSTODY

This section of the Operable Unit 3 QAPP applies to this addendum.

6. ANALYTICAL PROCEDURES

Soil/sediment and water samples will be analyzed for PCDDs, PCDFs, 2,3,7,8-TCDD, and 2,3,7,8-TCDF using the methodology based on SW-846 method 8280. The IT Technology Development Laboratory utilizes a methanol/hexane extraction of the soil samples, and a methylene chloride extraction of the water sample, followed by solvent exchange to hexane. After cleanup, the extracts are analyzed by high-resolution gas chromatography (GC)/low-resolution mass spectrometry (MS) analysis. The required minimum initial cleanup of the extract will consist of a silica gel/alumina column cleanup, followed by a carbon column cleanup. **These required cleanup procedures supersede any laboratory SOPs or practices to the contrary.** If GC/MS results are indeterminable by the laboratory, then optional cleanup procedures will be employed, specifically acid/base wash or additional column cleanup using silica gel, alumina, or carbon, as determined by the experienced analyst. In the case of extracts containing known or suspected (i.e., color) interferents, the acid/base wash may precede the column cleanup procedures. Table VI.1 lists the target compounds and the required detection limits.

Table VI.1. Dioxin/Furan Analytes, Quantitation Limits, and Analytical Methods

Analyte	Quantitation Limit[a]	
	Soil/Sediment (μg/kg)	Water (ng/L)
2,3,7,8-TCDD	1.0	10
Total TCDD	2.0	20
2,3,7,8-PeCDD	2.0	20
Total PeCDD	2.0	20
2,3,7,8-HxCDD	2.0	20
Total HxCDD	2.0	20
2,3,7,8-HpCDD	2.0	20
Total HpCDD	2.0	20
OCDD	2.0	20
2,3,7,8-TCDF	1.0	10
Total TCDF	2.0	20
2,3,7,8-PeCDF	2.0	20
Total PeCDF	2.0	20
2,3,7,8-HxCDF	2.0	20
Total HxCDF	2.0	20
2,3,7,8-HpCDF	2.0	20
Total HpCDF	2.0	20
OCDF	2.0	20

[a]Analyzed by USEPA SW-846 method 8280.

2,3,7,8-TCDD - tetrachlorodibenzo-p-dioxin
2,3,7,8-TCDF - tetrachlorodibenzofuran
TCDD - tetrachlorinated dibenzo-p-dioxin
TCDF - tetrachlorinated dibenzofuran
PeCDD - pentachlorinated dibenzo-p-dioxin
PeCDF - pentachlorinated dibenzofuran
HxCDD - hexachlorinated dibenzo-p-dioxin
HxCDF - hexachlorinated dibenzofuran
HpCDD - heptachlorinated dibenzo-p-dioxin
HpCDF - heptachlorinated dibenzofuran
OCDD - octachlorinated dibenzo-p-dioxin
OCDF - octachlorinated dibenzofuran

7. CALIBRATION PROCEDURES AND FREQUENCY

GC/MS will be used for the analysis of PCDDs and PCDFs according to methodology based on SW8280 (EPA 1986). A mixture of PCDD/PCDF isomers will be used to calibrate the GC/MS and must meet specific ion ratio criteria as established in method SW8280. Meeting these criteria is demonstrated each time the instrument is calibrated with a calibration check mixture. The tuning is also verified whenever a corrective action to the GC/MS system is taken that affects the tuning (e.g., ion source cleaning or repair).

Initial calibration of the GC/MS system is accomplished with a minimum of five concentrations of the target compounds. Relative response factors (RRFs) are calculated for the target compounds.The relative standard deviations for the RRFs must be less than 15% or initial calibration is not valid.

The initial calibration is verified every 12-hours with a column performance check sample mixture and a midpoint calibration standard. The column performance check mixture is used to verify column resolution for 2,3,7,8-TCDD. RRFs calculated from the midpoint calibration standard must be within ± 30% of the initial RRF mean value for the initial calibration to be valid. If these conditions are not met, the GC/MS system should be evaluated and corrective action taken (e.g., recalibrate).

