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QUALITY ASSURANCE PROJECT PLAN FOR REMEDIAL INVESTIGATION FMPC FERNALD, OHIO VOLUME 5

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VOLUME V

QUALITY ASSURANCE PROJECT PLAN

FOR

REMEDIAL INVESTIGATION

FEED MATERIALS PRODUCTION CENTER

FERNALD, OHIO

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REMEDIAL INVESTIGATION AND FEASIBILITY STUDY

FEED MATERIALS PRODUCTION CENTER
FERNALD, OHIO

TASK 2 REPORT
WORK PLAN REQUIREMENTS

REVISION 3 OF:

WORK PLAN

VOLUME I: SAMPLING PLAN

VOLUME II: HEALTH AND SAFETY PLAN

VOLUME III: COMMUNITY RELATIONS PLAN

VOLUME IV: DATA MANAGEMENT PLAN

VOLUME V: QUALITY ASSURANCE PROJECT PLAN

CONTAINED HEREIN

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1.0 INTRODUCTION

Advanced Sciences, Inc. (ASI) has been retained by the U.S. Department of Energy (DOE) to provide a site wide Remedial Investigation and Feasibility Study (RI/FS) for the Feed Materials Production Center (FMPC). This Quality Assurance Project Plan (QAPP) has been prepared by ASI and IT Corporation (IT) (a subcontractor to ASI) to document measures that will be undertaken by ASI and its subcontractors so that the work performed will be of needed quality to accomplish project objectives and will be responsive to requirements of Westinghouse Materials Company of Ohio (WMCO), the DOE, and the United States Environmental Protection Agency (U.S. EPA).

The QAPP is the governing document of quality assurance (QA) practices to be implemented for the FMPC remedial investigation. This document includes the quality objectives, the requirements for work performance to meet these objectives, and the means for verifying that the objectives have been met.

DOCUMENT CHANGE REQUEST NO. 23

REVISION OF THE WELL NUMBERING SYSTEM

Two revisions to the well numbering system for the RI/FS are being made. The first revision is to make the three digit designation a four digit designation. The second change is to assign depth and location designator well numbers to the existing wells that are identified by some other name, such as OS-1 or State 16.

Increase in Identifier Digits

The current practice is to give wells a series designation such as 100, 200, or 300, and a two-digit location such as 14, 64, or 89. The series number provides the depth designation of the well. For example, all 200-series wells are screened across the water table. All 100-series wells are in the till.

The location designation provides the a link between well series. The location designation means that all wells with the same last two digits were drilled in the same location. Thus, if a list of wells such as 215, 346, 315, 115 is provided, the reader instantly knows that three of the wells were drilled to different depths at one location and one well was drilled at another location to the same depth as the deepest well at the first location.

One limitation of this system is that there can be no more than 99 locations. There are only a few remaining location numbers left available in the present three-digit numbering system. This is insufficient to include all off-site wells and the anticipated additional wells that are likely before the RI/FS investigation is completed. The solution to this situation is to increase the number of digits in the location number from two to three. The effect is to shift the series number from the hundreds to the thousands column. Thus, former 100-series wells will become 1000-series, 200-series will become 2000-series, and so forth. Specifically, Well 115 will be 1015, Well 215 will be 2015, Well 315 will be 3015, and Well 415 will be 4015. This allows for the addition of 900 well location numbers, which should be adequate for the investigation and create minimum confusion in the transmission. There will be well numbers beginning with 1100, 2100, and 3100 in the near future that could create some short-term confusion.

DOCUMENT CHANGE REQUEST NO. 23

Assigning Additional Location Numbers

The current practice is to use well owner's names, Miami Conservancy District well names, or names that were established during the NLO Litigation Study as the identifier for wells that are off-site and were not funded by the DOE. This practice does not allow for the same simple well construction identification as the RI/FS well numbering system. These names are also of variable length and require special handling by the RI/FS Data Base Management System. Because of this, data from these wells may not be included in a particular search request. Table 1 lists the wells currently being used in the RI/FS and the new four-digit well designation that well be used in the future in the RI/FS. The old names are still in the computer system and searches can be made by individual name so that historical data is not lost. In the future, a data request for a map with all uranium values in 2000-series wells for a given sampling period will include the off-site wells. Similarly, maps will appear with the four-digit number so that an observer will know by the well number that the data he is looking at is from a well that samples a specific interval.

Implementation

These changes will become effective on September 9, 1988. This is between quarterly water sampling events and after the completion of the current off-site drilling program. Thus, the changes at this time will have a minimum effect on the day-to-day operations of the RI/FS investigation.

TABLE 1
RE-NAMED WELLS

NEW NAME	PREVIOUS NAME
1124	Cone House
1053	Argonne A-S
3053	Argonne A-D
1054	Argonne B-S
3054	Argonne B-D
1055	Argonne C-S
3055	Argonne C-D
4023* Corrected number	BU-13
2026	BU-101
2036* Corrected number	12-5
1040	RB
2050	Pallet Co.
2056	State 8
2057	State 16
1058	DH
1059	1A
2060	OS-1
1060	OS-1A
2061	OS-2
3062	OS-3
4101	P-1
4102	P-2
4103	P-3
3063	James Dill
3099	Robert James
3100	Charles Young
2121	BU-91
2122	BU-92
2123	12-3
2104	BPH
2105	State-10

2.0 PROJECT DESCRIPTION

The tasks that comprise the site characterization for the remedial investigation are as follows: description of current situation, work plan requirements, site investigation, site investigation analysis, laboratory and bench-scale studies, reports, community relations support, and assistance with the Federal Facility Compliance Agreement (FFCA).

The following ten laboratories, which are (1) CLP-certified (presently have a U.S. EPA contract), (2) participate in CLP (presently analyze Performance Evaluation Samples), or (3) participate in the U.S. EPA Cross-Check Program (radiological laboratories only), will be used to analyze FMPC samples.

Laboratory	Type of Analysis
Radiological Sciences Laboratory (RSL) (3) Oak Ridge, Tennessee	Radiological
Mixed Waste Laboratory (MWL) (2) Oak Ridge, Tennessee	Chemical
Middlebrook Pike Laboratory (1, organics only; 2) Knoxville, Tennessee	Chemical
Export Laboratory (1, organics only; 2) Pittsburgh, Pennsylvania	Chemical, Geotechnical
Special Analysis Laboratory (2) Knoxville, Tennessee	Dioxins
Santa Clara Valley Laboratory (1, pesticides only) San Jose, California	Organo-phosphorus pesticides
Cerritos Laboratory (1, organics only; 2) Cerritos, California	Chemical
Austin Laboratory (2) Austin, Texas	Chemical
PEI Laboratory (1, organics only; 2) Cincinnati, Ohio	Chemical
Edison Laboratory (2) Edison, New Jersey	Acute & Chronic

Laboratory-specific attachments of the laboratories are supporting procedures to the QAPP and are controlled documents that are considered proprietary information. Copies of applicable documents can be supplied to WMCO or others as directed by WMCO for this project, if requested.

2.1 Site Description and History

The FMPC is an industrial facility owned by the United States government and operated for the DOE under a management contract with WMCO. WMCO is administratively responsible to the Department of Energy -- Oak Ridge Operations Office (DOE-ORO).

The FMPC is located in a rural area of southwestern Ohio approximately ten miles northwest of Cincinnati and eight miles southwest of Hamilton. The site occupies 1,050 acres and is bounded by Highway 126 to the north, a transmission line to the east, Willey Road to the south, and Paddy's Run Road and the Ohio and Chesapeake Railroad to the west (see Figure 1-1, Work Plan).

The facility commenced operations in 1952; prior to 1986, it was operated by National Lead of Ohio, Inc. (NLO) under contract with DOE. The primary function of the FMPC is the production of metallic uranium fuel cores and other uranium compounds for use in United States defense programs. In addition, small amounts of

thorium have also been processed. As a result of these processes, the FMPC has generated both radioactive and nonradioactive wastes.

The FFCA was entered into on July 18, 1986, by the DOE-ORO and the U.S. EPA, pertaining to the FMPC. The FFCA was entered into pursuant to Executive Order 12088 (43 Federal Register [FR] 47707) to set forth compliance with existing environmental statutes and implementing regulations. The FFCA is intended to provide that the potential environmental impacts associated with past and present activities at the FMPC are thoroughly and adequately investigated and appropriate remedial response actions are taken, as required by the Resource Conservation and Recovery Act (RCRA) as amended, 42 USC 6901 et seq., and by the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA), 42 USC 9601 et seq., and by the Superfund Amendments and Reauthorization Act (SARA).

In accordance with the FFCA, this site characterization is being conducted at the FMPC to determine the nature and extent of releases, or threat of releases, of hazardous or radioactive substances, or pollutants at or from the FMPC. Additionally, data necessary to support a Remedial Investigation (RI) and Feasibility Study (FS) will be gathered.

Information developed for the RI will subsequently be used to support preparation of an FS. The purpose of the FS will be to develop and evaluate remedial action alternatives and recommend remedial action alternatives to the U.S. EPA in order to protect the public health, welfare, or the environment from releases or threatened releases of hazardous or radioactive substances, or pollutants at or from the FMPC.

A complete description of the site background and history is provided in Section 2.0 of the Work Plan. Section 3.0 of the

Work Plan discusses the nature and extent of problems at the FMPC.

2.2 PROJECT SCHEDULE

ASI anticipates beginning work on the FMPC 30 days after satisfactory review and approval of the Health and Safety Plan, the QAPP, and the Work Plan. Anticipated completion of the site characterization investigation will be approximately 21 months after site work begins.

2.3 INTENDED USE OF ACQUIRED DATA

The intended use of the acquired data is to assess the nature of the site and the degree and extent of potential problems resulting from past activities at the site, to evaluate the potential hazard to human health and the environment, and to identify and evaluate the alternative remedial actions. These objectives can be efficiently met by completing the following tasks:

- Characterizing the site in terms of wastes present and produced; lateral and vertical extent of contamination in surface waters, ground waters, sediments, and soils; rate and direction of waste migration; target receptors (population at risk, threatened resources, and sensitive ecosystems); and site geology and surface water/ground water hydrology.

- Developing and evaluating alternative production, handling, and disposal measures considering economic feasibility, technological feasibility, environmental impacts, regulatory constraints, and timeliness of the completion; and offering recommendations regarding the most cost-effective remedial alternatives.

3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

3.1 ORGANIZATION

As prime contractor, ASI is the project director and is directly responsible to DOE/WMCO for the success of this project. ASI has organized a project team with extensive experience in the technical and administrative disciplines required for this project. A major subcontractor to ASI is IT Corporation (IT), with analytical laboratory capabilities and certifications conforming to U.S. EPA specifications, mixed waste capabilities, health physics technology, and RI/FS experience in hydrogeology and feasibility studies.

The Fernald FMPC RI/FS Project, though basically divided into RI and FS phases, involves diverse technical disciplines, common to both phases. The project organization has been structured to include experienced professional specialists in these various disciplines. The project organization chart is shown in Figure 3-1.

3.2 AUTHORITY AND RESPONSIBILITY

3.2.1 PROJECT DIRECTOR

The Project Director has total responsibility for the completion and technical adequacy of the RI/FS. He is responsible for the accomplishment of the RI/FS on schedule and within allocated cost. The project director will have responsibility for technical, financial, and scheduling matters. Other duties, as necessary, include:

- Procurement, along with administrative personnel, and supervision of subcontractor services;
- Assignment of duties to the project staff and orientation of the staff to the needs and requirements of the project;
- Review of project-specific procedures and internally prepared plans, drawings, and reports;

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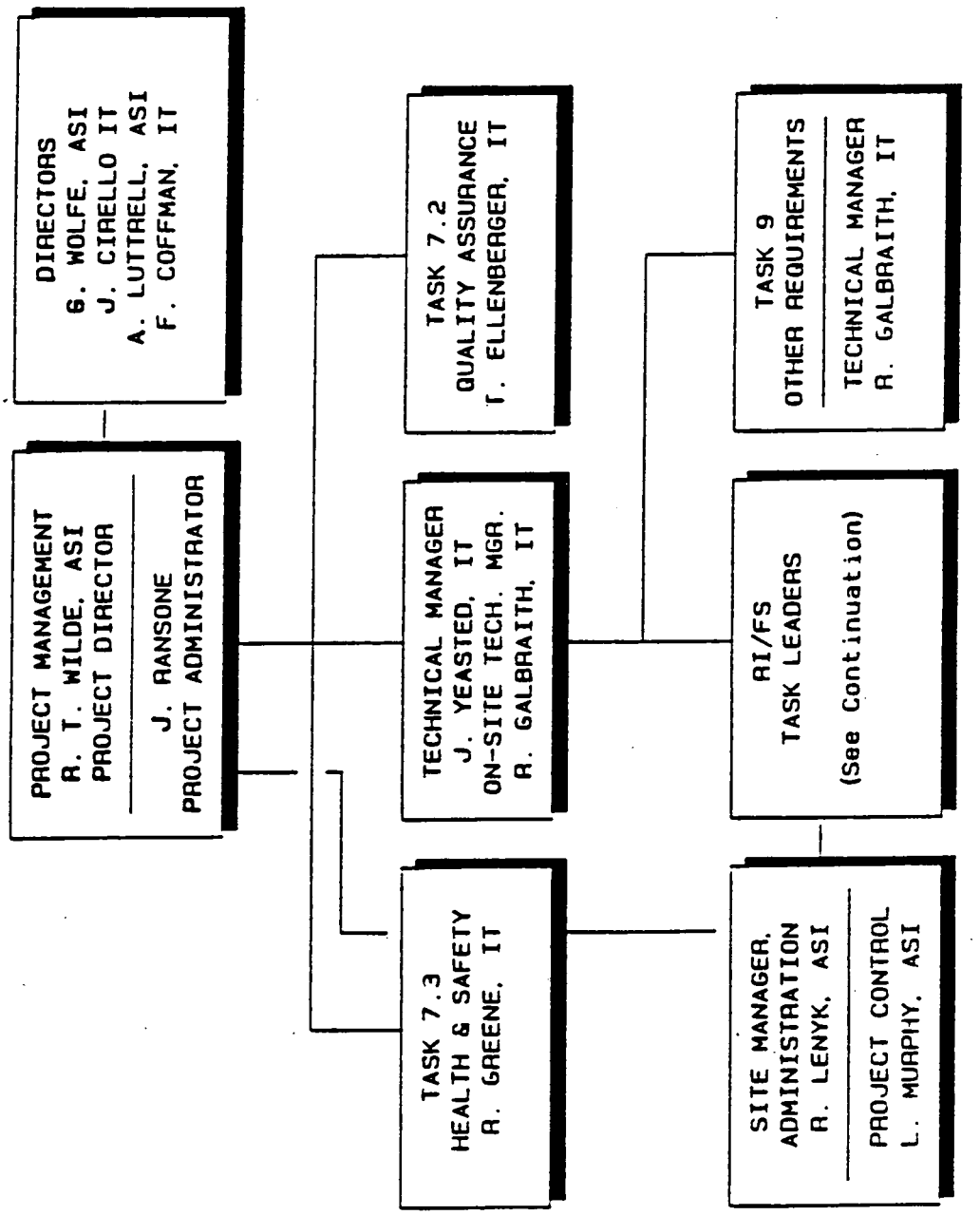

FIGURE 3-1 ORGANIZATION CHART

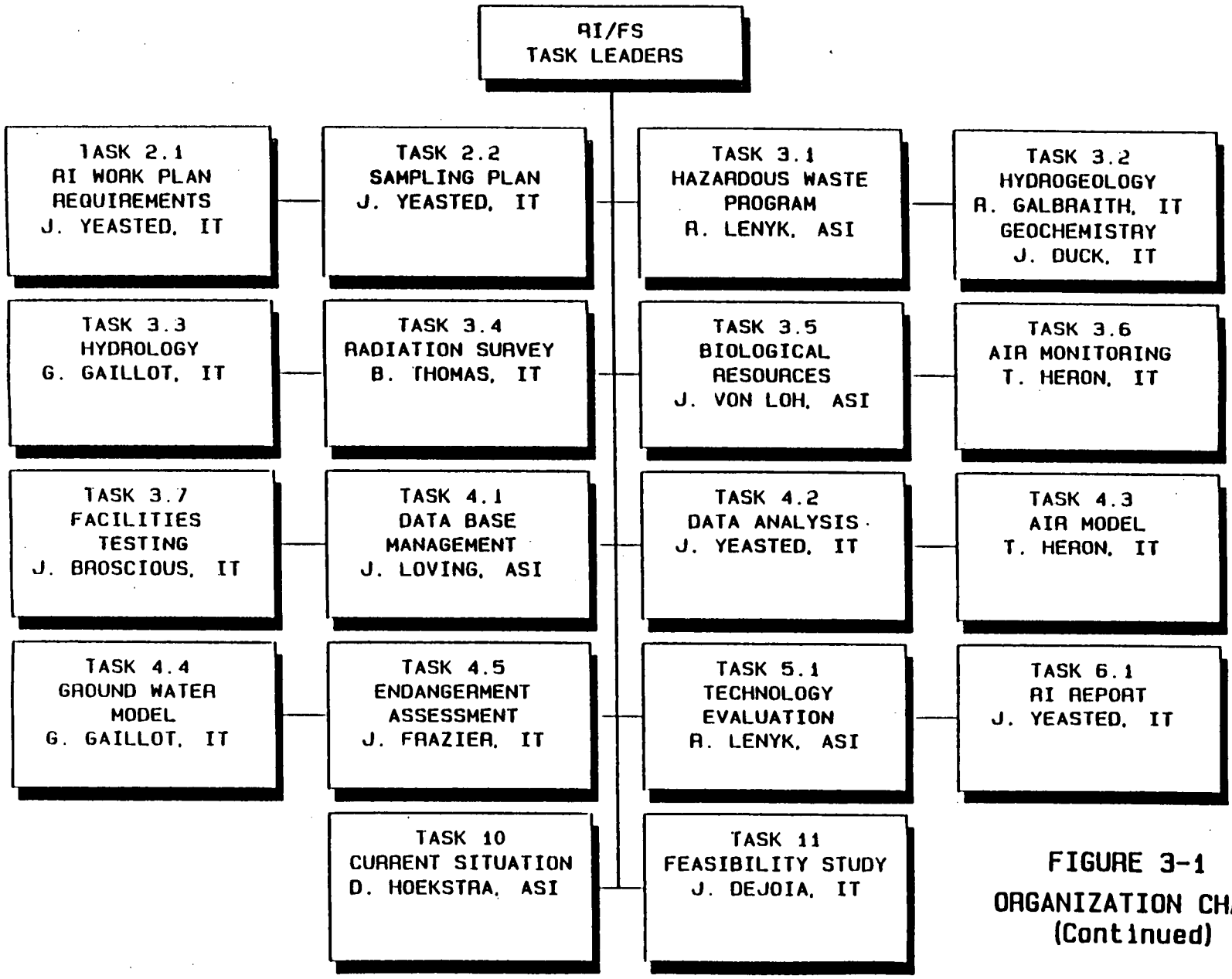

FIGURE 3-1
ORGANIZATION CHART
(Continued)

- Dissemination of project-related information to U.S. EPA and the public as requested by WMCO/DOE;
- Serving as liaison between the project staff and other internal groups, such as the laboratories, Quality Assurance (QA), and Health and Safety.

3.2.2 IT TECHNICAL MANAGER

The IT Technical Manager reports directly to the Project Director. He is responsible for the technical quality of all RI/FS tasks. The Technical Manager's responsibilities include:

- Technical review of procedures, plans, drawings, and reports;
- Supervision and coordination of technical RI/FS tasks and activities; and
- Reporting the project technical status and progress to the Project Director.

3.2.3 SITE MANAGER

The ASI Site Manager reports directly to the Project Director. He is responsible for coordination of all site activities related to performance and scheduling of field work including support personnel, materials procurement, and field subcontract administration. He coordinates with the IT On-Site Technical Manager in administrative matters and in maintaining the cost and schedule control of the technical activities. He is responsible for issuing technical cost, schedule, and progress reports as directed by the Project Director.

3.2.4 IT ON-SITE TECHNICAL MANAGER

The IT On-Site Technical Manager reports to the Project Director. He provides technical assistance and acts for the IT Technical Manager, when appropriate. These responsibilities include:

- Coordination of on-site IT personnel and resources;
- Schedule and cost-control of IT-led tasks; and
- Technical surveillance of all field activities.

3.2.5 OTHER REQUIREMENTS ADMINISTRATOR

The Other Requirements Administrator reports to the ASI Site Manager. He provides cost-estimating and schedule control for all Task 9 activities. Individual Task 9 leaders will report directly to the Project Director. The ASI Site Manager is responsible for staffing Task 9 activities so that they do not interfere with RI/FS activities.

3.2.6 QUALITY ASSURANCE OFFICER

The QA Officer reports to the Project Director and is responsible for guidance in the development of the QAPP and control of project QA/QC activities. The QA Officer will provide the necessary guidance to the project and laboratory staffs on quality-related matters and perform project audits and surveillance. The QA Officer has the authority and responsibility to identify quality problems and initiate, recommend, or provide corrective actions. The QA Officer verifies the implementation of the corrective actions. Other duties include:

- Notification ~~of~~ to personnel of nonconformances and changes in QA procedures and verifying the implementation of corrective action;
- Determination of audit and surveillance schedules and conducting the audit and surveillance;
- Review and approval of quality-related project documents;
- Serving as the official contact for all QA matters for the project;
- Identification of QA needs, resolving QA problems, answering requests for guidance or assistance, and verifying administration of nonconformances; and
- Verification that data meets the intended work objectives.

The QA Officer may take actions independent of the project group to stop the project if required for compliance with the QAPP.

3.2.7 HEALTH AND SAFETY OFFICER

The Health and Safety Officer is responsible for preparing and implementing a Health and Safety Plan which satisfies state and federal regulations and is consistent with site conditions. The Health and Safety Officer may take actions independent of the project group to stop the project, if required, for compliance with the Health and Safety Plan.

The Health and Safety Officer is responsible for the day-to-day implementation of the Health and Safety Plan during site activities. Specifically, the officer's responsibilities include:

- Issuing personnel protective equipment and proper dosimetry;
- Approval of personnel protective equipment and safety procedures specified in task plans or work procedures;
- Maintaining and using field monitoring equipment necessary to define on-site hazards associated with conducting RI activities;
- Designating personnel protection levels and deciding protection level upgrades; and
- Providing necessary guidance to the project and laboratory staffs so they can safely perform their functions in accordance with federal and state regulations.

3.2.8 TASK LEADERS

Task leaders are assigned for each Work Breakdown Structure (WBS) Level 2 sub-task (2.1, 3.3, 3.4, etc.). They report to the ASI Site Manager or the IT On-Site Technical Manager for administrative matters and report to the IT Technical Manager for technical issues. Task Leaders are responsible for:

- Technical, cost, and schedule planning;
- Maintaining cognizance of DOE and U.S. EPA requirements;

- Staffing, using both ASI and IT personnel; and
- Evaluation of progress and associated reporting.

Personnel staffing these key positions are identified in Table 3-1. Thirty days notice is provided to WMCC prior to any changes in this listing.

3.3 PROJECT COMMUNICATIONS

Incoming project-related materials in the form of correspondence, sketches, logs, authorizations, or other information will be routed to the project director, or designee, after the original is marked with the date received. The project director, or designee, will then determine which personnel will review the materials and route the materials accordingly.

As soon as practical, incoming correspondence originals will be placed in the project central file at the ASI/Fernald office. If the correspondence is required by the project personnel for reference, a copy should be made rather than holding the original. Correspondence which is of importance to the QAPP will be routed to the QA officer.

Project-related materials transmitted externally including correspondence, reports, drawings, and sketches will be appropriately reviewed, approved, and, if necessary, signed prior to transmittal. Project outgoing correspondence will, as a minimum, be signed by the project director, or a key level individual assigned this responsibility by the project director. If joint signatures are desirable, the originator of the correspondence, when different from management, may also sign. QA correspondence will be signed by the QA officer. Outgoing project correspondence and reports should be read by the project director, or designee, prior to issue. The office copy of project correspondence should bear routing information and be routed to the QA officer, if judged appropriate by the project director.

TABLE 3-1

RI/FS KEY PERSONNEL LIST

ADVANCED SCIENCES, INC. (ASI)

1.	R. T. Wilde	- Project Director
2.	R. G. Lenyk	- Site Manager, - Technology Evaluation (Task 5.1) Task Leader
3.	J. F. Ransone	- Project Administrator
4.	L. T. Murphy	- Project Control Program Manager - Other Requirements Administrator
5.	J. Von Loh	- Biological Resources (Task 3.5) Task Leader
6.	J. E. Loving	- Data Base Management (Task 4.1) Task Leader
7.	M. F. Nicol	- Corporate Quality Assurance Manager

TABLE 3-1 (Continued)

IT CORPORATION

1.	J. G. Yeasted	- Hydrology (Task 3.2) Task Leader - Technical Manager - RI Work Plan (Task 2.1) Task Leader - RI Sampling Plan (Task 2.2) Task Leader - Data Analysis (Task 4.2) Task Leader - RI Final Report (Task 6.1) Task Leader
2.	R. M. Galbraith	- On-Site Technical Manager
3.	T. L. Ellenberger	- Quality Assurance
4.	R. T. Green	- Health and Safety Officer
5.	G. Gaillot	- Hydrology (Task 3.3) Task Leader - Ground Water Modeling (Task 4.4) Task Leader
6.	J. J. Duck	- Geochemistry (Task 3.2) Task Leader
7.	J. A. Broscious	- Facilities Testing (Task 3.7) Task Leader
8.	J. R. Frazier	- Radiation Survey (Task 4.2) Task Leader - Endangerment Assessment (Task 4.5) Task Leader
9.	T. M. Heron	- Air Modeling (Task 4.3) Task Leader

Communications relative to the project which are initiated by third parties (e.g., media, interested individuals and groups) will be referred directly to designated WMCO representative(s) without comment, unless directed otherwise.

4.0 QUALITY ASSURANCE PROGRAM

The purpose of a QA program is to establish policies for the implementation of regulatory requirements and to provide an internal means for control and review so that the work performed is of the highest professional standard.

The responsibility for the overall direction of the QA program rests with the QA officer. The QA officer is responsible for maintaining the QA program and verifying its implementation through audits and surveillances.

4.1 Quality Assurance Documents

The QA program is documented in this QAPP and supporting procedures that direct quality-related activities. The policies and procedures specified by these manuals define acceptable practices to be used by personnel. The QAPP is project-specific and serves as the governing QA document for this project.

The QA manuals of IT Analytical Services and IT Engineering Services and Laboratory-Specific Attachments of RSL, Mixed Waste Laboratory, Middlebrook Pike Laboratory, Special Analysis Laboratory, Santa Clara Valley Laboratory, Cerritos Laboratory, Austin Laboratory, PEI Laboratory, Edison Laboratory, and the Export Geotechnical Laboratory are supporting procedures to the QAPP and are controlled documents that are considered proprietary information. Copies of applicable documents can be supplied to WMCO or others as directed by WMCO for this project, if requested.

4.2 Project Quality Assurance Objectives

Project quality assurance objectives are that:

- Scientific data will be of sufficient or greater quality to meet scientific and legal scrutiny;
- Data will be gathered or developed in accordance with procedures appropriate for the intended use of the data; and
- Data will be of known or acceptable precision, accuracy, completeness, representativeness, and comparability as required by the FMPC project.

Fundamental mechanisms that will be employed to achieve these quality goals can be categorized as prevention, assessment, and correction. These include:

- Prevention of errors by planning, documented instructions and procedures, and careful selection and training of skilled, qualified personnel;
- Quality assessment through a program of audits and surveillances to supplement continual informal review; and
- Correction for prevention of recurrence of conditions adverse to quality.

The QAPP has been prepared in direct response to these goals. This plan describes the QA Program to be implemented and the quality control (QC) procedures to be followed by ASI and its subcontractors during the course of the RI/FS for the FMPC site.

The QAPP describes the project organization structure and specifies the procedures, documentation requirements, sample custody requirements, acceptance criteria, audit and corrective action provisions, etc. to be applied to provide confidence that all operations and activities meet the intent of RCRA requirements. The QAPP is prepared in accordance with U.S. EPA guidance as presented in Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80.

The procedures contained or referred to herein have been taken from:

- IT Analytical Services Quality Assurance Manual;
- IT Engineering Services Quality Assurance Manual;
- IT/Radiological Sciences Laboratory Quality Assurance Manual;
- IT/Radiological Sciences Laboratory Radioanalytical Methodology and Procedures Manual;

- Laboratory-Specific Attachments for RSL, Mixed Waste Laboratory, Middlebrook Pike Laboratory, Special Analysis Laboratory, Santa Clara Valley Laboratory, Cerritos Laboratory, Austin Laboratory, PEI Laboratory, Edison Laboratory, and Export Geotechnical Laboratory;
- RCRA Ground-Water Monitoring Technical Enforcement Guidance Document (TEGD), September, 1986.
- Code of Federal Regulations (CFR), 40 CFR 261, Appendix II, "EP Toxicity Test Procedures;"
- U.S. EPA Quality Assurance Handbook, (U.S. EPA 600/9-76-005);
- Proposed Sampling and Analytical Methodologies for Addition to Test Methods for Evaluating Solid Waste Physical/Chemical Methods, U.S. EPA, (PB85-103026);
- American Society for Testing and Materials (ASTM) Standards, Section 11, Volumes 11.01 and 11.02, "Water," and Section 4, Volume 04.08, "Soil and Rock, Building Stones;"
- American Public Health Association, Standard Methods for the Examination of Water and Waste Water, 16th Edition, 1985;
- Methods for Chemical Analysis of Water and Wastes, (U.S. EPA-600/4-79-020);
- Federal Register, Volume 49, October 26, 1984, 40 CFR 136, pages 43234-43436;
- Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, U.S. EPA (SW-846);
- Handbook for Sampling and Sample Preservation of Water and Waste Water, U.S. EPA (PB83-124503);
- Field and Laboratory Methods Applicable to Overburdens and Mine Soil, (U.S. EPA-600/2-80-054);
- Prescribed Procedures for Measurement of Radioactivity in Drinking Water, (U.S. EPA-600/4-80-032);
- Code of Federal Regulations, 10 CFR 50, Appendix B, "Quality Assurance Criteria for Nuclear Power Plants and Fuel Processing Plants;"

- American National Standards Institute (ANSI) NQA-1, Quality Assurance Program Requirements for Nuclear Facilities;

- USNRC Regulatory Guide 4.15, Rev. 1, "Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment";
- Chemical Analytical Services for Multi-Media Multi-Concentration Metals and Inorganics, U.S. EPA (WA-87-K026);
- Chemical Analytical Services for Multi-Media Multi-Concentration Organics, GC/MS Techniques, U.S. EPA (WA-87-K236); and
- Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, U.S. EPA, (QAMS 005/80).

4.3 ANALYTICAL QUALITY ASSURANCE OBJECTIVES FOR DATA

QA objectives have been established for precision, accuracy, completeness, representativeness, and comparability for each major measurement parameter for the FMPC project. These objectives are presented in Tables 4-1, 4-2, and 4-3 and are pertinent to both the analytical laboratories and field procedures. Data will be calculated and reported in units consistent with Contract Laboratory Program (CLP) analytical procedures and other organizations reporting similar data to allow for comparability of data. Definitions for precision, accuracy, completeness, representativeness, and comparability are as follows:

- Precision - A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the standard deviation. Comparison of replicate values is best expressed as the relative percent difference (RPD). Various measures of precision exist depending upon the "prescribed similar conditions."
- Accuracy - The degree of agreement of a measurement (or an average of replicate measurements), 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 in a system.
- Completeness - A measure of the amount of valid data obtained from a measurement system compared to the

TABLE 4-1

GENERAL CHEMISTRY AND METALS
METHOD REFERENCES AND QUALITY CONTROL OBJECTIVES
FEED MATERIALS PRODUCTION CENTER
FERNALD, OHIO

PARAMETER	INSTRUMENT	FREQUENCY OF CALIBRATION CHECKS	TYPICAL PRECISION RPD %(a)	TYPICAL ACCURACY % OF RECOVERY	EXPECTED COMPLETENESS (%)	EXPECTED METHOD DETECTION LIMIT(i)	METHOD NO.	REFERENCE
Alkalinity	pH meter	Daily(b) (water)	±10	-	90	1000 ug/l(g)(water)	310.1	1
		Daily (solids)	±10	-	90	500 mg/kg(h)(solids)	3.2.3	2
Aluminum	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	100 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	20 mg/kg (solids)	200.7	12
Ammonia	Distillation with UV/VIS Spectrophotometer	Daily (water)	±10	±20	90	50 ug/l (water)	350.2	1
		Daily (solids)	±10	±20	90	2 mg/kg (solids)	33-2,350.2	11,1
	Specific Ion Meter/ Microprocessor	Daily (water)	±10	±20	90	50 ug/l (water)	350.2	1
		Daily (solids)	±10	±20	90	2 mg/kg (solids)	33-2,350.2	11,1
Antimony	Atomic Absorption Spectrophotometer	Daily (water)	±10	±20	90	10 ug/l (water)	204.1,204.2	1
		Daily (solids)	±10	±20	90	2 mg/kg (solids)	7040,7041	3
Arsenic	Atomic Absorption Spectrophotometer	Daily (water)	±10	±20	90	10 ug/l (water)	206.2	1
		Daily (solids)	±10	±20	90	2 mg/kg (solids)	7060	3

See footnotes at end of table.

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TABLE 4-1
(Continued)

PARAMETER	INSTRUMENT	FREQUENCY OF CALIBRATION CHECKS	TYPICAL PRECISION RPD %(a)	TYPICAL ACCURACY % OF RECOVERY	EXPECTED COMPLETENESS	EXPECTED METHOD DETECTION LIMIT(i)	METHOD NO.	REFERENCE
Barium	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	100 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	20 mg/kg (solids)	200.7	12
Beryllium	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	5 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	1 mg/kg (solids)	200.7	12
Boron	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	10 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	2 mg/kg (solids)	200.7	12
Cadmium	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	5 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	1 mg/kg (solids)	200.7	12
Calcium	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	100 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	20 mg/kg (solids)	200.7	12

See footnotes at end of table.

TABLE 4-1
(Continued)

PARAMETER	INSTRUMENT	FREQUENCY OF CALIBRATION CHECKS	TYPICAL PRECISION RPD %(a)	TYPICAL ACCURACY % OF RECOVERY	EXPECTED COMPLETENESS (%)	EXPECTED METHOD DETECTION LIMIT(i)	METHOD NO.	REFERENCE
Cation Exchange Capacity	Atomic Absorption Spectrophotometer	Daily (solids)	±10	±20	90	1 mg/kg (solids)	3.2.17	2
Chloride	Buret/Indicator	Daily (water)	±10	±20	90	500 ug/l (water)	325.3	1
		Daily (solids)	±10	±20	90	9 mg/kg (solids)	10-2,325.3	11,1
Chromium (Total)	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	10 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	2 mg/kg (solids)	200.7	12
Chromium VI	UV/VIS Spectrophotometer	Daily (water)	±10	±20	90	10 ug/l (water)	7196	3
		Daily (solids)	±10	±20	90	1 mg/kg (solids)	7196	3
Cobalt	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	10 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	2 mg/kg (solids)	200.7	12
Copper	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	10 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	2 mg/kg (solids)	200.7	12
Cyanide (Total)	Distillation with UV/VIS Spectrophotometer	Daily (water)	±10	±20	90	20 ug/l (water)	335.2	1
		Daily (solids)	±10	±20	90	0.50 mg/kg (solids)	9010	3
Dissolved Solids (Total)	Filtration/ Analytical Balance	3 months (water)	±10	-	90	1000 ug/l (water)	160.1	1

See footnotes at end table.

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TABLE 4-1
(Continued)

PARAMETER	INSTRUMENT	FREQUENCY OF CALIBRATION CHECKS	TYPICAL PRECISION RPD %(a)	TYPICAL ACCURACY % OF RECOVERY	EXPECTED COMPLETENESS	EXPECTED METHOD DETECTION LIMIT(i)	METHOD NO.	REFERENCE
Fluoride	pH meter with Specific Ion Electrode	Daily (water)	±10	±20	90	100 ug/l (water)	340.2	1
		Daily (solids)	±10	±20	90	1 mg/kg (solids)	10-2,340.2	11,1
Iron	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	100 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	20 mg/kg (solids)	200.7	12
Iron (Leachable)	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (solids)	±10	±20	90	1 mg/kg (solids)	200.7(e)	5,12
Lead	Atomic Absorption Spectrophotometer	Daily (water)	±10	±20	90	5 ug/l (water)	239.1,239.2	1
		Daily (solids)	±10	±20	90	1 mg/kg (solids)	7420,7421	3
Lithium	Atomic Absorption Spectrophotometer	Daily (water)	±10	±20	90	100 ug/l (water)	303A	6
		Daily (solids)	±10	±20	90	2 mg/kg (solids)	303A	6
Magnesium	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	100 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	20 mg/kg (solids)	200.7	12

See footnotes at end of table.

TABLE 4-1
(Continued)

PARAMETER	INSTRUMENT	FREQUENCY OF CALIBRATION CHECKS	TYPICAL PRECISION RPD %(a)	TYPICAL ACCURACY % OF RECOVERY	EXPECTED COMPLETENESS	EXPECTED METHOD DETECTION LIMIT(i)	METHOD NO.	REFERENCE
Manganese	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	10 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	2 mg/kg (solids)	200.7	12
Manganese (Leachable)	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (solids)	±10	±20	90	1 mg/kg (solids)	200.7(e)	5,12
Mercury	Atomic Absorption Spectrophotometer	Daily (water)	±10	±20	90	0.2 ug/l (water)	245.1	1
		Daily (solids)	±10	±20	90	0.10 mg/kg (solids)	7471	3
Molybdenum	Atomic Absorption Spectrophotometer	Daily (water)	±10	±20	90	10 ug/l (water)	246.1,246.2	1
		Daily (solids)	±10	±20	90	2 mg/kg (solids)	246.1,246.2	1
Nickel	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	30 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	6 mg/kg (solids)	200.7	12
Nitrate	UV/VIS Spectrophotometer	Daily (water)	±10	±20	90	100 ug/l (water)	352.1	1
		Daily (solids)	±10	±20	90	1 mg/kg (solids)	10-2,352.1	11,1

See footnotes at end of table.

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TABLE 4-1
(Continued)

PARAMETER	INSTRUMENT	FREQUENCY OF CALIBRATION CHECKS	TYPICAL PRECISION RPD %(a)	TYPICAL ACCURACY % OF RECOVERY	EXPECTED COMPLETENESS	EXPECTED METHOD DETECTION LIMIT(i)	METHOD NO.	REFERENCE
Organic Carbon (Total) (TOC)	Total Organic Carbon Analyzer	Daily (water)	±25	±20	90	1000 ug/l (water)	415.1	1
		Daily (solids)	±25	±20	90	1 mg/kg (solids)	(f)	8
Organic Halogens (Total) (TOX)	Total Organic Halide Analyzer	Daily (water)	±10	±20	90	10 ug/l (water)	9020	3
		Daily (solids)	±10	±20	90	0.1 mg/kg (solids)	9020	3
Organic Nitrogen (Total)	Distillation with UV/VIS Spectrophotometer	Daily (water)	±10	±20	90	1.0 mg/l (water)	351.3	1
		Daily (solids)	±10	±20	90	2 mg/kg (solids)	351.3	1
	Specific Ion Meter/ Microprocessor	Daily (water)	±10	±20	90	1.0 mg/l (water)	351.3	1
		Daily (solids)	±10	±20	90	2 mg/kg (solids)	351.3	1
pH	pH meter	Daily (water)	±10	-	90	0.05 pH unit (water)	150.1	1
		Daily (solids)	±10	-	90	0.05 pH unit (solids)	3.2.2	2
Phosphorus	UV/VIS Spectrophotometer	Daily (water)	±10	±20	90	10 ug/l (water)	365.2	1
		Daily (solids)	±10	±20	90	100 ug/kg (solids)	(f)	9
Potassium	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	100 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	20 mg/kg (solids)	200.7	12
Selenium	Atomic Absorption Spectrophotometer	Daily (water)	±10	±20	90	5 ug/l (water)	270.2	1
		Daily (solids)	±10	±20	90	1 mg/kg (solids)	7740	3
Silicon	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	100 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	20 mg/kg (solids)	200.7	12

See footnotes at end of table.

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TABLE 4-1
(Continued)

PARAMETER	INSTRUMENT	FREQUENCY OF CALIBRATION CHECKS	TYPICAL PRECISION RPD %(a)	TYPICAL ACCURACY % OF RECOVERY	EXPECTED COMPLETENESS (%)	EXPECTED METHOD DETECTION LIMIT(i)	METHOD NO.	REFERENCE
Silver	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	10 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	2 mg/kg (solids)	200.7	12
Sodium	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	100 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	20 mg/kg (solids)	200.7	12
Specific Conductance	Conductivity Meter	Daily (water)	±10	-	90	1 umho/cm (water)	120.1	1
		Daily (solids)	±10	-	90	1 umho/cm (solids)	10-2,120.1	11,1
Strontium	Atomic Absorption Spectrophotometer	Daily (water)	±10	±20	90	10 ug/l (water)	303A	6
		Daily (solids)	±10	±20	90	2 mg/kg (solids)	303A	6
Sulfate	UV/VIS Spectrophotometer	Daily (water)	±10	±20	90	2000 ug/l (water)	375.4	1
		Daily (solids)	±10	±20	90	10 mg/kg (solids)	3.2.6	2
Sulfide	Buret/Indicator	Daily (water)	±10	±20	90	500 ug/l (water)	376.1	1
		Daily (solids)	±10	±20	90	10 mg/kg (solids)	9030	3
Thallium	Atomic Absorption Spectrophotometer	Daily (water)	±10	±20	90	10 ug/l (water)	279.1,279.2	1
		Daily (solids)	±10	±20	90	2.0 mg/kg (solids)	7840,7841	4

See footnotes at end of table.

TABLE 4-1
(Continued)

PARAMETER	INSTRUMENT	FREQUENCY OF CALIBRATION CHECKS	TYPICAL PRECISION RPD %(a)	TYPICAL ACCURACY % OF RECOVERY	EXPECTED COMPLETENESS	EXPECTED METHOD DETECTION LIMIT(i)	METHOD NO.	REFERENCE
Tin	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	50 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	10 mg/kg (solids)	200.7	12
Vanadium	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	50 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	10 mg/kg (solids)	200.7	12
Zinc	Inductively Coupled Argon Plasma Emission Spectrometer	Daily (water)	±10	±20	90	10 ug/l (water)	200.7	12
		Daily (solids)	±10	±20	90	2 mg/kg (solids)	200.7	12

(a)RPD = relative percent difference.

(b)Each day samples are analyzed.

(c)The indicated relative percent difference is representative of the corresponding U.S Environmental Protection Agency (U.S. EPA) Contract Laboratory Program (CLP) Hazardous Substance List (HSL) compounds as a group.

(d)The indicated percent recovery range represents the minimum and maximum acceptable value for the indicated U.S. EPA CLP HSL compounds as a group. However, individual percent recovery ranges have been established for each matrix spike compound. Consult "Chemical Analytical Services for Multi-Media Multi-Concentration Organics, GC/MS Techniques," WA-87-K236, U.S. EPA, Washington, D.C., 1987 for more detailed quality assurance/quality control criteria.

TABLE 4-1
(Continued)

(e)A method number is not assigned to the iron and manganese leaching procedures cited from the corresponding reference.
(f)A method number is not assigned to the indicated analytical procedures cited from the corresponding reference.
(g)ug/l equals micrograms per liter or parts per billion (ppb)
(h)mg/kg equals milligrams per kilogram or parts per million (ppm).
(i)Method detection limits may vary with sample matrix.

REFERENCES:

(1) U.S. Environmental Protection Agency (EPA), March 1983, "Methods for Chemical Analysis of Water and Wastes," Publication EPA-600/4-79-020.

(2) U.S. Environmental Protection Agency (EPA), March 1978, "Field and Laboratory Methods Applicable to Overburdens and Mine Soils," EPA-600/2-78-054.

(3) U.S. Environmental Protection Agency (EPA), November 1986, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, revised 3rd edition.

(4) U.S. Environmental Protection Agency (EPA), 1984, "Proposed Sampling and Analytical Methodologies for Addition to Test Methods for Evaluating Solid Waste Physical/Chemical Methods," PB85-103026.

(5) Filipek, L. H., et al., 1981, "Factors Affecting the Partitioning of Cu, Zn, and Pb in Boulder Coatings and Stream Sediments in the Vicinity of a Polymetallic Sulfide Deposit," Chemical Geology, No. 33, pp. 45-64.

(6) APHA-AWWA-WPCF, 1985, Standard Methods for the Examination of Water and Waste Water, 16th Edition.

(7) Federal Register, October 26, 1984, "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater," Appendix A, 40 CFR 136, published in Volume 49, No. 209.

(8) Oceanographics International Corporation, no date, Operating Procedure Manual for 524B Total Carbon System, College Station, Texas.

(9) Walsh, L. M. and J. D. Blaton, 1973, "Soil Testing and Plant Analysis," Soil Science Society of America, Madison, Wisconsin.

(10) U.S. Department of the Interior, 1967, "Methods for Analyzing and Testing Coal and Coke," Bulletin 638, U.S. Bureau of Mines, Washington, D.C.

(11) American Society of Agronomists, 1982, "Methods of Soil Analysis," Part II, "Chemical and Microbial Properties," A. L. Page, Editor, 9, Madison, Wisconsin

(12) U.S. Environmental Protection Agency (EPA), 1987, "Chemical Analytical Services for Multi-Media Multi-Concentration Metals and Inorganics," WA-87-K026, Washington, D.C.

TABLE 4-2

CONTRACT LABORATORY PROGRAM (CLP)
HAZARDOUS SUBSTANCE LIST (HSL) COMPOUND
METHOD REFERENCES AND QUALITY CONTROL OBJECTIVES
FEED MATERIALS PRODUCTION CENTER
FERNALD, OHIO

PARAMETER	INSTRUMENT	FREQUENCY OF CALIBRATION CHECKS	MATRIX SPIKE PRECISION RPD%(a) Water	MATRIX SPIKE PRECISION RPD%(a) Solids	MATRIX SPIKE ACCURACY % OF RECOVERY Water	MATRIX SPIKE ACCURACY % OF RECOVERY Solids	SURROGATE SPIKE ACCURACY % OF RECOVERY Water	SURROGATE SPIKE ACCURACY % OF RECOVERY Solids	EXPECTED COMPLETENESS %	EXPECTED METHOD DETECTION LIMIT(b) Water ug/l(c)	EXPECTED METHOD DETECTION LIMIT(b) Solids ug/kg(d)	METHOD NO.	REFERENCE
Volatiles:	Gas Chromatograph/ Mass Spectrometers	Daily							90			Exhibit D	1
Chloromethane			-	-	-	-	-	-		10	10		
Bromomethane			-	-	-	-	-	-		10	10		
Vinyl Chloride			-	-	-	-	-	-		10	10		
Chloroethane			-	-	-	-	-	-		10	10		
Methylene Chloride			-	-	-	-	-	-		5	5		
Acetone			-	-	-	-	-	-		10	10		
Carbon Disulfide			-	-	-	-	-	-		5	5		
1,1-Dichloroethene			14	22	61-145	59-172	-	-		5	5		
1,1-Dichloroethane			-	-	-	-	-	-		5	5		
trans-1,2-Dichloroethene			-	-	-	-	-	-		5	5		
Chloroform			-	-	-	-	-	-		5	5		
1,2-Dichloroethane			-	-	-	-	-	-		5	5		
2-Butanone			-	-	-	-	-	-		10	10		
1,1,1-Trichloroethane			-	-	-	-	-	-		5	5		
Carbon Tetrachloride			-	-	-	-	-	-		5	5		
Vinyl Acetate			-	-	-	-	-	-		10	10		
Bromodichloromethane			-	-	-	-	-	-		5	5		
1,1,2,2-Tetrachloroethane			-	-	-	-	-	-		5	5		
1,2-Dichloropropane			-	-	-	-	-	-		5	5		
trans-1,3-Dichloropropene			-	-	-	-	-	-		5	5		
Trichloroethene			14	24	71-120	62-137	-	-		5	5		
Dibromochloromethane			-	-	-	-	-	-		5	5		
1,1,2-Trichloroethane			-	-	-	-	-	-		5	5		
Benzene			11	21	76-127	66-142	-	-		5	5		
cis-1,3-Dichloropropene			-	-	-	-	-	-		5	5		

See footnotes at end of table.

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TABLE 4-2
(Continued)

PARAMETER	INSTRUMENT	FREQUENCY OF CALIBRATION CHECKS	MATRIX SPIKE PRECISION RPD%(a) Water	MATRIX SPIKE PRECISION RPD%(a) Solids	MATRIX SPIKE ACCURACY % OF RECOVERY Water	MATRIX SPIKE ACCURACY % OF RECOVERY Solids	SURROGATE SPIKE ACCURACY % OF RECOVERY Water	SURROGATE SPIKE ACCURACY % OF RECOVERY Solids	EXPECTED COMPLETENESS %	EXPECTED METHOD DETECTION LIMIT(b) Water ug/l(c)	EXPECTED METHOD DETECTION LIMIT(b) Solids ug/kg(d)	METHOD NO.	REFERENCE
Volatiles (Continued):													
2-Chloroethyl Vinyl Ether			-	-	-	-	-	-		10	10		
Bromoform			-	-	-	-	-	-		5	5		
2-Hexane			-	-	-	-	-	-		10	10		
4-Methyl-2-pentanone			-	-	-	-	-	-		10	10		
Tetrachloroethene			-	-	-	-	-	-		5	5		
Toluene			13	21	76-125	59-139	-	-		5	5		
Chlorobenzene			13	21	75-130	60-133	-	-		5	5		
Ethylbenzene			-	-	-	-	-	-		5	5		
Styrene			-	-	-	-	-	-		5	5		
Total Xylenes			-	-	-	-	-	-		5	5		
SURROGATE COMPOUNDS:													
Toluene-d_3			-	-	-	-	88-110	81-117		-	-		
4-Bromofluorobenzene			-	-	-	-	86-115	74-121		-	-		
1,2-Dichloroethane-d_4			-	-	-	-	76-114	70-121		-	-		
Acid/Base-Neutral Extractables:	Gas Chromatograph/ Mass Spectrometers	Daily							90			Exhibit D	1
Phenol			42	35	12-89	26-90	-	-		10	330		
Bis(2-Chloroethyl)ether			-	-	-	-	-	-		10	330		
2-Chlorophenol			40	50	27-123	25-102	-	-		10	330		
1,3-Dichlorobenzene			-	-	-	-	-	-		10	330		
1,4-Dichlorobenzene			28	27	36-97	28-104	-	-		10	330		
Benzyl Alcohol			-	-	-	-	-	-		10	330		
1,2-Dichlorobenzene			-	-	-	-	-	-		10	330		
2-Methylphenol			-	-	-	-	-	-		10	330		
Bis(2-chloroisopropyl)ether			-	-	-	-	-	-		10	330		
4-Methylphenol			-	-	-	-	-	-		10	330		
N-Nitroso-di-n-propylamine			38	38	41-116	41-126	-	-		10	330		
Hexachloroethane			-	-	-	-	-	-		10	330		
Nitrobenzene			-	-	-	-	-	-		10	330		
Isophorone			-	-	-	-	-	-		10	330		

See footnotes at end of table.

TABLE 4-2
(Continued)

PARAMETER	INSTRUMENT	FREQUENCY OF CALIBRATION CHECKS	MATRIX SPIKE PRECISION RPD%(a) Water	MATRIX SPIKE PRECISION RPD%(a) Solids	MATRIX SPIKE ACCURACY % OF RECOVERY Water	MATRIX SPIKE ACCURACY % OF RECOVERY Solids	SURROGATE SPIKE ACCURACY % OF RECOVERY Water	SURROGATE SPIKE ACCURACY % OF RECOVERY Solids	EXPECTED COMPLETENESS %	EXPECTED METHOD DETECTION LIMIT(b) Water ug/l(c)	EXPECTED METHOD DETECTION LIMIT(b) Solids ug/kg(d)	METHOD NO.	REFERENCE
Acid/Base-Neutral Extractables: (Continued)													
2-Nitrophenol			-	-	-	-	-	-		10	330		
2,4-Dimethylphenol			-	-	-	-	-	-		10	330		
Benzoic Acid			-	-	-	-	-	-		50	1600		
Bis(2-chloroethoxy)methane			-	-	-	-	-	-		10	330		
2,4-Dichlorophenol			-	-	-	-	-	-		10	330		
1,2,4-Trichlorobenzene			28	23	39-98	38-107	-	-		10	330		
Naphthalene			-	-	-	-	-	-		10	330		
4-Chloroaniline			-	-	-	-	-	-		10	330		
Hexachlorobutadiene			-	-	-	-	-	-		10	330		
4-Chloro-3-methylphenol			42	33	23-97	26-103	-	-		10	330		
2-Methylnaphthalene			-	-	-	-	-	-		10	330		
Hexachlorocyclopentadiene			-	-	-	-	-	-		10	330		
2,4,6-Trichlorophenol			-	-	-	-	-	-		50	1600		
2,4,5-Trichlorophenol			-	-	-	-	-	-		10	330		
2-Chloronaphthalene			-	-	-	-	-	-		50	1600		
2-Nitroaniline			-	-	-	-	-	-		10	330		
Dimethylphthalate			-	-	-	-	-	-		10	330		
Acenaphthylene			31	19	46-118	31-137	-	-		50	1600		
3-Nitroaniline			-	-	-	-	-	-		10	330		
Acenaphthene			-	-	-	-	-	-		50	1600		
2,4-Dinitrophenol			-	-	-	-	-	-		50	1600		
4-Nitrophenol			50	50	10-80	11-114	-	-		10	330		
Dibenzofuran			-	-	-	-	-	-		10	330		
2,4-Dinitrotoluene			38	47	24-96	28-89	-	-		10	330		
2,6-Dinitrotoluene			-	-	-	-	-	-		10	330		
Diethylphthalate			-	-	-	-	-	-		10	330		
4-Chlorophenyl Phenyl Ether			-	-	-	-	-	-		10	330		
Fluorene			-	-	-	-	-	-		50	1600		
4-Nitroaniline			-	-	-	-	-	-		50	1600		
4,6-Dinitro-2-methylphenol			-	-	-	-	-	-		10	330		
N-Nitrosodiphenylamine			-	-	-	-	-	-		10	330		
4-Bromophenyl Phenyl Ether			-	-	-	-	-	-		10	330		
Hexachlorobenzene			50	47	9-103	17-109	-	-		50	1600		
Pentachlorophenol			-	-	-	-	-	-		10	330		
Phenanthrene													

See footnotes at end of table.

TABLE 4-2
(Continued)

PARAMETER	INSTRUMENT	FREQUENCY OF CALIBRATION CHECKS	MATRIX SPIKE PRECISION RPD2(a) Water	MATRIX SPIKE PRECISION RPD2(a) Solids	MATRIX SPIKE ACCURACY % OF RECOVERY Water	MATRIX SPIKE ACCURACY % OF RECOVERY Solids	SURROGATE SPIKE ACCURACY % OF RECOVERY Water	SURROGATE SPIKE ACCURACY % OF RECOVERY Solids	EXPECTED COMPLETENESS %	EXPECTED METHOD DETECTION LIMIT(b) Water ug/l(c)	EXPECTED METHOD DETECTION LIMIT(b) Solids ug/kg(d)	METHOD NO.	REFERENCE
Acid/Base-Neutral Extractables: (Continued)													
Anthracene			-	-	-	-	-	-		10	330		
Di-n-butylphthalate			40	47	11-117	29-135	-	-		10	330		
Fluoranthene			-	-	-	-	-	-		10	330		
Pyrene			31	36	26-127	35-142	-	-		10	330		
Butyl Benzyl Phthalate			-	-	-	-	-	-		10	330		
3,3'-Dichlorobenzidine			-	-	-	-	-	-		20	660		
Benzo(a)anthracene			-	-	-	-	-	-		10	330		
Bis(2-ethylhexyl)phthalate			-	-	-	-	-	-		10	330		
Chrysene			-	-	-	-	-	-		10	330		
Di-n-octylphthalate			-	-	-	-	-	-		10	330		
Benzo(b)fluoranthene			-	-	-	-	-	-		10	330		
Benzo(k)fluoranthene			-	-	-	-	-	-		10	330		
Benzo(a)pyrene			-	-	-	-	-	-		10	330		
Indeno(1,2,3-cd)pyrene			-	-	-	-	-	-		10	330		
Dibenzo(a,h)anthracene			-	-	-	-	-	-		10	330		
Benzo(g,h,i)perylene			-	-	-	-	-	-		10	330		
SURROGATE COMPOUNDS:													
Nitrobenzene-d_5			-	-	-	-	35-114	23-120		-	-		
2-Fluorobiphenyl			-	-	-	-	43-116	30-115		-	-		
p-Terphenyl-d_{14}			-	-	-	-	33-141	18-137		-	-		
Phenol-d_5			-	-	-	-	10-94	24-113		-	-		
2-Fluorophenol			-	-	-	-	21-100	25-121		-	-		
2,4,6-Tribromophenol			-	-	-	-	10-123	19-122		-	-		
Pesticides/Polychlorinated Biphenyls:	Gas Chromatographs	Daily							90			Exhibit D	1
alpha-BHC			-	-	-	-	-	-		0.05	8.0		
beta-BHC			-	-	-	-	-	-		0.05	8.0		
delta-BHC			-	-	-	-	-	-		0.05	8.0		
gamma-BHC			15	50	56-123	46-127	-	-		0.05	8.0		
Heptachlor			20	31	40-131	35-130	-	-		0.05	8.0		
Aldrin			22	43	40-120	34-132	-	-		0.05	8.0		

See footnotes at end of table.

TABLE 4-2
(Continued)

PARAMETER	INSTRUMENT	FREQUENCY OF CALIBRATION CHECKS	MATRIX SPIKE PRECISION RPD%(a) Water	MATRIX SPIKE PRECISION RPD%(a) Solids	MATRIX SPIKE ACCURACY % OF RECOVERY Water	MATRIX SPIKE ACCURACY % OF RECOVERY Solids	SURROGATE SPIKE ACCURACY % OF RECOVERY Water	SURROGATE SPIKE ACCURACY % OF RECOVERY Solids	EXPECTED COMPLETENESS %	EXPECTED METHOD DETECTION LIMIT(b) Water ug/l(c)	EXPECTED METHOD DETECTION LIMIT(b) Solids ug/kg(d)	METHOD NO.	REFERENCE
Pesticides/Polychlorinated Biphenyls: (Continued)													
Heptachlor Epoxide			-	-	-	-	-	-		0.05	8.0		
Endosulfan I			-	-	-	-	-	-		0.05	8.0		
Dieldrin			18	38	52-126	31-134	-	-		0.10	16.0		
4,4'-DDE			-	-	-	-	-	-		0.10	16.0		
Endrin			21	45	56-121	42-139	-	-		0.10	16.0		
Endosulfan II			-	-	-	-	-	-		0.10	16.0		
4,4'-DDD			-	-	-	-	-	-		0.10	16.0		
Endosulfan Sulfate			-	-	-	-	-	-		0.10	16.0		
4,4'-DDT			27	50	38-127	23-134	-	-		0.10	16.0		
Endrin Ketone			-	-	-	-	-	-		0.10	16.0		
Methoxychlor			-	-	-	-	-	-		0.5	80.0		
Chlordane			-	-	-	-	-	-		0.5	80.0		
Toxaphene			-	-	-	-	-	-		1.0	160.0		
Aroclor 1016			-	-	-	-	-	-		0.5	80.0		
Aroclor 1221			-	-	-	-	-	-		0.5	80.0		
Aroclor 1232			-	-	-	-	-	-		0.5	80.0		
Aroclor 1242			-	-	-	-	-	-		0.5	80.0		
Aroclor 1248			-	-	-	-	-	-		0.5	80.0		
Aroclor 1254			-	-	-	-	-	-		1.0	160.0		
Aroclor 1260			-	-	-	-	-	-		1.0	160.0		
SURROGATE COMPOUND:													
Dibutylchlorendate			-	-	-	-	24-154(e)	20-150(e)		-	-		

(a) RPD equals relative percent difference.

(b) The indicated detection limits are appropriate to low level water and solids samples. Medium water Contract Detection Limits (CRDL) for Volatile, Acid/Base-Neutral Extractable, and Pesticide HSL compounds are 100 times the individual low water CRDL. Medium solids CRDLs for Volatile, Acid/Base-Neutral Extractable, and Pesticide HSL compounds are 100, 60, and 15 times individual low solid CRDL, respectively.

(c) ug/l equals micrograms per liter or parts per billion (ppb).

(d) ug/kg equals micrograms per kilogram or parts per billion (ppb).

(e) The indicated surrogate spike percent recovery limits are for advisory purposes only. These limits are not used to determine whether a sample should be reanalyzed.

REFERENCES

(1) U.S. Environmental Protection Agency (EPA), 1987, "Chemical Analytical Services for Multi-Media, Multi-Concentration Organics, GC/MS Techniques," WA-87-K236, Washington, D.C.

TABLE 4-3

LABORATORY RADIOLOGICAL TESTING
QUALITY CONTROL PERFORMANCE SUMMARY
FEED MATERIALS PRODUCTION CENTER
FERNALD, OHIO

PARAMETER	INSTRUMENT	FREQUENCY OF CALIBRATION CHECKS	TYPICAL PRECISION % RSD(a)	TYPICAL ACCURACY % Bias(b)	COMPLETENESS (%)	EXPECTED METHOD DETECTION LIMIT(e)	METHOD NO.	REFERENCE
Gross Alpha	Tennelec Low Background Alpha/Beta Counting System	Daily (c)	±20	±30	90	1.0 pCi/l(f,h) (water)	RSL-308	1
	or							
	Ludlum Scaler Scintillation Counters Models 1000, 2000, and 2200	Daily	±20	±30	90			
Gross Beta	Tennelec Low Background Alpha/Beta Counting System	Daily	±20	±30	90	4.0 pCi/l(h) (water)	RSL-308	1
Radium-226	Alpha Spectrometer	Daily	±20	±30	90	1.0 pCi/l (water) 300 pCi/kg(g) (solids)	RSL-309 RSL-201, RSL-309	2,3,4,5 2,3,4,5
	and/or							
	Nuclear Data Computer Based Gamma Spectrometer Model ND9900 (Data System Only)	NA	NA(d)	NA	NA			
Radium-228	Tennelec Low Background Alpha/Beta Counting System	Daily	±20	±30	90	3.0 pCi/l (water) 500 pCi/kg (solids)	RSL-309 RSL-201, RSL-309	2,3,4,5 2,4,3,5
Strontium-90	Tennelec Low Background Alpha/Beta Counting System	Daily	±20	±30	90	5 pCi/l (water) 500 pCi/kg (solids)	RSL-305 RSL-201, RSL-305	6,7,8 6,7,8
Technetium-99	Packard Liquid Scintillation Spectrometer	Daily	±20	±30	90	30 pCi/l (water) 900 pCi/kg (solids)	RSL-310 RSL-201, RSL-310	9 9
Thorium, Isotopic	Alpha Spectrometer	Daily	±20	±30	90	1.0 pCi/l (water) 600 pCi/kg (solids)	RSL-304 RSL-201, RSL-304	2,3,4,5 2,3,4,5
	and							
	Nuclear Data Computer Based Gamma Spectrometer Model ND9900 (Data Systems Only)	NA	NA	NA	NA			

See footnotes at end of table.

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TABLE 4-3
(Continued)

PARAMETER	INSTRUMENT	FREQUENCY OF CALIBRATION CHECKS	TYPICAL PRECISION % RSD(a)	TYPICAL ACCURACY % Bias(b)	COMPLETENESS (%)	EXPECTED METHOD DETECTION LIMIT(e)	METHOD NO.	REFERENCE
Uranium, Isotopic Uranium, Total	Alpha Spectrometer	Daily	±20	±30	90	1.0 pCi/l (water) 600 pCi/kg (solids) 1 ug/l (total)	RSL-304 RSL-201, RSL-304	2,3,4,5 2,3,4,5
	and							
	Nuclear Data Computer Based Gamma Spectrometer Model ND9900 (Data Systems Only)	NA	NA	NA	NA			
Plutonium, Isotopic	Alpha Spectrometer	Daily	±20	±30	90	1.0 pCi/l (water) 600 pCi/kg (solids)	RSL-304 RSL-201, RSL-304	2,3,4,5 2,3,4,5
	and							
	Nuclear Data Computer Based Gamma Spectrometer Model ND9900 (Data Systems Only)	NA	NA	NA	NA			
Neptunium-237	Alpha Spectrometer	Daily	±20	±30	90	1.0 pCi/l (water) 600 pCi/kg (solids)	RSL-304 RSL-201, RSL-304	2,3,4,5 2,3,4,5
	and							
	Nuclear Data Computer Based Gamma Spectrometer Model ND9900 (Data Systems Only)	NA	NA	NA	NA			
Cesium-137 Gamma Spec	Intrinsic Germanium Detectors	Daily	±20	±30	90	20 pCi/l (water) 200 pCi/kg (solids)	RSL-201, RSL-112 RSL-201, RSL-112	1 1
	and							
	Nuclear Data Computer Based Gamma Spectrometer Model ND9900 (Data System Only)	NA	NA	NA	NA			

See footnotes at end of table.

TABLE 4-3
(Continued)

PARAMETER	INSTRUMENT	FREQUENCY OF CALIBRATION CHECKS	TYPICAL PRECISION % RSD(a)	TYPICAL ACCURACY % Bias(b)	COMPLETENESS (%)	EXPECTED METHOD DETECTION LIMIT(e)	METHOD NO.	REFERENCE
Ruthenium-106	Intrinsic Germanium Detectors	Daily	±20	±30	90	150 pCi/l (water) 1 nCi/kg (solids)	RSL-201, RSL-112 RSL-201, RSL-112	1 1
	and							
	Nuclear Data Computer Based Gamma Spectrometer Model ND9900 (Data System Only)	NA	NA	NA	NA			

(a)Precision is expressed as the percent relative standard deviation of the corresponding method.

(b)Accuracy is expressed as the percent bias of the analyzed versus actual spike concentration for the indicated method.

(c)Each day samples are analyzed.

(d)The indicated instrumentation is a system used for data acquisition and reduction. Therefore, analytical definitions of method precision and accuracy are not applicable.

(e)The indicated method detection limits (MDLs) represent the 95 percent confidence limit for false detection and false rejection discrimination.

(f)pCi/l = picocuries per liter.

(g)pCi/kg = picocuries per kilogram.

(h)For sample with solids <500 ppm.

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(6)Hume, D. N., N. E. Ballou, and L. E. Gendenin, June 1945, CN-2815, pp. 23-26.

(7)Swift, E. H., 1940, A System of Chemical Analysis, Prentice-Hall, New York, p. 376.

(8)Willard, H. H. and E. W. Goodspeed, 1936, "Separation of Strontium, Barium, and Lead from Calcium and Other Metals," Ind. Eng. Chem., Anal. Ed., 8, p. 414.

(9)Anders, Edward, November 1960, The Radiochemistry of Technetium, National Academy of Sciences - National Research Council.

amount that was expected to be obtained under correct normal conditions.

- Representativeness - Expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental concern.

- Comparability - Expresses the confidence with which one data set can be compared to another.

4.4 SAMPLE MATRIX

The QA objectives for sample data will be verified using field and laboratory QC samples. The sample groups, measurement parameter, number of samples per parameter, and field and laboratory QC sample number are presented as a sample matrix in Table 4-4.

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TABLE 4-4
SAMPLE MATRIX

SAMPLE GROUP	MEASUREMENT PARAMETER[a]	NUMBER OF SAMPLES	QC SAMPLES (% OF TOTAL)	
			FIELD (15%)	LAB (10%)
Biological	Org./Inorg.	10	2	1
Biological	Radiological (Rad)	121	18	12
Ground Water	Extended HSL	37	6	4
Ground Water	Dioxins	7	1	1
Ground Water	RCRA	628	95	63
Ground Water	Rad	628	95	63
Sediment	Grain Size	15	0	2
Sediment	Extended HSL	17	3	2
Sediment	Rad Full	39	6	4
Sediment	Rad Limited	38	6	4
Subsurface Soil	Geochemical	35	6	4
Subsurface Soil	Geotechnical	20	0	2
Subsurface Soil	HSL Org./Inorg.	30	5	3
Subsurface Soil	Rad	118	18	12
Surface Soil	Extended HSL	10	2	1
Surface Soil	Rad	281	42	28
Surface Soil	Rad Limited	84	13	9
Surface Water	H_2O General	49	8	5
Surface Water	Extended HSL	7	2	1
Surface Water	Rad Full	45	7	5
Surface Water	Rad Limited	55	9	6
Urinalysis	Isotopic Uranium	90	0	9

[a]Parameters are defined in the Sampling Plan.

5.0 FIELD PROCEDURES

The IT Manual of Practice, Subsurface Investigations, associated with the IT Engineering Services QA Manual, provides information on the advancement of geotechnical borings, geotechnical sampling, piezometer/well installation and monitoring, and permeability testing. This document will be used to supplement this plan, as necessary.

An IT technical procedure entitled, "Visual Classification of Subsurface Materials, Classification Log Legend," which describes the manner in which soil and rock core logging is performed and information obtained from subsurface borings is recorded, shall also be used on this project.

Information obtained from site exploration activities shall be recorded and documented in accordance with Section 5.2.2 of the IT Engineering Services QA Manual. Required documentation of field investigations and testing includes a daily log of project activities; any photographic records taken; and the appropriate subsurface logs, test data forms, piezometer/well installation forms, and field collection forms.

Members of the Project Staff working in field operations shall keep a daily log (Figure 5-1, Field Activity Daily Log) of the project activities. Items to be included in the daily log, as appropriate, are:

- Field activity subject;
- General work activity;
- Unusual events;
- Changes to plans and specifications;
- Visitors on site;

DAILY LOG			
DATE			
NO.			
SHEET		OF	

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FIELD ACTIVITY DAILY LOG

PROJECT NAME	PROJECT NO.
FIELD ACTIVITY SUBJECT:	
DESCRIPTION ON DAILY ACTIVITIES AND EVENTS:	
VISITORS ON SITE:	CHANGES FROM PLANS AND SPECIFICATIONS, AND OTHER SPECIAL ORDERS AND IMPORTANT DECISIONS.
WEATHER CONDITIONS:	IMPORTANT TELEPHONE CALLS:
PERSONNEL ON SITE:	
SUPERVISOR:	DATE:

FIGURE 5-1

- Subcontractor progress and specifications; and
- Communication with WMCO, regulatory agencies, or others.

Copies of the daily log entries should be sent to the site manager and on-site technical manager on an approximately weekly basis. If the logs are not submitted as required, it is the responsibility of the site manager to contact the field personnel.

Any photographs obtained are to be identified with the project number, date taken, and a brief description. This may be done individually on the back of the photographs or in an album in which the photographs are mounted. Album photographs must be provided with the individual descriptions and dates taken.

Appropriate test data forms shall also be prepared. They shall include, as appropriate, the test location (e.g., boring, depth, sampling station, elevation, field coordinates) and the items listed for laboratory test data forms. All requested information shall be addressed. If not applicable, requested information should be designated as such.

Originals of field records will be maintained in the project central file at the ASI/Fernald office. During performance of the field program, a copy of the field records will be sent periodically to the site manager and the on-site technical manager. These copies can provide adequate documentation of work activities should the originals be destroyed, lost, or stolen.

Procedures are described for the following field activities in the remainder of Section 5.0:

- Radiation measurement techniques;
- Drilling procedures;
- Monitoring well/piezometer design and installation;
- Monitoring well development;

- Borehole geophysical logging; and
- Aquifer testing.

5.1 RADIATION MEASUREMENT TECHNIQUES

This section describes applicable standards and practices for the performance of field radiation measurements. The following procedures can also be found in a separate project-specific document, titled "Radiation Measurements Task Procedures."

5.1.1 FIELD RADIATION SURVEY WITH FIDLER DETECTOR

This procedure describes the methods by which a Field Instrument for the Detection of Low Energy Radiation (FIDLER) is used to monitor ground surface deposits of uranium.

Included in the procedure are instructions for:

- Initial instrument setup;
- Daily instrument standardization using a sealed "check source";
- Determination of the instrument's response to terrestrial background radiation;
- Establishment of field calibration factors;
- Radiation survey measurements of ground surface areas; and
- Use of the instrument to estimate the uranium concentration in the surface soil.

Method references for the above techniques are presented in:

- UMTRA Procedure 10.17.3, In-Situ Ra-226 Measurement Using the Shielded Delta Procedure;
- Tinney, J.F. and Koch, J.J., An X-Ray Screening Meter for Plutonium Contamination, UCRL-5000-67-3, 1967; and
- Instrument manufacturer's instructions.

The FIDLER was developed in the 1960s as a portable instrument to monitor ground surfaces contaminated with plutonium. A five-inch

diameter, thin sodium iodide (NaI) crystal serves as the detector. It is optically coupled to a quartz light pipe and installed in a standard five-inch probe housing, which has an entrance window of beryllium. The principal use of this detector is for photons with energies less than about 75 KeV. Uranium is an alpha emitter; however, the radiation from its daughter products include low-energy photons, principally L x-rays from thorium. In the case of U-238, by far the predominant uranium isotope at FMPC, there are two low-energy photons from Th-234 which can be detected by the FIDLER. These are 63 KeV (3.5 percent abundant) and 93 KeV (4 percent abundant). If significant concentrations of natural thorium are present, radiation from Ac-228 will create interference in detecting the 93 KeV photon from Th-234. For this reason, the instrument will be adjusted for maximum response from the 63 KeV photon from Th-234.

5.1.1.1 Initial Instrument Setup

Before this instrument can be used to monitor low-energy radiation in the field, it is necessary to determine the maximum response for contamination by the uranium daughter, Th-234. This is done by using a source of Am-241 (60 KeV) (whose intensity is several times greater than background radiation levels), as follows:

- Inspect the FIDLER, ratemeter/scaler (RM/S), and interconnecting cable for obvious damage. If no damage is observed, connect the FIDLER and RM/S, and adjust the High Voltage to 50 volts or less.
- Depress the THRESH button. Turn the THRESH potentiometer clockwise until 0049 displays.
- With WINDOW IN/OUT switch IN, depress the WINDOW button. Turn the WINDOW potentiometer until 0020 appears on the display.
- Switch the WINDOW IN/OUT to OUT.
- Connect the probe and expose the Am 241 source.

- Increase HV (if HV potentiometer is at a minimum, it will take approximately 3 turns before any change is indicated). While increasing the HV, observe the log scale of the ratemeter. Increase HV until ratemeter indication occurs.
- Switch the WINDOW IN/OUT to IN.
- Turn the HV control until maximun reading occurs on the log scale. Increase HV until reading starts to drop off. Then decrease the HV for maximum reading.
- Turn RANGE selector switch to the X1K position.
- Press ZERO button. If meter does not read, switch to a lower range until a reading occurs.
- Carefully adjust the HV potentiometer until maximum reading is achieved on the range scale. The instrument is now peaked for daughters of uranium-238 on both the LOG and RANGE scales.

5.1.1.2 Daily Instrument Standardization

Following completion of the instrument setup procedure, establish a fixed source-detector geometry so that the source-to-detector distance is a matter of record and reproducible from one day to the next. Standardize the FIDLER as follows:

- Make five one-minute counts with the Am-241 source in its check position, find the average of these counts. Record each count on the "Instrument Checks" form (Figure 5.2).
- Over a period of ten (10) days, determine the standard deviation from the average of the five one-minute daily counts. Record this value (σ) and three times the value (3σ) in the instrument logbook.

  standard deviation $\sigma = \left[\frac{(\Sigma x^2) - (\Sigma x)^2}{n(n-1)}\right]^{0.5}$

- A control chart will be prepared using information from the previous two steps. This chart will be prepared at the beginning of the site characterization and will be used each day to record the FIDLER's response to the

Am-241 source. On a piece of linear graph paper, place consecutive calendar dates on the x-axis. Define a range along the y-axis which includes the average FIDLER source count ± 3 times the standard deviation. Draw three horizontal lines which intersect the y-axis at these three points.

LARGE VOLUME SCINTILLATION DETECTOR

INSTRUMENT CHECK
PROCEDURE RSL-2003

Scaler Model _____ Serial No. _____

Detector Model _____ Serial No. _____

Date	Battery Response	Bkgd (cpm)	Cesium Check Source (cpm)	Americium Check Source (cpm)	Surveyor's Initals

FIGURE 5-2

- On a daily basis, repeat the five-point standardization procedure before the FIDLER is used for field measurements.

Determine the average count rate; plot this value on the control chart for that day. If the average count rate falls within the boundary as defined above, the FIDLER may be used for field measurements. However, if the average count rate is not within the boundary, corrective action must be taken according to the following procedure.

- Repeat the procedure "Initial Instrument Setup."
- Turn off ratemeter and disconnect cable to FIDLER. Clean the cable and chassis connectors with ethyl alcohol and let dry.
- Reconnect cable, turn on ratemeter; check voltage setting.
- Check the source-to-detector distance; make necessary corrections.
- Confirm proper window setting.
- Repeat the five-point standardization procedure. If FIDLER response is within the average count rate $\pm 3\sigma$, the unit may be used. If the average count rate is still outside the control boundary, turn off the ratemeter and use a substitute ratemeter. Repeat the five-point standardization procedure. If problems still exist, repeat the initial instrument setup. Repeated failures will require the attention of a manufacturer-certified service technician.

5.1.1.3 Determination of Instrument Response to Background Radiation

As part of the site characterization, several background locations will be selected in the vicinity of the FMPC. Each instrument, while having the same discriminator settings, responds differently to radiation. It is important that all instruments be checked at the same time to document the differences in responses. The FIDLER response to radioactivity in the soil at each of these background locations will be determined as follows:

- At each background station, establish an area where measurements can be made with the FIDLER probe in contact with the surface. Mark the location so that additional (or repeat) measurements can be made at later dates.

- In a field logbook or on the Field Activity Daily Log (Figure 5-1), describe the area chosen for background measurements and list specific features (soil is sandy, rocky, damp, wet, etc.). Also record climatic conditions at the time measurements are made. Make five one-minute counts and record in the field logbook or Field Activity Daily Log.

- When all background location measurements have been completed, compare the average count rate at each station with the concentration of uranium in the soil samples collected at that point.

5.1.1.4 Establishment of Field Calibration

The response of a FIDLER to radioactivity in the soil is site specific and is governed by several parameters, such as the presence of radioactivity other than uranium, a layer of clean soil between a subsurface deposit of uranium and the ground surface, nearby radioactive sources such as radon in the air or a waste storage pit, etc. Therefore, the field calibration factors determined in this instruction can only be considered estimates. Use of this calibration serves as an indicator for the need to perform soil sample analyses.

Several areas within the FMPC site where surface radioactivity is evident will be chosen as test areas. These should range from near-background radiation levels to the highest expected level on site. Readings will be taken at the designated test areas as follows:

- Perform five one-minute measurements with the FIDLER in contact with the ground surface and record the count rate in a field logbook or the Field Activity Daily Log.

- Collect a sample of surface soil (0 to 2 inches); submit it to the analytical laboratory for a gamma-ray

spectral and total uranium analysis. Soil sample collection procedure and chain-of-custody requirements are described in detail in Sections 6.0 and 7.0.

- Following completion of the analysis of soil samples, compare the data from the five one-minute measurements to the soil concentration of uranium and prepare a table of values.

Based on the information above, using a least square fit, establish a calibration factor. Record this factor in the instrument logbook.

5.1.1.5 Radiation Survey Measurements of Ground Surface Areas

Surface scanning (whether on the ground, on equipment, or on building surfaces) with portable radiation detectors is normally carried out to determine the location of anomalous areas (radiation levels in excess of guideline values) within a measurement grid block or the average radiation level within a measurement grid block. The scan of ground surfaces at FMPC will be carried out to determine the average estimated soil concentration of uranium within small areas which have been staked by a licensed surveyor. This will be accomplished by moving the FIDLER probe in a serpentine pattern over the area to be surveyed. During this scan, the detector will be held approximately two inches above the surface. Although the beryllium window is protected by Lucite, care must be taken to protect the window during this operation. Radiation survey measurements will be performed according to the following procedure:

- Determine by checking the daily instrument standardization documentation that the FIDLER system has been checked and is ready for field measurements.

- Receive instructions from the Radiation Measurements Task Leader. These instructions should be specific as to:

  - The transit survey has been completed and 100-foot grids have been established;

- The number of people required (one technical to monitor and one to record data);
- Time requirements; and
- Special instructions.

- Areas to be scanned should be subdivided into square grid blocks approximately 100 feet by 100 feet.
- Grid block squares should be further subdivided into smaller square or rectangular grids, the total area of which can be scanned in several minutes. Areas 25 feet by 25 feet can be scanned in three minutes. The grids will be measured with a tape measure.
- Drainage paths will have been staked at 100-foot intervals. Scanning these areas with a FIDLER can only be done if the area is free of standing or flowing water. If the area meets these criteria, place a grid stake at 25-foot intervals between the 100-foot stakes.
- Attach a Surface Measurements Field Logbook Form (Figure 5-3) to a clipboard or other suitable writing surface; mark the grid block coordinates in the boxes along the left and top margins of the form.
- Scan measurements will be recorded as integral counts over the area to be scanned; that is, pulses from the FIDLER will be summed for the three-minute intervals necessary to scan each 25-foot by 25-foot area.
- Scans will begin at one corner of a grid block and will progress in a serpentine pattern over the entire block, ending at the diagonally opposite corner of the block. Push the count button as the scan begins. With practice, the timed count will end upon reaching the opposite corner. During the scan, the technician must listen to the audible signal. This may be achieved by using headphones. Although scanning speed should not be slowed when increased audible signals are heard, the presence of apparent "hot spots" should be noted when the integrated count for the scan is recorded.
- Upon completion of the scan for an individual grid block, record the integrated count in its respective position on the Surface Measurements Field Logbook Form.
- Return to the locations that were identified as having a "hot spot" and conduct a one-minute count over the area indicated as having the highest concentration. Record the count on the Surface Measurements Field Logbook Form.

DATE	
TIME	
PAGE ___ OF ___	
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Surface Measurements Field Logbook Form

Site: _____ Surveyors: _____

Area: _____ Recorded By: _____

Date: _____ Count Time: _____ Background: _____ CPM

_____ CPM

(-X)W←←←←Coordinates→→→→E(+X)

(-Y)S ←←←←Coordinates→→→→N(+Y)

Comments: _____ Scaler Model _____ Serial No. _____

_____ Probe Model _____ Serial No. _____

_____ Scaler Model _____ Serial No. _____

_____ Probe Model _____ Serial No. _____

_____ PIC Serial No. _____

FIGURE 5-3

- Repeat the survey of at least two subgrids (approximately 10 percent) for each 100-foot by 100-foot grid. The subgrid yielding the highest count, as well as a subgrid selected randomly, should be recounted. A calculator generating random numbers may be used to select the specific subgrid. Record the repeated data in the appropriate box on the Surface Measurements Field Logbook Form, under the first data point.

- When all grid blocks for each assignment have been completed, return signed Surface Measurement Field Logbook Forms to the radiation measurements task leader and return the FIDLER system to its storage area.

5.1.1.6 Use of the FIDLER to Estimate Soil Uranium Concentration

The determination of uranium contamination in soil is based on the laboratory analysis of soil samples to identify isotopes of uranium. The FIDLER serves to identify areas which have "elevated" readings and provides a systematic approach to a biased soil sampling program by collecting samples in areas where the instrument response is consistently higher. It is possible to develop a correlation between the FIDLER response and degrees of soil contamination by comparing instrument response and the laboratory results of soil samples.

The correlation is determined by first collecting soil samples from grids which exhibited elevated readings. A survey with the FIDLER is repeated for the 25-foot by 25-foot subgrid as described in Section 5.1.1.5, as well as for the specific location where the sample is to be collected. The FIDLER is placed directly on the soil over the sample location and the integral count recorded. The soil sample is then collected at several depths to determine the concentration on the surface versus the subsurface. The instrument response is plotted with the concentrations of uranium, and a correlation is calculated using a linear regression method.

The soil sampling program collects samples from areas previously surveyed during the radiation measurements task, especially those

identified to have elevated readings. The FIDLER is used to survey each sample location to provide additional data concerning instrument response to soil contamination.

5.1.2 IN-SITU GAMMA RAY MEASUREMENTS USING THE DELTA-GAMMA TECHNIQUE

The purpose of this procedure is to provide guidance for the in-situ measurement of gamma rays emitted from surface soil and to provide a technique for screening potentially contaminated soils in the presence of adjacent sources of air-scattered gamma radiation.

These procedures were developed for the Uranium Mill Tailings Remedial Action (UMTRA) Program for measuring in-situ Ra-226 concentrations in soil where adjacent sources of radiation influence probe measurements. This procedure will be used to screen for anomalous gamma radiation levels in areas where adjacent sources of radiation influence the use of an unshielded probe.

This procedure is referenced to UMTRA PROCEDURE 10.17.3, IN-SITU RA-226 MEASUREMENT USING THE SHIELDED DELTA PROCEDURE.

Screening of soils in areas of elevated gamma-ray intensity will be based on the results of the delta-gamma technique described below. This survey technique will provide a basis for detecting anomalous conditions in the subsurface soil below the measurement location.

Twenty background delta count measurements will be taken to provide a statistical basis for evaluating gamma-ray measurements of potentially contaminated locations as a function of site background levels. The average background delta count and its standard deviation will be determined. Measurements subsequently taken during the field work that have a delta count greater than the average background plus three standard deviations of the

background will be marked, and the area will be further characterized by taking additional delta-gamma measurements and sampling the surface soil according to the procedures in Section 6.0.

Figure 5-4 presents a field data sheet to be used in the laboratory for documenting background delta-gamma results, and to calculate the average and standard deviation of the background data. Figure 5-5 provides a field data sheet to be used for documenting field delta-gamma measurements.

The delta-gamma calibration will be performed at RSL. The calibration factor will account for Th-232 and K-40 concentrations. It will be initially developed for estimating the in-situ Ra-226 concentration. Site-specific calibration constants can be developed by performing delta counts at various locations having a uniform increase in surface radiation levels. At each location, a surface soil sample will be obtained and analyzed for gamma-emitting radionuclides. From this data, a correlation of delta count rate to gamma-ray contributing radionuclide concentrations can be established. In-situ gamma-ray measurements using the delta-gamma technique will be performed according to the following procedure:

- Daily before each sampling exercise, check the high voltage (as stated on detector probe), threshold setting (100), window setting (gross mode), and battery conditions (BAT indicator off) on the ratemeter/scaler.

- The measurement of in-situ gamma radiation is performed by making "up" (shield out) and "down" (shield in) counts over the area to be characterized.

- With the shield out, make and record three "up" counts with the rate meter/scaler set to integrate 1,000 counts ("c" scale). When 1,000 counts have been registered, the ratemeter/scaler will calculate the count rate (CPM) and display the value. Record each value on the field data sheet. With the shield in, make and record three "down" counts in the same fashion. Record each value on the field data sheet.

Shielded Delta-Gamma In-Situ Measurement

Background Assessment

Project Name:_____ Recorded By:_____

Date:_____ Time:_____

Delta Housing No.:_____ Daily Check Results:_____

Probe No.:_____ HV_____(1000V) Threshold_____(100)

Ratemeter/Scaler No.:_____ BAT_____(OK) Window_____(OUT)

Check Source No.:_____ Check Location:_____

Unchielded C_1*_____ C_2_____ C_3_____ Avg. Unshielded_____

Shielded C_1*_____ C_2_____ C_3_____ Avg. Shielded _____

Delta 1_____ 2_____ 3_____ Avg. Delta _____

Bkg. Meas.	Location	Unshielded C_1*	Unshielded C_2	Unshielded C_3	Shielded C_1*	Shielded C_2	Shielded C_3	Delta
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								

Average _____

Std. Dev._____

Ave. ± 3 Std. Dev._____

*Each count represents the counts per minute based on integration of 1000 cts. on the "C" scale of a ratemeter/scaler.

FIGURE 5-4

DATE	
TIME	
PAGE ____ OF ____	
PAGE	

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Shielded Delta-Gamma In-Situ Measurements
Field Radiation Measurements

Project Name: _____ Recorded By: _____

Shield Id. No.: _____ Calibration Factor: _____

Probe No.: _____ Comments: _____

Ratemeter/Scaler No.: _____ _____

Surveyors: _____ _____

_____ _____

Field Measurements

Measurement N-S	Location E-W	Unshielded Counts (CPM) C_1	C_2	C_3	Shielded Counts (CPM) C_1	C_2	C_3	Average Delta	Location For Further Consideration Yes	No

FIGURE 5-5

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- Compare the sample delta to the average background delta plus three standard deviations of the background delta. If the sample delta is greater, the location will be staked and the areal extent of material further characterized by making additional delta-gamma measurements and by obtaining surface soil samples according to the sampling procedures in Section 6.0.

5.1.3 EXPOSURE RATE MEASUREMENTS USING A PRESSURIZED ION CHAMBER (PIC)

The purpose of this instruction is to provide uniform measurement techniques when using a secondary standard instrument, the pressurized ion chamber, for determining external gamma exposure rate at a height of one meter above the soil or facility surfaces.