8. INTERNAL QUALITY CONTROL CHECKS

Internal quality control checks for PCDD/PCDF analyses are specified in the analytical method and in Table III.1 in this addendum. Field quality control checks include a duplicate for every 10 samples collected and an equipment (rinsate) blank for every 20 samples collected. Laboratory quality control checks shall include: method blanks, matrix spikes, matrix spike duplicates, calibration, use of internal standards, and system performance check mixtures. Detailed definitions of the quality control checks are given in section 8 of the Operable Unit 3 QAPP. The frequency of the quality control checks are specified in Table III.1.

9. DATA REDUCTION, VALIDATION, AND REPORTING

Data reduction and validation will be performed as specified in section 9 of the Operable Unit 3 QAPP. Laboratory data report packages for PCDD/PCDF analyses will consist, at a minimum, of the following items where applicable:

- a case narrative for each laboratory batch of samples analyzed;
- a cross-reference of laboratory sample identification numbers to the project sample identification numbers;
- a description of data qualifiers used in the laboratory report;
- a record of sample extraction/preparation for all field samples and laboratory quality control checks;
- a summary page stating the extraction and analysis dates for each field sample and laboratory quality control check;
- sample results;
- results of matrix spikes, matrix spike duplicates, method blank, initial calibration, continuing calibration checks, and replicate samples; and
- labeled and dated chromatograms/spectra of sample results and the laboratory quality control checks listed above.

The case narrative for each data report package will summarize the following information on the given set of samples analyzed:

- date of issue;
- the laboratory analysis performed;
- any deviations from the stated analytical method;
- the laboratory batch number;
- the number of samples and the sample matrices;
- a reference to the quality control procedures performed for the specific methods used, including the reference to the acceptance criteria used;
- the contents of the laboratory report;
- the project name and number;
- the state of the samples received (e.g., whether preserved and packed properly);

- whether the sample holding times were met, and identification of those for which they were not;

- any observations that may have had an impact on the analyses;

- any technical problems affecting the analysis and corrective actions taken;

- laboratory quality control checks that did not meet the project criteria (as specified in the QAPP) and/or laboratory criteria (include any corrective actions taken and any known possible reasons for the results); and

- the laboratory manager's signature approving the issuance of the data package.

A copy of the chain-of-custody with all relinquished signatures will accompany each data package.

10. PERFORMANCE AND SYSTEM AUDITS

This section of the Operable Unit 3 QAPP applies to the addendum.

11. PREVENTATIVE MAINTENANCE

Table XI.1 lists preventative maintenance requirements for major instruments utilized by this addendum.

Table XI.1. Preventative Maintenance Requirements for Major Instruments IT Laboratories

Applicable Analytical Method	Instrument	Items Checked/Serviced	Minimum Frequency
SW8280[a]	Gas chromatograph	Replace septa	Daily
	Mass spectrometer	Perform column maintenance	As needed
		Replace column	As needed
		Clean source	Quarterly, as needed
		Replace injection port liner	As needed
		Clean poles	Quarterly, as needed
		Clean fans	Monthly
		Printer maintenance	Monthly
		Printer ribbon replacement	As needed
		Retune	Quarterly, as needed
		Dip rods	Quarterly, as needed
		Change mechanical pump oil	Biannually
		Change turbo pump oil	Annually
		Change water filter	Annually
		Change gas line filter	As needed
		Cool flow maintenance	Biannually
		Calibrate instruments	Quarterly, as needed

Note: "As needed" under minimum frequency refers to the fact that the item is routinely checked during analyses, and the item is replaced/repaired when it fails or is found to be worn.

[a] "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, EPA, November 1986 or most recent version.

12. SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

This section of the Operable Unit 3 QAPP applies to this addendum.

13. CORRECTIVE ACTION PROTOCOLS

This section of the Operable Unit 3 QAPP applies to this addendum.

14. QUALITY ASSURANCE REPORTS TO MANAGEMENT

This section of the Operable Unit 3 QAPP applies to this addendum.

15. REFERENCES

This section of the Operable Unit 3 QAPP applies to this addendum.