One of the most accurate methods for measuring the gamma radiation exposure rate in the field is with a secondary standard instrument such as the Reuter-Stokes PIC. The PIC will be used to "field calibrate" or standardize other exposure rate measuring instruments such as the SPA-3 or hand-held scintillation instruments.

The procedure described below is referenced to the Reuter-Stokes PIC Operational Manual. However, other manufacturers' instructions for operation of the PIC may be used.

5.1.3.1 PIC Operational Procedure

Set up the PIC and perform the field measurement according to the following instructions:

- Check proper operation of the PIC according to the manufacturer's instructions.
- Place the PIC in the center of the survey grid block or at the survey grid point in accordance with the sampling plan.
- Attach the sensor head to the tripod and adjust the height of the chamber to one meter from the surface to be measured.

- Check that the electrometer and mode switches are both in the off position and connect the cable to the readout housing.

  - Turn the Display/Recorder switch to on.

  - Place ELECTROMETER switch to zero.

  - Place MODE switch to BATT position.

  - Depress the switch designated 300V.

  - If the digital display shows less than 85 (as a percent of charge), replace the 300 V battery.

- Check the charge on the -14V, +14V, and 12V lead-acid batteries by depressing the switch immediately below the voltmeters for each battery. If the needle is on or near the shaded area of the meter, recharge these batteries before proceeding.

- Place the ELECTROMETER switch in the ZERO position and wait 60 seconds.

- Place the ELECTROMETER switch in the READ position.

- Reset the mechanical counter and start the stopwatch simultaneously.

- After at least 60 minutes and when the counter integrates the next microR (μR), record elapsed time and exposure.

- Determine exposure rate in microR/hr (μR/hr) using the following formula:

  $$\frac{60 \text{ minutes/hr}}{\text{count time minutes}} \times \text{exposure recorded by PIC}$$

- Record the exposure rate on Figure 5-6.

5.1.3.2 Calibration of the Sodium Iodide Detector Using the PIC

Correlation of scintillation detector measurements with PIC measurements on a site-specific basis permits the determination of correction factors which can be applied to the scintillation detector measurement to evaluate gamma exposure rates. This technique permits the use of less expensive portable instruments to obtain exposure rate survey data. This procedure describes the methodology for correlating counts obtained using a gamma ray

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PAGE

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Gamma-Ray Exposure Rate Survey

Site:_____ Location:_____

Date:_____ Survey No.:_____ Surveyor:_____

Scaler Model:_____ Serial No.:_____ Count Time:_____

Probe Model:_____ Serial No.:_____ Background:_____CPM

Comments:_____ Calibration Factor:_____ uR/hr/cpm

Location	uR/hr at 1 meter	Location	uR/hr at 1 meter	Location	uR/hr at 1 meter	Location	uR/hr at 1 meter

FIGURE 5-6

scintillation probe coupled to a ratemeter/scaler, to the exposure rate measurements taken with the PIC.

The correlation is performed using 5 to 50 locations ranging in activity from normal background to approximately 500 uR/hr. The FMPC site will be subdivided into areas where, within the area, the variation in the photon spectrum will be relatively consistent. It is anticipated that several areas will be defined by the photon spectrum. Accordingly, each area will be correlated separately to develop area-specific correction factors.

Measurement locations will be chosen so that the exposure rate is relatively constant with slight differences in the positioning of the two detectors. Measurements will be taken alternately at each location with both instruments one meter from the ground or measurement surface. Results will be recorded on the Site-Specific Exposure Rate Correlation Form (Figure 5-7).

After performing measurements at a sufficient number of locations, the readings from both instruments will be correlated by determining the mathematical relationship between the responses of the two instruments, i.e., with a linear, least-squares regression analysis.

5.1.4 WALK-OVER SURVEYS USING THE LARGE-VOLUME SCINTILLATION DETECTOR

The purpose of this instruction is to provide uniform measurement techniques when using a large-volume scintillation detector and to provide uniform interpretation of the results from such measurements.

The most sensitive gamma-ray survey instrument is the large-volume, sodium iodide scintillation detector (e.g., the Eberline Model SPA-3 or equivalent). It is commonly used for locating gamma-ray sources in low background areas and for measurements of low-level exposure rates.

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Exposure Rate Correlation Data

Entry Form - Site-Specific

Site:_____ Location:_____

Date:_____ Survey No.:_____ Surveyor:_____

Location N-S	Location E-W	Section	PIC #_____ µR/hr at 1 meter	Inst #_____ CPM at 1 meter	Inst #_____ CPM at 1 meter

FIGURE 5-7

This procedure is referenced in the manufacturer's operation manual. Various other manufacturers' instructions for operation of the large-volume scintillation detector may be used.

Large-Volume Scintillation Detector Operational and Field Measurement Procedure

Set up the detector and perform the field measurement according to the following instructions:

- Check proper operation of the detector and its associated ratemeter/scaler according to the manufacturer's instructions.
- Perform correlation of the detector according to Section 5.1.3.2 each time a measurement is taken and record the results on the Gamma-Ray Exposure Rate Survey form (Figure 5-2).
- Perform daily source checks using a 0.1 μCi Cesium-137 check source and record the response on the Instrument Checks from (Figure 5-2).
- Perform daily background checks at the field office and record the response on the Instrument Checks form (Figure 5-2).
- For walkover surveys, proceed to the assigned grid area and reset the scaler on the ratemeter/scaler.
- Subdivide the 100-foot by 100-foot grid into 16 squares, each 25 feet by 25 feet.
- At one corner of the grid, start the scaler and timer and walk at a steady rate (approximately two feet per second) over the subgrid, beginning along one side and covering the entire grid in a rectilinear fashion until all areas have been surveyed.
- The scintillation detector should be suspended by a rope or strap and swung from side-to-side in a serpentine fashion. Each 25-foot by 25-foot subgrid should be surveyed in approximately two minutes.
- During the walkover, survey meter count rate may be monitored with earphones, if necessary. Any location with an elevated count rate (indicated by a higher

pitch in the earphones) in localized areas will be marked by dropping a weighted flag, and the survey will continue at the same pace.

- After the walkover of the grid is completed, the scaler and timer are stopped and the following items are recorded on the Surface Measurements Field Logbook Form (Figure 5-3): (1) grid ID number, (2) the total count, and (3) the elapsed time.

- Return to each location which has been "flagged" within the grid and perform a systematic survey beginning at the flag and working outward to determine the areal extent of the elevated reading.

- Record the highest count rate and the approximate location and areal extent; insert a marker at the location of highest reading.

5.2 Drilling Procedures

Soil borings are made to determine the nature, arrangement, thickness and extent of the soil strata. The depth of borings, frequency, and the type of testing and sampling required are dependent upon the purpose of the subsurface investigation.

Borehole advancement for monitoring wells will be performed using cable tool drilling techniques. The use of mud rotary or continuous flight hollow stem augers is prohibited for monitoring wells. A soil auger drilling rig will be used to collect undisturbed soil samples in clay layers that may be found in the till. With the cable tool techniques, the hammer is used to dislodge the soils and mix them with potable water for recovery and removal from the hole. Cable tool borehole drilling will be performed in accordance with Section 2.2.5, Percussion Drilling Borehole Advancement, IT Manual of Practice, Subsurface Investigations.

Comments from the IT Manual of Practice, Subsurface Investigations pertinent to the cable tool drilling technique follow:

- When advancing the boring using cable tool drilling techniques, a temporary steel casing will be

drilled, driven, or pushed as the borehole is advanced. **The temporary casing will be threaded, nominal ten-inch diameter for constructing 1000 series monitor wells. The temporary casing will be threaded, nominal eight-inch or nominal ten-inch diameter to allow for the construction of the monitoring well in the 2000, 3000, and 4000 series borings.** Additionally, the temporary casing in the 4000 series boring may be nominal ten-inch diameter to approximately 150 foot depth, then telescope to nominal eight-inch diameter to allow for the construction of the monitoring well. Cuttings will be removed from the borehole using a sand pump or dart valve bailer.

THE CONTENT OF CHANGE IS UNDERLINED ABOVE

- During the process of drilling monitoring wells, disturbed soil samples will be collected. Disturbed samples will be collected with a split-barrel or split-spoon sampler in accordance with ASTM D1586.

- Relatively undisturbed soil samples will be collected with thin-walled tubes or Shelby tubes in accordance with ASTM D1587. These samples will be collected with a soil auger drill rig. The soil auger drill rig will only be used for borings to collect samples for geochemical and geotechnical purposes. Undisturbed samples are not being collected from borings for monitoring wells because the heavy blows from cable tool (percussion) drilling will result in significant densification of cohesionless soils and remolding of cohesive soils, which will adversely affect sampling and testing operations.

- Drilling operations shall be performed in such a manner to minimize the introduction of contaminants into the subsurface soil and ground water. Accordingly, only clear, potable water will be used as a drilling fluid and only as a last resort. Water samples will be collected from the on-site source and each water tank hose and analyzed for the full list of organic, inorganic, and radiological parameters. If possible, all drilling operations will be performed dry without the use of water.

- The source(s) of any water to be used in drilling, grouting, and well and/or piezometer installation must be approved prior to field operations. Water used in aid of drilling will be analyzed and results reviewed prior to introduction into the borehole.

- Field Activity Daily Logs (Figure 5-1) will be completed daily to indicate drilling and sampling activities such as footage drilled, materials used, etc. Boring logs (Figure 5-8) will be generated to document subsurface conditions.

- The abandonment of borings and wells will be approved by the project director prior to any casing removal or sealing/backfilling. Once approved, these borings and/or wells shall be sealed by grouting from the bottom of the boring or well to ground surface. This shall be done by placing a grout pipe to the bottom of the boring or well and pumping grout through this until undiluted grout flows from the boring or well at ground surface. The grout shall consist of Volclay grout mixed properly with the recommended activator. After the grout has set (about 72 hours), the contractor shall check the abandoned site for grout settlement. 84

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VISUAL CLASSIFICATION OF SOILS

PROJECT NUMBER:	PROJECT NAME:	
BORING NUMBER:	COORDINATES:	DATE:
ELEVATION:	GWL: Depth Date/Time	DATE STARTED:
ENGINEER/GEOLOGIST:	Depth Date/Time	DATE COMPLETED:
DRILLING METHODS:		PAGE OF

DEPTH ( )	SAMPLE TYPE & NO.	BLOWS ON SAMPLER PER ( )	RECOVERY ( )	DESCRIPTION	USCS SYMBOL	MEASURED CONSISTENCY (TSF)	REMARKS

NOTES:

FIGURE 5-8

Any depression in the grout shall then be filled with additional grout. Methods other than those set forth herein may be implemented as dictated by appropriate state and/or local agencies.

- The use of dug sumps is not allowed. The use of drums or a plastic-lined, above-ground holding structure is required for cuttings and water removed from the borehole.

- No dyes, tracers, or other substances shall be used or otherwise introduced into borings, wells, piezometers, grout, backfill, ground water, or surface water unless specifically required by the contract.

- Drilling equipment that enters a boring will be cleaned using a high-pressure, hot-water sprayer between borings to prevent cross-contamination.

- Each operating drill rig shall have a geologist assigned to it who will be responsible for logging the samples, preparing the boring logs (Figure 5-8) and well sketches, and installing the well. No geologist will be responsible for more than one operating rig per project site or installation involving soil sampling, rock coring, or monitoring well installation functions.

- Soils will be sampled continuously to the base of the till, then at five-foot intervals or at every lithologic change thereafter. These samples are to be obtained with a driven split-spoon or pushed thin-wall Shelby tube sampler or both in each boring. A sufficient number of continuous cores will be completed to adequately characterize the geology of the site.

The contractor shall be responsible for securing any and all boring or well drilling permits required by state or local authorities and for complying with any and all state or local regulations with regard to the submission of well logs, samples, etc.

The contractor shall also be responsible for complying with any and all regulations (to include placement) for drilling safety and underground utility detection.

5.3 MONITORING WELL/PIEZOMETER DESIGN AND INSTALLATION

To provide proper quality control of monitoring well design and installation, the following standard operating procedures have been set forth, along with the applicable forms, to successfully complete a field drilling investigation that obtains hydrogeological and future water quality information.

All field measurements and comments will be recorded on the appropriate forms (Figures 5-9 through 5-13). Per data reporting format and protocols, all lines on the forms will be completed. The letter designation "NA" for not applicable or "NK" for not known will be used in all blank spaces. Also acceptable is a neat and precisely drawn arrow through applicable spaces from an "NA" or "NK." If some steps or procedures were not performed as described, the reason must be stated as completely as possible on the appropriate form or submitted as an attachment thereto.

All monitoring wells/piezometers will use the following design and materials:

- Well casing will be four-inch inside diameter, 316 stainless steel with flush-thread joints for monitoring wells.
- Five, ten, and fifteen foot sections of commercial 0.01-inch-slot stainless steel screens will be used (minimum three square inches open area per foot of screen).
- Screen gravel pack material will be a well-sorted medium or coarse quartz sand. A representative sample will be retained for documentation.
- Sodium bentonite pellets and Volclay grout will be used in the backfill for 100- and 200-series wells. Only Volclay grout will be used in 300- and 400-series wells. Samples of all grouts will be retained for documentation.
- Wells (100 and 200 series) will be backfilled with a sand pack to a height of two to four feet above the screen, then a five foot bentonite pellet plug will be placed on top of the sand pack and, finally, grout to the surface. The 300- and 400-series wells will be

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PIEZOMETER INSTALLATION SHEET

PROJECT NAME _____ FIELD ENG./GEO. _____ DATE _____
PROJECT NO. _____ CHECKED BY _____ DATE _____
BORING NO. _____
PIEZOMETER NO. _____ DATE OF INSTALLATION _____

BOREHOLE DRILLING

DRILLING METHOD _____	TYPE OF BIT _____
DRILLING FLUID (S) USED:	CASING SIZE (S) USED:
FLUID _____ FROM _____ TO _____	SIZE _____ FROM _____ TO _____
FLUID _____ FROM _____ TO _____	SIZE _____ FROM _____ TO _____

PIEZOMETER DESCRIPTION

TYPE _____	RISER PIPE MATERIAL _____
DIAMETER OF PERFORATED SECTION _____	RISER PIPE DIAMETERS:
PERFORATION TYPE:	O.D. _____ I.D. _____
SLOTS ☐ HOLES ☐ SCREEN ☐	LENGTH OF PIPE SECTIONS _____
AVERAGE SIZE OF PERFORATIONS _____	JOINING METHOD _____
TOTAL PERFORATED AREA _____	

PROTECTION SYSTEM

RISER PROTECTIVE PIPE LENGTH _____	OTHER PROTECTION _____
PROTECTIVE PIPE O.D. _____	

ITEM	DISTANCE ABOVE/BELOW GROUND SURFACE ( )		ELEVATION ( )	
TOP OF RISER PIPE				
GROUND SURFACE	0.0			
BOTTOM OF PROTECTIVE PIPE				
BOREHOLE FILL MATERIALS:				
GROUT/SLURRY	TOP	BOTTOM	TOP	BOTTOM
BENTONITE	TOP	BOTTOM	TOP	BOTTOM
SAND	TOP	BOTTOM	TOP	BOTTOM
GRAVEL	TOP	BOTTOM	TOP	BOTTOM
PERFORATED SECTION	TOP	BOTTOM	TOP	BOTTOM
PIEZOMETER TIP				
BOTTOM OF BOREHOLE				
GWL AFTER INSTALLATION				

WAS THE PIEZOMETER FLUSHED AFTER INSTALLATION? YES ☐ NO ☐
WAS A SENSITIVITY TEST PERFORMED ON THE PIEZOMETER? YES ☐ NO ☐
REMARKS _____

FIGURE 5-9

DATE						
TIME						
PAGE ____ OF ____						
PAGE						

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PIEZOMETER SENSITIVITY TEST

PROJET NAME_____TESTED BY_____DATE_____
PROJECT NO._____

BORING NO. _____
PIEZOMETER NO. _____
TEST TRIAL NO. _____
GWL BELOW TOP OF PIPE PRIOR TO TEST _____(M-scope, 0.01 foot accuracy)
TIME READING TAKEN _____

THE PIEZOMETER WAS FILLED WITH WATER. THE FOLLOWING DATA SHOW THE DROP OF WATER FROM THE TOP OF THE PIPE AS TIME ELAPSED.

ELAPSED TIME (MINUTES)	DROP OF WATER (FEET)

BASED ON THE ABOVE DATA, IT IS CONCLUDED THAT THE PIEZOMETER IS:

_____FUNCTIONING PROPERLY

_____NOT FUNCTIONING PROPERLY

FIGURE 5-10

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PIEZOMETER DATA SHEET

PAGE ____ OF ____

PAGE

PROJECT NAME _____ PROJECT NO. _____

(1) DEPTH TO THE WATER LEVEL FROM THE TOP OF PIPE
(2) ELEVATIONS SHOWN ARE TOP OF PIEZOMETER PIPE

DATE & RECORDER	PIEZOMETER READINGS - FEET (1)							ELEVATION OF WATER LEVEL - FEET			
	# EL (2)	# EL.	# EL.	# EL.	# EL.	# EL.	# EL.	# EL.	# EL.	# EL.	# EL.

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FIGURE 5-11

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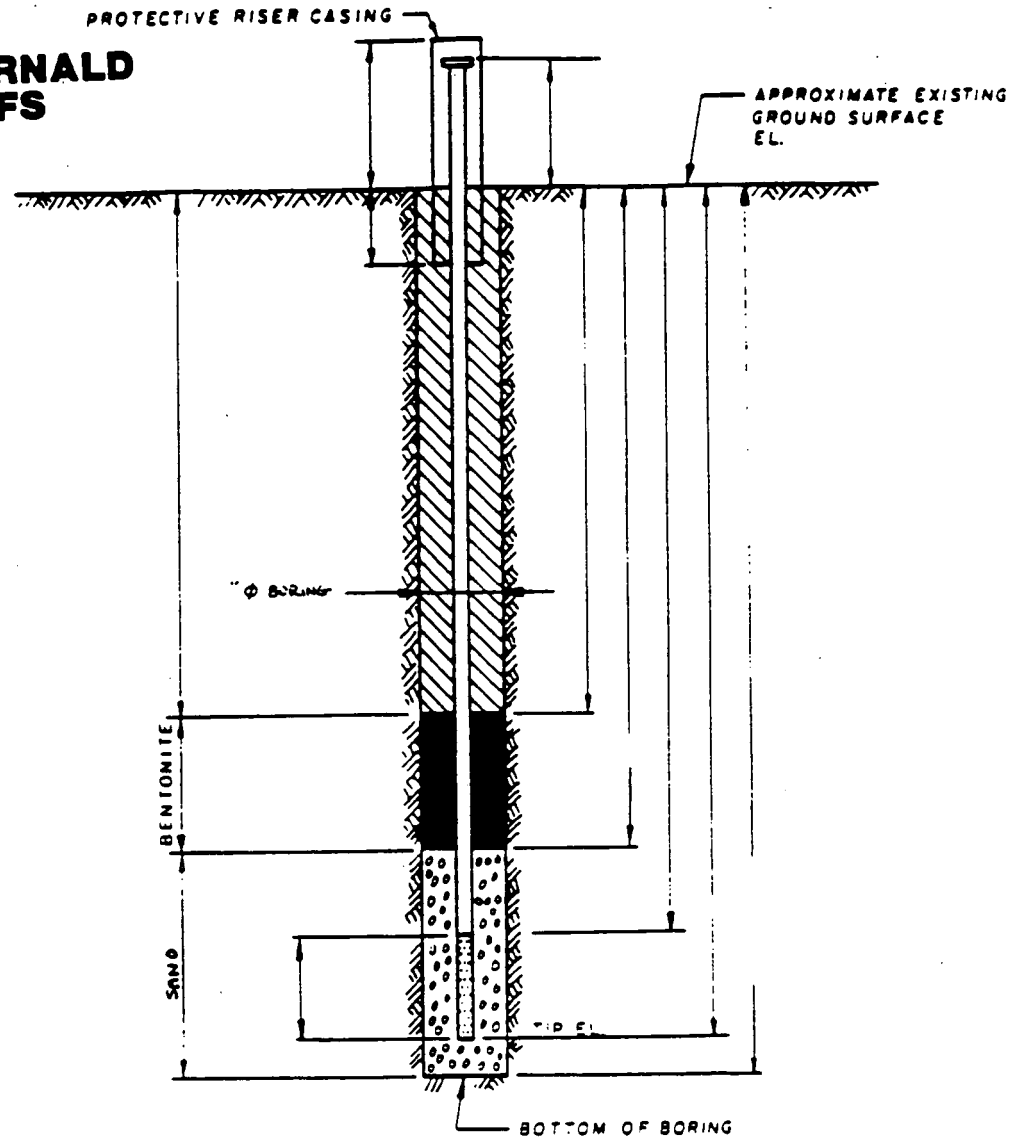

DRAWING NUMBER
CHECKED BY
APPROVED BY
DRAWN BY

NOTES:

1. RISER PIPE IS IN I.D SCHEDULE PIPE, THREADED FLUSH-JOINTED
2. SCREEN IS IN I.D PIPE CONTINUOUS SLOT SCREEN (0.0 0 IN SLOT SIZE)
3. LOWER END OF SCREEN IS CAPPED.
4. ELEVATION OF WATER LEVEL
5. WATER LEVEL READING ON

FIGURE 5-12
MONITORING WELL INSTALLATION DETAILS

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PAGE ___ OF ___					
PAGE					

FMPC RI/FS
MONITORING WELL COMPLETION CHECKLIST
PROJECT 682

WELL NUMBER

ITEM											COMMENTS.
1. A steel protective casing with steel lid and hasp welded to casing has been installed with a lock.											
2. The protective casing has been painted satisfactorily.											
3. The well identification no. has been welded into the top of the protective casing.											
4. The well I.D. number has been painted with enamel type paint on the inside of the lid of the protective casing.											
5. The grout seal has been installed in the annulus between the well riser and the outer protective casing to a height of about 6" above ground surface.											
6. A weep hole has been drilled through the protective casing at the proper location.											
7. A 3 foot by 3 foot by 4 inch concrete pad has been installed around the protective casing.											
8. The disturbed area around the well has been regraded as per project specifications.											
9. The well riser is finished with a vented stainless steel cap.											
10. A measuring point notch is filed on the top of the well riser for vertical control.											
11. 3" diameter steel posts, located 4' radially around the well, placed 2' below the ground and having 4' minimally above the ground are concreted in place.											

Y = YES
N = NO
NA = NOT APPLICABLE

DATE: _____

SIGNED: _____

FIGURE 5-13

backfilled with a sandpack to a height of five feet above the screen and then grouted to the surface. The grout will consist of a slurry of American Colloid Company Volclay grout. A sample of the backfill material will be collected for analysis to identify the presence of contaminants of interest.

- Annular space grout seals will be composed of a Volclay grout mixed with potable water and activator as per manufacturer's instructions.

- All grout material will be placed using pumps and tremie line methods.

- A five-foot length (minimum thickness 1/4 inch) of ten-inch inside diameter black iron pipe will be used as a protective casing. Each protective casing will be fitted with a hinged cap, hasp, and lock. A drain hole will be drilled in the oversleeve one foot above the land surface.

- A mixture of approximately 1:2 cement:sand and approved water will be placed between the well riser and the outer protective casing to a height just below the drain hole mentioned above. This design will allow water which enters the annulus to drain.

- The top of the inside casing will be finished with a vented stainless steel cap or an airtight cap and a vent hole not more than six inches from the top of the casing.

- All wells and piezometers will be identified with a number welded into the lid of the protective casing. All protective casings will be painted high-visibility orange.

- A measuring point notch will be filed on the inner casing of each well and will be identified on the notes and well sketches.

- All disturbed drilling areas will be regraded and restored to near original conditions upon completion.

A one-quart or one-liter representative sample of each type of proposed granular backfill shall be submitted by the contractor for approval prior to use. Each sample shall be described in terms of lithology, grain size distribution, and source (both company from whom purchased and pit or quarry of origin). This

material shall be clean, inert, and siliceous. Typically, graded sand meeting the requirements of ASTM C-33 for fine aggregate (concrete sand) is sufficient.

A sketch of any well installation (see Figures 5-14 and 5-15) including piezometers, shall be recorded and shall show (by depth from surface grade) the bottom of the boring, screen location, granular backfill, seals, grout, cave-in, centralizers, if used, and height of riser above ground surface. The actual composition of the grout, seals, and granular backfill shall also be recorded. Also indicated shall be the screen, slot size (in inches), and slot configuration. Any use of polyvinylchloride (PVC) solvents, glues, or cleaners is prohibited.

Well sketches shall also include details of the protective casing.

Protective casing shall be installed around all monitoring wells. Exceptions will be on a case-by-case basis, as determined. Minimum elements in the protection design include:

- A five-foot minimum length of black 10-inch-diameter iron pipe or galvanized pipe extending approximately 2.5 feet above the ground surface and set in Volclay grout and concrete apron.
- A locking cover and lock shall be provided and secured to the top of each protective casing.
- The location identification shall be labeled on the well protector in two places using two methods: (1) painted on the inside of the cover with enamel type paint; and (2) welded into the top of the locking cover. Location identification shall conform with format and shall be designated by the site manager.
- The cover must keep precipitation out of the protective casing and shall be secured to that casing with a padlock.
- All padlocks at a given site shall be able to be opened by the same key.
- Protective casings are to be painted high-visibility orange.

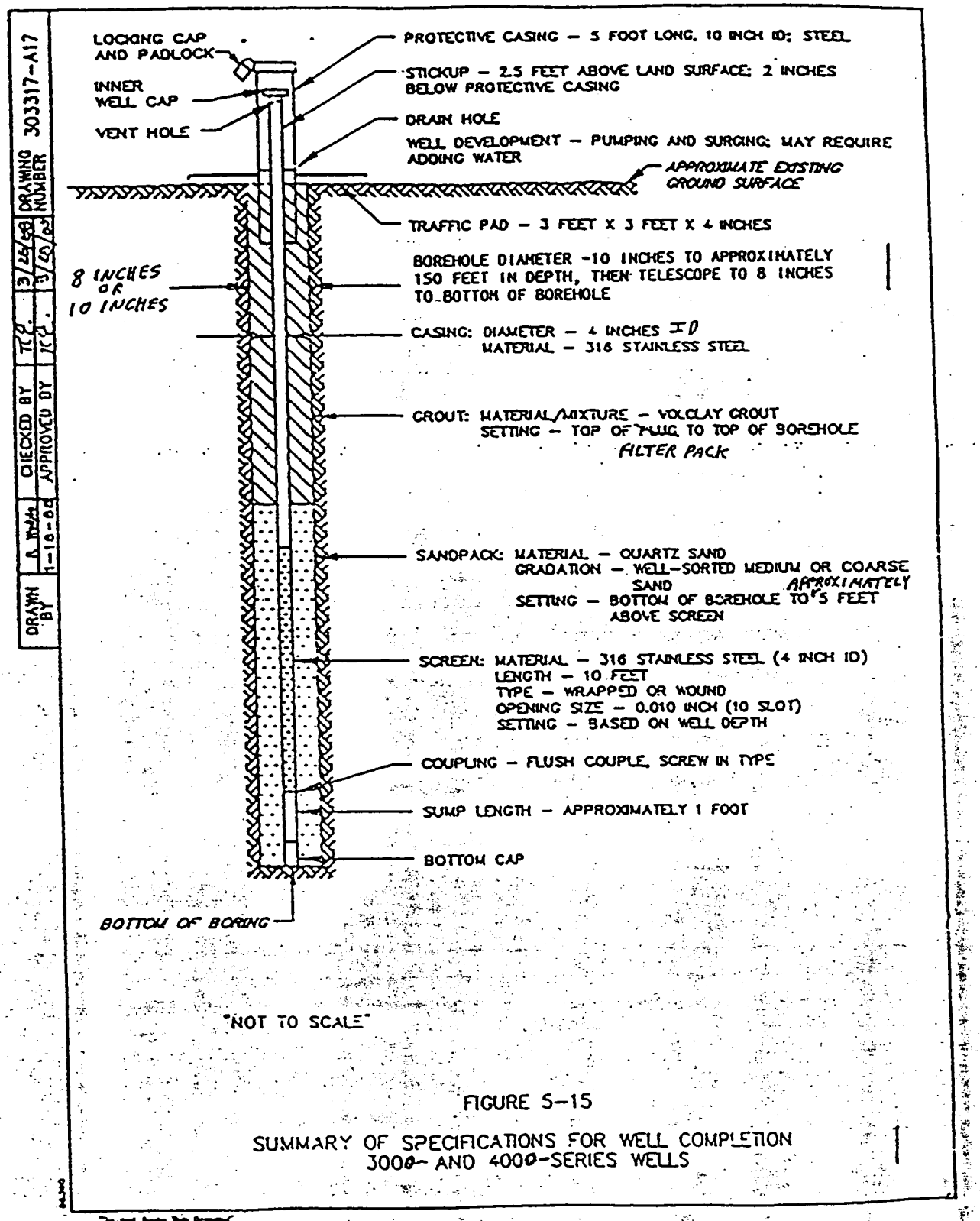

FIGURE 5-15

SUMMARY OF SPECIFICATIONS FOR WELL COMPLETION 3000- AND 4000-SERIES WELLS

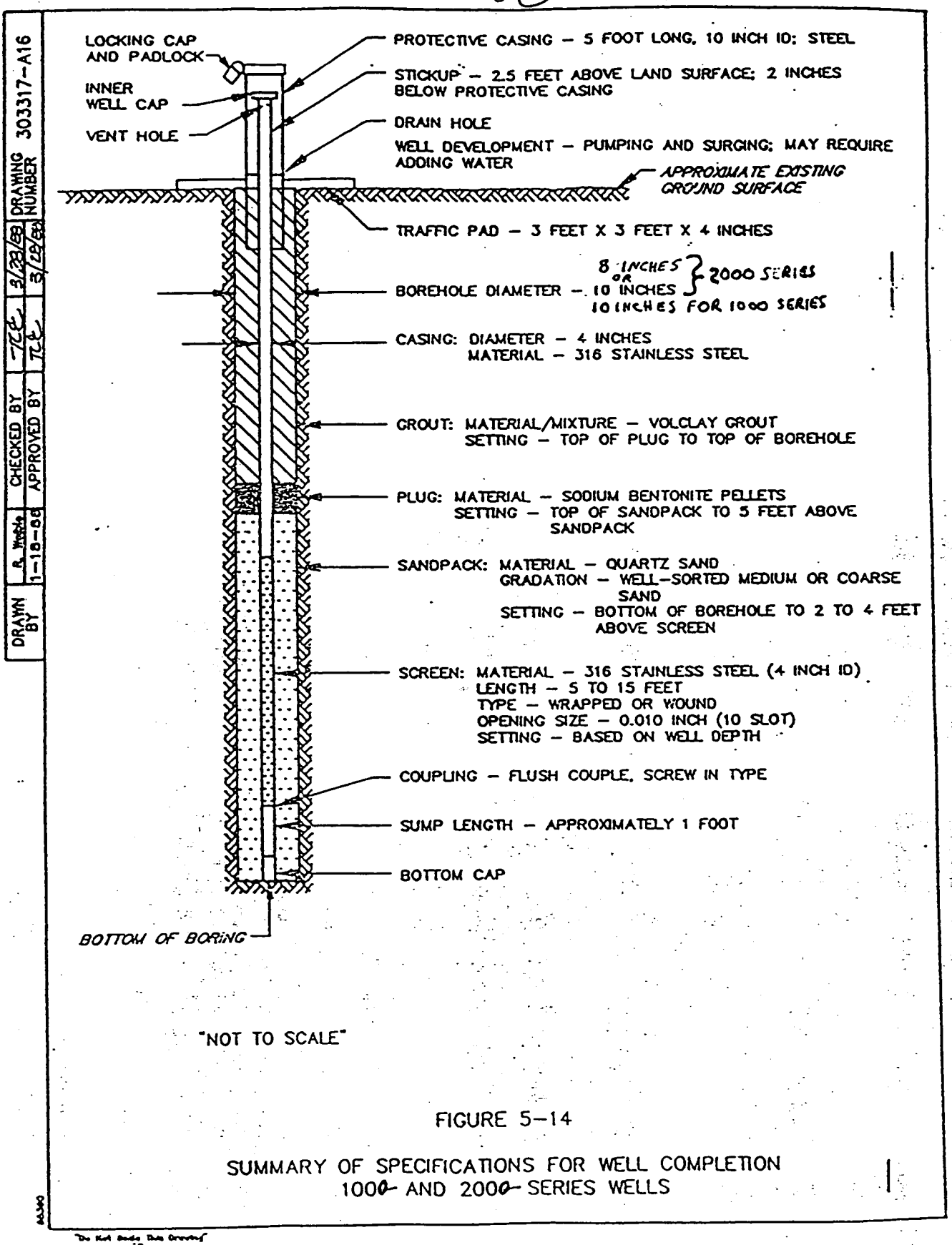

FIGURE 5-14

SUMMARY OF SPECIFICATIONS FOR WELL COMPLETION
1000- AND 2000- SERIES WELLS

- Three-inch diameter steel posts, each radially located four feet around each well, placed two feet below ground surface and having four feet minimally above ground surface will be concreted in place (optional).

Once the boring has been advanced to the desired depth, the well/piezometer will be constructed by the following procedure:

- Place the desired length of screen and casing inside the temporary casing.
- Place the sand pack in the annular space between the screen/casing and temporary casing.
- Withdraw the temporary casing slowly while placing sand pack material.
- After placing the sand pack to a level of at least two feet but not more than four feet above the screen, a five-foot-thick bentonite pellet layer (only for 100- and 200-series wells) is placed on top of the sand pack as the temporary casing continues to be withdrawn slowly. Periodic measurements will be made to check the uniform placement of the sand pack.
- The remaining annular space will be filled to the surface using Volclay grout.
- The temporary casing will be removed gradually as the backfill materials are placed in such a manner that the bottom of the temporary casing is kept below the top of the backfill material.
- The protective casing will be installed and concreted in place.
- A three-foot by three-foot by four-inch concrete apron will be installed around the protective casing.

5.4 MONITORING WELL DEVELOPMENT

The following procedure is presented for the proper development of monitoring wells and piezometers for ground water sampling purposes.

All field measurements and comments will be recorded on the appropriate data reporting form (Figure 5-16). All lines on the form will be completed. The letter designation "NA" for not

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TASK 3.2

MONITORING WELL DEVELOPMENT

PAGE ___ OF ___

WELL NUMBER _____

DATE(S) OF WELL INSTALLATION _____

WELL DEVELOPMENT START DATE/TIME _____ COMPLETION DATE/TIME _____

		BEFORE DEVELOPMENT	AFTER DEVELOPMENT
STATIC WATER LEVEL			
DEPTH OF OPEN HOLE			
CHANGES IN PHYSICAL CHARACTER OF WATER REMOVED	TURBIDITY		
	COLOR		
	PARTICULATES		
	ODOR		

TOTAL CALCULATED VOLUME OF WATER TO BE REMOVED (IN GALLONS) _____

TOTAL VOLUME OF WATER ACTUALLY REMOVED DURING DEVELOPMENT (GALLONS) _____

TYPE AND SIZE OF PUMP _____ TYPE AND SIZE OF BAILER _____

PHYSICAL CHARACTER OF REMOVED SEDIMENTS (LITHOLOGY AND GRAIN SIZE)

DESCRIPTION OF SURGE TECHNIQUES (IF USED)

METER CALIBRATION

pH TEMP	pH STD.	pH STD.	SPECIFIC CONDUCT.	uMHOS/CM	uMHOS/CM	TURBIDITY	
						NTU	NTU

METER MODEL/ SERIAL NO.	pH METER	SPECIFIC CONDUCTANCE METER	TURBIDITY METER	DISSOLVED OXYGEN METER

FIELD ENGINEER/GEOLOGIST _____ DATE _____

DRILLER/HELPER _____

CHECKED/VERIFIED BY _____ DATE _____

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FIGURE 5-16

DATE AND TIME SAMPLE COLLECTED	GALLONS OF WATER PURGED FROM WELL	TEMP.	SPECIFIC CONDUCTANCE	pH	DO	TURBIDITY	REMARKS

REMARKS/OBSERVATIONS

FIGURE 5-16
(CONTINUED)

MWDEV2 (2/88)

applicable or "NK" for not known will be used in all blank spaces. Also acceptable is a neat and precisely drawn arrow through applicable spaces from an "NA" or "NK." If some steps or procedures are not performed as described, the reason must be stated as completely as possible on the appropriate form or submitted as an attachment.

Monitoring well development will be performed as soon as possible after well installation, but no sooner than 48 hours after grouting is completed.

Well development equipment may include, but is not limited to, bailers, surge blocks, pumps, and hoses. Equipment and materials used for well development will be properly decontaminated the same way as drilling equipment, before and between each use. Decontamination will normally be high-pressure hot-water washing (steam cleaning) of equipment used to develop the well. However, if, during well development, the equipment becomes visibly contaminated, decontamination will be a high-pressure/hot-water wash, methanol wash followed by two separate deionized water rinses. In the case of visible inorganic contaminants (visibly contaminated with soil/sediment) decontamination will be high-pressure/ hot-water wash, dilute-hydrochloric-acid wash followed by two separate deionized water rinses. The internal mechanisms of pumps will be flushed using either dilute solutions of methanol and deionized water or dilute hydrochloric acid and deionized water when contaminants are visible. Development shall be continued with a sump and/or bottom discharge bailer, possibly supplemented with a surge block, until the well water is clear and sediment within the well is removed to the fullest extent practical. The natural hydraulic conductivity of the formation should have been restored and foreign sediment removed to provide turbid-free ground water samples. If the well continues to yield turbid ground water samples, the "Decision Chart for Turbid Ground-Water Samples" procedure described in the TEGD will be followed in an attempt to

achieve turbidity-free water. The recommended acceptance/rejection value of five nephelometric turbidity units (NTU) is based on the need to minimize biochemical activity and possible interferences with ground water sample quality. As a minimum:

- For those wells where the boring was made without the use of drilling fluid water, remove five times the standing water volume in the well (well screen and casing plus saturated annulus). Should recharge be so slow that five volumes could not be removed in one day or the water remains discolored or contains greater than one percent particulates after this five volume removal, the hydrogeology task leader will direct an alternate procedure based on the judgment of the field geologist.

- For those wells where the boring was made or enlarged with the use of drilling fluid (water), remove five times the measured amount of total fluids lost while drilling plus five times the standing water volume as above. The same procedure also applies here as above for cases of slow recharge, discolored, or particulate-laden water.

- No water will be added to the well to assist development without prior approval. No dispersing agents, acids, or disinfectants will be used.

- During development, an effort will be made to remove the standing water from points near the bottom of the well screen as well as from the top of the water column.

- If problems are encountered during development, the task leader will be promptly notified by telephone.

- The volume of water collected for analysis will depend on the analytical needs of the project.

The following data will be recorded on the Monitoring Well Development form (Figure 5-16) as part of development:

- Well designation (location ID);

- Date(s) of well installation;

- Date(s) and time of well development;

- Static water level before and after development;

- Quantity of water removed and time of removal;
- Depth of open hole inside the well before and after development;
- Physical character of the removed water, to include changes during development of turbidity, color, and odor;
- Physical character of removed sediments, to include lithology and grain size;
- Type and size/capacity of pump and/or bailer used;
- Description of surge techniques, if used; and
- Field characteristics of water removed including pH, specific conductance, temperature, and dissolved oxygen.

5.5 BOREHOLE GEOPHYSICAL LOGGING

This section provides a procedure for consistently performing and documenting geophysical logging activities, if required. The following procedure shall be followed by the hydrogeologist or designated field team leader (FTL).

Prior to any geophysical logging operation, the hydrogeologist or designated FTL will review the logging program and supply the information necessary to: (1) transport all applicable equipment to the drill site when required and (2) provide an acceptable logging operation. All electrodes and cables will be properly cleaned and decontaminated prior to the downhole logging operation and between each well.

The geophysical team will calibrate all logging equipment and provide documentation of all before and after survey calibrations. The FTL will forward copies of all calibrations and log runs to the hydrogeologist. The hydrogeologist will be responsible to see that all logs are appropriately filed at the field office.

When the geophysical team arrives at the drill site, the FTL will review the logging program and provide information necessary to complete the log heading of the Field Log Quality Control Check List (FLQCCL) (Figure 5-17). Field prints of all logging activities will be provided to the FTL, including calibration steps. The FTL will monitor and record the progress of logging activities.

Prior to completing the geophysical logging, the FTL will check the electrodes and cables to see that they have been properly cleaned and decontaminated and will check the FLQCCL for completeness. Unchecked items will be discussed and recorded in the remarks section. When the FTL is satisfied with the performance of the logging operation, he/she and a member of the geophysical team will sign and date the FLQCCL.

At the conclusion of the logging operations, copies of data generated will be transferred to the hydrogeologist for distribution, analysis, and archiving. The geophysical team will secure original log data. Magnetic recordings, if generated during the course of downhole logging, will be transmitted to the field office. This will be done at the convenience of and in a recording format specified by the technical manager.

5.6 AQUIFER/PERMEABILITY TESTING

The purpose of this section is to define the procedures to be followed by hydrologists when conducting hydraulic tests to characterize hydrogeologic units in the project area. The testing will be performed to estimate the hydrologic characteristics of a given hydrogeologic interval. The equipment used in the test will depend on the rate of flow of fluid encountered during drilling and testing. Several different test tool arrangements are planned to be able to perform various tests.

FERNALD RI/FS

FIELD BOREHOLE GEOPHYSICAL LOG QUALITY CONTROL CHECKLIST

SITE I.D. _____ LOGGING COMPANY _____

LOCATION I.D. _____ LOG DATE _____

BOREHOLE DEPTH, AS MEASURED FROM LAND SURFACE — DRILLER _____ FEET; LOGGER _____ FEET

LOG RUNS

__	NATURAL GAMMA	__	GAMMA-GAMMA DENSITY
__	NEUTRON POROSITY	__	SPECTRAL GAMMA-RAY
__	SPONTANEOUS POTENTIAL	__	INDUCED POLARIZATION
__	SHORT-NORMAL RESISTIVITY	__	INDUCTION
__	CALIPER	__	THERMAL NEUTRON
__	TEMPERATURE	__	LONG-NORMAL RESISTIVITY

LOGGING ENGINEER SHALL CHECK ALL ITEMS 1 THROUGH 43 APPLICABLE TO THE LOGGING OPERATION. ITEMS UNCHECKED SHALL BE DISCUSSED AND RECORDED IN THE "REMARKS" SECTION. ITEMS NOT APPLICABLE SHALL BE DESIGNATED "N/A." LOGGING ENGINEER AND FTR SHALL SIGN AND DATE THE CHECKLIST UPON COMPLETION OF THE LOGGING OPERATION AND THIS CHECKLIST.

NO.		GENERAL
1		LOGGING ENGINEER HAS COPIES OF NEARBY DRILL HOLES FOR COMPARISON
2		LOGGING ENGINEER HAS COPIES OF PREVIOUS LOG RUNS OF THIS DRILL HOLE
3		LOG HEADINGS COMPLETELY FILLED OUT
4		APPROPRIATE LOGGING SCALES CHOSEN AND RECORDED FOR ALL LOGS
5		DEPTHS AT WHICH ANY MAJOR CHANGES IN MUD PROPERTIES OCCURRED ARE INCLUDED IN REMARKS SECTION OF LOG HEADING
6		DEPTHS AT WHICH ANY HOLE OR CASING SITE CHANGES OCCUR ARE INCLUDED IN REMARKS SECTION OF LOG HEADING
7		DEPTHS AT WHICH ANY UNUSUAL HOLE CONDITIONS WERE ENCOUNTERED ARE INCLUDED IN REMARKS SECTION OF LOG HEADING
8		MUD SAMPLES TAKEN FROM FLOW LINE IMMEDIATELY AFTER CALCULATING BOTTOMS UP AND BEFORE COMING OUT OF THE HOLE
9		Rm, Rmf, Rmc AND CORRESPONDING TEMPERATURES MEASURED AND RECORDED
10		CASING I.D. SIZE, BIT SIZE AND HOLE SIZE RECORDED
11		LOGGING SPEEDS RECORDED AND CONFORM TO CLIENTS REQUEST OR SERVICE COMPANY SPECIFICATIONS
12		ALL SCALE CHANGES NOTED ON LOG AT CORRESPONDING DEPTHS
13		DEPTHS TO RECOGNIZABLE MARKER BEDS AGREE ON ALL LOGS
14		TOOL DESCRIPTION AND DESIGNATION INCLUDED IN LOG HEADING
15		DATE OF LAST SHOP CALIBRATION RECORDED
16		BASE OF CASING DETECTED AND LABELED ON ALL LOGS
17		CALIBRATION BEFORE AND AFTER EACH SURVEY
18		ALL CURVES LABELED

NO.		GAMMA RAY LOG
19		BACKGROUND GAMMA RAY RECORDED
20		CALIBRATION SENSITIVITY RECORDED
21		LOGGING SPEED LESS THAN 20 FEET/MINUTE

FIGURE 5-17

FERNALD RI/FS

FIELD BOREHOLE GEOPHYSICAL LOG QUALITY CONTROL CHECKLIST

SITE CODE _____ LOCATION I.D. _____ DRILLER _____

DEPTH LOGGING COMPANY _____

DATE _____ LOGGER _____

NO.		NEUTRON LOG
22		CALIPER RECORDED
23		BACKGROUND GAMMA RAY RECORDED
24		LOGGING SPEED LESS THAN 20 FEET/MINUTE
25		POROSITY (%) AND RATIO CURVES RECORDED
26		NEUTRON MATRIX LABELED

NO.		SPONTANEOUS POTENTIAL
27		GROUND ELECTRODE IN A STABLE POSITION
28		ADEQUATE SCALE FOR SP SENSITIVITY APPROXIMATELY 5 DIVISIONS DEFLECTIONS FROM SHALE ZONE TO CLEAN SAND ZONE
29		SP BASELINE SHIFTS GREATER THAN ONE DIVISION CORRECTED ABRUPTLY AND LABELED
30		IF SP ABNORMAL, RERUN WITH SURVEY CURRENT OFF AND GROUND ELECTRODE CHECKED
31		WHILE ON BOTTOM PRIOR TO LOGGING SP RECORDED AND LABELED WITH CURRENT ON AND OFF
32		WHILE ON BOTTOM PRIOR TO LOGGING SP CALIBRATION SIGNAL RECORDED AND LABELED
33		WHILE STOPPED IN HOLE PRIOR TO LOGGING SP GALVANOMETER CHECKED FOR DRAFT OR CALIBRATION
34		LOGGING SPEED LESS THAN 20 FEET/MINUTE

NO.		RESISTIVITY LOGS
35		RESISTIVITY CURVES DO NOT READ LESS THAN ZERO
36		LOGGING SPEED LESS THAN 20 FEET/MINUTE
37		LATEROLOG HAS SYMMETRICAL CURVE SHAPE
38		BACKGROUND GAMMA RAY RECORDED

NO.		CALIPER LOG
39		CHANGES OF 1/8 INCH CAPABLE OF BEING DISTINGUISHED ON SELECTED SCALE
40		RECORDING MADE IN CASING AS A CHECK OF CALIBRATION
41		LOGGING SPEED LESS THAN 20 FEET/MINUTE

NO.		TEMPERATURE LOG
42		LOG RECORDED WHILE GOING IN HOLE AT LESS THAN 20 FEET/MINUTE
43		FLOW LINE MUD PIT AND AIR TEMPERATURE MEASURED AND RECORDED

NO.		GAMMA-GAMMA DENSITY LOG
44		CALIPER RECORDED
45		BACKGROUND GAMMA RAY RECORDED
46		LOGGING SPEED LESS THAN 20 FEET/MINUTE
47		BULK DENSITY (G/cc) AND POROSITY (%) CURVES RECORDED
48		MATRIX DENSITY AND FLUID DENSITY LABELED
49		CORRECTION CURVES (G/cc) RECORDED

FIGURE 5-17
(CONTINUED)

FERNALD RI/FS

FIELD BOREHOLE GEOPHYSICAL LOG QUALITY CONTROL CHECKLIST

SITE CODE _____ LOCATION I.D. _____ DRILLER _____

DEPTH LOGGING COMPANY _____

DATE _____ LOGGER _____

NO.		SPECTRAL GAMMA RAY LOG
50		LOGGING SPEED LESS THAN 20 FEET/MINUTE
51		FLOW AND T CURVES LABELED
52		BACKGROUND GAMMA RAY RECORDED

NO.		INDUCED POLARIZATION
53		LOGGING SPEED LESS THAN 20 FEET/MINUTE

NO.		INDUCTION LOG
54		RESISTIVITY CURVES DO NOT READ LESS THAN ZERO
55		LOGGING SPEED LESS THAN 20 FEET/MINUTE
56		INDUCTION LOG AND SHORT NORMAL HAVE SYMMETRICAL SHAPE
57		INDUCTION RESISTIVITY CURVE CHECKS CONDUCTIVITY CURVE
58		AMPLIFIED SHORT NORMAL RECORDED
59		SHORT NORMAL DOES NOT READ LESS THAN ZERO
60		CONDUCTIVITY DOES NOT READ LESS THAN ZERO

NO.		THERMAL NEUTRON
61		LOGGING SPEED LESS THAN 20 FEET/MINUTE
62		BACKGROUND GAMMA RAY RECORDED

NO.		GENERAL	HOUR	DATE
63		WHEN SERVICE COMPANY NOTIFIED		
64		WHEN SERVICE COMPANY REQUESTED		
65		WHEN SERVICE COMPANY ARRIVED		
66		WHEN HOLE READY		
67		WHEN LOGGING STARTED		
68		WHEN LOGGING COMPLETED		
69		WHEN SERVICE COMPANY RELEASED		

OPERATIONAL EFFICIENCY CHECK: ITEMS 63 THRU 69 SHALL BE FILLED OUT BY THE FTR

FIGURE 5-17
(CONTINUED)

FERNALD RI/FS

FIELD BOREHOLE GEOPHYSICAL LOG QUALITY CONTROL CHECKLIST

SITE CODE _____ LOCATION I.D. _____ DRILLER _____

DEPTH LOGGING COMPANY _____

DATE _____ LOGGER _____

LOG TYPE	DEPTH (FT.)		TIME (HOUR)	
	FROM	TO	ELAPSED	LOST

NOTE: ELAPSED TIME DOES NOT INCLUDE LOST TIME

REMARKS

NO.		

_____ DATE

_____ (SIGNATURE OF FTR)

_____ DATE

_____ SIGNATURE OF SERVICE COMPANY REPRESENTATIVE AND COMPANY

The data obtained during field aquifer or permeability tests will include:

- Static water level;
- Pumping well water discharge rate;
- Drawdown or pressure versus time data for pumping and/or monitoring wells;
- Water temperature and specific conductance; and
- Test interval.

An aquifer or permeability test data form (Figures 5-18 or 5-19) will be completed for each test to document test results. This form will be the lead page of the data records of the test. The complete original test data records will be kept at the field office. Additionally, a copy of these records will be made for the hydrogeologist and one retained by the field representative in charge of the test.

A diagram of the equipment actually used in each aquifer or permeability test will be prepared by the hydrogeologist or received from the subcontractor responsible for the testing. The diagram will show the tubing dimensions, the depth of water intake, and the location of gauges and packers. The diagram will become part of the data records of each test.

All gauges and flowmeters used in the pump-out procedures will be calibrated before arrival on site. Copies of the documentation of instrument calibration will be obtained from the instrument or testing service company by the hydrogeologist. The calibration records will consist of laboratory measurements and, if performed, any on-site zero adjustment and/or calibration.

Water pumped from the well during the aquifer or permeability test will be disposed of in the General Sump and treated in the WMCO wastewater system.

Figure 5 - 18

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DOCUMENT CHANGE REQUEST NO. 17

FIELD PERMEABILITY TESTS

FALLING HEAD *

PROJECT ______________________ TESTED BY ______________ DATE ______________

PROJECT NO. ______________________ CALCULATED BY ______________ DATE ______________

BORING NO. ______________________ CHECKED BY ______________ DATE ______________

RADIUS OF TEST BORING, R ______________________ PERMEABILITY, K ______________

HEIGHT OF CASING (REF. LEVEL) ABOVE GROUND SURFACE, H_c ______________________

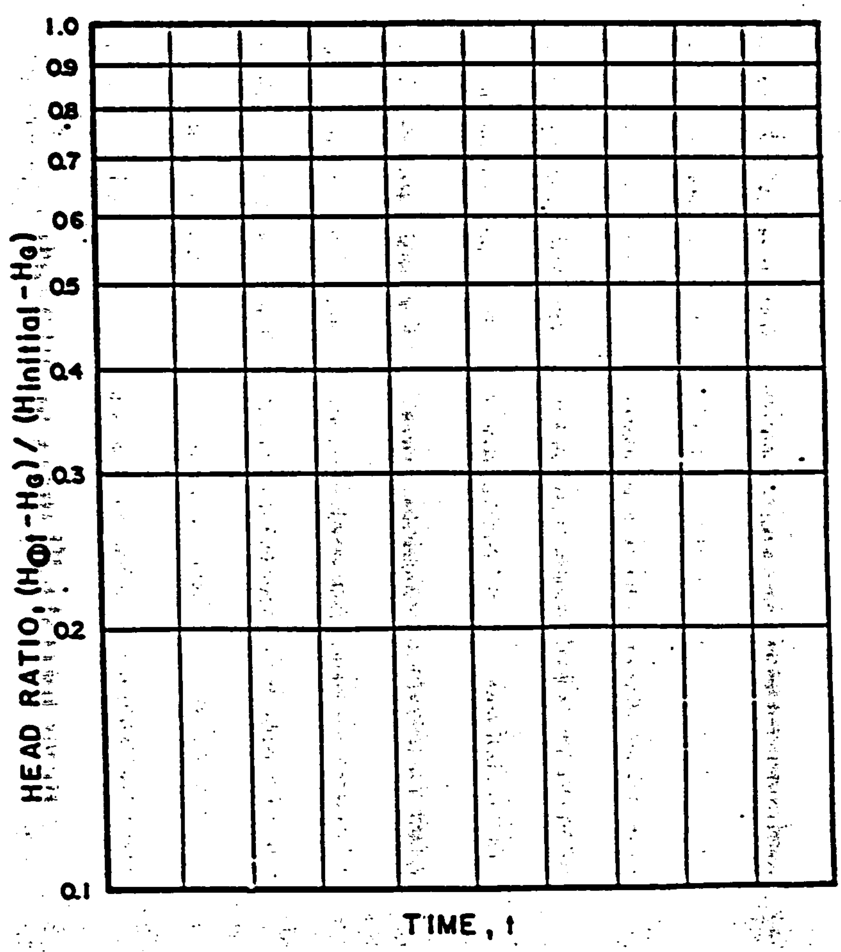
DEPTH TO BOTTOM OF TEST POCKET, L_1 ______________________

DEPTH TO TOP OF TEST POCKET, L_2 ______________________

DEPTH TO GROUNDWATER TABLE FROM TOP OF CASING, H_G ______________________

*CASED HOLE, UNCASED OR PERFORATED EXTENSION OF LENGTH, $L_1 - L_2$.

ELAPSED TIME t SEC.	DEPTH TO WATER IN CASING H Meters



H_1 = DEPTH TO WATER IN CASING FROM TOP OF CASING AT TIME, t_1

H_2 = DEPTH TO WATER IN CASING FROM TOP OF CASING AT TIME, t_2

$$K = \frac{R^2}{2(L_1 - L_2)(t_2 - t_1)} \ln\left[\frac{L_1 - L_2}{R}\right] \ln\left[\frac{H_1 - H_G}{H_2 - H_G}\right]$$

NOTE: THE EQUATION ASSUMES THE HORIZONTAL AND VERTICAL PERMEABILITIES ARE EQUAL.

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FIELD PERMEABILITY TESTS CONSTANT HEAD*

PROJECT_____ TESTED BY_____ DATE_____
PROJECT NO._____ CALCULATED BY_____ DATE_____
BORING NO._____ CHECKED BY_____ DATE_____

HEIGHT OF CASING ABOVE GROUND SURFACE _____
INSIDE RADIUS OF CASING, R _____
DEPTH TO GROUNDWATER TABLE FROM GROUND SURFACE, H_G _____
HEIGHT TO TOP OF WATER IN CASING FROM GROUND SURFACE, H_W _____
DEPTH OF CASING BELOW GROUND SURFACE, H_D _____
LENGTH OF TEST POCKET, L _____

*CASED HOLE, UNCASED EXTENSION OF LENGTH, L

TIME 24 hr. SYSTEM	ELAPSED TIME, t SEC.	VOLUMETER READING, V	FLOW RATE $Q = V/(t_2 - t_1)$	PERMEABILITY K CM / SEC

NOTES:
ANY CONSISTENT SET OF UNITS CAN BE USED IN THE EQUATIONS BELOW.
THE EQUATIONS ASSUME THE HORIZONTAL AND VERTICAL PERMEABILITIES ARE EQUAL.

FOR TESTS BELOW GROUNDWATER TABLE:

$$K = \frac{Q}{2\pi L (H_G + H_W)} \ln \left[\frac{L}{2R} + \sqrt{1 + \left(\frac{L}{2R}\right)^2} \right]$$

FOR TESTS ABOVE GROUNDWATER TABLE:

$$K = \frac{Q}{2\pi L (H_D + H_W)} \ln \left[\frac{L}{2R} + \sqrt{1 + \left(\frac{L}{2R}\right)^2} \right]$$

FIGURE 5-19

In cases where a weir or an orifice are used to measure flow volumes or rates, these devices will be checked on site using a vessel of known volume and a stopwatch. Their accuracy will be documented before testing proceeds.

Equipment will be installed and demonstrated to the technical representative's satisfaction that the system is in proper working order and performing to specifications before the proposed start of the pumping test.

5.6.1 BOREHOLE HYDRAULIC TESTING FOR AQUIFER CHARACTERISTICS

This section provides guidelines and procedures for determining the hydraulic parameters of aquifers using slug tests and pumping tests. The hydraulic conductivity (K) of an aquifer is a measure of the aquifer's ability to conduct water. The greater the value of hydraulic conductivity, the more rapidly the aquifer conducts water. The transmissivity (T) of an aquifer is defined as the ability of the aquifer to transmit water through its entire thickness and is equal to the hydraulic conductivity (K) times the saturated thickness of the aquifer (b); this can be stated as $T = Kb$, for cases in which K is constant with depth. Otherwise, transmissivity is equal to the integration of hydraulic conductivity over depth, $T = \int K(Z)dz$.

Specific yield of an aquifer is the ratio of the volume of water drained by gravity per unit volume of aquifer material. The storage coefficient of an aquifer is the volume of water in storage released from an aquifer column of unit cross section under a unit decline of head.

5.6.1.1 Slug Test

The purpose of this section is to outline procedures for the conduct and evaluation of slug tests, which are a quick and inexpensive method of estimating the hydraulic conductivity or transmissivity of many aquifer systems.

A slug test will generally work in hydrogeologic units where K is less than or equal to 10^{-2} cm/sec. In hydrogeologic units of greater hydraulic conductivity, the water level may return to static level before a sufficient number of water level readings can be made. In hydrogeologic units of low K (10^{-4} to 10^{-7} cm/sec), slug tests are generally a reliable field method of determining hydraulic conductivity or transmissivity near the screened zone of the well.

The advantages of using slug tests to estimate hydraulic conductivities are:

- Estimates can be made in situ and errors incurred in laboratory testing of disturbed samples are avoided.
- Tests can be performed quickly at relatively low cost because a pumping well and observation wells are not required.
- Hydraulic conductivity measurements of small discrete portions of an aquifer can be made (i.e., sand layers in a clay).

The disadvantages to using slug tests must be evaluated before the test is conducted. These include:

- Only the hydraulic conductivity of the area immediately surrounding the well is estimated. These results may not be representative of the average hydraulic conductivity of the area.
- Often only a range of hydraulic conductivity values rather than an average value can be deduced from test results.
- Certain assumptions are made in the analysis process. If the assumptions made are inappropriate for the geologic conditions at the site, the results may be erroneous.
- The storage coefficient (S) usually cannot be determined.
- Data sufficient for analysis may not be collected if the hydraulic conductivity is relatively high.

Design, Drilling, and Construction of a Slug Test Well

Several factors enter into the design of a well for the performance of slug testing. These include depth and diameter of the test well, length of screened interval, and size of screen openings. Some of these factors can be chosen as drilling proceeds or be predetermined from previous borings. Well design is generally determined from a combination of drill cuttings, borings, and geophysical logging.

The depth and screened interval of the hole are determined by the location of the zone of interest. The diameter of the hole and casing to be inserted in the hole should be large enough so that the volume of the slug does not cause water to overflow the top of the casing. This may be a problem when the water level in the borehole is relatively close to the ground surface. The screened interval should consist of screen with a percentage of open intervals large enough so as not to restrict water flowing from the borehole to the subsurface materials tested.

The construction of the test well involves the assembly of the appropriate diameter casing for insertion into the borehole. A preslotted screened section is prepared and attached to the casing string, with a cap on the bottom (to prevent material from entering the bottom of the screen), and the whole assembly is lowered into the hole. If a gravel pack is necessary as a filter because the aquifer contains very fine unconsolidated sand, then it should be placed opposite the screened interval. A bentonite seal is placed between the gravel pack and the grout. The annulus is then grouted from the top of the bentonite seal to the ground surface in order to allow hydraulic connection only between the test well and the zone to be tested. The hole should be flushed or bailed before grouting if drilling fluid remains in the hole. If the ground water in the hole is highly contaminated, special safety precautions should be taken to control the discharge water before test well development.

Equipment necessary to conduct a slug test includes:

- Borehole;
- Water level measuring device;
- Known volume of the slug that will fit into the well or borehole or container to add or remove a known volume of water;
- Watch with second hand;
- Semi-log graph paper; and
- Indelible pen or pencil and paper.

A number of water-level measuring devices can be used for measuring the depth to water in a well. An excellent device for measuring the depth to water during a slug test is the pressure transducer. Accurate readings may also be obtained with electric water-level indicators or weighted tapes. Water-level measurement by the weighted-tape method should be avoided as it may be difficult to obtain a sufficient number of readings for analysis of the test if the water level recovers quickly in a short period of time.

Slug Test Procedure

Before beginning the slug test, the following information should be recorded:

- Site identification and well or borehole location;
- Location and elevation of the reference point from which water depth measurements are made;
- Elevation of ground water with respect to the reference point;
- Date and time of test;
- Well depth, screen length, riser pipe radius, well screen radius, radius of the gravel pack plus the well screen or borehole depth and radius;
- Aquifer or ground water zone being tested;

- Volume of water added or withdrawn or volume of the solid cylinder (slug);
- Type of measuring device used; and
- Names of the personnel conducting the test.

The following procedures will apply to most slug tests. The field procedure required for a particular slug test may be slightly different, however, and the procedures may be modified as necessary.

- Determine the static water level in the well by periodically measuring the depth to water for several minutes and taking the average of the readings.
- Instantaneously introduce or remove a known volume of water to/from the well. Another method is to introduce a solid cylinder of known volume to displace and raise the water level, allow the water level to restabilize and remove the cylinder. It is important to remove or add the volumes as quickly as possible because the analysis assumes as instantaneous change in volume is created in the well.
- With the moment of volume addition/subtraction assigned time zero, measure and record the depth to water and the time of each reading. Depths should be measured to the nearest 0.01 foot. The number of depth-time measurements necessary to complete the test are variable. It is critical to make as many measurements as possible in the early part of the test.
- Continue measuring and recording depth-time measurements until the water level returns to equilibrium conditions or a sufficient number of readings have been made to clearly show a trend on a semi-log plot of time versus depth.

The time required for slug test completion is a function of the volume of the "slug," the hydraulic conductivity of the formation, and the type of well completion. The slug volume should be large enough that a sufficient number of water level measurements can be made before the water level returns to equilibrium conditions. The length of the test may range from less than a minute to several hours.

If the well is to be used as a monitoring well, precautions should be taken that the well is not contaminated by the material introduced into the well. If water is added to the monitoring well, it should be from an uncontaminated tested and/or approved source and transported in a clean, approved container. Bailers or measuring devices should be cleaned prior to the test. If tests are performed on more than one monitoring well, care must be taken to avoid cross contamination of the wells.

Slug Test Data Analysis

The analysis of slug test data is based on a modification of well-known ground water flow equations (the Theim equation, the Theis equation, or subsequent modifications). Several authors have presented analytical solutions for the analysis of slug test data. Most solutions require a semi-logarithmic plot of the data collected: dimensionless head (logarithmic scale) or residual head (logarithmic scale) versus time (arithmetic scale).

Hvorslev (1951) was one of the first researchers to publish techniques of analysis of either constant or slug (falling head) tests in near-surface saturated soils. His analysis for slug tests involves a semi-logarithmic plot of the falling head (water level) divided by the initial head against time. Basic algebraic equations are presented for different configurations of the soil relative to the test hole. In general, the permeability is proportional to a "shape factor" and inversely proportional to the "time lag." The "shape factor" is determined from the test well characteristics or dimensions. "Time lag" is determined from the semi-logarithmic plot.

Cooper et al. (1967) and Papadopoulos et al. (1973) developed a set of type curves for analyzing slug test data, particularly for tests run in materials which are confined (under artesian pressure). Field data are plotted as dimensionless head (arithmetic scale) versus time (logarithmic scale) and matched to a set of

type curves. The match point values are substituted into simple algebraic equations formulated to obtain a value for K.

Bower and Rice (1976) and Bower (1978) developed a technique for analyzing slug test data collected from completely or partially penetrating wells in unconfined aquifers. Their analysis involves a plot of residual head (logarithmic scale) versus time (arithmetic scale). A straight line is applied to the early-time data and used to calculate a value for K.

Pressurized slug test methods have been developed for testing extremely low conductivity (10^{-8} cm/sec or lower) materials (Bredehoeft and Papadopoulos, 1980). Basically, the pressurized slug technique is a modification of the conventional slug test discussed previously. The advantage of the pressurized slug technique is the reduction of time required to perform a test in tight formations. This method involves creating an instantaneous pressure surcharge on drawdown in the test zone, then closing a valve in the well. Based on the rate of decay of the pressure slug and the geometry of the test zone, the transmissivity, hydraulic conductivity, and storage coefficient may be calculated.

The specific equations to be used to calculate the permeability of the aquifer materials are presented in Figures 5-17 and 5-18.

5.6.1.2 Aquifer Pumping Test

The purpose of this section is to briefly describe the use of aquifer pumping tests to evaluate aquifer characteristics. Aquifer pumping tests, commonly referred to as pump tests, are used to determine the hydraulic characteristics and properties of water-bearing zones. Pumping tests provide results that are often more representative of the aquifer's characteristics than the results of slug tests.

Aquifer characteristics which may be obtained from pumping tests include the hydraulic conductivity (K), transmissivity (T)k, and specific yield (S_y) for unconfined aquifers and the storage coefficient (S) for confined aquifers.

Advantages of pumping tests include:

- A greater portion of the aquifer is tested and the results obtained may be more reliable and representative of the aquifer characteristics.
- The coefficient of storage and specific yield values can be determined.

Disadvantages of pumping tests include:

- In low permeability aquifers, long-term pumping may be required to complete the test and obtain reliable aquifer characteristics.
- Disposal of discharged water may require special provisions if the ground water is contaminated.
- Pumping tests may be very expensive when compared to slug tests.

Equipment, personnel, and time commitments needed to conduct pumping tests are greater than those required for slug tests. Briefly, a pumping test consists of pumping one well and recording the drawdown in the pumping well and in other nearby observation wells.

There are several types of pumping tests. The most common type is the constant rate discharge test (Todd, 1980). Variable rate tests are also employed under some conditions. Although the analysis is more complicated, any sort of temporal variations in flow rate can be accounted for by assuming the law of superimposition holds. Usually this is a valid assumption. The most widely used variable rate tests are the step-drawdown test, the constant head test, and the air lift pump test (Kruseman and DeRidder, 1976).

Another useful technique is injection testing. Injection tests, both constant rate and variable rate, are analytically identical to pumping tests except for consideration of the flow rate rather than a withdrawal as a surcharge to the aquifer. The data quality is also similar. Injection tests are commonly used in the petroleum industry (Earlougher, 1977) and numerous applications exist in hazardous waste management practice. Obviously, water sampling for geochemical characterization of the aquifer must be conducted prior to application of this technique. The injection fluid should be free of suspended solids and should be of equal or higher quality than the ground water at the test site.

One major advantage of this type of test is that contaminated ground water is not removed from the formation and, thus, is not a disposal problem. A potential disadvantage of the injection test is that in certain cases the injection well would have to be able to withstand some induced hydraulic pressure. Cases where the injection rate is large enough to elevate the water level above the well casing would require a sealed well head configuration and the proper well construction to prevent leakage of injected fluid on the ground surface. These requirements may be relatively expensive and difficult to operate in the field without the necessary precautions. Once an injection system is working at a constant rate of flow, the data collected are analyzed in much the same way as data from an aquifer pumping test, except that water level buildup instead of drawdown is used in the analysis.

This procedure briefly describes equipment and other factors which must be considered for constant-rate pumping tests. Sources which provide additional information are presented in the list of references. Ground Water Wells (Johnson, 1966) is a field guide with many suggestions on how to perform the test. Ground Water Hydrology (Todd, 1980) and other references, such as Kruseman and DeRidder (1976), provide analytical techniques and

example problems of pumping tests conducted under different geologic conditions.

Aquifer Pumping Test Equipment

The amount of equipment necessary to perform a constant-rate pumping test is greater than that used in a slug test. Equipment and wells needed for a pumping test include:

- Pumping well and pump; Pumps are commonly of the submersible or turbine type. The pumping well should be properly developed prior to testing.
- One or more observation wells hydraulically connected to the pumped aquifer and completed to the proper specifications for the particular test;
- An orifice, weir, flow meter, container or other type of water measuring device to accurately measure and monitor the discharge from the pumping well;
- Sufficient pipe to transport the discharge from the pumping well away from the area to prevent infiltration in the area of influence of the pump zone;
- Gate valve on the discharge pipe to control the pumping rate;
- Outlet near the well head for water quality determinations and sampling;
- Depth-to-water measuring devices for each observation well and the pumping well (this may include steel tapes, electric sounding probes, Stevens recorders, or pressure transducers);
- A thermometer and other necessary water quality equipment;
- Watches capable of reading to the nearest second for all observers, or a stopwatch;
- 3-cycle by 5-cycle log and 5-cycle semi-log graph paper;
- Indelible pens or pencils and form for recording times and drawdown measurements at each well;
- Appropriate references and calculator for determinations in the field; and

- A barometer or recording barograph (for tests conducted in confined aquifers).

Aquifer Pumping Test Pump Well Design

Design of the pump well is an important consideration in aquifer testing. In some cases, an existing well will be used for pumping. When sufficient funds and conditions permit, a pumping well can be designed and constructed specifically for the test. The pumping well should be screened throughout the thickness of the aquifer to be tested with a standard well screen. The well should be gravel packed, if necessary, to minimize sand production in unconsolidated fine-grained aquifers. Standard well construction techniques are discussed in Johnson (1966). The well should be correctly sealed from overlying and underlying units that are not to be directly pumped and so that leakage down the well annulus cannot occur; such leakage could interfere with the interpretation of the test. The completed pumping well should be developed by the appropriate methods to remove drilling fluid from the surrounding aquifer materials. Proper development of the well may prevent an unexpected variation in the pumping rate during the constant discharge test which, if the variation occurred, could lead to inconsistent drawdown data from the pumping well.

Aquifer Pumping Test Observation Well Siting

The location and number of the observation wells depends on several factors:

- Whether the aquifer tested is confined or unconfined;
- The thickness of the aquifer;
- The anisotropy of the aquifer;
- The location of the screened interval of the pumping well relative to the total aquifer thickness;
- The location of aquifer boundaries, whether positive (lake or stream) or negative (impermeable boundaries); and
- Practical and economic considerations.

Any number of observation wells may be considered. Often, four wells are desirable, three on a line passing through the center of the pumped well and one on a line normal to that line and also passing through the pumping well. A number of guidelines for location of observation wells are presented in the Ground water Manual prepared by the U.S. Department of Interior and Kruseman and DeRidder (1976).

As a general rule for tests performed in both confined and unconfined aquifers, the observation wells should be screened or completed in a substantial portion of the aquifer thickness in approximately the median depth of the test zone. In some cases, special tests require that observation wells be selectively completed in several depth zones in order to accurately determine aquifer characteristics such as anisotropy and vertical hydraulic conductivity.

The location of the observation well from the pumping well is partially controlled by the aquifer conditions, whether confined or unconfined. The location of observation wells generally depends on four aquifer conditions:

- For most aquifers with fully penetrating pumping wells, observation wells should be located at a distance estimated by using the Theis formulation (Theis, 1935). Use of this formulation is described by Walton (1970). Assumed aquifer parameters are used to determine a location which will give the amount of drawdown required for proper analysis.

- Thin aquifers with fully penetrating wells: for confined aquifers, the nearest observation well should be located at least 25 feet from the pumping well; for unconfined aquifers, observation wells should generally be located 15 to 100 feet from the pumping well.

- For thick isotropic aquifers with a partially penetrating pumping well, observation wells should be located one and one-half to two times the aquifer thickness from the pumping well.

- For thick anisotropic aquifers with a partially penetrating well, observation wells should be located at a minimum distance from the pumping well equal to twice the thickness of the aquifer times the square root of the ratio of the horizontal to the vertical hydraulic conductivity.

Aquifer Pumping Test Procedures

The water level must be monitored over time in the pumping well and in each observation well as the pumping well is discharged at a constant rate.

The following data must be recorded accurately at the time the test is performed:

- Well identification number or location of pumping well and each observation well;
- Location and elevation of each well;
- Location and elevation of reference point from which water depth measurements are made and elevation of ground surface with respect to the reference point;
- Date and time of test; and
- Well depth, pump depth, screen length, well radius and radius of gravel pack plus well screen for each well.

All gauges, transducers, flow meters, etc., used in conducting pumping tests, shall be calibrated before use at the site. Copies of the documentation of instrumentation calibration should be obtained by the hydrogeologist and later filed with the test data records. The calibration records will consist of laboratory measurements and any on-site zero adjustment and/or calibration, if performed. In cases where a weir or an orifice is used to measure flow rates, the device will be checked on site using a container of measured volume and stopwatch. Accuracy of the meters must be verified before testing proceeds.

Changes in barometric pressure are recorded during the test (preferably with an on-site barograph) to correct water levels

for any possible fluctuations which may occur due to changing atmospheric conditions.

During an aquifer test, water levels should be measured to give at least 10 observations of drawdown within each log cycle of time. During the early part of the test, sufficient personnel should be available to have at least one person at each observation well. After the first two hours, two people are usually sufficient to continue the test. It is not necessary to take readings at the wells simultaneously. It is very important that depth-to-water readings be measured accurately and readings recorded at the exact time measured.

The duration of the test is determined by the needs of the project and the aquifer properties. One simple test for determining adequacy of data is when the log-time versus drawdown for the most distant observation well begins to plot as a straight line on the semi-log graph paper. There are several exceptions to this simple rule of thumb; therefore, it should be considered a minimum criteria. Different hydrogeologic conditions can produce straight-line trends on log-time versus drawdown plots. In general, longer tests produce more definitive results. Unconfined aquifers and partially penetrating wells may have shorter test durations. A knowledge of the local hydrology, combined with a clear understanding of the overall objectives of the project, is necessary in interpreting just how long the test should be conducted. There is no need to continue the test if the water level becomes constant with time. This normally indicates that a hydrogeologic source has been intercepted and that additional useful information will not be collected by continuing the pumping portion of the test.

Measurement of recovering water levels after pumping stops is often performed to verify results obtained from the pumping portion of the test. The recovering water levels in the pumping

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well and the observation wells are measured for a period immediately following cessation of pumping. Monitoring of the recovery period should, by rule of thumb, be at least one-half the length of the pumping portion of the aquifer test. This length of time usually allows the collection of useful data for additional analysis.

Aquifer Pumping Test Data Analysis

Numerous techniques of analysis have been developed by researchers to evaluate water level data collected from constant discharge aquifer pumping tests. Many of the analyses use the graphical curve matching technique which involves the matching of theoretical type curves to plot of log-drawdown versus log-time from the observation wells. Other analyses rely on other graphical techniques such as application of a straight line to plots of drawdown versus log-time.

Theis (1935) developed a theoretical formula which related the lowering of the potentiometric level in an aquifer due to the constant withdrawal of water from the aquifer. This classic formulation has been widely used to predict water level response in aquifers due to specified pumping stresses. Several authors have described the use of this formula in estimating transmissivity and storativity from aquifer test data: Lohman (1972), Walton (1970), Todd (1980) and Freeze and Cherry (1979). In brief, the method involves superimposing the Theis solution (or "type" curve) on a log-log plot of drawdown versus time data from a test well. The coordinate axis of the type curve must be kept parallel to the field data plot axes. Data from the "match point" are used to solve two algebraic equations which give the value of transmissivity (T) and storativity (S) (or storage coefficient). Hydraulic conductivity (K) may be computed by dividing T by the aquifer thickness (b).

The same aquifer parameters can also be determined from a semi-log plot of drawdown versus time for either the pump well or observation wells. Cooper and Jacob (1946) analytical data when plotted as described above, compares with Theis's T, S and K values.

Hydrogeolgic judgment should be used when applying straight line (Cooper-Jacob method) fits to these plots because different hydrogeologic conditions can produce similar trends in the data plot. For example, bounded aquifers produce straight line trends that may yield apparent transmissivity values that are too low. Knowledge of the hydrogeology of the area and the type of aquifer conditions is essential for correct analysis of test data.

Since the development of the Theis equation, several other formulas have been published which attempt to describe the response of certain aquifers to a constant pumping stress. Hantush (1955, 1956) developed a formula and a corresponding set of type curves for an aquifer which is overlain and/or underlain by a confining bed which has significant storage and experiences vertical flow when the adjacent aquifer is pumped. The same curve matching procedure is used here as with the Theis type curve, except that several type curves are available for matching to the data, depending on the degree of leakage into the pumped aquifer. Hydraulic characteristics of the confining unit can be calculated using the set of Hantush type curves.

Another commonly used set of type curves was developed by Boulton (1954, 1963) and is used when the aquifer tested is unconfined and may exhibit a phenomenon known as delayed yield. Delayed yield is a result of retardation of drainage due to capillary tension. Boulton's type curves include a set of early-time type curves and late-time type curves. The early-time data and late-time data are respectively matched to the early-time and late-time type curves to give values for transmissivity, storage

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coefficient, and specific yield. The particular curve used in the set of "type" curves yields additional information concerning how the unconfined aquifer responds to pumping. Nueman (1972, 1974, 1975) also developed a set of type curves for the response of unconfined aquifers to pumping, but used different assumptions concerning the physical processes in effect. In practice, Nueman and Boulton curves have been shown to give similar results.

The specific equations to be used to calculate the permeability of the aquifer materials from the pumping test data are presented in Figures 5-18 and 5-19.

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6.0 SAMPLE COLLECTION PROCEDURES

Detailed project sampling plans have been prepared to document the scope and methodology of exploration, radiation measurement, and sampling activities for the FMPC.

The following considerations form the basis for the site-specific sampling program:

- Frequency of sampling;
- Location and number of monitoring stations to be sampled;
- Methods of sampling to be employed;
- Media to be sampled;
- Number of samples to be collected;
- Volume of samples to be collected;
- Type and kind of analyses to be performed in the field;
- Type and kind of analyses to be performed at the laboratories;
- Procedures and precautions to be followed during sampling; and
- Methods of preservation and shipment.

Sampling will be frequent enough to identify materials and to describe important material changes. Methods of sampling employed shall preserve the integrity of material parameters.

The following guidelines are applicable to the implementation of a field sampling program:

- Prepare and study the site map showing the location of sample monitoring stations.
- Review the sample collection program to become familiar with the overall scope of the study and also sampling procedures and equipment, sample handling procedures, and shipping requirements.

- Determine the characteristics of the material to be sampled, become familiar with the safety precautions and practices, and obtain the necessary safety equipment.

- Obtain equipment and materials necessary to perform field sampling and analyses.

- Review manufacturer's instructions on equipment calibration and sensitivity.

- Calibrate all field equipment prior to field work according to manufacturer's instructions and calibration procedures. Note: Certain equipment may require calibration in the field before each measurement.

- Fill out the appropriate sample field collection reports (Sample Collection Log, Figure 7-3; Water Quality Field Collection Report, Figure 6-1; or Ecological Field Survey Sample Collection Log, Figure 6-2) completely prior to leaving the field location.

- Label all sample containers with appropriate information. This includes project number and name, sample number, location, sampling date and time, preservatives added, and sampler's initials.

- Complete chain-of-custody records and laboratory request for analysis forms which will accompany all samples during shipment.

The following guidelines are applicable for sample packaging to avoid breakage or cross contamination:

- Sample container lids are never to be mixed. Sample lids will stay on the original container until time of sampling (only open sample container at the time of sampling). The original sample containers will arrive in the field in packages with custody tape affixed at the appropriate access points. When sample container packages are received in the field, the chain-of-custody form for shipping the empty containers to the field will be appropriately marked to state if the custody seal was affixed when the package arrived.

- After a sample is placed in a plastic/glass container, the sample container will be secured with a custody seal and placed in a plastic bag to minimize the potential for contamination from vermiculite or other

DOCUMENT CHANGE REQUEST NO. 12

FERNALD
RI/FS

Date: _____
Time: _____
Page: ____ of ____
Page: _____

WATER QUALITY FIELD COLLECTION REPORT

PROJECT NAME _____ SAMPLE LOCATION _____
PROJECT NUMBER _____
DATE COLLECTED _____
TIME COLLECTED _____ SAMPLE NUMBER _____
COLLECTED BY _____ SAMPLE TYPE _____

SAMPLING INFORMATION

AIR TEMPERATURE _____
DO SATURATION IN AIR _____
WATER TEMPERATURE _____
DEPTH OF SAMPLE _____
WATER LEVEL _____

FIELD READINGS

	READ 1	READ 2	READ 3
pH			
Spec. Cond. uMHOS/cm			
D.O. MG/L			

METER CALIBRATION

pH Temp.	pH Std.	pH Std.	D.O. Temp.	D.O. Zero	D.O. Full Sc	D.O. Calib. O_2	Spec. Cond Temp.	Spec. Cond. Low	Spec. Cond. High
OK ✓			OK ✓				OK ✓		

WEATHER CONDITIONS _____
ADDITIONAL REMARKS _____

TEST EQUIPMENT LIST

EQUIPMENT NUMBER	EQUIPMENT NAME

NOTE: ONLY EQUIPMENT SUBJECT TO CALIBRATION NEED BE LISTED.

Figure 6.1

DATE						
TIME						
PAGE ____ OF ____						
AGE						

FERNALD RI/FS

ECOLOGICAL FIELD SURVEY
SAMPLE COLLECTION LOG

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PROJECT NAME _____ PROJECT NO. _____

COLLECTED BY _____ DATE _____ TIME _____

STATION NAME _____ STATION NO. _____

LOCATION _____

WEATHER _____ AIR TEMPERATURE _____

SAMPLE NO.	SAMPLE TYPE	COLLECTION METHOD/ SAMPLER	DEPTH	COLLECTION DIMENSIONS	SAMPLE PRESERVATIVE

LOCATION SKETCH

HABITAT:

TYPE _____

AVG. WIDTH _____ PERCENT SHADING _____

AVG. DEPTH _____ FLOW VELOCITY _____

TURBIDITY _____ POOL/RIFFLE RATIO _____

COVER _____

SUBSTRATE _____

GRADIENT _____

BANKS _____

GENERAL DESCRIPTION OF:

LAND USE/ TOPOGRAPHY _____

POLLUTION SOURCES _____

FLOODPLAIN _____

MACROPHYTES _____

CONDITIONS/COMMENTS _____

DEPTH	DISSOLVED OXYGEN	WATER TEMP.
SURF.		

FIGURE 6-2

packing material. Sample containers may be placed between cardboard inserts and will not need to be placed in plastic bags (e.g., subsurface soil sample jars are placed back in their original shipping container, a cardboard box with cardboard inserts).

- Upon arrival at the laboratory, the QC coordinator, or designee will examine the contents of the shipping container and document on the chain-of-custody record if any sample containers do not have the custody tape affixed.

- Shipping coolers with plastic/glass containers will be filled initially with approximately three inches of vermiculite or suitable substitute (non-combustible, absorbent packing material). Under no circumstances will locally obtained material (sawdust, sand, etc.) be used. Do not use earth or ice as packing materials.

- The secured sample containers will be placed in the cooler in such a way so they do not touch one another. Use additional container protection (e.g., new, unused paint cans), if necessary. (Keep cooler closed except when placing sample in cooler).

- Commercially available artificial icing material will be used. If unavoidable, ice may be used, provided that it is placed in a plastic bag. Ice is not to be used as a substitute for packing material.

- Remaining space in the cooler will be filled with inert packing material.

- The original chain-of-custody and request-for-analysis records will be placed in a plastic bag and taped to the bottom of the cooler lid. (Chain-of-custody and request-for-analysis procedures are in Section 7.0.)

Samples will be properly packaged and labeled for shipment and dispatched to the appropriate laboratory for analysis. Separate chain-of-custody and request-for-analysis records will be prepared for each laboratory. The following requirements for shipping containers will be followed:

- United States Department of Transportation (DOT) regulations covering the transport of hazardous materials are contained in the Code of Federal Regulations (CFR) Title 49, Parts 170-179. Field personnel should acquaint themselves with the general provisions of these requirements and with IT's Manual of Practice: Sample Packaging and Shipment.

- Shipping containers are to be padlocked or custody-sealed for shipment, as appropriate. The packing custody seal should consist of filament tape wrapped around the container, with a custody seal affixed at appropriate access points. In this way, access to the container can be gained only by cutting the filament tape and breaking the seal.

- Shipping cooler containers will be secured by field personnel with a proper custody seal, marked with indelible pen or ink, and addressed to the appropriate laboratory.

- Field personnel will make arrangements for transportation of samples. When custody is relinquished to a shipper, field personnel will telephone the receiving laboratory custodian to report the expected time of arrival of the sample shipment and the existing time constraints (holding times) for sample analysis.

The FMPC sampling program is based on the project objectives coupled with a review of existing data for the site vicinity. The sampling program includes the following:

- Ground water sampling;
- Surface water sampling;
- Surface soil sampling;
- Sediment sampling;
- Subsurface soil sampling; and
- Radon sampling.

6.1 GROUND WATER SAMPLING TECHNIQUES

The primary consideration in ground water sampling is to obtain a representative sample of the ground water body. To safeguard against collecting nonrepresentative stagnant water from a well or piezometer, the following guidelines and techniques should be observed during sample withdrawal:

- As a general rule, all monitoring wells will be pumped or bailed prior to collecting a sample. Evacuation of at least three well volumes of water is recommended for a representative sample.

- For wells that can be pumped or bailed dry, the well will be evacuated and allowed to recover prior to

sample withdrawal. If the recovery rate is fairly rapid, and if time allows, evacuation of more than one volume of water is recommended.

- As soon as the well recovers, samples will be collected in accordance with the stability and volatility of the parameters to be tested and the Water Quality Field Collection Report (Figure 6-1) will be completed. For instance, samples for HSL volatile organic compounds, pH, specific conductance, and temperature will be collected first. Parameters which are not sensitive to pH or volatilization should be drawn last.

- For wells that cannot be pumped or bailed dry, at least three well volumes of water will be removed before collecting samples.

- Care will be taken to avoid excessive pumping of a monitoring well. Excessive pumping can lead to an increase or decrease in the concentrations of a contaminant at the sampling point of interest.

- A stainless steel submersible pump will be used to purge the monitoring wells prior to sample collection. A water-level measurement will be initially taken to determine the depth to ground water in the casing. The submersible pump will be lowered to a depth of five to ten feet below the water level, always above the well screen. The well will be initially pruged from this depth so that fresh water from the screened interval will move upward through the casing and completely flush the well. The pumping rate will be less than 20 gallons per minute (gpm) and will continue until field pH, temperautre, and specific conductance readings hav estabilized.

- The purge pump and lines will be decontaminated between wells using procedures as specified below.

- If drawdown is significant, the submersible pump will be lowered during purging to keep the pump five to then feet below the water level in the casing and the pumping rate will be reduced.

- Once the well has been purged and allowed to recharge and stabilize, samples will be collected through the stainless steel sampling valve. In the case where samples are being collected to be analyzied for TOC, TOX, or volatile organics, samples will be collected using either a positive gas displacement stainless steel and/or Teflon bladder pump or a teflon bailer.

- During sampling, the pump will be operated continuously and the flow rate reduced to one liter per minute for the collection of volatile organic compound samples.

- When the pump and lines are removed from a well, they will be placed on plastic sheeting to avoid contact with the ground.

- Prior to re-use, the pump and lines will be drained and the outside surfaces will be decontaminated with a water wash and deionized water rinse. The internal surfaces will be decontaminated by pumping deionized water through the pump system. Purging of the next monitoring well will further reduce the possibility of cross-contamination. Should the equipment become visibly contaminated, it will be disassembled and cleaned by the following procedures:

  - In the case of inorganic contaminants, the equipment will first be washed with a nonphosphate detergent and then rinsed with dilute (0.1 N) hydrochloric acid followed by two separate deionized water rinses.

  - In the case of organic contaminants, the equipment will first be washed with a nonphosphate detergent and then rinsed with tap water, methanol, and two separate deionized water rinses.

  - As described above, the equipment will be thoroughly rinsed with two deionized water rinses to remove traces of hydrochloric acid, detergent, and methanol (acetone may be substituted for methanol if volatile organic compounds are not being determined).

  - Sampling equipment will not be placed directly on the ground or on other contaminated surfaces prior to insertion into the well, but will be placed on a clean plastic sheet adjacent to or around the well.

  - The final deionized water rinse will be sampled and analyzed at a frequency of one per every set of twenty locations, or fraction thereof, to check for cross-contamination between monitoring wells.

  - Decontamination of the submersible sampling pump(s) and other sampling equipment will be performed at a designated central staging area at the FMPC. If this is not possible due to extenuating circumstances, the sampling equipment may be decontaminated in the field.

- Existing wells with limited access will be sampled from the pump discharge.

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- Field measurements will be performed on unpreserved samples immediately after sample collection. Samples collected for field measurements will be kept separate from samples preserved for shipment to the laboratory.

- A sufficient amount of sample will be collected to perform all the required analyses. The sample bottles containing the appropriate preservatives will be filled to the bottle neck with the sample, without overflowing. Sample container lids will be tight and secure.

- Samples collected will be stored in the field in a cooler and maintained at approximately 4 degrees Celsius using blue ice or other approved, sealed cooling materials.

- Samples will be properly labeled and chain-of-custody records, water quality field collection report (Figure 6-1), sample collection log, and laboratory request for analysis forms will be properly filled out.

6.1.1 PARAMETER-SPECIFIC SAMPLING PROCEDURES

Water sample collection for volatile organic compounds, acid and base-neutral extractable compounds, dissolved metals, and general chemistry parameters should be performed in accordance with the following procedures:

- Volatile Organic Compounds

  - Two screw-cap vials with Teflon-lined silicone rubber septa (U.S. EPA-approved vials) will be filled to overflowing and sealed without any entrapped air bubbles. These vials will be 40 milliliters or larger.

  - Each vial will be visually checked for air bubbles and placed in a cooler.

  - A field/trip blank per each sample set will accompany the samples back to the laboratory. The field/trip blank will consist of organic-free deionized water used in the field to decontaminate the sampling equipment. The field/trip blank will monitor for ambient contamination levels in the cooler during transport. Field/trip blanks will be analyzed at a frequency of one per every set of twenty samples or fraction thereof.

  - Rinsate samples will be collected from sample collection tools following each well sampling to

monitor for potential cross contamination of samples between wells or laboratory cleaned sampling equipment will be used for each well. Rinsate samples will be analyzed at a frequency of one per every set of twenty samples or fraction thereof.

- A sample collection log, a chain-of-custody record, a laboratory request for analysis form, and a sample label will be completed in the field. All these forms, except for the sample collection log, will accompany the samples to the laboratory.

- The cooler will be packed with artificial icing material to maintain the samples at 4 degrees Celsius during shipment, sealed, and transported to the laboratory.

- Acid and Base-Neutral Extractable Compounds and Pesticides/PCBs

  - Appropriate glass sample bottles complete with teflon-lined caps will be filled to capacity and sealed.

  - The sample bottles will be placed in a cooler with sufficient packing to prevent breakage during shipment.

  - A field/trip blank per each sample set will accompany the samples back to the laboratory. The field/trip blank will consist of organic-free deionized water used in the field to decontaminate the sampling equipment. Field/trip blanks will be analyzed at a frequency of one per every set of twenty samples or fraction thereof.

  - Rinsate samples will be collected following each well sampling to monitor for potential cross contamination of samples between wells or laboratory cleaned sampling equipment will be used in each well. Rinsate samples will be analyzed at a frequency of one per every set of twenty samples or fraction thereof.

  - A sample collection log, a chain-of-custody record, a laboratory request for analysis form, and a sample label will be filled out in the field. All of these forms, except for the sample collection log will accompany the samples to the laboratory.

  - The cooler will be packed with artificial icing material to maintain the samples at 4 degrees Celsius during shipment, sealed, and transported to the laboratory.

- Dissolved Metals

  - When it is desired to determine concentrations of dissolved metals in a water system, the sample should be filtered in the field, as soon as possible after collection, through a 0.45-micron filter using a Millipore filtration apparatus or equivalent equipped with a hand or electrical vacuum pump.

  - The filtering apparatus or equivalent should be cleaned or rinsed thoroughly with deionized water before filtering each sample.

  - It is recommended that the first 100 to 150 milliliters of filtrate from each sample be discarded to rinse the filter and filtration apparatus of any contaminating substances.

  - The filtered sample is immediately transferred to a 500-milliliter plastic bottle containing one milliliter of concentrated nitric acid preservative per 100 milliliters of sample.

  - The sample pH will be checked with pH paper or a pH meter to check that the pH is less than two. The bottle will be sealed with a plastic cap fitted with a polyethylene liner.

  - A sample collection log, a chain-of-custody record, a laboratory request for analysis form, and a sample label will be completed in the field. All of these forms, except for the sample collection log, will accompany the sample to the laboratory.

- General Chemistry Parameters

  - Sample bottles will be either polyethylene or glass and will contain the appropriate chemical preservatives. Sample bottle closures will be plastic with polyethylene liners.

  - Sample bottles will be filled to capacity and sealed.

  - A field/trip blank per each sample set will accompany the samples back to the laboratory. The field/trip blank will consist of organic-free deionized water used in the field to decontaminate the sampling equipment. Field/trip blanks will be analyzed at a frequency of one per every set of twenty samples or fraction thereof.

  - Rinsate samples will be collected following each well sampling to monitor for potential cross contamination

of samples between wells or laboratory cleaned sampling equipment will be used for each well. Rinsate samples will be analyzed at a frequency of one per every set of twenty samples or fraction thereof.

- The sample bottles will be placed in a cooler with sufficient packing to prevent breakage during shipment.

- Collected samples will be stored in the field in an ice chest filled with blue ice and maintained at approximately 4 degrees Celsius.

- Samples will be properly labeled and sample collection logs, chain-of-custody records, and laboratory request for analysis form will be properly completed.

- Radionuclide Parameters

  - In general, samples for radionuclide analyses can be collected in the same manner as samples for other chemical analyses.

  - Flush sample lines and sampling equipment with sample medium to minimize radionuclide adsorption effects.

  - Sample containers made of Teflon, polyethylene, or polyvinyl chloride (PVC) will be used to minimize radionuclide losses by adsorption. (Glass and metal containers tend to adsorb radionuclides.)

  - Floating debris and bottom sediments will be avoided when sampling surface waters. Avoid suspended sediments when sampling ground water.

  - The pump or Teflon bailer will be decontaminated in accordance with the procedures in Section 6.1, Ground Water Sampling Techniques, to minimize contamination of subsequent samples.

  - Water samples will be filtered in the field through an 0.45-micron membrane filter as soon as possible after sample collection when specific dissolved radionuclides are to be determined. Containers without preservative will be used to initially collect samples prior to filtration. Transfer samples to containers with preservative after filtration has been performed. When samples are collected for analysis of total radionuclide concentrations, containers with preservative will be used to collect the sample and filtration will not be required.

- Sample container lids will be tightly secured.
- Samples will be properly labeled and chain-of-custody records, sample collection logs, and laboratory request for analysis forms will be properly completed.

6.1.2 WATER LEVEL MEASUREMENTS

The following procedures should be followed for collecting water level data from wells:

- Obtain permission from owner to measure water level in well if required off site.
- In the case of domestic wells, ask the owner not to use water for short time so pump does not activate and affect water level. Make sure water level is stable.
- Remove well cap if an access hole is not available. (Be sure not to take water-level measurements in the riser pipe from the pump discharge line.)
- Lower M-scope probe until water is reached (this will be indicated by a buzzer, meter needle deflection, or light); raise probe above the water level and slightly shake; then lower again and recheck. Be careful not to get tangled with electrical wiring from pump, if present.
- Note depth to water to 0.01 foot from the measuring point, i.e., top of casing, top of sanitary seal.
- Enter water level and measuring point in log with date and time or on Figure 5-11, Piezometer Data Sheet.
- Replace cover on well.

6.2 FIELD ANALYTICAL PROCEDURES FOR GROUND WATER SAMPLES

Immediately following sample collection, temperature, pH, specific conductance, and dissolved oxygen will be measured in the field and documented on the Water Quality Field Collection Report (Figure 6-1). The following field procedures pertain to these tests. All determinations will be performed on unpreserved samples. In adverse weather conditions, field determinations may be performed indoors in an area maintained at 20 to 25 degrees Celsius. Ground water field measurements may also be taken in

situ (downhole) to avoid changes which might occur if the sample is removed from the well.

6.2.1 TEMPERATURE

Scope and Application

The temperature of surface and ground water is important for numerous applications. It is required to normalize data from other analytical determinations such as pH, specific conductance, alkalinity, dissolved oxygen etc.

Summary of Method

Temperature readings are obtained by partially immersing the thermometer in a sample and allowing it to equilibrate for about two minutes. A minimum of two consecutive readings are obtained on each sample.

Apparatus

A metal-cased, direct-reading thermocouple with a normal range of zero to 50 degrees Celsius measured in two-degree intervals is used..

Measurement Practices

- Temperature readings should be taken immediately after sample collection on unpreserved samples.
- Readings should be taken until two identical values are obtained.
- The average readings for each sample will be documented immediately on field measurement or collection forms.

Reference

1. American Public Health Association, et al., 1985, Standard Methods for the Examination of Water and Wastewater, 16th Edition, Method 212, pp. 126-127.

6.2.2 pH

Scope and Application

pH is defined as the negative logarithm of hydrogen-ion activity in a sample and is a measure of effective hydrogen-ion concentration. The pH values are very useful for assessing the acidic or basic nature of a body of water and also for elucidating complex chemical reactions occurring in water.

Summary of Method

The pH of water is normally determined by immersing a combination electrode (glass and reference electrode) in the solution and measuring the potential difference with a pH meter.

Apparatus

- Standard pH meter (battery operated) with an expanded scale capable of measuring pH to the nearest 0.01 unit.
- Combination electrode.
- Three standard buffer solutions
  - pH 4.0;
  - pH 7.0; and
  - pH 10.0.
- A metal-cased direct-reading thermocouple with a normal range of zero to 50 degrees Celsius measured in two-degree intervals.

Calibration

- Expose filling hole by lowering rubber sleeve.
- Make sure that the electrode is filled with potassium chloride (KCl) solution.
- Rinse electrodes with distilled water.
- Set pH meter to proper ambient water temperature. Temperature correction of readings is automatic.

- Calibrate pH meter with at least two appropriate buffers before starting pH measurement and also after every fifth sample. Follow manufacturer's instructions for calibration.

Sample Measurement

- Rinse electrodes with distilled water.
- Transfer sample into a 100-milliliter beaker.
- Insert electrode into the sample solution and let it equilibrate for a few minutes. Stir the sample during measurement.
- Read the pH values on the pH meter to the nearest 0.1 unit.

Measurement Practices

- Measure temperature and set meter to the correct temperature setting.
- Rinse electrodes with distilled water between each measurement.
- Make sure that the line cord is properly attached to the instrument.
- Expose filling hole on the electrode by lowering rubber sleeve.
- Electrode tip needs to be immersed only one inch to obtain accurate readings.
- Never move or touch connecting cables during pH measurement.

References

1. American Public Health Association, et al., 1985, Standard Methods for the Examination of Water and Wastewater, 16th Edition, Method 423, pp. 429-437.

2. American Society for Testing and Materials (ASTM), 1985, Water, Standard D1293-84, pp. 253-264.

3. Instruction Manual for Orion's 407A Specific Ion Meter.

6.2.3 SPECIFIC CONDUCTANCE

Scope of Application

The ability of a solution to carry an electric current under specific conditions is reflected by specific conductance values. These values also indicate the concentration of dissolved solids in the natural water. Since specific conductance of a sample can change with time, this determination will be carried out in the field. The field measurement can also aid in assessing whether the sample is representative of the site.

Summary of Method

A conductivity cell is immersed in a sample of water and the conductance in micromhos per centimeter (μmhos/cm) is measured directly from the meter. The readings are corrected for temperature and are recalculated for 25 degrees Celsius.

Apparatus

- Conductivity cell or probe;
- Conductivity meter; and
- Thermometer.

Calibration

- Switch on the instrument and let it warm up for a few minutes.
- Calibrate according to the manufacturer's instructions.

Sample Measurement

- Insert the probe completely into the sample.
- Switch the meter to the appropriate scale and record the readings in μmhos/cm.
- Measure the temperature of the sample with a thermometer.
- Record all readings on the field measurement form.

- Correct the specific conductances values for temperature using appropriate temperature correction factors.

Measurement Practice

- Replace battery when the read line adjustment cannot be accomplished.
- Rinse probe with distilled water between each measurement.

References

1. American Public Health Association, et al., 1985, Standard Methods for the Examination of Water and Wastewater, 16th Edition, Method 205, pp. 76-80.

2. Instruction Manual for YSI Model 33 S-C-T Meter, Yellow Springs Instrument Company.

6.2.4 DISSOLVED OXYGEN

Scope and Application

Adequate dissolved oxygen is necessary to maintain the life of aquatic organisms. The dissolved oxygen concentration may also have an effect on the redox potential of surface and ground water and the chemical behavior of aqueous constituents. Physical, chemical, and biochemical activities in water may affect the dissolved oxygen levels.

Summary of Method

Dissolved oxygen is normally measured in the field by immersing a membrane electrode into the water. Oxygen gas molecules diffuse through a membrane into a measuring cell at a rate proportional to the concentration of molecular oxygen in the water. Inside the sensor, the oxygen reacts with an electrolyte and is reduced spontaneously or by an applied voltage, depending on the instrument. The current that is generated is directly proportional to the concentration of molecular oxygen in the water outside the sensor. The instrument reading is converted to the concentration

- Frequently change and calibrate membrane electrode to eliminate interference from gases other than oxygen.
- Rinse probe with distilled water between each measurement.
- Store the probe in a water-saturated air environment.

References

1. American Public Health Association, et al., 1985, Standard Methods for the Examination of Water and Wastewater, 16th Edition, Method 421F, pp. 422-426.

6.2.5 Alkalinity

Scope and Application

Alkalinity is a measure of the acid buffering capacity of a sample and is formally defined as the equivalent sum of bases that are titratable with a strong acid. The carbonate species, bicarbonate, HCO_3^- and carbonate, CO_3^{-2}, are the dominant bases which contribute to alkalinity in most natural waters. Since alkalinity concentration is affected by changes in temperature, pH, and degassing, the alkalinity analysis should be performed in the field as soon as possible after collection. Uranium and other radionuclides form carbonate complexes; therefore, an accurate determination of carbonate concentration is necessary to quantify radionuclide mobility in ground water.

Summary of Method

Bicarbonate and carbonate concentrations are determined by electrometric titration. A strong acid of known concentration is added to a water sample while the pH of the sample is monitored. Using the acid volume needed to reach the end point, the concentration of bicarbonate and carbonate is calculated.

Apparatus

- pH meter that can be read to 0.01 units
- pH probe
- pH buffer solutions
  - pH 4.0
  - pH 7.0
- 50 milliliter (ml) buret
- Volumetric pipet (Class A)
- Magnetic stirrer and small teflon stir bar
- Buret stand and clamp
- 250 ml beaker or flask
- Standardized sulfuric or hydrochloric acid, approximately 0.02 normal

Calibration

Follow the manufacturer's instructions for calibration of the pH meter. Calibrate the meter daily before starting titrations.

Sample Measurement

- Fill buret with acid; open stopcock and drain out some acid to remove all air bubbles.
- Place a clean dry teflon stir bar into a clean dry 250 ml beaker.
- Pipet 50.0 ml of sample into the beaker.
- Place beaker onto center of stirring plate and gently place the clean, dry, pH probe into the beaker. Position buret over the beaker.
- Record the initial pH of the sample to the nearest 0.01 pH unit. Record to the nearest 0.01 ml the initial acid volume in the buret.
- Turn on stirring mechanism to a gentle or slow setting.

- Titrate acid into the sample. If the sample pH is not near an endpoint, i.e., pH greater than 9 or pH between 5 and 8, acid can be titrated in volumes sufficient enough to lower the pH by 0.2 to 0.5 units. However, when the pH of the sample is near an endpoint, i.e., between pH 8 and 9, or between pH 4 and 5, titrate dropwise, as very small additions of acid will cause large changes in sample pH.

- Turn off the stirring mechanism after allowing sufficient time for the sample to mix thoroughly.

- Record the acid volume and pH.

- Repeat titration of acid into the sample until the pH of the sample is in the range of 3.0 to 3.5.

Measure Practices

- Keep buret full between titrations. This will prevent a film from developing on the inside of the buret.

- Clean glassware and stir bar in a soap bath and rinse three times with deionized water. Rinse the pH probe with deionized water only.

References

1. American Public Health Association, American Water Works Association and Water Pollution Control Federation, 1985, "Standard Methods for the Examination of Water and Wastewater," (16th ed.); Washington, D.C., American Public Health Association, 1268 pp.

2. Skougstad, M.W., M.J. Fishman, L.C. Friedman, D.E. Erdmann, and S.S. Duncan, (eds.), 1979, "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments: Techniques of Water - Resources Investigations of the U.S. Geological Survey," Book 5, Chapter A1, 626 pp.

3. U.S. Environmental Protection Agency, 1983, "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, Cincinnati, Ohio, U.S. EPA, Environmental Monitoring and Support Laboratory, Office of Research Development, 460 pp.

6.26 Eh (redox Potential

Scope and Application

Eh (redox potential) is a measure of the aqueous electron concentration. Eh is controlled by reactions involving elements which are present in more than one oxidation state. The chemical behavior and mobility of many aqueous constituents are strongly influenced by the redox potential of surface or ground water. Physical, chemical, and biochemical processes in water will affect Eh.

Summary of Method

Eh is measured in the field by immersing a combination electrode into water. The electrode consists of a platinum redox and a silver/silver chloride reference electrode combined in one body. Eh measurement is based on potential difference between the constant voltage reference electrode and variable voltage of the platinum electrode. The voltage of the platinum electrode is dependent on the concentration of electrons in solution. For ground water, Eh must be measured using a flow box or similar device which prevents atmospheric contamination of the water sample.

Apparatus and Reagents

- pH/mV meter; Orion Research Model 407 A/F or equivalent
- Combination redox electrode; Orion Research Model 96-78 or equivalent
- Electrode filling solution; Orion Research Model 900001 or equivalent
- Deionized water for cleaning and decontamination
- Kimwipes or equivalent
- Zobell calibration solutions

  - Solution A:
    a. 4.22 grams potassium ferrocyanide
    b. 1.65 grams potassium ferricyanide
    c. Combine and dilute to 100 milliliters (ml) with deionized water

  - Solution B:
    a. 0.42 gram potassium ferrocyanide
    b. 1.65 grams potassium ferricyanide
    c. 2.09 grams potassium fluoride
    d. Combine and dilute to 100 ml with deionized water

Calibration

Turn selector dial on pH/mV meter to "battery check." The meter should read in the "battery OK" range, if not, charge or replace batteries before using. If battery is charged, turn selector dial to "mV," remove protective cap from redox electrode, and place electrode in Zobell A solution. The meter should read 200-300 mv. Record the reading, remove the electrode from the Zobell A solution, rinse the electrode with deionized water and dry with a Kimwipe. Place the electrode in Zobell B solution; the meter is given for each Zobell solution since the Eh of the solution is a function of temperature.

Sample Measurement

- Water temperature measurements must accompany the Eh measurement. Record the water temperature to the nearest tenth of a degree centigrade.
- Turn the selector dial on pH/mV meter to "mV" and remove the protective cap. from the redox electrode.
- Place electrode into the rubber stopper or gasket on the top of the flow box. Approximately half of the electrode should remain out of the flow box, since the level of the filling solution must be higher than the water level. Also, the small hole in the electrode body must breathe.d
- Turn on water flow, allow the box to fill, and make certain no air remains trapped at the top of the flow box.
- Reduce flow in the box to as low as possible while maintaining positive pressure in the flow box.
- Allow sufficient time for thermal equilibration and Eh stabilization before taking the reading. Gently move the probe, as if stirring the water, to speed up the stabilization of the Eh reading.
- Read the "mV" or millivolt scale of the pH/mV meter (blue scale on the Orion 407 A/F). After Eh has stabilized, record the value to the nearest millivolt. Make certain to include the sign of the reading, since positive or negative potentials are possible.
- Remove the redox electrode from the flow box and rinse with deionized water. Place the protective cap on the electrode tip, and turn the selector dial of the meter to "off."

- In measuring Eh of surface water, follow the same procedure. However, the flow box is not necessary. Approximately one half of the electrode must remain above the water surface; do not fully immerse the electrode.

Measurement Practice

- Remove salts or precipitates from the exterior of the redox electrode by rinsing with deionized water.

- Filling hole of the electrode can be sealed with scotch tape. Do not cover the smaller breathing hole; it must stay open for proper operation.

- If the platinum tip of the electrode becomes coated with any substance, cleaning is necessary. Cleaning with deionized water and Kimwipes. If this does not remove the coating or film, use a nonabrasive cleaner, deionized water and Kimwipes. If the coating or film is still present, contact the manufacturer. Do not use cleaners which contain petroleum products on the electrode.

- To change the filling solution, turn the electrode upside down briefly to moisten the O-ring, and push the sleeve into the cap to drain the electrode. Fill the electrode with new solution, slowly push the sleeve into the cap until some solution drains from the electrode. Add more filling solution, if necessary, and retape the filling hole.

- Store the redox electrode dry. Drain filling solution and rinse interior and exterior with deionized water before storing.

References

1. American Society for Testing and Materials, 1986, "Annual Book of ASTM Standards," Volume 11.01, Philadelphia, American Society for Testing and Materials, pp. 261-267.

2. Langmuir, D., 1971, "Eh-pH determination," In "Procedures in Sedimentary Petrology," ed. R. E. Carver; New York, John Wiley, pp 597-634.

3. Orion Research Incorporated, 1983, "Instruction Manual Platinum Redox Electrodes," Form IM9778/7801; Boston, Orion Research Incorporated, 13 pp.

6.3 SURFACE WATER SAMPLING

The procedures and practices described herein are applicable to the collection of water samples from streams, ponds, lakes, rivers, springs, and seeps. Basically, there are two different techniques for collecting surface water samples. They are:

- Grab sampling; and
- Composite sampling.

Whether the technique of sampling is grab or composite sampling, the following practices will be observed:

- The sample container will be clean and uncontaminated. Appropriately cleaned sample containers with or without preservatives are prepared by the laboratory prior to shipment to the field. Sample containers with preadded preservatives will be filled to the top without overflowing to prevent loss of the preservatives. Do not rinse sample containers.
- The point of sampling will be chosen with care so that a representative sample of the water to be tested is obtained. Choose the location of the sampling point with respect to the information desired and in conformity to local conditions.
- Avoid sampling when there is visible surface debris or when artificial turbulence is present in the stream.
- Do not use containers with preservatives to collect water samples directly from a body of water. Use a separate container (grab bottle) for sample collection and transfer the water sample into containers with preservatives.

- Stand downstream from the collection point to prevent any possible contamination during sample collection.

- Grasp the grab bottle securely at the base with one hand and plunge it mouth down into the water, avoiding surface scum. Position the bottle opening towards the current flow and away from the hand of the collector, the shore, the side of sampling platform, or boat. The sampling depth will be 15 centimeters (6 inches) below the water surface. If the water body is static, an artificial current can be created by moving the bottle horizontally in the direction it is pointed and away from the sampler. Tip the bottle slightly upwards to allow air to exit and the bottle to fill. After removal of the bottle from the stream, transfer the sample to the appropriate container with preservative.

- Collect a sufficient amount of sample to perform all the required analyses. Fill the sample bottles all the way up to the neck with the sample, without overflowing. Use the grab bottle for transfer of sample from the surface water to the sample bottles.

- VOA vials will be filled by placing an inverted glass bottle without preservative about 12 inches below the surface of the water and then turning the bottle over to allow it to fill below the water surface. The glass container is removed from the water, and the VOA vials are immediately filled from the container.

- When more than one grab bottle volume of sample is required to fill all the necessary sample containers, the portions will be composited in a holding container prior to filling the individual sample containers. This will provide for homogeneity of the collected sample. However, composited samples should be transferred to sample containers with appropriate preservatives as soon as sufficient sample is available to fill all the sample bottles.

- Perform field measurements (Section 6.2) or sample filtration on unpreserved sample immediately after sample collection.

- Make sure that the sample container lids are tight and secure.

- Store collected samples in the field in an ice chest filled with ice and maintained at approximately 4 degrees Celsius.

- Make sure samples are properly labeled and chain-of-custody records, sample collection logs, water quality field collection reports (Figure 6-1), and laboratory request for analysis forms are properly filled out.

6.4 Surface Soil Sampling

Surface material or soil will be collected using a hand trowel, scoop, or coring device in accordance with the Work Plan and the Surface Soil Sampling Task Procedures. The following procedures will be used for soil sampling:

- Samples will be collected using a trowel, scoop, or coring device in accordance with the sampling plan.
- Decontamination of all sampling equipment will be performed at a designated central staging area at the FMPC. Decontamination in the field will only be performed if circumstances prevent equipment decontamination at the staging area. The trowel, scoop, or coring device, and other sampling equipment such as pans, gloves, etc. will be decontaminated between each sample collection by cleaning with a nonphosphate detergent and a bottle brush, rinsing with tap water, methanol (acetone may be substituted if volatile organic compounds are not being determined), and two separate deionized water rinses.
- The final deionized water rinse following equipment decontamination will be sampled and analyzed at a frequency of one per every set of twenty locations or fraction thereof to check for potential cross-contamination between surface soil sample locations.
- Sufficient sample and rinsate volumes will be collected to perform all required analyses.
- Samples will be transferred directly from the hand trowel, scoop, or coring device to the appropriate sample containers.
- If soil samples are being collected for volatile organics analysis (VOA), the sample must be transferred to standard VOA vials. Vials should be completely filled to minimize any head space in the containers.

- Soil samples collected for other organic or inorganic analyses should be transferred to glass containers with screw cap closures.

- A field/trip blank for each sample set will accompany the samples back to the laboratory. The field/trip blank will consist of organic-free deionized water used in the field to decontaminate the sampling equipment.

- Collected samples will be stored in the field in an ice chest filled with blue ice and maintained at approximately 4 degrees Celsius.

- Samples will be properly labeled and chain-of-custody records, field collection reports, and laboratory request for analysis forms will be properly completed.

6.5 SEDIMENT SAMPLING

The following practices will be observed for sediment sampling:

- Prior to sampling sediments in a stream, the sampling device, usually a vertical pipe core sampler, will be rinsed with stream water downstream from the sampling location.

- When traverse sampling of rivers and large streams is necessary, a clamshell or dredge may be used for bottom sample collection.

- Sediment samples will be collected to a depth of approximately six inches below the sediment/water interface.

- The vertical pipe core sampler and/or dredge will be decontaminated between each sample collection by cleaning with river water and a brush. Should the equipment become heavily contaminated, it will be cleaned by the following procedures:

  - In the case of inorganic contaminants, the equipment will first be washed with a nonphosphate detergent and then rinsed with dilute (0.1 N) hydrochloric acid followed by two separate deionized water rinses.

  - In the case of organic contaminants, the equipment will be washed with a nonphosphate detergent and then rinsed with tap water, methanol, and two separate deionized water rinses.

  - As described above, the equipment shall be thoroughly rinsed with two deionized water rinses to remove traces of hydrochloric acid, detergent, and methanol (acetone may be substituted if volatile organic compounds are not being determined).

- Sampling equipment will not be placed directly on the ground or other contaminated surfaces prior to use, but will be placed on a clean plastic sheet.

- Samples of the final deionized water rinse will be collected and analyzed at a frequency of one per every set of twenty locations or fraction thereof to check for cross-contamination between sampling points.

Decontamination of the sampling equipment will be performed at a designated central staging area at the FMPC. If this is not possible due to extenuating circumstances, the sampling equipment may be decontaminated in the field, but only as a last resort.

- o A sufficient amount of samples will be collected to perform all required analyses.

- o Samples will be transferred directly to a stainless steel pan for thorough mixing of the sample prior to transfer to the proper sample container.

- o Collected samples will be stored in the field in an ice chest filled with commercially available icing material and maintained at approximately 4 degrees Celsius.

- o Samples will be properly labeled and chain-of-custody records, field collection reports, and laboratory request for analysis forms will be properly completed.

6.6 SUBSURFACE SOIL SAMPLING

6.6.1 COLLECTION OF SUBSURFACE SOIL SAMPLES

Subsurface material or soil will be collected using a split-spoon sampler or thin-wall tube in accordance with the Work Plan. Once collected, sampling equipment will be decontaminated and samples will be transferred to appropriate containers (see Section 6.4).

Subsurface materials or soils will be collected from soil borings. The borings will be advanced using cable tool drilling for monitoring wells and a soil auger drill rig for geotechnical and geochemical borings. Other suitable means may be used for the geotechnical borings depending on the characteristics of the

subsurface material. Subsurface material or soil will be collected at specified intervals in accordance with the Work Plan.

Because of its ability to penetrate greater depths and to maintain the physical integrity of the samples, a coring tool is commonly used to collect soil samples. The most common procedures use a thin-wall steel tube (Shelby tube) which is forced into the undisturbed soil at the bottom of a borehole. This is sometimes referred to as drive sampling. Shelby tubes are generally from one to three inches in diameter and 12 to 24 inches long. When the Shelby tube is retrieved, friction will usually retain the sample inside the tube, at least in most unsaturated materials.

Split-tube or split-spoon samples can also be used for sample collection when undisturbed samples are not required. Subsurface soil samples should be collected in accordance with the following procedures:

- The appropriate sampling device, consisting of a threaded coupling to fit a standard drill rod and a replaceable, thin-walled seamless steel tube or split-spoon sampler, is lowered down the borehole and driven into the undisturbed soil at the bottom of the boring.
- The sampling device is removed from the boring and either sealed and shipped to the laboratory as in the case of Shelby tubes, or the split tube, or split-spoon sampler is opened and the sample is removed and transferred to appropriate sample containers for shipment to the laboratory.
- Care should be taken to avoid collection of caving materials at the base of the borehole. Undisturbed soils should be collected.
- Subsurface soil samples collected for volatile organics analysis will be taken from the interior of the sample and will be transferred as soon as possible to standard VOA vials. Vials should be completely filled to minimize any headspace in the containers.

- Subsurface soil samples collected for other organic or inorganic analyses should be transferred to glass containers with screw cap lids.

- Samples to be tested for physical characteristics requiring undisturbed soil should not be transferred but should be left in the Shelby tube. The ends of the Shelby tube are capped, taped, and sealed with wax.

- A sample label, chain-of-custody form, field collection report, and a laboratory request for analysis form will be filled out in the field.

- The split-spoon sampler and other sampling equipment such as trowels, pans, gloves, etc. will be decontaminated between each collected sample by cleaning with tap water and a bottle brush, rinsing with deionized water, methanol, and deionized water again. Should the sampling equipment become heavily contaminated, it will be cleaned according to the following procedures:

  - In the case of inorganic contaminants, the equipment will first be washed with a nonphosphate detergent and then with dilute (0.1 N) hydrochloric acid, followed by two separate deionized water rinses.

  - In the case of organic contaminants, the equipment will first be washed with a nonphosphate detergent and then rinsed with tap water, methanol, and two separate deionized water rinses.

  - As described above, the equipment will be thoroughly rinsed with two deionized water rinses to remove traces of hydrochloric acid, detergent, and methanol (acetone may be substituted for methanol if volatile organic compounds are not being determined).

  - Sampling equipment will not be placed directly on the ground or other contaminated surfaces prior to insertion into the boring, but will be placed on a clean plastic sheet adjacent to or around the boring.

  - Samples of the final deionized water rinse will be collected in sample bottles containing the appropriate preservatives and analyzed at a frequency of one per set of twenty locations or fraction thereof to check for cross-contamination between borings.

Decontamination of the sampling equipment will be performed at a designated central area at the FMPC. If this is not possible due to extenuating circumstances, the sampling equipment may be decontaminated in the field, but only as a last resort.

6.6.2 FIELD SCREENING OF SUBSURFACE SOIL SAMPLES FOR RADIOACTIVE CONTAMINATION

The purpose of this section is to provide uniform measurement techniques for screening subsurface soil samples for radioactive contamination and to determine which sample will be chosen for full radiological analysis.

The following procedure describes the technique for screening subsurface soils for intermediate and high-energy gamma ray emitters. The screening level is chosen for instrument gross count rates which exceed the background count rate by three standard deviations when the sample is counted in a low-background area.

The screening will be preformed with an ESP-1, Model 141, SPA-3, Sodium Iodine Scintillation Detector survey instrument.

The operation and calibration of this instrument are found in Section 5.1.1 and Section 5.1.4. Table 8-3 also provides calibration references and preventive maintenance programs.

The following assay procedure is performed with the survey instrument:

- Perform a background check with the survey instrument. Compare the count rate with the accepted background count rate for the area. If a larger than the acceptable background count rate is present, move to an area with a lower background rate. (Note: A shielded enclosure will be used by the sample coordinator at the sample coordinator's trailer.) Perform five separate 30-second background counts for the instrument. Calculate the average; record data.

- Position the sample (in sealed container) in contact with the survey instrument and perform a thirty-second count per sample.
- Record the count rate.
- Submit results to the field task leader (geologist).
- Subsurface soil samples will be selected for radiological analysis based on the results of screening. The sample selected for laboratory analysis will be that which exhibits the highest relative reading for the given location and horizon (at least one sample per horizon, i.e., tills, upper sand and gravel, and lower sand and gravel per location will undergo radiological analysis).

6.7 RADON SAMPLING

6.7.1 RADON SAMPLING IN SOIL (FLUX MEASUREMENTS)

This procedure provides instruction for the determination of radon flux from ground surfaces using charcoal canisters as described in W. A. Goldsmith, et al., Radon Flux Measurements, RASCA Procedures Manual, Procedure No. 13.11, Revision 8, ORNL-6000, September 1982.

Radon that emanates across a soil/air interface will vary as ambient weather conditions change. Flux is a function of Ra-226 in subsurface soil, but is affected by soil porosity, soil moisture, and ambient pressure and temperature. While determinations of radon flux are not exact, a series of canisters placed at various points in an area where ambient conditions are the same, should provide information about the relative magnitude of the radon source.

Sampling will be performed at the frequency and locations specified in the Work Plan according to the following procedures:

- Charcoal canisters to be used for sample collection will be preconditioned for 24 hours at 100 degrees Celsius.

- After the canisters have cooled, they are placed individually in a plastic bag and sealed. Also prior to use in the field, a background count must be made and recorded.

- Each charcoal canister will be placed on a smooth ground surface after grass has been removed. Remove one canister from its plastic bag and twist the canister's thin wall cylinder about 0.5 cm into the ground. Soil should be packed around the canister as a seal.

- Record the location and time of emplacement on the sample collection log sheet.

- Continue the above procedure until charcoal canisters have been installed at all the sampling locations specified in the Work Plan.

- The appropriate exposure time for the canisters is a function of the local radon flux. To determine this approximate period, it will be necessary to deploy several canisters at selected sites in order to establish an exposure time. This will be done by placing canisters in an area believed to represent near background conditions and one which is believed to represent a near maximum radon flux. At each location, four canisters will be placed on the ground and sealed as described. One canister will be removed from each location following exposures of 2, 6, 24, and 32 hours. As each canister is removed, it will be sealed in a clean plastic bag and transported to the laboratory trailer. Following a period of at least four hours, the canister will be counted using gamma-ray spectrometry techniques. An evaluation of data from this exercise will be used to establish optimum exposure periods.

- Following exposure for the established optimum period, canisters will be retrieved. When the canister is removed from the ground, a clean rag or paper wipe will be used to remove loose soil, mud, etc., from the canister rim in contact with the ground.

- Place each canister in an individual plastic bag, complete all information blanks on a sample container label and the sample collection log sheet and attach the label to the plastic bag. Record the sample identification on the chain-of-custody record.

- Transport the samples to the laboratory for analysis. 165
  Note: If a field laboratory is used to perform the radon analysis, the samples must be stored at least

four hours prior to analysis to allow equilibration of the radon daughters.

6.7.2 LONG-TERM RADON SAMPLING IN AIR

This procedure provides instructions for the collection of samples for the determination of long-term radon-222 (Rn-222) concentrations in air under ambient outdoor conditions as described in H. W. Alter and R. L. Fleisher, 1981, "Passive Integrating Radon Monitor for Environmental Monitoring," Health Physics, Volume 40, p. 693 and Terradex Corporation, "Instruction for Track-Etch[R] Type F Radon Detectors," Walnut Creek, California.

As radon and radon progeny decay, the resultant alpha particles produce radiation-damage tracks in thin plastic films exposed to the air. The film detector is mounted inside the bottom of a plastic shield. A special filter installed over the mouth of the cup filters out radon daughters, as well as dust and dirt, so that only radon gas enters the cup. The detectors are chemically treated after exposure to make the tracks visible. The number of tracks in a specified area is directly proportional to the integrated alpha exposure from the decay of the radon to which the detector was exposed. The detection range is from 0.2 to 20,000 pCi/l-months for outdoor measurements.

Sampling will be performed at the frequency and locations specified in the work plan according to the following procedures:

- Prior to the planned installation of the film detectors, submit a purchase order for the purchase and readout of the detectors. Specify a delivery date two weeks prior to installation.
- Specify the quantity of detectors desired. This should include enough detectors to install a minimum of two detectors per location and at least ten detectors for known exposures and background determinations.
- Request that the detectors in this order be produced from the same batch of film. The detectors must all be Type F configuration.

- Specify maximum readout area for outdoor monitoring. For Terradex film detectors, this is 19.3 mm^2.

- Specify that results should be reported in pCi/l and in either tracks/mm^2 or total tracks.

- Inspect the shipment of film detector cups when they arrive from Terradex.

- Check the aluminum bags for holes or tears.

- Check the contents of each bag by feeling along the entire length of the aluminum bag for crushed cups. DO NOT open bag.

  IMPORTANT: DO NOT open the sealed aluminum bags until they are to be installed at the monitoring location.

- Attach the protective canister (sheild) to a post or other suitable location at a height of two to three meters. The shield should be firmly mounted with no obstructions to the free flow of air past the detector. In general, canisters should be mounted on the northern sides of the mounting post. Building walls, highway structures, or large signs should be avoided.

- Alternatively, the detector with the protective canister surrounding the plastic cup may be placed inside a larger stand-alone shelter. Canisters should be installed so that the entire detector is at least six inches from any obstruction. Install the canister with the open mouth facing down. The stand-alone shelter should be open and allow the free flow of air through it. The detectors must be mounted firmly to avoid movement within the shelter.

- At the monitoring location, open one aluminum bag and remove sufficient film detector cups to enable installation of the appropriate number of replicates, making sure to close the bag after removing the set.

- Visually inspect the film detector to check to see that it is securely fastened to the bottom of the cup. If the film is not secured to the bottom of the cup, secure it. Extreme care should be taken not to touch the film.

- If the film is touched, use a different cup and detector. On the field data sheet, write down the serial

number of the breached cup and indicate what happened in the comments section.

- Using one filter, cover the mouth of the cup. The shiny side of the filter should be facing out.

- Using one red covering and holding the filter taut, firmly push the cover ring over the two sets of ribs of the cup.

- Check that the cover is fully seated around the cup rim. Inspect the fully assembled detector to check to see that the filter covers the entire mouth of the cup, there are no large wrinkles in the filter and that the filter has not been torn or punctured by assembly.

- Install the film detector cup at the monitoring location inside the protective canister.

- Enter the serial number of the film detector cup, the date the bag was opened, the date installed, site number, and location on the field data sheet.

- Schedule the installation of the film detector cups such that when a bag is opened, all cups can be installed immediately. Do not use film detector cups that have been stored in an opened bag overnight. The aluminum bags should be saved for use when the film detector cups are retrieved.

- Remove the film detector cup from the canister and wipe off any dust or dirt from the outside of the cup. Remove the red cover ring and filter from the mouth of the cup.

- Inspect the filter by holding it up to the light. Note any damage to the filter in the comments section of the data sheet (e.g., pinholes, scratches, tears, etc.)

- Visually inspect the film on the bottom of the cup. Note any discoloration or damage to the film in the comments section of the data sheet.

- Enter the date removed next to the correct serial number on the field data sheet and place the film detector cup in the aluminum bag.

- Stack the exposed cups. Close aluminized bag and proceed to install new cups. After an aluminized bag is filled (10 cups, maximum), insert the empty cup provided into the last film detector cup with a yellow plastic separator sheet. If an end cup is not provided, place the yellow plastic separator sheet over

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the mouth of the last film detector cup and secure it with one of the red coverings.

- Fold the open end of the aluminum bag and seal using tape. Remember, do not exceed 10 cups per bag.
- When all exposed detectors have been retrieved, proofread the field data sheets to check to see that all applicable information is recorded in the proper column and that all comments are legible. (Detector serial number, date opened, date installed, date removed, site number, location.)
- Check that all aluminum bags containing exposed detectors are sealed and have no pinholes or tears.
- Transcribe all data from the field data sheets to the vendor's data sheets.
- Complete the vendor's preshipping checklist. Specify the readout sensitivity with any special handling instructions and include with the data sheets and detectors.
- Ship detectors to the vendor for readout.

6.7.3 SHORT-TERM RADON SAMPLING IN AIR

The purpose of this procedure is to describe the methods used to collect samples of ambient air and soil gas.

Two basic types of sampling are used in radon measurements, gas bag samples, and soil gas samples (radon flux). Gas bags provide integrated samples of ambient radon in air while soil gas accumulators provide samples of radon in emanated soil gas. Instantaneous air samples may be collected using an evacuated SC-6 scintillation cell.

The sampling procedures described in this method are referenced to DOE Order 5480.1, 10 CFR 20, and U.S. EPA standards 40 CFR 192. Sampling will be performed at the frequency and locations specified in the Work Plan according to the following procedures:

- Gas Sampling (24, 48, or 72 hours Integrated Sample) of Ambient Air

- Place radon gas bag at sampling location and turn on pulse pump. Record date and time of start-up on sample collection log and on the gas bag. To fill 44-liter bag, use the following pump cycles:

Sample Time (Hours)	Pump Interval (Seconds)
24	5
48	12
72	15

  Secure and protect sampling equipment from inclement weather conditions and vandalism.

- After completion of sampling period, turn off pulse pump and record date and ending time on sample collection log and on the gas bag.

- Take the gas bag to the counting room for transfer of the air sample to an SC-6 scintillation cell.

- Using a small piece of tygon tubing, attach an evacuated SC-6 cell to the valve on the gas bag in series with a 0.45-micron filter and transfer the air into the cell by opening the valves. Transfer at least three cell volumes to provide a representative sample. Record the date and time of the sample transfer on the label of the SC-6 cell and on the sample collection log.

- Place the SC-6 cell in the sample holding area for counting in a SAC-R5 alpha counter. Results of the count will be used to determine the radon gas concentration expressed in pCi/l in the integrated sample. See the calibration and counting procedure instructions for the use of the SAC-R5 and SC-6 scintillation cell radon counting system.

• Soil Gas Sampling (Radon Flux Measurements)

- Position the special 55-gallon drum section over the sampling point and, with a downward twisting motion, countersink the barrel one to two inches into the soil, or otherwise seal the drum to the ground.

- Attach the 50 cc syringe to the valve assembly, open the valve, and rapidly pump the syringe 10 times.

- Close the valve, remove the syringe, and attach an evacuated SC-6 cell by means of a piece of tygon tubing with in-line 0.45-micron filter.

- Open the valve on the drum, then open the valve on the SC-6 and fill the SC-6 cell. Close the valve on

the SC-6 cell and close the valve on the barrel. Record the time and the date for the sample. Also record the cross-sectional area of the drum as the sample area.

A 55-gallon drum with a diameter of 22.5 inches has an area equivalent to 0.256 m^2.

- After 45 minutes, repeat steps 2 through 4 using a second evacuated SC-6 cell.

  NOTE: The first sample is considered to be a background sample. The second sample represents the radon buildup over the 45-minute sampling time.

- Submit the samples to the counting lab for counting. The radon concentration will be determined by counting in a SAC-R5 alpha counter. Results will be used to detemine the radon flux ratio expressed in pCi/m^2-sec.

• Instantaneous Air Sampling

- Instantaneous or "spot" air samples are collected using an evacuated SC-6 scintillation cell.

- At the desired sampling location, collect a sample by opening the valve on a SC-6 cell which has an in-line 0.45-micron filter connected to the valve by means of a small piece of tygon tubing. Record the location, date, and time of collection on the SC-6 cell label.

- Transfer the filled SC-6 cell to the counting lab for counting in a SAC-R5 alpha counter. Results in pCi/l will be used to determine the concentration of radon in the sample.

6.8 FIELD STORAGE AND SHIPMENT OF SAMPLES

Samples will be stored in a cool place away from direct sunlight. As soon as samples are collected, filtered (if necessary), and preserved, they should be stored in an ice chest packed with plenty of blue ice. Field personnel should make sure that sample container lids are tight and secure before storing them in an ice chest. Samples must be promptly shipped to the appropriate laboratory so that sample holding times are not exceeded.

Samples collected during site characterization operations will have to be transported to IT laboratories in Tennessee and Pennsylvania for analysis. Transportation of samples must be accomplished not only in a manner designed to protect the integrity of the sample, but also to prevent any detrimental effects from the potentially hazardous nature of the samples. Regulations for packaging, marking, labeling, and shipping of hazardous materials, substances and wastes are promulgated by the U.S. Department of Transportation (DOT) and described in the 49 CFR 171 through 177 (in particular, 172.402h, Packages Containing Samples). In general, these regulations were not intended to cover the shipment of samples collected at hazardous waste sites. However, the U.S. EPA has deemed it prudent to package, mark, label, and ship samples observing these DOT procedures. The information contained in this section is for general guidance and, although factual, should not be misconstrued as identical to DOT regulations for transportation of hazardous materials.

Samples collected at the FMPC site shall be classified as either environmental or hazardous substance (or waste) samples. In general, environmental samples are collected off site, such as water samples from the various drainages, and are not expected to be grossly contaminated with high levels of toxic substances. Samples of soil or water from certain locations in the waste storage area will be considered hazardous substances. A distinction between the two types of samples, environmental and hazardous, must be made for two major reasons: (1) to determine the appropriate procedures for the transportation of the samples; and (2) to protect the health and safety of the laboratory personnel receiving the samples. Special precautions, procedures, and secondary containment areas within laboratories will be used when samples other than environmental samples are received. If there is any doubt as to the classification of a sample, it will be considered a hazardous sample and shipped accordingly.

6.8.1 ENVIRONMENTAL SAMPLES

Samples judged to be environmental are not considered hazardous material; however, environmental samples will be packaged and shipped according to the following procedures.

Environmental samples will be packaged following the procedures for samples classified as "flammable liquids" or "flammable solids," excluding packages being placed inside metal cans as required for "flammable liquids." Sample containers properly identified and with a sealed lid will be placed in sealed polyethylene bags, then packed in fiberboard containers or metal picnic cooler-type containers. Sufficient incombustible, absorbent cushioning material will be used to minimize the possibility of sample container breakage.

Sample containers will have a completed sample identification tag. The outside container will be marked "Environmental Sample." No DOT marking and labeling are required.

No DOT shipping papers are required, and there are no DOT restrictions on mode of transporation.

6.8.2 HAZARDOUS SUBSTANCE SAMPLES

FMPC samples not determined to be environmental samples or samples known or expected to contain hazardous material must be considered hazardous substance samples and transported according to the following requirements. If the material in the sample is known or can be identified, then it should be packaged, marked, labeled, and shipped according to the specific instructions for that material if listed in the DOT Hazardous Materials Table, 49 CFR 172.101. For those samples of hazardous substances where the contents are unknown, the selection of the appropriate transportation category will be based upon the DOT Hazardous Material Classification, a prioritized system of transportation categories. This system is presented in Table 6-1.

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TABLE 6-1

DOT HAZARDOUS MATERIALS CLASSIFICATION

1. Radioactive Material
2. Poison "A"
3. Flammable Gas
4. Non-Flammable Gas
5. Flammable Liquid
6. Oxidizer 13. ORM-B
7. Flammable Solid
8. Corrosive Materials (liquid)
9. Poison B
10. Corrosive Material (solid)
11. Irritating Materials
12. Combustible liquid (in containers having capacity exceeding 110 gallons)
13. ORM-B
14. ORM-A
15. Combustible liquids (in containers having capacity of 100 gallons or less)
16. ORM-E

For purposes of packaging safety, liquid and solid samples will be classified no lower than "flammable" as per Table 6-1.

The selection of the correct category for an unknown sample is through the process of elimination utilizing the DOT classification system. Unless known or demonstrated otherwise (through the use of radiation survey instruments), the sample will be considered radioactive and appropriate shipping regulations for radioactive material will be followed. If radioactive material is eliminated, the sample will be considered to contain Poison "A" materials, the next material on the list. Poison "A" is defined by DOT as extremely dangerous poisonous gases or liquids of such a nature that a very small amount of gas, or vapor of the liquid, mixed with air, is dangerous to life. The class "A" poisons listed in 49 CFR 172.101 and their physical state at normal temperatures are presented in Table 6-2.

If poison "A" is eliminated as a shipment category, the next two classifications are "flammable" and "non-flammable" gases. Since few gas samples will be collected, flammable liquids would be the next category. The elimination of the classification of radioactive, Poison "A," flammable gas, and non-flammable gas which require more stringent shipping procedures, permits the sample to be classified as a flammable liquid (or solid) and shipped accordingly. The shipping requirements for flammable liquids would also suffice for shipping any other samples classified below flammable liquids on the DOT classification schedule. For samples containing unknown materials, other categories listed below flammable liquids/solids on the table will not be considered.

6.8.3 SAMPLES CLASSIFIED AS RADIOACTIVE

Federal regulations governing the transport of radioactive sample material are extremely complex and reflect overlapping jurisdiction of many federal agencies. The important features of

TABLE 6-2

CLASS "A" POISONS AND THEIR PHYSICAL STATE AT ROOM TEMPERATURES

Compound	Physical State
arsine	gas
bromoacetone	liquid
chloropicrin & methyl chloride mixture	gas
chloropicrin & non-flammable, non-liquified compressed gas mixture	gas
cyanogen chloride containing less than 0.9% water	gas
cyanogen gas	gas
gas identification set	gas
gelatin dynamite (H.E. Germaine)	gas
grenade (with poison "A" gas charge)	gas
hexaethyl tetraphosphate & compressed gas mixture	gas
hydrocyanic acid (prussic solution)	liquid
hydrocyanic acid, liquified	gas
insecticide liquified gas, containing poison "A" or poison "B" materials	gas
methyldichloroarsine	liquid
nitric oxide	gas
nitrogen dioxide	liquid
nitrogen peroxide	liquid
nitrogen tetroxide	liquid
organic phosphate	gas
parathion & compressed gas mixture	gas
phosgene (diphosgene)	gas
phosphine	gas
poisonous liquid or gas n.o.s.	liquid or gas
tetraethyl dithiopyrophosphate	gas
tetraethyl pyrophosphate	gas

the DOT regulations (49 CFR 173, dated 1 November 1983) are summarized with their application to samples contaminated with uranium and associated daughter radionuclides.

In general, most samples collected at the FMPC will not be classified as radioactive for transport purposes and will, therefore, require no special attention to be considered in compliance. On the other hand, certain samples may fall into categories for which special packaging and shipping restrictions are mandated. Guidelines for determining the category to which a particular sample shipment belongs and for selecting a suitable mode of shipment and appropriate packaging are described below.

The following definitions are summarized from those presented in 49 CFR 173.403:

- Natural Thorium Thorium characterized by the naturally occurring distribution of thorium isotopes (essentially 100 weight-percent thorium-232);

- Natural Uranium Uranium characterized by the naturally occurring distribution of uranium isotopes (approximately 0.711 weight-percent uranium-235 and the remainder essentially uranium-238);

- Specific Activity In reference to a radionuclide, the activity of the nuclide per unit mass of the nuclide. The specific activity of a material in which a radionuclide is uniformly distributed is expressed as the activity per unit mass of the material;

- Radioactive Material Any material having a specific activity greater than 0.002 microcurie per gram;

- Special Form Radioactive Material Radioactive material that is either a single, solid piece or is obtained in a sealed capsule;

- Normal Form Radioactive Material Radioactive material that is not "Special Form Radioactive Material"; and

- Transport Index The dimensionless number (rounded up to the first decimal place) marked on the label of a package to designate the degree of control to be exercised by the carrier during transport. Generally, the transport index is the number expressing the maximum

radiation level, in millirem per hour, occurring at distance of 1 meter (3.3 feet) from the external surface of the package. Other considerations may apply to certain fissile materials.

6.8.3.1 Quantities of Radioactivity in Packages

The regulations impose limits on the total activity (i.e., specific activity times the weight of the package) contained within a package of radioactive material. With respect to Type A packages, the limits are expressed as two quantities, A1 and A2, which refer to the maximum permissible activity for radionuclides in Special Form and Normal Form radioactive materials, respectively. Since the samples from FMPC fall into the latter category, the A2 value sets the activity limits for packages of such samples. In those cases where contaminated material shipments are designated Low Specific Activity or Limited Quantity, some fraction of the A2 value will normally apply.

Table 6-3 lists the A1 and A2 values cited in 49 CFR 173.435 for radionuclides of the uranium decay series. Values for radionuclides not listed in the regulations (e.g., lead-214, bismuth-214, polonium-214) have been assigned in accordance with the specifications set forth in 49 CFR 173.433(a)(2).

Determination of permissible quantities for samples that are contaminated with FMPC waste is more complicated than simple reference to the A2 values, since such material contains a mixture of radionuclides. For such mixtures, two rules apply. A mixture of radionuclides from a single decay chain is considered to be a single radionuclide if the following criteria are met: (1) the parent and daughters are present in their naturally occurring proportions (for example, in secular equilibrium); (2) no daughter has a half-life longer than 10 days; and (3) no daughter has a half-life longer than the parent radionuclide. For such a mixture, the A2 quantity to be applied is that of the parent radionuclide (49 CFR 173.433(b)(2)). For those mixtures

TABLE 6-3

A_1 AND A_2 QUANTITIES FOR RADIONUCLIDES OF THE URANIUM DECAY SERIES

Isotope	Atomic Number	Decay	Half-Life[a]	Quantity in Curies A1	A2
U-238	92	Alpha	4.5×10^9 y	Unlimited	Unlimited
Th-234	90	Beta	24 d	10	10
U-234	92	Alpha	2.5×10^5 y	100	0.1
Th-230	90	Alpha	7.5×10^4 y	3	0.003
Ra-226	88	Alpha	1622 y	10	0.05
Rn-222	86	Alpha	3.8 d	10	2.0
Pb-210	82	Beta	22 y	100	0.2
Bi-210	83	Beta	5 d	100	4.0
Po-210	84	Alpha	138 d	200	0.2

[a]Abbreviations: y = years; d = days; m = minutes; s = seconds

in which the identity and activity of radionuclides is known, the permissible quantity for each radionuclide must be such that the following equation is true:

$$F_1 + F_2 + \ldots + F_k \ldots F_n \leq 1$$

where F_k is the ratio of the total activity of the kth radionuclide to its A1 or A2 quantity [49 CFR 173.433(b)(3)].

6.8.3.2 Low Specific Activity Materials

Low Specific Activity (LSA) materials include uranium and thorium ores, physical and chemical concentrates of these ores (e.g., yellow cake), unradiated natural or depleted uranium or thorium, nonradioactive material externally contaminated with radioactivity that is not readily dispersible, and material in which the radioactivity is essentially uniformly distributed and does not exceed certain prescribed concentration limits. These limits for radionuclides of the uranium decay series, beginning with thorium-230, are listed in Table 6-4. In general, these concentrations will not be exceeded in FMPC samples.

Details for shipping LSA materials are described in 49 CFR 173.425. The chief advantage of shipping under the LSA category arises when the shipment is consigned Exclusive Use; that is, under the supervision or direction of a single consignor from point of origin to final destination [49 CFR 173.403(i)]. When packaged shipments of LSA materials are consigned as Exclusive Use, the shipment is exempt from specification packaging, labeling and marking. Requirements that must be met include the following [49 CFR 173.425(b)]:

- The shipment must be consigned Exclusive Use, unloaded from the conveyance in which it was originally loaded, and accompanied by written instructions for the maintenance of Exclusive Use shipping controls. LSA materials cannot be shipped by air.

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TABLE 6-4

LOW SPECIFIC ACTIVITY (LSA) CONCENTRATION LIMITS FOR RADIONUCLIDES OF THE URANIUM DECAY SERIES
[FROM 49 CFR 173.403(N)(4)]

Nuclide	A2 Value (curies)	Estimated Average Concentration	
		Microcuries/Gram	Curies/Kilogram
Th-230	0.003	0.1	0.0001
Ra-226	0.05	0.1	0.0001
Rn-222	2.0	300	0.3
Pb-210	0.2	5.0	0.005
Bi-210	4.0	300	0.3
Po-210	0.2	5.0	0.005

- The material must be packaged in strong, tight packages that will not leak under normal conditions of transport, and each package must be marked "Radioactive - LSA."

- The package does not exceed the limits for removable radioactive contamination and radiation level.

- The transport vehicle must be appropriately placarded, unless the shipment consists solely of unconcentrated uranium and thorium ores.

6.8.3.3 Limited Quantities of Radioactive Material

Limited quantity shipments of radioactive material must meet the requirements specified in 49 CFR 173.421 and 173.423. If the activity per package does not exceed 10^{-3} A2 quantity of the radionuclide, then it is exempt from specification packaging and from the associated shipping-paper, marking, and labeling requirements. Instead, the package need only comply with the following requirements:

- The material must be packaged in strong, tight packages that will not leak under normal conditions of transport. The outside of the inner packaging, or the outside of the packaging itself when there is no inner packaging, must be marked "Radioactive."

- The package does not exceed the limits for removable radioactive contamination and radiation level.

- The package must be certified as being acceptable for transport by having a notice enclosed in or attached to the package, stating the name of the consignor and including this statement: "This package conforms to conditions and limitations specified in 49 CFR 173.421 for excepted radioactive material, limited quantity, n.o.s., UN2910."

One advantage of limited quantity shipments is that there are no restrictions on air transport. [Guidelines specified in the International Civil Aviation Organization (ICAO) regulations should be consulted for air shipments of radioactive materials.] The major disadvantage is the low concentrations and quantity weights permissible for mixtures of different radionuclides.

6.8.4 RADIATION AND CONTAMINATION CONTROL

Measurements of radiation level (dose rate) and of nonfixed (removable) radioactive contamination are conducted on radioactive-material shipments to control exposure to radioactivity. Inspectors of such shipments can readily determine whether the packages comply with the regulations summarized below.

6.8.4.1 Radiation Limits

Radiation level is the radiation dose-equivalent rate expressed in millirem per hour (mrem/h) [49 CFR 173.403(v)]. Permissible radiation levels for various shipping categories are described below:

- Limited Quantity Packages The radiation level at any point on the external surface of the package may not exceed 0.5 mrem/h [49 CFR 173.421(b)].

- LSA Packages The radiation level of the LSA packages transported as Exclusive Use shipments shall not at any time during transport exceed any of the following limits [49 CFR 173.441(b)]:

  - 200 mrem/h on the accessible external surface of the package; or 1,000 mrem/h if the following criteria are met: the shipment is made in a closed transport vehicle, the package is secured so that its position remains fixed during transport, and no unloading/loading operations occur between the beginning and end of transport;

  - 10 mrem/h at any point on the outer surface of the transport vehicle;

  - 10 mrem/h at any point 2 meters (6.6 feet) from the outer surfaces of the transport vehicle; and

  - 2 mrem/h at any normally occupied position in the transport vehicle. This provision does not apply to private motor carriers when personnel operate under a radiation protection program.

- Other Packages The radiation level must not exceed 200 mrem/h at any point on the external surface of the package, and the transport index must not exceed 10.

6.8.4.2 Contamination Control

Nonfixed (removable) radioactive contamination is defined as radioactive contamination that is easily removed from the surface by wiping with an absorbent material [49 CFR 173.403(r)]. The maximum permissible limits for removable radioactive contamination, as set forth in 49 CFR 173.443(a), are summarized below; these limits apply to any area of 300 square centimeters.

- 10^{-5} micro Ci/cm^2 or 22 disintegrations per minute (dpm)/cm^2, for beta/gamma-emitting radionuclides, all radionuclides with half-lives less than 10 days, natural uranium, natural thorium, uranium-235, uranium-238, thorium-232, and thorium-228 and thorium-230 when contained in ore and physical concentrates; and
- 10^{-6} micro Ci/cm^2 or 2.2 dpm/cm^2, for all other alpha-emitting radionuclides.

Exclusive use consignments of radioactive material may not exceed these limits at the beginning of transport, and may not exceed 10 times the limits at any time during transport (49 CFR 173.443).

6.8.5 SAMPLES CLASSIFIED AS POISON "A"

6.8.5.1 Packaging

Collect samples in a polyethylene or glass container which is of an outer diameter narrower than the valve hole in a DOT Spec. #3A1800 or #3A1800 metal cylinder. Fill sample container allowing sufficient ullage (approximately 10 percent by volume) so it will not be liquid-full at 130°F. Seal sample container.

Attach a properly completed identification tag to sample container.

With a string or flexible wire attached to the neck of the sample container, lower it into a metal cylinder which has been partially filled with incombustible, absorbent loose packaging material (vermiculite or diatomaceous earth). Allow sufficient absorbing material between the bottom and sides of the container and the

metal cylinder to prevent breakage and absorb leakage. After the cylinder is filled with cushioning material, drop the ends of the string or wire into the cylinder valve hole. Only one sample may be placed in a metal cylinder.

Replace valve, torque to 250 feet/pound (for one-inch opening) and replace valve protector on metal cylinder, using teflon tape.

One or more cylinders may be placed in a DOT-approved outside container.

6.8.5.2 Marking and Labeling

Abbreviations will be used only when specified. The following information will be placed on the side of the cylinder, or on a tag wired to the cylinder valve protector, either hand printed or in label form: "Poisonous Liquid, n.o.s." or "Poisonous Gas, n.o.s. NA9035," laboratory name and address. Place the following DOT label on the cylinder: "Poisonous Gas" (even if the sample is liquid).

If the metal cylinders are placed in an outside container, both the container and cylinders inside will have the same markings and labels as above. In addition, "Laboratory Sample" and "Inside Packages Comply With the Prescribed Specifications" will be marked on the top and/or front side of the outside container. "THIS SIDE UP" marking will be placed on the topside of the container, with upward pointing arrows on all four sides.

6.8.5.3 Shipping Papers

The bill of lading will be completed and the certification statement signed (if the carrier does not provide a certification statement, a standard industry form will be used) with the following information in the order listed. One form may be used for more than one exterior container. Abbreviations will be used only as specified: "Poisonous Liquid, n.o.s. NA9035, "Limited

Quantity" or "Ltd. Qty.", net weight or net volume. The net weight or net volume must be placed just before or just after the "Poisonous Liquid, n.o.s." marking.

A chain-of-custody record will also be properly executed and included in the container or with the cylinder, if legal use of samples is required or anticipated.

Unless samples are driven to the laboratory, an employee will accompany shipping container to the transport carrier and, if required, open the outside container for freight inspection. Alternately, samples may be shipped through the WMCO Transportation Department.

Using the word "poisonous" does not convey the certain knowledge that a sample is in fact poisonous, or to what degree it may be poisonous; however, it is intended to describe the class of packaging in order to comply with DOT regulations.

6.8.5.4 Transportation

Poison "A" categorized packages may not be transported by Federal Express Corporation (air cargo) or other common carrier aircraft, or by rental, non-government aircraft. Samples may be shipped by ground transport or government-owned aircraft.

6.8.6 SAMPLES CLASSIFIED AS FLAMMABLE LIQUIDS OR FLAMMABLE SOLIDS PROCEDURES

6.8.6.1 Packaging

Samples will be collected in a glass container with non-metallic, Teflon-lined screw cap allowing sufficient ullage (approximately 10 percent by volume) so that the container is not liquid full at 130°F. If an air space in the innermost container cannot be tolerated in order to maintain sample integrity, the sample will be placed within a second container to provide the required ullage.

A properly completed sample identification tag will be attached to the sample container.

The sample container closure will be sealed and the sample placed in a two-millimeter-thick (or thicker) polyethylene bag, one sample per bag. The sample identification tags will be positioned to enable it to be read through the bag.

The sealed bag will be placed inside an appropriate size metal can with enough incombustible, absorbent, cushioning matter (e.g., vermiculite, diatomaceous earth) to prevent breakage and absorb liquid; one bag per can. The can will be pressure closed using clips, tape, or other positive means to hold the lid securely, tightly, and effectively.

One or more metal cans will be placed (or single one-gallon bottle) surrounded with incombustible, absorbing packaging material for stability during transport into a strong outside container, such as a cooler or inside an approved fiberboard box.

6.8.6.2 Marking and Labeling

Abbreviations will be used only when specified. The following information will be placed on the metal can (or 1-gallon bottle), either hand printed or in label form: laboratory name and address, "Flammable Liquid n.o.s. UN1993" or "Flammable Solid n.o.s. UN1325." Not otherwise specified (n.o.s.) is not used if the flammable liquid or solid is identified. In that case, the name of the specific material is used and listed before the category (e.g., "Flammable Liquid").

The following DOT labels will be placed on the outside of the can (or bottle): "Flammable Liquid" or "Flammable Solid." The "Dangerous When Wet" label will be used with the "Flammable Solid" label if the material meets the definition of a water reactive material. The "Cargo Aircraft Only" label will be used if the

net quantity of the sample in each package is greater than one quart (for "Flammable Liquid," n.o.s.) or 25 pounds (for "Flammable Solid" n.o.s.). [49 CFR 172.402, 172.101].

If the cans are placed in an exterior container, both that container and inside can (or bottle) will have the same markings and labels as above.

The words, "This End Up" or "This Side Up," will be clearly printed on the top of the outer package. Upward pointing arrows will be placed on the sides of the package. The words, "Laboratory Samples," will also be printed on the top of the package.

6.8.6.3 Shipping Papers

Abbreviations will be used only when specified: "Flammable Liquid n.o.s. UN1993" or "Flammable Solid n.o.s. UN1324"; Limited Quantity (or Ltd. Qty.); net weight or net volume (abbreviations of weight or volume are acceptable). Further descriptions such as "Laboratory Samples" or "Cargo Aircraft Only" (if applicable) are allowed if they do not contradict the required information. The net weight or net volume will be placed just before or just after the "Flammable Liquid, n.o.s." or "Flammable Solid, n.o.s." description. The bill of lading will be completed and the certification statement signed (if the carrier does not provide, a standard industry form will be used) with the following information in the order listed. One form may be used for more than one exterior container.

A chain-of-custody record, properly executed, will be included in the exterior container.

6.8.6.4 Transportation

Unknown hazardous substances classified as flammable liquids may be transported by rented or common carrier truck, bus, railroad,

or by Federal Express Corporation, but they should not be transported by any other common carrier air transport, even "cargo only" aircraft. (DOT regulations permit "cargo only" aircraft such as TWA, United, etc.; however, difficulties with regular airline transportation suggests avoiding these types of transportation.)

These procedures are designed to enable air cargo shipment by entities such as Federal Express; however, they should not be construed as an endorsement of a particular commercial carrier.

If samples are transported by any type of government-owned vehicle, including aircraft, DOT regulations are not applicable. However, personnel will use the packaging procedures described. The bill of lading with a certification is an exception and does not have to be executed.

6.9 SAMPLE BOTTLE PREPARATION AND SAMPLE PRESERVATION

Sample containers and appropriate preservatives are presented in Tables 6-5 and 6-6 for specific chemical and radiological parameters. Sample bottles containing premeasured amounts of the appropriate chemical preservatives are prepared in the laboratory and are shipped to the field. Sample bottles are either purchased precleaned in accordance with U.S. EPA specifications or are cleaned in the laboratory to U.S. EPA specifications. Volatile organic analysis (VOA) vials are purchased precleaned. Glass containers for other organic analyses are washed with a nonphosphate detergent, rinsed with tap water, rinsed with methanol, rinsed with deionized water, and allowed to air dry. Plastic containers for metals analyses are washed with a nonphosphate detergent, rinsed with tap water and deionized water, and then rinsed with dilute nitric acid. Plastic containers for other general chemistry and radiological procedures are washed with a nonphosphate detergent and rinsed with tap water and deionized water.

TABLE 6-5
SAMPLING AND PRESERVATION REQUIREMENTS
FOR WATER SAMPLES
FEED MATERIALS PRODUCTION CENTER
FERNALD, OHIO

PARAMETER	CONTAINER(a)	VOLUME REQUIRED (ml)	PRESERVATION(b)	MAXIMUM HOLDING TIMES(c)
Inorganics				
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 Chloride	P,G	50	None required	28 days
o Cyanide (Total)	P,G	1,000	Cool 4°C, NaOH to pH >12, 0.6g Ascorbic Acid(d)	14 days(e)
o Fluoride	P	300	None required	28 days
o Redox Potential (Eh)	P,G	100	None required	Analyze immediately
o Hydrogen Ion (pH)	P,G	100	None required	Analyze immediately
o Hexavalent Chromium	P,G	50	Cool 4°C	24 hours
o Mercury(f)	P,G	100	HNO_3 to pH <2	28 days
o Metals, Except Hexavalent Chromium and Mercury(f)	P,G	200	HNO_3 to pH <2	6 months
o Nitrate	P,G	100	Cool 4°C	48 hours
o Organic Carbon (Total) (TOC)	G	25	Cool 4°C, HCl or H_2SO_4 to pH <2	28 days
o Organic Halogen (Total) (TOX)	G	100	Cool 4°C, H_2SO_4 to pH <2	14 days
o Organic Nitorgen (Total)	P,G	500	Cool 4°C, H_2SO_4 to pH <2	28 days

See footnotes at end of table.

TABLE 6-5
(Continued)

PARAMETER	CONTAINER(a)	VOLUME REQUIRED (ml)	PRESERVATION(b)	MAXIMUM HOLDING TIMES(c)
o Oxygen, Dissolved	G bottle and top	300	None required	Analyze immediately
o Phenols (Total)	G	500	Cool 4°C, 0.008% $Na_2S_2O_3$(d)	7 days until extraction, 40 days after extraction
o Phosphorus (Total)	P,G	50	Cool 4°C, H_2SO_4 to pH <2	28 days
o Residue, Filterable (TDS)	P,G	100	Cool 4°C	7 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 Temperature	P,G	1,000	None required	Analyze immediately
HSL Organic Compounds(g)				
o Volatiles	G, Teflon-lined septum	40	Cool 4°C, 0.008% $Na_2S_2O_3$(d)	7 days(i)
o PCBs	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 Pesticides	G, Teflon-lined cap	1,000	Cool 4°C, NaOH or HCl to pH 5-9(h), 0.008% $Na_2S_2O_3$(d)	7 days until extraction, 40 days after extraction
o Acid/Base Neutral Extractables	G, Teflon-lined cap	1,000 each	Cool 4°C, 0.008% $Na_2S_2O_3$(d)	7 days until extraction, 40 days after extraction
o Polychlorinated dibenzo-P-Dioxins and Polychlorinated Dibenzofurans	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$(d)	7 days until extraction, 40 days after extraction

See footnotes at end of table.

TABLE 6-5
(Continued)

PARAMETER	CONTAINER(a)	VOLUME REQUIRED (ml)	PRESERVATION(b)	MAXIMUM HOLDING TIMES(c)
Radiological Samples				
o Gross Alpha	P,G	1,000	HNO_3 to pH <2	6 months
o Gross Beta	P,G	1,000	HNO_3 to pH <2	6 months
o Radium-226	P,G	1,000	HNO_3 to pH <2	6 months
o Radium-228	P,G	1,000	HNO_3 to pH <2	6 months
o Strontium-90	P,G	1,000	HNO_3 to pH <2	6 months
o Technetium-99	P,G	1,000	HNO_3 to pH <2	6 months
o Cesium-137, Gamma Spec	P,G	1,000	HNO_3 to pH <2	6 months
o Ruthenium-106	P,G	1,000	HNO_3 to pH <2	6 months
o Plutonium, Isotopic	P,G	1,000	HNO_3 to pH <2	6 months
o Neptunium-237	P,G	1,000	HNO_3 to pH <2	6 months
o Thorium, Total	P,G	1,000	HNO_3 to pH <2	6 months
o Thorium, Isotopic	P,G	1,000	HNO_3 to pH <2	6 months
o Uranium, Total	P,G	1,000	HNO_3 to pH <2	6 months
o Uranium, Isotopic	P,G	1,000	HNO_3 to pH <2	6 months

See footnotes at end of table.

TABLE 6-5
(Continued)

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

(b)Sample preservation will be performed immediately upon sample collection.

(c)Samples will be analyzed as soon as possible after collection. The times listed are maximum times that samples should be held in the laboratory before analysis.

(d)Will only be used in the presence of residual chlorine. Sodium thiosulfate is only proposed as a sample preservative for HSL organics when residual free chlorine is known to be present. Sodium thiosulfate will be omitted in all other cases.

(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.

(f)Samples will be filtered immediately following collection on site before adding preservatives for dissolved metals.

(g)Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

(h)The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

(i)If acid preservation (HCl to pH<2) is used, the maximum holding time can be extended from 7 days to 14 days.

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. HSL organic compound requirements are promulgated in "Chemical Analytical Services for Multi-Media Multi-Concentration Organics, GC/MS Techniques," WA-87-K236, U.S. Environmental Protection Agency, Washington, D.C., 1987.

TABLE 6-6
SAMPLING AND PRESERVATION REQUIREMENTS
FOR SEDIMENT AND SOIL SAMPLES
FEED MATERIALS PRODUCTION CENTER
FERNALD, OHIO

PARAMETER	CONTAINER	SAMPLE WEIGHT REQUIRED (gm)	PRESERVATIVE	REFERENCE	MAXIMUM HOLDING TIMES(a)
Radionuclides	Plastic	100(b)	None	1	Indefinite
Inorganics	Plastic, Glass	500(c)	Cool, 4°C	2,3,4,5	Indefinite
Volatile Organics	Glass, Teflon-lined septum(d)	40	Cool, 4°C	6	10 days
Semivolatile Organics(e)	Glass, Teflon-lined cap	400	Cool, 4°C	6	10 days until extraction, 40 days after extraction
Pesticides/PCBs	Glass, Teflon-lined cap	400	Cool, 4°C	6	5 days until extraction 40 days after extraction
Cation Exchange Capacity (Total)	Plastic, Glass	400	Cool, 4°C	4	Indefinite

(a)Samples will be analyzed as soon as possible after collection. The times listed are maximum times that samples should be held before analysis.

(b)Check with laboratory for the volume of samples required for different radionuclide analyses.

(c)The collected sample can be divided for wet chemistry, metals, etc.

(d)40-ml VOA vial.

(e)Semivolatile organics include acid and base-neutral extractables and pesticides/polychlorinated biphenyls.

TABLE 6-6
(Continued)

REFERENCES:

(1)U.S. Environmental Protection Agency (EPA), September 1982, "Handbook for Sampling and Sample Preservation of Water and Wastewater," EPA-600/4-82-029.

(2)U.S. Environmental Protection Agency (EPA), April 1984, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, 2nd ed.

(3)U.S. Environmental Protection Agency (EPA), 1984, "Proposed Sampling and Analytical Methodologies for Addition to Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," PB85-103026.

(4)U.S. Environmental Protection Agency (EPA), 1978, "Field and Laboratory Methods Applicable to Overburdens and Mine Soil," EPA-600/2-80-054.

(5)U.S. Environmental Protection Agency (EPA), 1987, "Chemical Analytical Services for Multi-Media Multi-Concentration Metals and Inorganics," WA-87-K026, U.S. EPA, Washington, D.C.

(6)U.S. Environmental Protection Agency (EPA), 1987, "Chemical Analytical Services for Multi-Media Multi-Concentration Organics, GC/MS Techniques," WA-87-K236, U.S. EPA, Washington, D.C.

7.0 CHAIN-OF-CUSTODY PROCEDURES

7.1 FIELD SAMPLING

The following will be used in the chain-of-custody process for sample tracking and field activities:

- Sample identification and labeling;
- Sample chain-of-custody form;
- Sample collection log; and
- Laboratory request for analysis form.

7.1.1 SAMPLE IDENTIFICATION AND LABELING

All samples will be adequately marked for identification from the time of collection and packaging through shipping and storage. Marking will generally be made on the sample container (jar, bottle, etc.) but may be applied directly to the sample, or on a tag or label attached to the sample or container, depending on the type of sample and its intended use. Sample identification will include, as appropriate:

- Project name and number;
- Sample number;
- Sample location (e.g., boring, test pit, depth or sampling interval, and field coordinates);
- Sampling date and time;
- The initials of the individual(s) performing the sampling; and
- Sample preservative used.

An example of a standard sample label is illustrated in Figure 7-1. A sample numbering system will be established for the purpose of identifying the samples according to location and type as described in Sampling Plan, Volume 1, Introduction.

7.1.2 CHAIN-OF-CUSTODY RECORD

Documentation of the sample chain of custody is provided by the use of a chain-of-custody record that includes the sampling

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IT CORPORATION

Project Name _____ Project No. _____

Sample Location _____ Number _____

Boring/Well No. _____ Date _____

Collector's Name _____ Time _____

Sample Type: ____ Ground Water ____ Surface Water

____ Soil ____ Sludge/Waste

Parameters _____ Preservative _____

Bottle _____ of _____ ____ Filtered ____ Nonfiltered

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FIGURE 7-1

EXAMPLE SAMPLE LABEL

location, the type and amount of samples collected, the date and time of sample collection, the name(s) of the person(s) responsible for sample collection, the date and time of all custody transfers, the signature of the person relinquishing and accepting sample custody, and other pertinent information.

Chain-of-custody procedures document sample possession from the time of collection to disposal. A sample is considered in custody if it is:

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

A chain-of-custody record will be initiated in the field and will accompany each group of samples during shipment to the laboratory. Each time custody of the sample changes, the new custodian will sign the record and indicate the dates of transfer. An example of an IT chain-of-custody record is included in Figure 7-2.

If the samples are shipped to the laboratory by commercial carrier, the original 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 original 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. (The carrier and waybill number will be written on the chain-of-

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CHAIN-OF-CUSTODY RECORD

R/A Control No. _____

C/C Control No. 00150

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

FIGURE 7-2

custody form. If the original chain-of-custody form is sealed in the shipping container before the waybill number is received, then this number will be written on the copy of the chain-of-custody form.)

Upon receipt in the laboratory, the Quality Control Coordinator, or representative, shall open the shipping containers, compare the contents with the chain-of-custody record, and sign and date the record. The Quality Control Coordinator will also record the carrier and waybill number on the original chain-of-custody form, if it is not already present.

All original chain-of-custody records, analytical data, and other project documentation will be maintained in a project file. Project files will be stored in a central filing system pending disposition by WMCO.

A legible copy of the field chain-of-custody record will be maintained in the ASI/Fernald office on site. Once samples are received in the laboratory, chain-of-custody records will be signed by a designated representative of the laboratory and copies of the signed chain-of-custody records will be submitted to the ASI/Fernald office or other designated representative.

7.1.3 SAMPLE COLLECTION LOG

A sample collection log is prepared for each sample to record information pertaining to the location and collection of a sample. The following information is required on the sample collection log, as appropriate:

- Unique sample number;
- Sample location;
- Collector initials;
- Date and time sample collected;

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- Sample coordinates (Ohio State Planar System); and
- Sample identification (type, media, sequence, blank, spike, duplicate, split).

An example of a sample collection log is provided in Figure 7-3.

7.1.4 LABORATORY REQUEST FOR ANALYSIS FORM

The laboratory request for analysis form is prepared to indicate the testing program required for the collected samples. The following information is recorded on the laboratory request for analysis form:

- Project name and number;
- Date samples shipped;
- Required report date and turnaround times for analysis;
- Contact with telephone number for receipt of the analytical report and billing invoices;
- Sample identification numbers; and
- Sample volume collected and appropriate preservatives.

An example of a laboratory request for analysis form is provided in Figure 7-4.

7.2 ANALYTICAL LABORATORY

7.2.1 LABORATORY SAMPLE RECEIPT

Upon sample receipt, the QC coordinator or designee shall:

- Examine all samples and determine if proper temperature has been maintained during shipment. The receiving temperature will be recorded on the chain-of-custody record. If samples have been damaged during shipment, the remaining samples shall be carefully examined to determine whether they were affected. Any samples suspected of being affected shall also be considered damaged. It will be noted on the chain-of-custody record what specific samples were damaged and that the samples were removed from the sampling program. Field

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SAMPLE COLLECTION LOG

UNIQUE SAMPLE NUMBER	SAMPLE LOCATION	COLLECTOR INITIALS	COLLECTION DATE			COLLECTION TIME		COORDINATES		SAMPLE DEPTH INTERVAL	SAMPLE TYPE	SAMPLE MEDIA	PRESERVATIVE	SEQUENCE	BLANK	SPIKE	DUPLICATE	DUPLICATE OF SAMPLE NUMBER	SPLIT	SPLIT OF SAMPLE NUMBER	INITIAL
			MO	DY	YR	HR	MIN	NORTH	EAST												

FIGURE 7-3

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REQUEST FOR ALYSIS

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 _____

WHITE - Original, to accompany samples
YELLOW - Field copy

FIGURE 7-4

personnel will be notified in writing as soon as possible of an estimate of the cause of damage, that samples were damaged and that they must be resampled, or the testing program changed.

- Compare samples received against those listed on the chain-of-custody record.

- Verify that sample holding times have not been exceeded. If the sample holding time has been exceeded, the QC coordinator, or designee, will notify in writing to the field personnel that this has occurred and will prepare a Nonconformance Report (Section 16.0).

- Sign and date the chain-of-custody record and attach the waybill to the chain-of-custody record.

- Place the samples in adequate laboratory storage.

- Enter the samples in the laboratory sample log-in book which contains the following information:

  - Project identification number;
  - Sample numbers;
  - Type of samples; and
  - Date received in laboratory;

- Notify the laboratory manager or group leaders of sample arrival;

- Place the completed chain-of-custody records in the project file.

If samples collected arrive without chain-of-custody or with incorrect chain-of-custody records, the following actions shall be undertaken by the QC coordinator:

- If the chain-of-custody record is incorrect, a memorandum to the project director and field personnel is prepared stating the deviations. The memorandum must be signed and dated by the person originating the chain-of-custody and by the QC coordinator. The memorandum will serve as an amendment to the chain-of-custody. If the information on the chain-of-custody record cannot be corrected by the QC coordinator or the field personnel, the samples affected shall be removed from the sampling program.

- If the chain-of-custody record is not shipped with the samples, the field personnel shall be contacted and a memorandum prepared 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 record.

7.2.2 LABORATORY STORAGE OF SAMPLES

The primary considerations for sample storage are:

- Maintaining the prescribed temperature, if required, which is typically 4 degrees Celsius; and
- Extracting and/or analyzing samples within the prescribed holding time for the parameters of interest.

The requirements of Tables 6-5 and 6-6 for temperatures and holding times shall be used. Placing of samples in the proper storage environment is the responsibility of the QC coordinator or designee, who should notify the laboratory manager or group leaders if there are any samples which must be analyzed immediately because of holding time requirements.

7.2.3 INITIATION OF TESTING PROGRAM

As stated in Section 7.1.4, 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 QC coordinator shall immediately notify the manager responsible for the work for definition of the analysis program.

The laboratory manager and group leaders are responsible for prioritizing samples on the basis of holding time and of the required time needed to include the samples into the laboratory sample stream.

7.2.4 SAMPLE DISPOSAL

The chain of custody for the sample is completed as part of sample disposal. 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.

The laboratory manager shall determine disposition of samples if not specified on the request for analysis form (Figure 7-4).

In general, IT laboratories will not maintain samples and extracts longer than six months beyond completion of analysis, unless otherwise specified.

8.0 EQUIPMENT CALIBRATION/MAINTENANCE

Measuring and test equipment used in the field and laboratory will be controlled by a formal calibration program (e.g., Section 5.5 of the IT Engineering Services QA Manual, Section 6.1 of the IT Analytical Services QA Manual, Section 7 of the IT/RSL QA Manual, and in Laboratory-Specific Attachments for the Mixed Waste, Middlebrook Pike, Special Analysis Laboratory, Santa Clara Laboratory, Cerritos Laboratory, Austin Laboratory, PEI Laboratory, Edison Laboratory, and Geotechnical Laboratories). The program will provide equipment of the proper type, range, accuracy, and precision to provide data compatible with the specified requirements and desired results. Calibration of measuring and test equipment may be performed internally using in-house reference standards traceable to national Bureau of Standards (NBS), or some other nationally-reliable recognized standard, or externally by agencies or manufacturers.

8.1 Responsibilities

The responsibility for the calibration of laboratory equipment rests with the applicable laboratory manager. The site manager is responsible for the calibration of field equipment and field equipment provided by subcontractors.

8.2 Calibration Procedures

Documented and approved procedures will be used for calibrating measuring and test equipment. Whenever possible, widely-accepted procedures, such as those published by ASTM, or the U.S. EPA, or procedures provided by manufacturers in equipment manuals, will be adopted. Procedures for measuring and test equipment routinely calibrated are discussed in the IT Analytical Services QA Manual, the IT/RSL QA Manual, the IT/RSL Radioanalytical Methodology and QA Procedures Manual, and in Laboratory-Specific

Attachments for the Mixed Waste, Middlebrook Pike, Special Analysis Laboratory, Santa Clara Laboratory, Cerritos Laboratory, Austin Laboratory, PEI Laboratory, Edison Laboratory, and Geotechnical Laboratories. Procedures for the calibration of major equipment that may be used for this project are presented in Tables 8-1 through 8-3. Calibration procedures specific to the analytical methods referenced in Tables 4-1 through 4-3 are

discussed with the appropriate analytical procedures in Section 9.0 of the QAPP.

Calibrated equipment shall be uniquely identified by using either the manufacturer's serial number, a calibration system (Section 5.5.7 of the IT Engineering Services QA Manual) identification number, or other identifier(s). A calibration sticker will be attached to the appropriate equipment or record noted in calibration log so project personnel can recognize the most recent date of calibration and/or when the next calibration is due.

It is the responsibility of all personnel to check calibration status prior to using the equipment.

Measuring and test equipment shall be calibrated at prescribed intervals and/or as part of operational use. Frequency shall be based on the type of equipment, inherent stability, manufacturer's recommendations, values given in national standards, intended use, and experience (Tables 8-1 through 8-3). Equipment shall be calibrated, whenever possible, using reference standards having 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 calibration shall be documented.

Reference standards (physical, radiological, and chemical) shall be used only for calibration. Physical standards shall be stored separately from working measuring and test equipment. 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 to the satisfaction of the laboratory manager or site manager, as applicable. Equipment that cannot be repaired shall be replaced.

DOCUMENT CHANGE REQUEST NO. 11

Records will be prepared and maintained for each piece of calibrated measuring and test equipment to indicate that established calibration procedures have been followed. Records for subcontractor field equipment used only for this specific project will be kept in the project files. Records for equipment controlled by the IT calibration system (Section 6.0 of the IT Analytical Services QA Manual, Section RS12.0 of the IT/RSL QA Manual, and Mixed Waste, Middlebrook Pike, Special Analysis Laboratory, Santa Clara Valley Laboratory, Cerritos Laboratory, Austin Laboratory, PEI Laboratory, Edison Laboratory, and Geotechnical Laboratory-Specific Attachments) will be maintained by the appropriate IT laboratory.

TABLE 8-1

ENVIRONMENTAL LABORATORY AND FIELD EQUIPMENT
CALIBRATION AND MAINTENANCE PROCEDURES
FEED MATERIALS PRODUCTION CENTER
FERNALD, OHIO

INSTRUMENT/EQUIPMENT	CALIBRATION METHOD/PROCEDURE	REFERENCE	CALIBRATION FREQUENCY	MAINTENANCE SCHEDULE
INORGANIC ANALYSIS				
Atomic Adsorption Spectrophotometer (AA)	Metals(a) 206.2 CLP-M, 239.2 CLP-M, 245.1 CLP-M, 245.2 CLP-M, 270.2 CLP-M	(1)	Daily or with each use	Complete routine preventive maintenance program exists with daily, weekly, monthly, quarterly, semiannual, and annual activities.
Inductively Coupled Argon Plasma Emission Spectrometer (ICP)	200.7 CLP-M	(1)	Daily or with each use	Equipment tuneup conducted annually involving replacement of worn-out parts, calibration for optimum response, and performance.
UV/VIS Spectrophotometer	Cyanide and Nitrate(a) 335.2 CLP-M, 352.1	(1,2)	Daily or with each use	Routine preventive maintenance program, annual visit by service personnel for tuneup.
Specific Ion Meter/Microprocessor	pH and Fluoride(b) 150.1 and 340.2	(2)	Daily or with each use	Routine preventive maintenance program, annual visit by service personnel for tuneup.
Analytical and Top-Loading Balances	Section 5.1(c)	(3)	Three months	Routine preventive maintenance program, semiannual visit by service personnel for calibration.

See footnotes at end of table.

TABLE 8-1
(Continued)

INSTRUMENT/EQUIPMENT	CALIBRATION METHOD/PROCEDURE	REFERENCE	CALIBRATION FREQUENCY	MAINTENANCE SCHEDULE
Thermometer	Section 5.2(c)	(3)	Six months	Traceable to National Bureau of Standards
Conductivity Meter	Conductivity(d) 120.1	(2)	Daily or with each use	Routine preventive maintenance program, annual visit by service personnel for tuneup.
Exotox 50 Portable Gas Monitor	Standard calibration gases Chapter 6 of users manual	Neotronics Exotox 50 Portable Gas Monitor User Manual, 1986	Once/year at factory	Performance test every three months. Recharge battery after each use.
Draeger Multi-Gas Detector	Leak test per page 16 of instructions	Draeger Multi-Gas Detector, Instructions for Use	Before each use	Operational check before each use.
SKC Model 224-PCXR7 Sample Pump	Use flowmeter per page 13 of instructions	Operation Instructions for Universal Flow Sample Pump Model 224-PCXR7 SKC Inc.	Before each use	Recharge batteries after each use.
Dissolved Oxygen Meter	Probe in calibration chamber, switch instrument to CALIB O_2 and adjust CALIB until meter indicates local altitude	YSI Model 51B Dissolved Oxygen Meter Instruction Manual	Daily or before each use	Replace batteries after 1,000 hours or if full scale adjustment can not be made, Membrane replacement 2-4 weeks. Store probe in humid environment.
Water Level Indicator	Section 4.5(c)	(3)	Six months	Serviced after each field use.

See footnotes at end of table.

TABLE
(Contin.

INSTRUMENT/EQUIPMENT	CALIBRATION METHOD/PROCEDURE	REFERENCE	CALIBRATION FREQUENCY	MAINTENANCE SCHEDULE
ORGANIC ANALYSIS				
Gas Chromatograph/Mass Spectrometer (GC/MS)	Exhibit D(e)	(4)	Daily or with each use	Complete routine preventive maintenance program exists with daily, weekly, monthly, quarterly, semiannual, and annual activities.
	Exhibit D(e)	(4)	Daily or with each use	Equipment tuneup conducted semi-annually involving replacement of worn-out parts and calibration for optimum response and performance.
Gas Chromatograph (GC)	Exhibit D(f)	(4)	Daily or with each use	Complete routine preventive maintenance program exists with manufacturer service contract support.
Total Organic Carbon Analyzer	9060	(5)	Daily or with each use	Complete routine preventive maintenance program exists with daily, weekly, monthly, quarterly, semiannual, and annual activities.
Total Organic Halide Analyzer	9020	(5)	Daily or with each use	Complete routine preventive maintenance program exists with daily, weekly, monthly, quarterly, semiannual, and annual activities.
HNu Photoionization Detector	Calibrate with Isobutylene	HNu Systems, Inc. Instruction Manual for Model PI101 Photio-ionization Analyzer, 1975	Routine Calibration once/10 days Factory Calibration once/year	Monthly cleaning of UV light source window and ion chamber. Recharge battery after each use.

See footnotes at end of table.

TABLE 8-1
(Continued)

References:

(1) U.S. Environmental Protection Agency (EPA), 1985, "Chemical Analytical Services for Multi-Media Multi-Concentration Metals and Inorganics," WA-87-K236, Washington, D.C.

(2) U.S. Environmental Protection Agency (EPA), 1983, "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, Cincinnati, Ohio.

(3) IT Engineering Services Quality Assurance Manual, Chapters 4.0 and 5.0.

(4) U.S. Environmental Protection Agency (EPA), 1985, "Chemical Analytical Services for Multi-Media Multi-Concentration Organics, GC/MS Techniques," WA-87-K026, Washington, D.C.

(5) U.S. Environmental Protection Agency (EPA), 1986, 3rd ed., "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846

NOTES

(a) Generally, the calibration procedure consists of constructing a four-point standard curve consisting of a blank and three calibration standards within the instrument's working range. After the ICP or AA systems have been initially calibrated, the accuracy of the initial calibration is verified and documented for each metal by analysis of an initial calibration verification solution prepared from a source different from the calibration standards. Continuing calibration verification is performed every 10 samples or two hours, whichever is more frequent, as well as at the beginning and end of a sample run. The initial and continuing calibration verification solution (standard) must read within 90 to 110 percent of its true value. A calibration blank is analyzed each time the instrument is calibrated, at the beginning and the end of the run, and at a frequency of 10 percent during the run. The calibration blank must be below the analytical method detection limit. Calibration of the UV/VIS spectrophotometer follows the same procedure as described for AA.

(b) pH calibration consists of standardizing the meter using National Bureau of Standards (NBS) traceable buffer solutions at pH 4.01, 7.00, and 10.00. The procedure consists of constructing a four-point standard curve consisting of one standard at the method detection limit and three calibration standards within the working range of the specific ion electrode. Initial and continuing calibration verification solutions are analyzed as described for metals above.

TABLE 8-1
(Continued)

NOTES (Continued)

(c) Analytical balances are checked for calibration within the laboratory on a quarterly basis in accordance with the IT Engineering Services Quality Assurance Manual, Chapter 5.0 and are recalibrated at least every six months by outside service personnel. The indicated sections referenced for thermometers and water level indicators are in the IT Engineering Services Quality Assurance Manual, Chapters 4.0 and 5.0.

(d) The specific conductivity meter is calibrated with standard potassium chloride (KCl) solutions over the range of the sample readings. The KCl solutions used for calibration shall bracket the sample readings.

(e) Initial calibration of the GC/MS consists of a five-point standard curve at Contract Laboratory Program (CLP) prescribed concentration levels. The minimum average response factor and the percent relative standard deviation of the response factors must be within acceptance criteria. Continuing calibration must be performed every 12 hours using a single standard, and acceptance criteria must be met or the five-point standard calibration must be repeated.

(f) Initial calibration of the GC consists of a four-point linearity check curve consisting of a blank and three evaluation standards at CLP-prescribed concentration levels for aldrin, endrin, 4,4'-DDT and dibutylchlorendate. Single-point calibration standard mixtures are used to calibrate the GC for all pesticides and PCBs following the evaluation standards. During the sample analyses, the midpoint evaluation standard is alternated with the single-component pesticides calibration standard every five samples. The pesticide/PCB analysis sequence must end with the single-component pesticides calibration standard.

TABLE 8-2

GEOTECHNICAL LABORATORY EQUIPMENT
CALIBRATION PROCEDURES[a]
FEED MATERIALS PRODUCTION CENTER
FERNALD, OHIO

INSTRUMENT	PROCEDURE	FREQUENCY
Balance	Section 5.1	3 Months
Oven	Section 5.3	3 Months
No. 200 Sieve	Section 5.7	6 Months
Thermometer	Section 5.2	1 Year
Timing Device	Section 5.5	1 Year
Pressure Transducer	Section 5.21	6 Months
Caliper	Section 5.9	6 Months
Liquid Limit Device	Section 5.4	6 Months
Hydrometer	Section 5.6	1 Year
Pycnometer	Section 5.20	1 Year
Consolidometer	Section 5.11	1 Year
Extensometer	Section 5.10	1 Year
Proving Ring	Section 5.12	1 Year
Unconfined Compression Machine	Section 5.14	1 Year
Field Vane Shear	Section 4.13	b

[a]Manuals of Practice associated with the IT Engineering Services Quality Assurance Manual.

[b]Prior to use on project.

TABLE 8-3

RADIOLOGICAL LABORATORY AND FIELD EQUIPMENT CALIBRATION PROCEDURES FEED MATERIALS PRODUCTION CENTER FERNALD, OHIO

INSTRUMENT/EQUIPMENT	MANUFACTURER	CALIBRATION REFERENCE	CALIBRATION FREQUENCY
Low Background Alpha/Beta Counting System	Tennelec	1,2,3,4,5,6,7,8	Daily(a)
Scaler Scintillation Counter	Ludlum	1,2,3,4,5,6,7,8	Daily
Alpha Spectrometer	Tennelec, Canberra	2,3,4,5	Daily
Computer Based Gamma Spectrometer, (Data System Only)	Nuclear Data, Model ND9900	2,3,4,5	Monthly as a single unit
Liquid Scintillation Spectrometer	Packard	9	Daily
Intrinsic Germanium Detectors	EG&G ORTEC	1	Daily
Field Instrument for the Detection of Low Energy Radiation (FIDLER)	Bicron/Ludlum	Section 5.1.1.1 of QAPP	Weekly
Pressurized Ion Chamber (PIC)	Reuter - Stokes	Instrument Manual	Annually by Manufacturer
Large-Volume Scintillation Detector (SPA-3/ESP-1)	Eberline/Eberline	Instrument Manual	Weekly

See footnotes at end of table.

TABLE 8-3 CONTINUED

TABLE 8-3
(cont)

INSTRUMENT/EQUIPMENT	MANUFACTURER	CALIBRATION REFERENCE	CALIBRATION FREQUENCY
Ludlum Model 19 Micro R Meter	Per Section 5 Ludlum Instruction Manual	Ludlum Model 19 Micro R Meter Instruction Manual	6 Months
Ludlum Model 12 Portable Survey Meter with 44-9 Pancake CM detector or 43-5 alpha scintillation detector	Per Section 5 Ludlum Instruction Manual	Ludlum Model 12 Survey Meter Instruction Manual	6 Months
Eberline BC-4 Beta counter	Known Beta Gamma Source, Section IV-D of Instruction Manual	Eberline BC-4 Beta Counter Instruction Manual	6 Months
Eberline SAC-4 Scintillation Alpha	Known Alpha Source Section IV-D of Instruction Manual	Eberline SAC-4 Scintillation Alpha Counter Instruction Manual	6 Months

(a) Each Day Samples Are Analyzed.
(b) Operational check before each use.

TABLE 8-3
(Continued)

REFERENCES

(1) Krieger, H. L. and E. L. Whittaker, August 1980, "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/4-80-032 (1980 Update), U.S. Environmental Protection Agency (U.S. EPA).

(2) Kraus, K. A. and G. E. Moore, 1955, "The Separation of Pa (V) and Fe (III) with HCl Mixtures," Journal of American Chemical Society 77, 1383.

(3) Wish, Leon, March 1959, "Quantitative Radiochemical Analysis by Ion Exchange," Analytical Chemistry 31, 326-329.

(4) Campbell, E. E. and W. D. Moss, October 10-11, 1963, "Determination of Plutonium in Urine by Ion Exchange," Proceedings of the Ninth Annual Conference on Bioassay and Analytical Chemistry, San Diego, California.

(5) Henley, L. C., May 1978, Radiochemical Procedures of the Industrial Safety and Applied Health Physics Division, Oak Ridge National Laboratory, ORNL/TM-6372.

(6) Hume, D. N., N. E. Ballou, and L. E. Gendenin, June 1945, CN-2815, 23-26

(7) Swift, E. H., 1940, A System of Chemical Analysis, Prentice-Hall, New York, 376.

(8) Willard, H. H. and E. W. Goodspeed, 1936, "Separation of Strontium, Barium, and Lead from Calcium and Other Metals," Ind. Eng. Chem., Anal. Ed., 8, 414.

(9) Anders, Ed, November 1960, The Radiochemistry of Technetium, National Academy of Sciences - National Research Council.

9.0 LABORATORY ANALYTICAL PROCEDURES

The laboratory analytical procedures required for the FMPC Work Plan are described in the following sections. The analytical procedures for the specific sampling plans are provided in the IT Analytical Services QA Manual, the IT/RSL QA Manual, and the Laboratory-Specific Attachments for RSL, Mixed Waste, Middlebrook Pike, Special Analysis Laboratory, Santa Clara Valley Laboratory, Cerritos Laboratory, Austin Laboratory, PEI Laboratory, Edison Laboratory, and Geotechnical Laboratories.

9.1 Laboratory Program Flow Chart

The generation of project chemical data and results will follow the standard IT laboratory analytical program management scheme as discussed in the above-referenced manuals and attachments. The laboratory analysis flow chart (Figure 9-1) outlines the management scheme, which consists of five major areas:

- Project initiation;
- Handling of collected samples;
- Laboratory testing program initiation;
- Data verification; and
- Report preparation.

These areas are described in Sections 9.0 and 10.0.

9.1.1 Project Initiation

Prior to initiation of laboratory testing, a planning session with the appropriate laboratory and project staffs will be conducted to discuss the specific aspects of the following project tasks that must be completed at this time:

- Define project requirements, including equipment, parameters, sampling procedures (Section 6.0), QC samples, and analytical methods (Section 9.3) selection.
- Request sample bottles from laboratory's sample custodians.

- Prepare sample bottles with appropriate labels and preservatives (Sections 6.6 and 7.1.1).
- Provide blank chain-of-custody and request for analysis forms with sample bottles; these will be shipped to the site.

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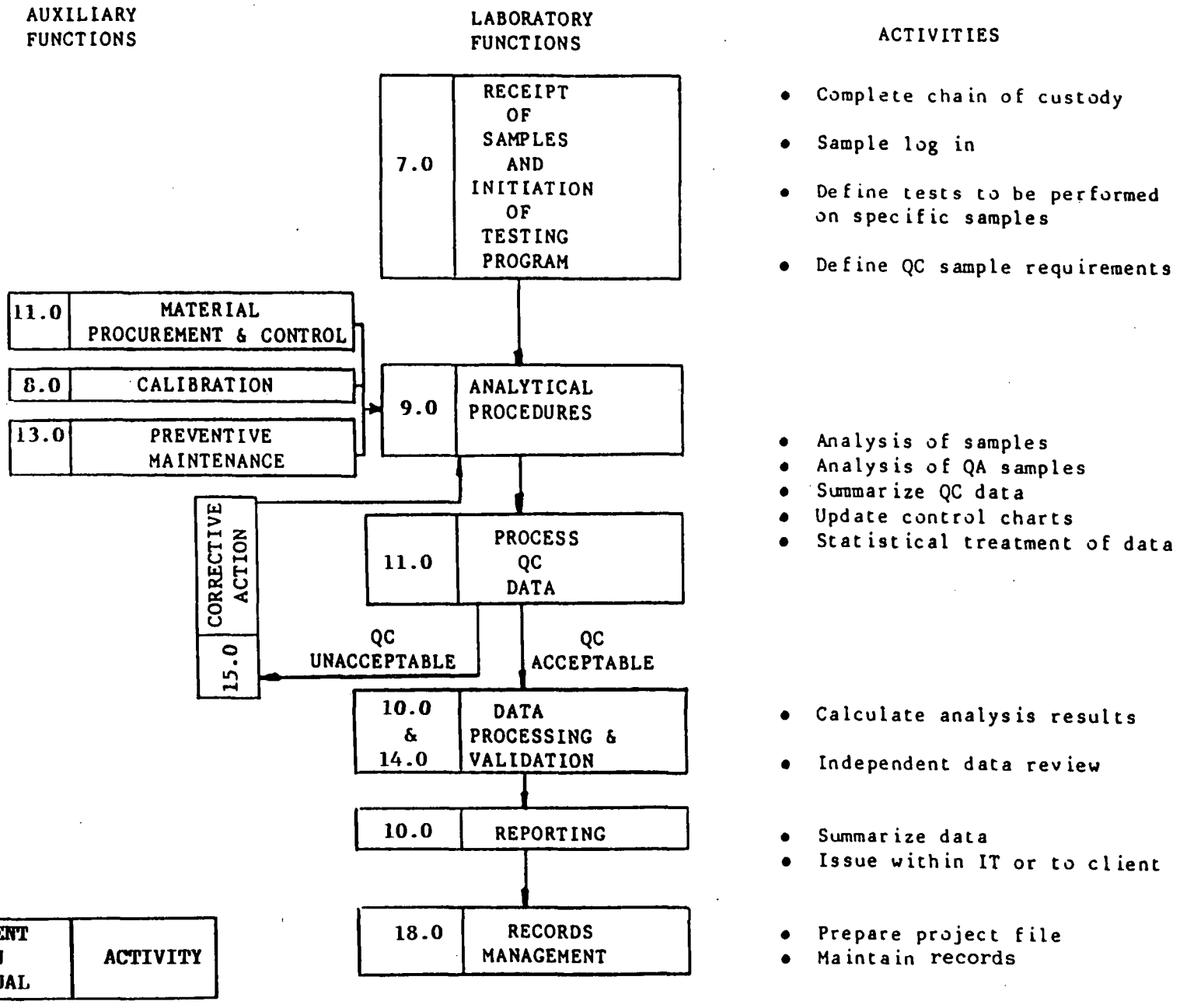

LABORATORY ANALYSIS FLOW CHART

FIGURE 9-1

9.1.2 SAMPLE PROCESSING PROCEDURES

The following procedures are implemented by field and laboratory personnel for handling of collected samples:

- Summarize field data collection on field sheets (Section 7.1) and initiate chain-of-custody records.
- Transport collected samples to the laboratory under suitable environmental conditions (Section 6.5).
- Follow U.S. DOT regulations for sample transport.
- At the laboratory, review received samples and process chain-of-custody records. If necessary, code samples with a unique number upon receipt in the laboratory (Section 9.2.1).
- Place samples in proper storage.
- Log samples in laboratory log. Open the chemistry project file and indicate parameters on sample control board (Section 9.2.2).

9.2 LABORATORY TESTING PROGRAM INITIATION

9.2.1 RADIOLOGICAL SCREENING

Samples from the Fernald RI/FS will undergo a radiological screening at IT/RSL to classify the samples into one of three hazard categories. All samples are counted for gross alpha and gross beta activity. In addition, samples that contain or are suspected of containing low energy beta emitters are analyzed by liquid scintillation counting. The purpose of the screening is threefold: (1) to determine if the samples can be shipped to other nonlicensed IT laboratories; (2) to verify that the radioactivity content is below the IT/RSL license limit; and (3) to evaluate the radiological hazard associated with the sample

preparation and analysis procedure. A description of the three hazard categories is given below:

- Category I - There are no radiological hazards associated with these samples. Category I samples may be shipped to other laboratories.

- Category II - Category II samples must be analyzed at IT/RSL. Standard safe laboratory procedures are sufficient to protect the health of laboratory personnel.

- Category III - Category III samples must be analyzed at IT/RSL. Screening results are reviewed on a case-by-case basis in order to specify handling and monitoring requirements and to verify that the radionuclide content of the sample does not exceed the license limit.

9.2.2 RECEIPT OF SAMPLES

Upon receipt of the samples, the laboratory QC coordinator shall:

- Examine all samples and determine if proper temperature has been maintained during shipment. If samples have been damaged during shipment, the remaining samples shall be carefully visually examined to determine whether they were affected. Any samples affected shall be also considered damaged. It will be noted on the chain-of-custody record 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 to avoid such damage in the future.

- Compare samples received against those listed on the chain-of-custody record.

- Verify that sample holding times have not been exceeded.

- Sign and date the chain-of-custody record and attach any waybill to the chain of custody.

- Place the samples in appropriate laboratory storage.

- Enter the samples in the laboratory sample log-in book which contains the following information:
  - Project identification number;
  - Sample numbers;
  - Type of samples; and
  - Date received in laboratory.
- Notify the laboratory manager of sample arrival.
- Place the chain-of-custody records in the laboratory project file.

9.3 LABORATORY TESTING PROCEDURES

Samples from the FMPC site will be analyzed using the chemical and radiological methods listed in Tables 4-1, 4-2, and 4-3. Geotechnical samples will be analyzed using methods listed in Table 9-4 (Section 9.4.5). The actual references are maintained in the laboratory for use by laboratory personnel.

9.4 ANALYTICAL METHODS

The analytical testing program for the samples collected from the FMPC site can be broadly divided into five analytical categories:

- Sample preparation;
- Inorganic analysis;
- Organic analysis;
- Radiological analysis; and
- Geotechnical analysis.

9.4.1 SAMPLE PREPARATION

If required by the project work plan, selected samples will be extracted according to Method 1310, Extraction Procedure (EP) Toxicity Test Method, as described in U.S. EPA, 1984, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, 2nd ed., revised, U.S. EPA, Washington, D.C. A method blank will be processed with each set of samples or with every 20 samples, whichever is more frequent. Additionally, a duplicate EP toxicity extraction will also be performed with each set of samples or with every 20 samples, whichever is more frequent. SW-846 requires that all EP toxicity leachate analyses

for metals be performed using the Method of Standard Additions described in Section 11.4.1.11. Accordingly, a single-point Method of Standard Additions (percent recovery spike) will be performed for each analytical parameter on each sample. If the percent recovery of the sample spike is not within the 85 to 115 percent range, a three-point Method of Standard Addition procedure as described in Section 11.4.1.11 will be performed. Other sample preparation procedures are described with the appropriate analytical procedures under Sections 9.4.2 and 9.4.3.

9.4.2 INORGANIC ANALYSIS

The inorganic analytical procedures which will be used to perform the laboratory program are specified below for:

- Metals (ICP);
- Arsenic and selenium;
- Mercury;
- Hexavalent chromium;
- Cyanide;
- Fluoride; and
- Nitrate.

9.4.2.1 Inductively Coupled Argon Plasma Spectrometry - U.S. EPA Method 200.7 CLP-M

Metals analyses will be performed by inductively coupled argon plasma emission spectrometry (ICP). However, ICP is not suitable for all metals, and graphite furnace atomic absorption spectrophotometry will be used to analyze for arsenic, selenium, and lead. Mercury will be analyzed by cold vapor atomic absorption spectrophotometry. A summary of the referenced analytical procedures (Table 4-1) is presented below.

Method Summary

The method describes a technique for the simultaneous or sequential multi-element determination of trace elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the

plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio frequency ICP. The spectra are dispersed by a grating spectrometer and the intensities of the line are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement on either or both sides of the analytical line will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate corrections made.

Interference

Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as follows:

- Spectral Interferences can be categorized as overlap of a spectral line from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena, and background contribution from stray light from the line emission of high concentration elements. The first of these effects can be compensated for by utilizing a computer correction of the raw data, requiring the monitoring and measurement of the interfering element. The second effect may require selection of an alternate wavelength. The third and fourth effects can usually be compensated by a background correction adjacent to the analyte line. In addition, users of simultaneous multielement instrumentation must assume the responsibility of verifying the absence of spectral interference from an element that could occur in a sample but

for which there is no channel in the instrument array. Listed in Table 9-1 are some interference effects for the recommended wavelengths. The data in Table 9-1 are intended for use only as a rudimentary guide for the indication of potential spectral interferences. For this purpose, linear relations between concentration and intensity for the analytes and the interferences can be assumed. The interference information, which was collected at the Ames Laboratory (See WA-87-K026), is expressed as analyte concentration equivalents (i.e., false analyte concentrations) arising from 100 milligrams per liter (mg/ℓ) of the interferent element. The suggested use of this information is as follows: Assume that arsenic (at 193.696 nanometers) is to be determined in a sample containing approximately 10 mg/ℓ of aluminum. According to Table 9-1, 100 mg/ℓ of aluminum would yield a false signal for arsenic equivalent to approximately 1.3 mg/ℓ. Therefore, 10 mg/ℓ of aluminum would result in a false signal for arsenic equivalent to approximately 0.13 mg/ℓ. The reader is cautioned that other analytical systems may exhibit somewhat different levels of interference than those shown in Table 9-1 and that the interference effects must be evaluated for each individual system. Only those interferents listed were investigated, and the blank spaces in Table 9-1 indicate that measurable interferences were not observed.

- Physical interferences are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies, especially in samples that may contain high dissolved solids and/or acid concentrations. The use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem which can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate, causing instrumental drift. Wetting the argon prior to nebulization, the use of a tip washer, or sample dilution have been used to control this problem. Also, it has been reported that better control of the argon flow rate improves instrument performance. This is accomplished with the use of mass flow controllers.

- Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not

TABLE 9-1

EXAMPLES OF ANALYTE CONCENTRATION EQUIVALENTS
ARISING FROM INTERFERENTS
FEED MATERIALS PRODUCTION CENTER
FERNALD, OHIO

ANALYTE	WAVELENGTH (nm)	INTERFERENT (100 mg/l)(a)									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	-	-	-	-	-	-	0.21	-	-	1.4
Antimony	206.833	0.47	-	2.9	-	0.08	-	-	-	0.25	0.45
Arsenic	193.696	1.3	-	0.44	-	-	-	-	-	-	1.1
Barium	455.403	-	-	-	-	-	-	-	-	-	-
Beryllium	313.042	-	-	-	-	-	-	-	-	0.04	0.05
Boron	249.773	0.04	-	-	-	0.32	-	-	-	-	-
Cadmium	226.502	-	-	-	-	0.03	-	-	0.02	-	-
Calcium	317.933	-	-	0.08	-	0.01	0.01	0.04	-	0.03	0.03
Chromium	267.716	-	-	-	-	0.003	-	0.04	-	-	0.04
Cobalt	228.616	-	-	0.03	-	0.005	-	-	0.03	0.15	-
Copper	324.754	-	-	-	-	0.003	-	-	-	0.05	0.02
Iron	259.940	-	-	-	-	-	-	0.12	-	-	-
Lead	220.353	0.17	-	-	-	-	-	-	-	-	-
Magnesium	279.079	-	0.02	0.11	-	0.13	-	0.25	-	0.07	0.12
Manganese	257.610	0.005	-	0.01	-	0.002	0.002	-	-	-	-
Molybdenum	202.030	0.05	-	-	-	0.03	-	-	-	-	-
Nickel	231.604	-	-	-	-	-	-	-	-	-	-
Selenium	196.026	0.23	-	-	-	0.09	-	-	-	-	-
Silicon	288.158	-	-	0.07	-	-	-	-	-	-	0.01
Sodium	588.995	-	-	-	-	-	-	-	-	0.08	-
Thallium	190.864	0.30	-	-	-	-	-	-	-	-	-
Vanadium	292.402	-	-	0.05	-	0.005	-	-	-	0.02	-
Zinc	213.856	-	-	-	0.14	-	-	-	0.29	-	-

(a)"mg/l" = milligrams per liter or parts per million (ppm).

pronounced with the ICP technique; however, if observed, they can be minimized by careful selection of operating conditions (that is, incident power, observation position, etc.), buffering of the sample, matrix matching, and standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element.

Instrumentation and Operating Conditions

A spectrometer will be used for ICP analyses. Because of the differences between various makes and models of satisfactory instruments, detailed operating instructions are not provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must 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 QC data confirming instrument performance and analytical results.

Materials and Reagents

Acids used in the preparation of standards and for sample processing must be ultrahigh purity grade or equivalent. Redistilled acids are acceptable. Volumetric glassware will be Type A and traceable to National Bureau of Standards (NBS) standards.

Standards

Commercially available certified standards will be used. Alternatively, laboratory stock standards may be prepared in accordance with U.S. EPA Method 200.7 CLP-M when stock standards are not commercially available. The following standards will be generated from the stock standards:

- Mixed Calibration Standard Solutions
  Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks. Add 2 milliliters of (1+1) nitric acid (HNO_3) and 10 milliliters of (1+1) hydrochloric acid (HCl) and dilute to 100 milliliters with deionized, distilled water. Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interference or the presence of impurities. Care should be taken when preparing the mixed standards that the elements are compatible and stable. Transfer the mixed standard solutions to a fluorocarbon or unused polyethylene bottle for storage. Fresh mixed standards should be prepared as needed with the realization that concentration can change on aging. Calibration standards must be initially verified using a QC sample and monitored weekly for stability.

- Instrument Check Standard
  A multielement standard of known concentrations prepared by the analyst to monitor and verify the calibration stability of the instrument.

- Interference Check Sample
  A multielement standard containing both interfering and analyte elements of known concentrations that can be used to verify background and interelement correction factors. This standard may be prepared by the analyst or, if available, obtained from the U.S. EPA.

Analysis

The following summary briefly describes the procedure for analysis of samples:

- Set up instrument with proper operating parameters established in Instrument and Operating Conditions. The instrument must be allowed to become thermally stable before beginning. This usually requires at least 30 minutes of operation prior to calibration.

- Initiate the appropriate operating configuration via the computer.

- Profile and calibrate instrument according to instrument manufacturer's recommended procedures, using mixed calibration standard solutions such as those described in Standards. Flush the system with the calibration blank between each standard. Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error. The

calibration curve will consist of a blank and three calibration standards within the working range of the instrument.

- Begin the sample run, flushing the system with the calibration blank solution between each sample. Analyze the instrument check standard and the calibration blank each ten samples.

Calculations

Calculations of analytical results should be performed in accordance with the manufacturer's instruction for computerized data reduction and in consideration of the following:

- Reagent blanks (method/preparation blanks) should be treated as specified in Exhibit E of WA-87-K026.
- If dilutions were performed, the appropriate factor must be applied to sample values.
- Data must be reported in micrograms per liter (µg/ℓ) or micrograms per kilogram (µg/kg).

Quality Control

The major QC elements of the analytical procedure are summarized below:

- Two types of blanks are required for the analysis. The calibration blank is used in establishing the analytical curve while the preparation blank is used to correct for possible contamination resulting from varying amounts of the acids used in the sample processing.

  - Calibration Blank - This blank is prepared by diluting 2 milliliters of (1+1) HNO_3 and 10 milliliters of (1+1) HCl to 100 milliliters with deionized, distilled water. Prepare a sufficient quantity to be used to flush the system between standards and samples.

    Analyze the calibration blank at a frequency of 10 percent. The results documented on Form III (Appendix A) should be within ± contract required detection levels (Table 4-1). If the result is not within the control level, terminate the analysis, correct the problem, and recalibrate the instrument.

- Reagent Blank (Method/Preparation Blank) - This blank must contain all the reagents and in the same volumes as used in the processing of the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

- In addition to the calibration standards, an instrument check standard, an interference check sample, and a QC sample are also required for the analyses.

  - Instrument Check Standard - The standard is prepared by the analyst by combining compatible elements at a concentration equivalent to the midpoint of their respective calibration curves.

    For continuing calibration verification, analyze an appropriate instrument check standard containing the elements of interest at a frequency of 10 percent and document on Form II, Part 1 and Part 2 (Appendix A). This check standard is used to determine instrument drift. If agreement is not within ±10 percent of the expected values, the analysis is out of control. The analysis must be terminated, the problem corrected, the instrument recalibrated, and the preceding 10 samples reanalyzed.

  - Interference Check Sample - This sample consists of a solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

    To verify interelement and background correction factors, analyze the ICP interference check sample at the beginning and end of the sample run or at a minimum of twice per eight-hour work shift, whichever is more frequent. The check sample must be analyzed initially at least five times to establish a mean value and standard deviation. Results documented on Form IV (Appendix A) must fall within the established control limits. If not, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze the samples.

  - QC Sample - This sample consists of a solution obtained from an outside source having known concentration values to be used to verify the calibration standards. The QC sample should be prepared in the same acid matrix as the calibration standards and in accordance with the instructions provided by the supplier.

A QC sample must be used daily for the initial calibration verification. A fresh dilution of this sample shall be analyzed and documented on Form VII (Appendix A) every week thereafter to monitor its stability. If the results are not within ±10 percent of the true value listed for the control sample, prepare a new calibration standard and recalibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

- Serial Dilution Interference Check
  A serial dilution interference check must be performed for each group of samples of a similar matrix type and concentration or for each 20 samples received, whichever is more frequent.

  If the analyte concentration is sufficiently high (minimally a factor of 10 above the instrument detection limit after dilution), an analysis of a 1:4 dilution must agree within 10 percent of the original determination. Serial dilution results must be reported on QC Report Form IX (Appendix A). Samples identified in field blanks cannot be used for serial dilution analysis.

  If the dilution analysis is not within 10 percent, a chemical or physical interference effect should be suspected and the data must be flagged with an "E."

9.4.2.2 Graphite Furnace Atomic Absorption Spectrophotometry for Arsenic, U.S. EPA Method 206.2 CLP-M and Selenium, U.S. EPA Method 270.2 CLP-M

Method Summary

A 20-microliter aliquot of the prepared sample solution is injected into the atomization chamber of the graphite furnace. The sample is dried, ashed to remove organic constituents, and finally atomized at high temperature. Light from a hollow cathode lamp, with a cathode made of the element to be determined, is directed through the flame into a monochromator and a detector that measures the amount of light absorbed. Absorption depends upon the presence of free unexcited ground state atoms in the graphite furnace. Since the wavelength of the light beam is characteristic of the metal being determined, the light energy

absorbed by the flame is a measure of the concentration of that metal in the sample. This principle is the basis of atomic absorption spectrophotometry.

Interferences

The graphite furnace procedure is subject to chemical and matrix interferences. Accordingly, procedures for the identification of matrix interferences and appropriate corrective actions are described in Section 11.0.

Arsenic and selenium are relatively volatile metals and low analytical results can result from volatilization during graphite furnace analysis. Accordingly, 100 microliters of a 5 percent nickel nitrate solution should be added to 5 milliliters of the sample prior to analysis.

Instrumentation and Operating Conditions

An atomic absorption spectrophotometer with a graphite furnace and autosampler will be used for arsenic and selenium analyses. General operating conditions are presented below:

- Arsenic
  - Optimum concentration range - 5 to 100 µg/ℓ;
  - Contract required detection limit - 10 µg/ℓ;
  - Drying time and temperature - 30 seconds at 125°C;
  - Ashing time and temperature - 30 seconds at 1,100°C;
  - Atomizing time and temperature - 10 seconds at 2,700°C;
  - Purge gas atmosphere - argon; and
  - Wavelength: 193.7 nanometers.
- Selenium
  - Optimum concentration range - 5 to 100 µg/ℓ;
  - Contract required detection limit - 5.0 µg/ℓ;

- Drying time and temperature - 30 seconds at 125°C;
- Ashing time and temperature - 30 seconds at 1,200°C;
- Atomizing time and temperature - 10 seconds at 2,700°C;
- Purge gas atmosphere - argon; and
- Wavelength: 196.0 nanometers.

Materials and Reagents

All volumetric glassware will be Type A and traceable to NBS standards. Reagents and chemicals used for standard preparation and sample processing will be ultrahigh purity grade or American Chemical Society (ACS) reagent grade as appropriate.

Standards

Commercially available certified standards will be used. Alternatively, stock standard solutions may be prepared in the laboratory in accordance with appropriate CLP procedures. The following standards are prepared from the stock standard solution:

- Working Standards
  Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The calibration standards must be prepared using the same type of acid and at the same concentration as will result in the sample to be analyzed after sample preparation. Withdraw appropriate aliquots of the stock solution, add 1 milliliter of concentrated HNO_3, 2 milliliters of 30 percent hydrogen peroxide (H_2O_2), and 2 milliliters of the 5 percent nickel nitrate solution. Dilute to 100 milliliters with deionized distilled water.

- Matrix Spike and/or Method of Standard Addition Spike Standard
  Prepare dilutions of the stock solution to obtain the appropriate concentrations.

- Instrument Check Standard
  Prepare dilutions of the stock solution to obtain a concentration equivalent to the midpoint of the calibration curve.

- Initial Calibration-Verification Solution
  Obtain from the U.S. EPA and prepare according to the accompanying instructions.

Analysis

The following summary briefly describes the procedure for analysis of samples:

- Set up instrument with proper operating parameters established in Instrument and Operating Conditions. Background correction is required for all analyses.

- Calibrate the instrument according to the instrument manufacturer's recommended procedures using a calibration curve consisting of a blank and three calibration standards within the working range.

- Begin the sample run by reanalyzing the calibration curve and analyzing the U.S. EPA initial calibration verification solution. Analyze the instrument check standard and the calibration blank (zero standard) every ten samples. Reanalyze the entire calibration curve at the end of the sample run.

Calculations

Calculation of arsenic and selenium concentrations is automated and sample aliquot concentrations are read directly from the instrument. Calculation of final sample concentrations is performed on the computer and output on the summary report.

Quality Control

The major QC elements of the graphite furnace atomic absorption spectrophotometry analytical procedures for arsenic and selenium are summarized below:

- Two types of blanks are required for the analysis. The calibration blank is used in establishing the analytical curve while the preparation blank is used to correct for possible contamination resulting from the acids used in the sample processing.

- Calibration Blank is prepared by diluting two milliliters of (1+1) HNO_3 to 100 milliliters with deionized, distilled water.

  Analyze the calibration blank at a frequency of 10 percent. The results documented on Form III (Appendix A) should be within ± contract-required detection levels. If the result is not within the control level, terminate the analysis, correct the problem, and recalibrate the instrument.

- Reagent Blank (Method/Preparation Blank). This blank must contain all the reagents and in the same volumes as used in the processing of the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

- Instrument Check Standard
  The standard is prepared by the analyst at a concentration equivalent to the midpoint of the respective calibration curve.

  For continuing calibration verification, analyze an appropriate instrument check standard containing the element of interest at a frequency of 10 percent and documented on Form II (Appendix A). This check standard is used to determine instrument drift. If agreement is not within ±10 percent of the expected values, the analysis is out of control. The analysis must be terminated, the problem corrected, the instrument recalibrated, and the preceding 10 samples reanalyzed.

- U.S. EPA Initial Calibration Verification Sample
  The sample consists of a solution obtained from an outside source having known concentration values to be used to verify the calibration standards. The sample should be prepared in the same acid matrix as the calibration standards and in accordance with the instructions provided by the supplier.

  A U.S. EPA initial calibration verification sample must be used daily or whenever samples are run for the initial calibration verification. If the results are not within ±10 percent of the true value listed for the control sample, prepare a new calibration standard and recalibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

- Matrix Interference Check for Graphite Furnace
  A postdigestion analytical spike of each sample must be prepared prior to analysis by adding a known quantity of the analyte to an aliquot of the digested sample. Spiking should be performed such that no greater than a 10 percent increase in volume occurs in the spiked sample aliquot. This is necessary to prevent dilution of potential matrix interferences. The spike concentration in the sample aliquot must be twice the Contract Required Detection Limit (CRDL) for the parameter of interest. If the spike recovery is outside the acceptance criteria of 85 to 115 percent documented on Form V B (Appendix A), a three-point method of standard additions must be performed at increments of 50, 100, and 150 percent of the sample absorbance to quantitate the sample.

9.4.2.3 Cold Vapor Atomic Absorption Spectrophotometry for Mercury, U.S. EPA Methods 245.1 CLP-M and 245.2 CLP-M

Method Summary

The flameless atomic absorption procedure is a physical method based on the absorption of radiation at 253.7 nanometers by mercury vapor. The mercury is reduced to the elemental state and purged from solution. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.

In addition to inorganic forms of mercury, organic mercurials may also be present. These organomercury compounds will not respond to the flameless atomic absorption technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but recent studies have shown that a number of organic mercurials, including phenyl mercuric acetate and methyl mercuric chloride, are only partially oxidized by this reagent. Potassium persulfate has been found to give approximately 100 percent recovery when used as the oxidant with these compounds. Therefore, a weighed portion of the sample is acid digested for two minutes at 95 degrees Celsius followed

by oxidation with potassium permanganate and potassium persulfate. Mercury in the digested sample is then measured by the conventional cold vapor technique.

Interference

The following conditions can interfere with the determination of mercury in water and solids:

- Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 mg/ℓ of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.

- Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/ℓ had no effect on recovery of mercury from spiked samples.

- Sea waters, brines, and industrial effluents high in chlorides require additional permanganate (as much as 25 milliliters). During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation of 253 nanometers. Care must be taken to make sure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 milliliters). In addition, the dead air space in the biochemical oxygen demand (BOD) bottle must be purged before the addition of stannous sulfate. Both inorganic and organic mercury spikes have been quantitatively recovered from the sea water using this technique.

- Interference from certain volatile organic materials which will absorb at this wavelength is also possible. A preliminary run without reagents should determine if this type of interference is present.

- Formation of a heavy precipitate in some wastewaters and effluents has been reported upon addition of concentrated sulfuric acid. If this is encountered, the sample cannot be analyzed by this method.

- Samples containing solids must be blended and then mixed while being sampled if total mercury values are to be reported.

Instrumentation and Operating Conditions

Atomic absorption spectrophotometer with a cold vapor apparatus for flameless mercury determination will be used for mercury analysis. General operating conditions for mercury are presented below:

- Optimum Concentration Range - 0.2 to 20.0 µg/ℓ in water; 0.2 to 5.0 mg/ℓ in solids;
- Wavelength - 253.7 nanometers;
- Purge Gas - argon or nitrogen; and
- Purge Gas Flow Rate - 1.0 liter per minute or per manufacturer's instructions.

Materials and Reagents

All volumetric glassware will be Type A and traceable to NBS standards. Reagents and chemicals used for standard preparation and sample processing will be ultrahigh purity grade or ACS reagent grade as appropriate.

Standards

Commercially available certified standards will be used. Alternatively, stock standard solutions may be prepared in the laboratory in accordance with appropriate CLP procedures. The following standards are prepared from the stock standard solution:

- Working standards
  Make successive dilutions of the stock mercury standard to obtain a working standard containing 0.1 microgram per milliliter (µg/mℓ). This working standard and the dilution of the stock mercury solutions should be prepared fresh daily. Acidity of the working standard should be maintained at 0.15 percent nitric acid. This acid should be added to the flask as needed before the addition of the aliquot.

  Transfer 0-, 0.5-, 1.0-, 5.0-, and 10-milliliter aliquots of the working mercury solutions containing 0 to 1.0 microgram of mercury to a series of 300-milliliter BOD bottles and process through the digestion procedure with the samples.

- Instrument Check Standard
  Prepare dilutions of the stock standard to obtain a working standard at the midpoint of the calibration curve.

- Matrix Spike Standard
  Prepare dilutions of the stock standard to obtain a matrix standard at the appropriate concentration to achieve a 1.0 µg/ℓ concentration in the final digest of the spiked sample. The increase in sample volume from spiking should not exceed 10 percent.

Analysis

The following summary briefly describes the procedure for the analysis of samples:

- Set up instrument in accordance with the operating parameters described in the CLP analytical procedure and the manufacturer's instructions.

- Transfer 100 milliliters, an aliquot diluted to 100 milliliters or, in the case of solids, 0.2 gram of sample containing not more than 1.0 microgram of mercury, to a 300-milliliter BOD bottle. Add 5 milliliters of sulfuric acid and 2.5 milliliters of concentrated nitric acid, mixing after each solution. Add 15 milliliters of potassium permanganate solution to each sample bottle. For sewage samples, additional permanganate may be required. Shake and add additional portions of potassium permanganate solution, if necessary, until the purple color persists for at least 15 minutes. Add 8 milliliters of potassium persulfate solution to each bottle and heat for two hours in a water bath at 95 degrees Celsius.

- Calibrate the instrument according to the CLP-recommended procedures using a calibration curve consisting of a blank and four calibration standards at 0.05, 0.10, 0.50, and 1.0 total microgram of mercury.

- Begin the sample run by analyzing the U.S. EPA initial calibration verification solution. Analyze the instrument check standard (midpoint calibration standard) and the calibration blank (zero standard) every 10 samples.

  Reanalyze the entire calibration curve at the end of the sample run.

Calculations

Calculation of mercury is automated on the Perkin-Elmer 5000 and sample aliquot concentrations are read directly from the instrument. Calculation of final sample concentrations is performed on the computer and output on a summary report.

Quality Control

The major QC elements of the cold vapor atomic absorption spectrophotometry technique for the determination of mercury are summarized below:

- Two types of blanks are required for the analysis. The calibration blank is used in establishing the analytical curve while the preparation blank is used to correct for possible contamination resulting from the acids used in the sample processing.

  - Calibration Blank - This blank is prepared by diluting 2 milliliters of (1+1) HNO_3 to 100 milliliters with deionized, distilled water.

    Analyze the calibration blank at a frequency of 10 percent. The result should be within ± contract required detection levels. If the result is not within the control level, terminate the analysis, correct the problem, and recalibrate the instrument.

  - Reagent Blank (Method/Preparation Blank) - This blank must contain all the reagents in the same volumes as used in processing the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

- Instrument Check Standard
  The standard is prepared by the analyst at a concentration equivalent to the midpoint of the calibration curve.

  For continuing calibration verification, analyze an appropriate instrument check standard containing the element of interest at a frequency of 10 percent and documented on Form II (Appendix A) This check standard is used to determine instrument drift. If agreement is not within ±20 percent of the expected values, the analysis is out of control. The analysis must be terminated, the problem corrected, the instrument recalibrated, and the preceding 10 samples reanalyzed.

- U.S. EPA Initial Calibration Verification Sample
  This sample consists of a solution obtained from an outside source having a known concentration value to be used to verify the calibration standards. The QC sample should be prepared in the same matrix as the calibration standards and in accordance with the instructions provided by the supplier. A QC sample must be used daily or whenever samples are run for the initial calibration verification. If the results are not within ±20 percent of the true value listed for the control sample, prepare a new calibration standard and recalibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

9.4.2.4 Alkaline Extraction and Colorimetric Determination of Hexavalent Chromium, U.S. EPA Methods 3060 and 7196

Method Summary

The method uses an alkaline digestion of the solid sample to solubilize both water-insoluble and water-soluble hexavalent chromium compounds. Solid samples are extracted with hot 3 percent sodium carbonate-2 percent sodium hydroxide solution to dissolve hexavalent chromium and to protect the extracted chromium from reduction to trivalent chromium. Water samples are processed without the alkaline extraction procedure.

Dissolved hexavalent chromium is determined colorimetrically by reaction with diphenylcarbazide in acid solution. A red-violet color of unknown composition is produced. Addition of an excess of diphenylcarbazide yields the red-violet product, and its absorbance is measured photometrically at 540 nanometers.

Interferences

The following conditions can interfere with the determination of hexavalent chromium in water and solids:

- Wastes containing high amounts of buffering capacity may require additional digestion solution to properly digest the sample.

- The chromium reaction with diphenylcarbazide is usually free from interferences. However, certain substances may interfere if the chromium concentration is relatively low. Hexavalent molybdenum and mercury salts also react to form color with the reagent; however, the red-violet intensities produced are much lower than those for chromium at the specified pH. Concentrations of up to 200 mg/ℓ of molybdenum and mercury can be tolerated. Vanadium interferes strongly, but concentrations up to 10 times that of chromium will not cause interference.

- Iron in concentrations greater than 1 mg/ℓ may produce a yellow color. However, no difficulty is normally encountered if the absorbance is measured photometrically at the appropriate wavelength.

Instrumentation and Operating Conditions

A spectrophotometer will be used for hexavalent chromium analysis. A one-centimeter path length at a wavelength of 540 nanometers will be used.

Materials and Reagents

All volumetric glassware will be Type A and traceable to NBS standards. Reagents and chemicals used for standard preparation and sample processing will be ultrahigh purity or ACS reagent grade as appropriate.

Standards

Commercially available certified standards will be used. Alternatively, stock standard solutions may be prepared in the laboratory in accordance with appropriate SW-846 procedures. The following standards are prepared from the stock standard solution:

- Working Standards
  Make successive dilutions of the stock chromium standard to prepare a set of three calibration standards within the working range of the spectrophotometer.

- Instrument Check Standard
  Prepare dilutions of the stock standard to obtain a working standard at the midpoint of the calibration curve.

- Matrix Spike Standard
  Prepare dilutions of the stock standard to obtain a matrix spiking standard at the appropriate concentration to achieve a spike concentration approximately twice the original sample concentration.

Analysis

The following summary briefly describes the procedure for the analysis of samples:

- Set up the spectrophotometer in accordance with the manufacturer's instructions and the appropriate analytical procedure.

- Water samples may be analyzed directly by the colorimetric procedure. Solid samples must be processed by the alkaline extraction procedure below:

  - Place 100 grams wet weight of the waste into a 600-milliliter beaker.

  - Determine the percent moisture in the sample on a separate aliquot.

  - Add 400 milliliters of digestion solution. Cover the beaker with the watch glass and heat it to near boiling on a hot plate with constant mixing for 30 to 45 minutes. Do not allow the sample to go to dryness because hexavalent chromium may be lost due to side reactions in the waste.

  - Cool the solution and transfer it quantitatively to the filtration apparatus with deionized, distilled water rinses and filter. Rinse the inside of the filter flask and filter pad with deionized, distilled water and transfer the filtrate and the rinses to a one-liter volumetric flask.

  - If the sample will not be immediately analyzed, it should be stored at a high pH. Just prior to analysis, place a magnetic stirring bar into the flask, place the flask on a stirrer, and, with constant stirring, slowly add concentrated nitric acid to the flask in small aliquots. Bring the pH of the solution to between 7 and 8.

    CAUTION: Carbon dioxide will be evolved.

  - Remove the stirring bar and rinse the bar into the flask. Dilute the contents of the flask to the mark with deionized, distilled water.

- Calibrate the instrument according to the SW-846 recommended procedures using a calibration curve consisting of a blank and three calibration standards within the working range.

- Begin the sample run by analyzing the U.S. EPA initial calibration verification solution. Analyze the instrument check standard (midpoint calibration standard) and the method (preparation) blank every 10 samples. Reanalyze the entire calibration curve at the end of the sample run.

- Analyze the samples and standards by the following technique. Transfer 95 milliliters of the extract to be tested to a 100-milliliter volumetric flask. Add 2 milliliters of diphenylcarbazide solution and mix. Add the sulfuric acid (H_2SO_4) solution to give a pH of 2 ±0.5, dilute to 100 milliliters with Type II water, and let stand 5 to 10 minutes for full color development. Transfer an appropriate portion of the solution to a one-centimeter absorption cell and measure its absorbance at 540 nanometers. Use Type II water as a reference. Correct the absorbance reading of the sample by subtracting the absorbance of a method (preparation) blank carried through the entire analytical procedure. From the corrected absorbance, determine the mg/ℓ of chromium present by reference to the calibration curve.

Calculations

Correct all absorbance readings for the method blank absorbance and read the equivalent concentration from the calibration curve. Correct solids readings for dilution during the alkaline extraction. Report results in mg/ℓ for water and milligrams per kilogram (mg/kg) dry weight for solids.

Quality Control

The major QC elements of the alkaline extraction and colorimetric determination of hexavalent chromium are summarized below:

- Method (Preparation) Blank
  This blank must contain all the reagents in the same volumes as used in processing the samples. The method blank must be carried through the complete procedure used for analysis of the samples. One method blank should be prepared for every batch of samples or every 20 samples of the same matrix, whichever is more frequent.

- Calibration Standards
  To compensate for possible slight losses of chromium during digestion or other operations of the analysis, treat the chromium standards by the same procedure as the sample. Accordingly, for solids prepared by the alkaline extraction procedure, the calibration standards should be carried through the alkaline extraction procedure to yield the appropriate standard concentrations when diluted to the same final volume as the samples.

- Instrument Check Standard
  The standard is prepared by the analyst at a concentration equivalent to the midpoint of the calibration curve and is analyzed at a frequency of 10 percent throughout the sample run. The check standard is used to determine instrument drift. If agreement is not within 10 percent of the expected value, the analysis is out of control. The analysis must be terminated, the problem corrected, the instrument recalibrated, and the preceding 10 samples reanalyzed.

- U.S. EPA Initial Calibration Verification Solution
  This sample consists of a solution obtained from an outside source having a known concentration value to be used to verify the calibration standards. The sample should be prepared in the same matrix as the calibration standards and in accordance with the instructions provided by the supplier. A U.S. EPA initial calibration verification sample must be used daily or whenever samples are run for the initial calibration verification. If the results are not within ±10 percent of the true value listed for the control sample, prepare a new calibration standard and recalibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

9.4.2.5 Digestion/Distillation and Titrimetric/Colorimetric Determination of Cyanide, U.S. EPA Method 335.2 CLP-M

Method Summary

Cyanide as HCN is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by volumetric titration or colorimetrically.

In the colorimetric measurement, the cyanide is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at a pH less than eight without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-pyrazolone or pyridine-barbituric acid reagent. The absorbance is read at 620 nanometers when using pyridine-pyrazolone and at 578 nanometers when using pyridine-barbituric acid. To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.

The titrimetric measurement uses a standard solution of silver nitrate to titrate cyanide in the presence of a silver-sensitive indicator.

Interference

The following conditions can interfere with the determination of total cyanide in water and solids:

- Sulfides adversely affect the colorimetric and titration procedures. If a drop of the distillate on lead acetate test paper indicates the presence of sulfides, treat 25 milliliters more of the sample than that required for the cyanide determination with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker and, from the filtrate, measure the sample to be used for analysis. Avoid a large excess of cadmium carbonate and a long contact time in order to minimize loss by complexation or occlusion of cyanide on the precipitated material. Sulfides should be removed prior to preservation with sodium hydroxide.

- The presence of surfactants may cause the sample to foam during refluxing. If this occurs, the addition of an agent such as Dow Corning 544 antifoam agent will prevent the foam from collecting in the condenser. Fatty acids will distill and form soaps under alkaline titration conditions, making the end point almost impossible to detect. When this occurs, one of the spectrophotometric methods should be used.

- Generally, interferences can be eliminated or reduced by using the digestion/distillation procedure referenced in U.S. EPA Method 335.2 CLP-M.

Instrumentation and Operating Conditions

A spectrophotometer will be used for cyanide analysis. A one centimeter path length at 578 or 620 nanometers will be used in accordance with the manufacturer's instructions.

Materials and Reagents

All volumetric glassware will be Type A and traceable to NBS standards. Reagents and chemicals used for standard preparation and sample processing will be ultrahigh purity or ACS reagent grade as appropriate.

Standards

Commercial certified standards will be used whenever available. Alternately, stock standard solutions may be prepared in the laboratory in accordance with CLP procedures. The following standards are prepared from the stock standard solution:

- Working Standards
  Make successive dilutions of the stock cyanide standard to prepare a set of three calibration standards within the working range of the spectrophotometer.

- Instrument Check Standard
  Prepare dilutions of the stock standard to obtain a working standard at the midpoint of the calibration curve.

- Distillation Check Standard
  Prepare dilutions of the stock standard to obtain a standard of appropriate concentration to be equivalent to the midpoint calibration standard after being carried through the digestion/distillation procedure. It is not imperative that all calibration standards be distilled in the same manner as the samples. At least one standard (midrange) must be distilled and compared to similar values on the curve to ensure that the distillation technique is reliable. If the distilled standard does not agree within ±15 percent of the undistilled standards, the operator should find and correct the cause of the apparent error before proceeding.

- Matrix Spike Standard
  Prepare dilutions of the stock standard to obtain a matrix spiking standard at the appropriate concentration to achieve a spike concentration of 100 µg/ℓ in the final digestate of the spiked sample.

Analysis

The following summary briefly describes the procedure for the analysis of samples:

- Digestion/Distillation

  - Place 500 milliliters of sample, an aliquot diluted to 500 milliliters, or, in the case of solids, five grams of sample in the one liter boiling flask. Add 50 milliliters of sodium hydroxide to the absorbing tube and dilute, if necessary, with distilled water to obtain an adequate depth of liquid in the absorber. Connect the boiling flask, condenser, absorber, and trap in the train.

  - Determine the percent moisture in the sample on a separate aliquot for later correction of solids results to a dry weight basis.

  - Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately one bubble of air per second enters the boiling flask through the air inlet tube.

  - The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It will be necessary to readjust the air rate occasionally to prevent the solution in the boiling flask from backing up into the air inlet tube.

  - Slowly add 25 milliliters concentrated sulfuric acid through the air inlet tube. Rinse the tube with distilled water and allow the air flow to mix the flask contents for three minutes. Pour 20 milliliters of magnesium chloride solution into the air inlet and wash down with a stream of water.

  - Heat the solution to boiling, taking care to prevent the solution from backing up into and overflowing from the air inlet tube. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, disconnect absorber and close off the vacuum source.

- Drain the solution from the adsorber into a 250 milliliter volumetric flask and bring up to volume with distilled water washings from the absorber tube.

- Titrimetric Determination

  - If the sample contains more than one milligram of cyanide, transfer the distillate, or a suitable aliquot diluted to 250 milliliters, to a 500 milliliter Erlenmeyer flask. Adds 10 to 12 drops of the benzalrhodanine indicator.

  - Titrate with standard silver nitrate to the first change in color from yellow to brownish-pink. Titrate a distilled water blank using the same amount of sodium hydroxide and indicator as in the sample.

  - The analyst should familiarize himself/herself with the end point of the titration and the amount of indicator to be used before actually titrating the samples. A five- or ten-milliliter microburet may be conveniently used to obtain a more precise titration.

- Colorimetric Determination

  - Set up the spectrophotometer in accordance with the manufacturer's instructions and the appropriate analytical procedure.

  - Calibrate the instrument according to U.S. EPA Method 335.2 CLP-M recommended procedures using a calibration curve consisting of a blank and three calibration standards within the working range.

  - Begin the sample run by analyzing the U.S. EPA initial calibration verification solution. Analyze the instrument check standard (midpoint calibration standards) and the methods (preparation) blank every 10 samples. Analyze the distillation check standard (midpoint calibration standard carried through the digestion/distillation procedure) with its corresponding set of samples. Reanalyze the entire calibration curve at the end of the sample run.

- Prepare a standard curve by plotting standard absorbance against the cyanide concentration in micrograms per 250 milliliters of original sample.

Calculations

Calculate the cyanide concentration according to the following equations from the CLP Method in WA-87-K026 depending upon the analytical method used and the sample matrix (water or solids):

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- Titrimetric

  - Water:

$$CN,\ mg/l = \frac{(A-B)\ 1{,}000\ ml/l}{ml\ orig.\ sample} \times \frac{250\ ml}{ml\ of\ aliquot\ titrated}$$

  - Solids:

$$CN,\ mg/kg = \frac{(A - B) \times \frac{250\ ml}{ml\ aliquot\ titrated} \times 1000\ g/kg}{C \times \frac{\%\ solids}{100}}$$

where

A = milliliters of silver nitrate ($AgNO_3$) for titration of sample (1 milliliter = 1 milligram Ag)

B = milliliters of $AgNO_3$ for titration of blank (1 milliliter = 1 milligram Ag)

C = wet weight of original sample in grams.

and

250 milliliters = volume of distillate, and

1,000 grams per kilogram = conversion factor grams to kilograms

mg/l aliquot titrated

- Colorimetric:

  - Water:

$$CN,\ \mu g/l = \frac{A \times 1{,}000\ ml/l}{B} \times \frac{50\ ml}{C}$$

where

A = µg CN read from standard curve (per 250 milliliters)

B = milliliters of original sample for distillation, and

C = milliliters taken for colorimetric analysis

and

50 milliliters = volume of original sample aliquot.

  - Solids:

$$CN,\ mg/kg = \frac{A \times \frac{50\ ml}{B}}{C \times \frac{\%\ solids}{100}}$$

where

A = µg CN read from standard curve (per 250 milliliters)

B = milliliters of distillate taken for colorimetric determination

C = wet weight of original sample in grams

and

50 milliliters = volume of standard taken for colorimetric determination.

Quality Control

The major QC elements of the titrimetric and colorimetric determination of cyanide are summarized below:

- Method (Preparation) Blanks
  This blank must contain all the reagents in the same volumes as used in processing the samples. The method blank must be carried through the complete procedure used for analysis of the samples. One method blank should be prepared for every batch of samples or for every 20 samples, whichever is more frequent.

- Instrument Check Standard
  This standard is prepared by the analyst at a concentration equivalent to the midpoint of the calibration curve and is analyzed at a frequency of 10 percent throughout the sample run. The check standard is used to determine instrument drift. If agreement is not within 85 to 115 percent of the expected value, the analysis is out of control. The analysis must be terminated, the problem corrected, the instrument recalibrated, and the preceding 10 samples reanalyzed.

- U.S. EPA Initial Calibration Verification Sample
  This sample consists of a solution obtained from an outside source having a known concentration value to be used to verify the calibration standards. The sample should be prepared in the same matrix as the calibration standards and in accordance with the instructions provided by the supplier. An initial calibration verification solution must be used daily or whenever samples are run for the initial calibration verification. If the results are not within ±10 percent of the true value listed for the control sample, prepare a new calibration standard and recalibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

- Distillation Check Standard
  It is not imperative that all calibration standards be distilled in the same manner as the samples. At least one standard (midrange) must be distilled and compared to similar values on the curve to ensure that the distillation technique is reliable. If the distilled standard does not agree within ±15 percent of the undistilled standards, the operator should find and correct the cause of the apparent error before proceeding.

9.4.2.6 Bellack Distillation and Specific Ion Electrode Determination of Fluoride, U.S. EPA Method 340.2

Method Summary

Following distillation to remove interferences, fluoride is determined potentiometrically using a fluoride electrode in conjunction with a standard, single-junction, sleeve-type reference electrode and a pH meter having an expanded millivolt scale or a selective ion meter having a direct concentration scale for fluoride.

Interference

Extremes of pH interfere; sample pH should be between five and nine. Polyvalent cations of Si^{+4}, Fe^{+3}, and Al^{+3} interfere by forming complexes with fluoride. The degree of interference depends upon the concentration of the complexing cations, the concentration of fluoride, and the pH of the sample. The addition of a pH 5.0 buffer containing a strong chelating agent preferentially complexes aluminum (the most common interference), silicon, and iron and eliminates the pH problem.

Instrumentation and Operating Conditions

An Orion 901 Ion analyzer with a fluoride-specific ion electrode will be used in accordance with the manufacturer's instructions for fluoride analysis of water and solids.

Materials and Reagents

All volumetric glassware will be Type A and traceable to NBS standards. Reagents and chemicals used for standard preparation and sample processing will be ultrahigh purity or ACS reagent grade as appropriate.

Standards

Commercial certified standards will be used whenever available. Alternately, stock standard solutions may be prepared in the laboratory in accordance with CLP procedures. The following standards are prepared from the stock standard solution:

- Working Standards
  Make successive dilutions of the stock cyanide standard to prepare a minimum of three calibration standards within the working range of the specific ion meter.

- Instrument Check Standard
  Prepare dilutions of the stock standard to obtain a working standard at the midpoint of the calibration curve.

- Distillation Check Standard
  Prepare dilutions of the stock standard to obtain a standard of appropriate concentration to be equivalent to the midpoint calibration standard after processing through the distillation procedure. It is not imperative that all calibration standards be distilled in the same manner as the samples. At least one standard (midrange) must be distilled and compared to similar values on the curve to ensure that the distillation technique is reliable. If the distilled standard does not agree within ±15 percent of the undistilled standards, the operator should find and correct the cause of the apparent error before proceeding.

- Matrix Spike Standard
  Prepare dilutions of the stock standard to obtain a matrix spiking standard at the appropriate concentration to achieve a spike concentration of 1.0 mg/l in the final digestate of the spiked sample. The spiking concentration may be adjusted if problems are encountered with the 1.0 mg/l spike concentration.

Analysis

The following summary briefly describes the procedure for the analysis of samples:

- Bellack Distillation for Solids

  - Place 400 milliliters distilled water in the distilling flask.

  - Carefully add 200 milliliters concentrated H_2SO_4 and swirl until contents are homogeneous. Add 25 to 35 glass beads, connect the apparatus making sure all joints are tight.

  - Heat slowly at first, then as rapidly as the efficiency of the condenser will permit (distillate must be cool) until the temperature of the flask contents reaches exactly 180 degrees Celsius. Discard the distillate. This process removes fluoride contamination and adjusts the acid-water ratio for subsequent distillations.

  - Cool to 120 degrees Celsius or below.

  - Add 300 milliliters sample or 5 to 10 grams for solids, mix thoroughly, distill until temperature reaches 180 degrees Celsius. Do not heat above 180 degrees Celsius to prevent sulfate carryover.

  - Add silver sulfate (Ag_2SO_4) at a rate of five milligrams per milligram (mg/mg) Cl when high-chloride samples are distilled.

  - Use the sulfuric acid solution in the flask repeatedly until the contaminants from the samples accumulate to such an extent that recovery is affected or interferences appear in the distillate. Check periodically by distilling standard fluoride samples. High-fluoride samples may require that the still be flushed by using distilled water and combining distillates.

  - Determine the percent moisture of the solid sample on a separate sample aliquot.

- Specific Ion Electrode Determination

  - Set up the specific ion meter in accordance with the manufacturer's instructions and the analytical procedure.

  - Calibrate the specific ion meter according to U.S. EPA Method 340.2 recommended procedures using a

calibration curve consisting of a blank and three calibration standards within the working range.

- Place 50 milliliters of sample or standard solution and 50 milliliters of buffer in a 150-milliliter beaker. Place on a magnetic stirrer and mix at medium speed.

- Immerse the electrodes in the solution and observe the meter reading while mixing. The electrodes must remain in the solution for at least three minutes or until the reading has stabilized. At concentrations under 0.5 mg/l fluoride, it may require as long as five minutes to reach a stable meter reading; high concentrations stabilize more quickly.

- If a pH meter is used, record the potential measurement for each unknown sample and convert the potential reading to the fluoride ion concentration of the unknown using the standard curve. If a selective ion meter is used, read the fluoride level in the unknown sample directly in mg/l on the fluoride scale.

Calculations

The fluoride concentration is read directly from the meter in mg/l. Solids samples must be corrected for the weight of sample carried through the distillation procedure and reported in mg/kg on a dry weight basis.

Quality Control

The major QC elements of the Bellack distillation specific ion electrode determination of fluoride are summarized below:

- Method (Preparation) Blanks
  This blank must contain all the reagents in the same volumes as used in processing the samples. The method blank must be carried through the complete procedure used for analysis of the samples. One method blank should be prepared for every 20 samples of the same matrix or batch of samples, whichever is more frequent.

- Instrument Check Standard
  This standard is prepared by the analyst at a concentration equivalent to the midpoint of the calibration curve and is analyzed at a frequency of 10 percent throughout the sample run. The check standard is used to determine instrument drift. If agreement is not within 85 to 115 percent of the expected value, the analysis is out of control. The analysis must be terminated, the problem corrected, the instrument recalibrated, and the preceding 10 samples reanalyzed.

- U.S. EPA Initial Calibration Verification Sample
  This sample consists of a solution obtained from an outside source having a known concentration value to be used to verify the calibration standards. The sample should be prepared in the same matrix as the calibration standards and in accordance with the instructions provided by the supplier. An initial calibration verification solution must be used daily or whenever samples are run for the initial calibration verification. If the results are not within ±10 percent of the true value listed for the sample, prepare a new calibration standard and recalibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

- Distillation Check Standard
  It is not imperative that all calibration standards be distilled in the same manner as the samples. At least one standard (midrange) must be distilled and compared to similar values on the curve to ensure that the distillation technique is reliable. If the distilled standard does not agree within ±15 percent of the undistilled standards, the operator should find and correct the cause of the apparent error before proceeding.

9.4.2.7 Colorimetric Determination of Nitrate Nitrogen, U.S. EPA Method 352.1

Method Summary

This method is based upon the reaction of the nitrate ion with brucine sulfate in a 13 N H_2SO_4 solution at a temperature of 100 degrees Celsius. The color of the resulting complex is measured at 410 nanometers. Temperature control of the color reaction is extremely critical.

Interference

The following conditions can contribute to analytical interferences:

- Dissolved organic matter will cause an off color in 13 N H_2SO_4 and must be compensated for by additions of all reagents except the brucine-sulfanilic acid reagent. This also applies to natural color present not due to dissolved organics.

- The effect of salinity is eliminated by addition of sodium chloride to the blanks, standards, and samples.
- All strong oxidizing or reducing agents interfere. The presence of oxidizing agents may be determined with a total residual chlorine test kit.
- Residual chlorine interference is eliminated by the addition of sodium arsenite.
- Ferrous and ferric iron and quadrivalent manganese give slight positive interferences, but in concentrations less than 1 mg/l, these interferences are negligible.
- Uneven heating of the samples and standards during the reaction time will result in erratic values. The necessity for absolute control of temperature during the critical color development period cannot be too strongly emphasized.

Instrumentation and Operating Conditions

A spectrophotometer will be used for nitrate determinations in accordance with the manufacturer's instructions. Measurements will be performed with a one-centimeter path length of 410 nanometers.

Materials and Reagents

All volumetric glassware will be Type A and traceable to NBS standards. Reagents and chemicals used for standard preparation and sample processing will be ultrahigh purity or ACS reagent grade as appropriate.

Standards

Commercial certified standards will be used whenever available. Alternately, stock standard solutions may be prepared in the laboratory in accordance with CLP procedures. The following standards are prepared from the stock standard solution:

- Working Standards
  Make successive dilutions of the stock standard to prepare a minimum of three calibration standards within the working range of the spectrophotometer.

- Instrument Check Standard
  Prepare dilutions of the stock standard to obtain a working standard at the midpoint of the calibration curve.

- Matrix Spike Standard
  Prepare dilutions of the stock standard to obtain a matrix spiking standard at the appropriate concentration to achieve a spike concentration of 1.0 mg/l in the final digestate of the spiked sample. The spiking concentration may be adjusted if problems are encountered with the 1.0 mg/l spike concentration.

Analysis
The following summary briefly describes the procedure for the analysis of the samples:

- Adjust the pH of the samples to approximately seven by using acetic acid or sodium hydroxide. If necessary, filter to remove turbidity.

- Set up the required number of sample tubes in the rack to handle the method blank, standards, and samples. Space tubes evenly throughout the rack to allow for even flow of bath water between the tubes. This should assist in achieving uniform heating of all tubes.

- If it is necessary to correct for color or dissolved organic matter which will cause color on heating, a set of duplicate samples must be run to which all reagents except the brucine-sulfanilic acid have been added.

- Pipet 10.0 milliliters of standards and samples or an aliquot of the samples diluted to 10.0 milliliters into the sample tubes.

- If the samples are saline, add two milliliters of the 30 percent sodium chloride solution to the method blank, standards, and samples. For fresh water samples, sodium chloride solution may be omitted. Mix contents of tube by swirling and place rack in cold water bath (zero to 10 degrees Celsius).

- Pipet 10.0 milliliters of sulfuric acid solution into each tube and mix by swirling. Allow tubes to come to thermal equilibrium in the cold bath. Be sure that temperatures have equilibrated in all tubes before continuing.

- Add 0.5 milliliter of brucine-sulfanilic acid reagent to each tube (except the interference control tubes), and carefully mix by swirling, then place the rack of tubes in the 100 degree Celsius water bath for exactly 25 minutes. Caution: Immersion of the tube rack into

the bath should not decrease the temperature of the bath more than one to two degrees Celsius. To keep this temperature decrease to an absolute minimum, flow of bath water between the tubes should not be restricted by crowding too many tubes into the rack. If color development in the standards reveals discrepancies in the procedure, the operator should repeat the procedure after reviewing the temperature control steps.

- Remove rack of tubes from the hot water bath, immerse in the cold water bath and allow to reach thermal equilibrium (20 to 25 degrees Celsius). Read absorbance against the reagent blank at 410 nanometers using a one centimeter or longer cell.

Calculations

Obtain a standard curve by plotting the absorbance of standards run by the above procedure against milligrams of nitrate-nitrogen per liter (NO_3-N/l). (The color reaction does not always follow Beer's Law.) Subtract the absorbance of the sample without the brucine-sulfanilic reagent from the absorbance of the sample containing brucine-sulfanilic acid and determine milligrams NO_3-N/l. Multiply by an appropriate dilution factor if less than 10 milliliters of sample are taken.

Quality Control

The major QC elements of the colorimetric determination of nitrate-nitrogen are summarized below:

- Method (Preparation) Blanks
  This blank must contain all the reagents in the same volumes as used in processing the samples. The method blank must be carried through the complete procedure used for analysis of the samples. One method blank should be prepared for every 20 samples of the same matrix or batch of samples, whichever is more frequent.

- Instrument Check Standard
  This standard is prepared by the analyst at a concentration equivalent to the midpoint of the calibration curve and is analyzed at a frequency of 10 percent throughout the sample run. The check standard is used to determine instrument drift. If agreement is not within 85 to 115 percent of the expected value, the analysis is out of control. The analysis must be terminated, the problem corrected, the instrument recalibrated, and the preceding 10 samples reanalyzed.

- U.S. EPA Initial Calibration Verification Solution
  This sample consists of a solution obtained from an outside source having a known concentration value to be used to verify the calibration standards. The sample should be prepared in the same acid matrix as the calibration standards and in accordance with the instructions provided by the supplier. An initial calibration verification solution must be used daily or whenever samples are run for the initial calibration verification. If the results are not within ±10 percent of the true value listed for the solution, prepare a new calibration standard and recalibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard, and repeat the calibration.

9.4.3 ORGANIC ANALYSIS

Samples specified for organic analysis will be analyzed for the U.S. EPA HSL organic compounds. These compounds are divided by the analytical procedures into the following three categories:

- Volatile organics;
- Semivolatile organics; and
- Pesticides and PCBs.

The analytical program will follow the methodologies outlined under Exhibit D - U.S. EPA, 1985, "Chemical Analytical Services for Multi-Media Multi-Concentration Organics, GC/MS Techniques," WA-87-K236, Washington, D.C., and will include the following conditions:

- Scans will be conducted for the HSL compounds identified in Table 4-2 of this QAPP.

- Unknown peaks with heights greater than 10 percent of the nearest internal standard will be tentatively identified by a library search of the U.S. EPA/ National Institute of Health (NIH) mass spectral library.

9.4.3.1 Volatile Organics

Method Summary

An inert gas is bubbled through a 5-milliliter water sample or a known weight of soil sample suspended in water which is contained

in a specially designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the purgeables are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer. The HSL volatile compounds are identified in Table 9-2.

Interference

Impurities in the purge gas, organic compounds outgassing the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems.

Samples can be contaminated by diffusion of volatile organics through the septum seal into the sample during shipment and storage.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carry over, the purging device and sample and purging of the entire systems may be required after unusually high amounts of volatiles are analyzed.

Analysis Rate

Instrumental analysis by gas chromatography/mass spectrometry (GC/MS), including blanks, duplicates, and standards, will allow four samples to be analyzed in an eight-hour period.

Instrumentation and Operating Conditions

The following instrumentation and operating conditions will be used to perform HSL volatile compound analyses:

TABLE 9-2

VOLATILE INTERNAL STANDARDS WITH CORRESPONDING
HSL ANALYTES ASSIGNED FOR QUANTIFICATION
FEED MATERIALS PRODUCTION CENTER
FERNALD, OHIO

BROMOCHLOROMETHANE	1,4-DIFLUOROBENZENE	CHLOROBENZENE-d_5
Chloromethane	2-Butanone	2-Hexanone
Bromomethane	1,1,1-Trichloroethane	4-Methyl-2-Pentanone
Vinyl Chloride	Carbon Tetrachloride	Tetrachloroethene
Chloroethane	Vinyl Acetate	1,1,2,2-Tetrachloroethane
Methylene Chloride	Bromodichloromethane	Toluene
Acetone	1,2-Dichloropropane	Chlorobenzene
Carbon Disulfide	trans-1,3-Dichloropropene	Ethylbenzene
1,1-Dichloroethene	Trichloroethene	Styrene
1,1-Dichloroethane	Dibromochloromethane	Total Xylenes
trans-1,2-Dichloroethene	1,1,2-Trichloroethane	Bromofluorobenzene(a)
Chloroform	Benzene	
1,2-Dichloroethane	cis-1,3-Dichloropropene	Toluene-d_8(a)
1,2-Dichloroethane-d_4(a)	2-Chloroethyl Vinyl Ether	
	Bromoform	

(a)The indicated compound is a surrogate standard.

- GC/MS, including

  - GC/MS glass jet interface;

  - Data system capable of continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program; and

  - Tekmar LSC-2 purging system.

- Tekmar Purge/Trap Conditions

  - Purge time - 9 minutes at ambient temperature
  - Dry purge - 4 minutes
  - Desorb time - 5 minutes at 125 degrees Celsius
  - Bake time - 30 minutes at 180 degrees Celsius
  - Purge gas - Helium, 50 milliliters per minute
  - Sample size - 1 microliter to 5 milliliters.

- GC/MS Conditions

  - GC Column - 6-foot-by-2-millimeter inside-diameter (ID) glass column packed with 1 percent SP-1000 on 60/80 mesh Carbopack B

  - Column Program - 45 degrees Celsius for three minutes, then programmed at 8 degrees Celsius per minute to 220 degrees Celsius and held for 15 minutes

  - Column Flow - 30 milliliters per minute

  - Injector Temperature - 180 degrees Celsius

  - Transfer Line Temperatures - 180 degrees Celsius

  - Source Temperature - according to manufacturer's specifications

  - Detection Mode - electron impact

  - Scan Range - 35 to 260 amu at 3 seconds per scan

  - Ionization Energy - 70 ev.

Materials and Reagents

All volumetric glassware will be Type A and traceable to NBS standards. All analytical standards and chemical reagents used for the analysis of HSL volatile compounds will be ultrahigh purity or ACS reagent grade as appropriate.

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Standards

The following procedures summarize the appropriate CLP protocol for generating calibration, internal, surrogate, matrix spike, and tuning standards:

- Stock Volatile Standards (1 µg/µl)
  Stock standard solutions are prepared from pure standard materials. The gases are purchased as certified solutions. Place about 45 milliliters of methanol in a 50-milliliter ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about five minutes or until all alcohol wetted surfaces have dried. Stock standard solutions are prepared from pure standard materials. The gases are purchased as certified solutions. Place about 45 milliliters of methanol in a 50-milliliter ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about five minutes or until all alcohol wetted surfaces have dried. Using a 50-microliter syringe, add several microliters of each compound to the methanol, making sure that the material falls directly into the alcohol. This procedure is performed on an analytical balance and the weight of the added compound is recorded for calculation of the compound concentration in the stock standard. Dilute to volume and transfer the stock standard solution into a Teflon-sealed screw-cap bottle and store at -10 to -20 degrees Celsius. Allow a minimum of headspace and protect from the light. This standard may be kept for as long as one month. Each standard is recorded in a bound standards log with a unique ID number assigned to each bottle. Concentrations of each analyte are based on the weight added and the final volume of the solution.

  Alternately, stock standards may be obtained from either the U.S. EPA Quality Assurance Materials Bank or a commercial source, but must be traceable to the U.S. EPA's Las Vegas, Nevada Environmental Monitoring and Support Laboratory (EMSL).

- Secondary Dilution Standard
  The secondary standard is prepared by diluting the stock standard with methanol in a 100-milliliter volumetric flask. Dilute to volume and transfer the solution into a teflon-sealed screw-top bottle. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation. Record in the bound standards logbook.

- Working Standards

The working standard is prepared by adding the appropriate amount of the secondary dilution standard into 5 milliliters of deionized water. For convenience, the 5 milliliters have already been placed in the 5-milliliter syringe used for injection of samples into the Tekmar purge and trap device. Initial calibration is required at 20, 50, 100, 150, and 200 µg/l per each compound. Continuing calibration requires the analysis of a 50 µg/l composite standard. The internal standards used for quantitating specific HSL volatile compounds are presented in Table 9-2.

- Internal and Surrogate Stock Standards
  These may be prepared separately or together in the same bottle and in the same manner as outlined above in "Stock Volatile Standards." Typically, three surrogates and three internal standards are used. Surrogate compounds are 1,2-dichloroethane-d_4, p-bromofluorobenzene, and toluene-d_8. Internal standards are bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d_5. Record standards in the bound standards log book.

- Internal and Surrogate Standard Spiking Solutions
  These solutions may be prepared together or separately as outlined in "Secondary Dilution Standard." The surrogate standard spiking solution contains 250 micrograms per 10 milliliters of each compound in methanol. The internal standard spiking solution is prepared to contain 25 µg/ml of each internal standard in methanol. When 10 microliters of each of these solutions is spiked into 5 milliliters of water, the following concentrations are obtained: 1,2-dichloroethane-d_4 (50 µg/l), p-bromofluorobenzene (50 µg/l), toluene-d_8 (50 µg/l), bromochloromethane (50 µg/l), 1,4-difluorobenzene (50 µg/l), and chlorobenzene-d_5 (50 µg/l).

  When 10 microliters of each of these solutions is added to 5 grams of solid sample, the equivalent concentration for each internal and surrogate standard compound is 50 µg/kg.

- Organic Matrix Standard Spiking Solution
  Prepare a spiking solution in methanol that contains the following compounds at a concentration of 250 microliters per 10.0 milliliters:

  - 1,1-Dichloroethene;
  - Trichloroethene;
  - Chlorobenzene;
  - Toluene;
  - Benzene.

Matrix spikes also serve as duplicates. When 10 microliters of the matrix spike solution is added to a 5-milliliter water sample or a 5-gram solids sample, the equivalent concentration is equal to 50 µg/l or 50 µg/kg for each matrix spike compound, respectively.

- Bromofluorobenzene (BFB) Standard
  Prepare a 25 nanograms per microliter (ng/µl) solution of BFB in methanol.

Calibration

The following procedures summarize the GC/MS calibration process:

- Tune up GC/MS system as outlined in Section 11.4.2.1.
- Calibrate using the five calibration standards as outlined in Section 11.4.2.2.
- Calculate response factors (RF) and evaluate calibration and system performance check compound responses as outlined in Section 11.4.2.3.

GC/MS Quantitation of HSL Volatile Compounds

Calculate the compound concentrations in the sample as determined using the following equations from the CLP methods in WA-87-K236.

- Water

$$\text{Concentration } \mu g/l = \frac{(A_x)(I_s)}{(A_{is})(RF)(V_o)}$$

where

A_x = area of the characteristic ion for the compound to be measured

A_{is} = area of the characteristic ion for the internal standard

I_s = amount of internal standard injected in nanograms

V_o = volume of water extracted in milliliters, and

RF = the response factor calculated for the compound in interest.

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- Sediment/Soil

$$\text{Concentration } \mu g/l = \frac{A_x(I_s)}{(A_{is})(RF)(W_s)(D)}$$

where

A_x, I_s, A_{is}, RF = same as given for water, above

$$D = \frac{100 - \text{percent moisture}}{100}$$

W_s = Weight of sample extracted (grams).

Tentative Identification of Non-HSL Volatile Compounds

A library search will be executed for non-HSL sample components for the purpose of tentative identification. For this purpose, a recent version of the EPA/NIH Mass Spectral Library will be used. Computer-generated library search routines will not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Up to 10 non-HSL substances of greatest apparent concentration will be tentatively identified via a forward search of the U.S. EPA/NIH mass spectral library. (Substances with responses less than 10 percent of the internal standard are not required to be searched in this fashion.) Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification according to the following criteria:

- Relative intensities of major ions in the reference spectrum (ions greater than 10 percent of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20 percent. (Example: For an ion with an abundance of 50 percent of the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.)
- Molecular ions present in reference spectrum should be present in sample spectrum.

- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds. Data system library reduction programs can sometimes create these discrepancies.

If, in the opinion of the mass spectral specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (i.e., unknown aromatic, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, include them. An estimated concentration for non-HSL components tentatively identified will be quantified by the internal standard method. For quantification, the nearest internal standard free of interferences shall be used.

The formula for calculating concentrations is the same as in GC/MS Quantitation of HSL Volatile Compounds above. Total area counts from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A response factor (RF) of one is to be assumed. The value from this quantitation shall be qualified as estimated. This estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

9.4.3.2 Semivolatile Organics

Method Summary

The method is used for the determination of acid and base-neutral extractable compounds that are partitioned into methylene chloride and are amenable to GC/MS analysis. The HSL acid and base-neutral extractable compounds are identified in Table 4-2.

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A measured volume of water, approximately one liter, is serially extracted with methylene chloride at a pH greater than 11 and again at a pH less than 2, using a separatory funnel or a continuous extractor. The methylene chloride extracts are dried and concentrated separately to a volume of 1 milliliter. In the case of solids, a 30-gram portion is mixed with anhydrous sodium sulfate and extracted with 1:1 methylene chloride/acetone using an ultrasonic probe. Separate 30-gram portions will be extracted for HSL acid and base-neutral extractable compounds and for HSL pesticide and PCB compounds. The HSL acid and base-neutral extract, following any screening or cleanup procedures, is concentrated to a final volume of 1 milliliter in methylene chloride. An aliquot (0.5 milliliter) of the initial 10-milliliter HSL pesticide and PCB extract is cleaned up using a micro alumina column, taken to a final volume of 1 milliliter in hexane, and analyzed by gas chromatography using an electron capture detector (ECD).

Interference

Impurities in solvent, reagent, and glass contamination may lead to elevated baselines in total ion current profiles (TICPs). Co-extracted matrix contaminants may cause interferences.

Analysis Rate

Instrumental analysis by GC/MS, including blanks, duplicates, and standards, will allow four samples to be analyzed in an eight-hour period.

Instrumentation

The following instrumentation and operating conditions will be used to perform HSL acid and base-neutral extractable compound analyses:

GC/MS, including

- GC/MS glass jet interface;

- Data system capable of continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program; and

- GC/MS conditions

  - GC Column - 30 meters by 0.25-millimeter ID fused silica capillary column coated with DB-5 at a 1.0 micron film thickness

  - Column Program - 30 degrees Celsius for 4 minutes, then programmed to 300 degrees Celsius at 8 degrees Celsius per minute and held for 10 minutes

  - Column Flow - Helium at 30 cm/s

  - Injector Type - Splitless, grob type

  - Injector Temperature - 250 to 300 degrees Celsius

  - Transfer Line Temperature - 250 to 300 degrees Celsius

  - Source Temperature - According to manufacturer's specifications

  - Detection Mode - Electron impact

  - Scan Range and Time - 35 to 500 amu at one second per scan

  - Ionization Energy - 70 ev.

Materials and Reagents

All volumetric glassware will be Type A and traceable to NBS standards. All analytical standards and chemical reagents used for the analysis of the Target Compound List (TCL) will be ultrahigh purity or ACS reagent grade as appropriate. The glassware, equipment, and reagents specified in U.S. EPA WA-87-K236 will be used.

Standards

The following procedures summarize the appropriate CLP protocol for generating calibration, internal, surrogate, matrix spike, and tuning standards:

- Calibration Standard
  Prepare calibration standards in methylene chloride at a minimum of five concentration levels. Each calibration standard should contain each compound of interest and each surrogate standard. Recommended GC/MS calibration standard concentrations are 20, 50, 80, 120, and 160 total nanograms. Continuing calibration requires the analysis of the 50 nanogram standard. The internal standards used for quantitating specific HSL acid and base-neutral compounds are presented in Table 9-3.

- Internal Standard
  The following compounds are used as internal standards: 1,4-dichlorobenzene-d_4, napthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, perylene-d_{12}. An internal standard solution is prepared by dissolving 200 milligrams of each compound in 50 milliliters of methylene chloride. It may be necessary to use 5 to 10 percent benzene or toluene in this solution and a few minutes of ultrasonic mixing in order to dissolve all the constituents. The resulting solution will contain each standard at a concentration of 4,000 ng/µℓ. A 10-microliter portion of this solution should be added to each 1 milliliter of sample extract. This will give a concentration of 40 ng/µℓ of each constituent.

- Surrogate Spiking Standards
  The compounds specified as surrogate standards are phenol-d_6, 2,4,6-tribromophenol, 2-fluorophenol, nitrobenzene-d_5, terphenyl-d_{14} and 2-fluorobiphenyl. Prepare a methanol solution containing these compounds at a concentration of 100 µg/mℓ for base-neutral surrogates and at a concentration of 200 ug/mℓ for acid (phenolic) surrogate standards. Add 1.0 milliliter of the surrogate spiking solution to every water sample extracted. Add 0.5 milliliter of the surrogate spiking solution to every solids sample extracted. This will be equivalent to 50 ug/ml and 100 ug/ml in the final extract volume for the base-neutral and acid surrogate standard compounds, respectively.

- Matrix Spiking Standards
  Prepare a spiking solution in methanol that contains the following compounds at a concentration of 100 µg/ℓ for base-neutrals and 200 µg/mℓ for acids:

TABLE 9-3
SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING
HSL ANALYTES ASSIGNED FOR QUANTIFICATION
FEED MATERIALS PRODUCTION CENTER
FERNALD, OHIO

1,4-DICHLOROBENZENE-d_4	NAPHTHALENE-d_8	ACENAPHTHENE-d_{10}	PHENANTHRENE-d_{10}	CHRYSENE-d_{12}	PERYLENE-d_{12}
N-Nitrosodimethylamine	Nitrobenzene	Hexachlorocyclopentadiene	4,6-Dinitro-2-methylphenol		Di-n-octyl phthalate
Phenol	Isophorone	2,4,6-Trichlorophenol	N-nitrosodiphenylamine	Pyrene	Benzo(b)fluoranthene
Aniline	2-Nitrophenol	2,4,5-Trichlorophenol	1,2-Diphenylhydrazine	Butylbenzyl phthalate	Benzo(k)fluoranthene
bis(2-chloroethyl) ether	2,4-Dimethylphenol	2-Chloronaphthalene	4-Bromophenyl phenyl ether	3,3'-Dichlorobenzidine	Benzo(a)pyrene
2-Chlorophenol	Benzoic acid	2-Nitroaniline	Hexachlorobenzene	Benzo(a)anthracene	Indeno(1,2,3-cd)pyrene
1,3-Dichlorobenzene	bis(2-Chloroethoxy)methane	Dimethyl phthalate	Pentachlorophenol	bis(2-ethylhexyl) phthalate	Dibenz(a,h)anthracene
1,4-Dichlorobenzene	2,4-Dichlorophenol	Acenaphthylene	Phenanthrene	Chrysene	Benzo(g,h,i)perylene
Benzyl alcohol	1,2,4-Trichlorobenzene	3-Nitroaniline	Anthracene	Terphenyl-d_{14}(a)	
1,2-Dichlorobenzene	Naphthalene	Acenaphthene	Di-n-butyl phthalate		
2-Methylphenol	4-Chloroaniline	2,4-Dinitrophenol	Fluoranthene		
bis(2-Chloroisopropyl) ether	Hexachlorobutadiene	4-Nitrophenol			
4-Methylphenol	4-Chloro-3-methylphenol	Dibenzofuran			
N-Nitroso-di-n-propylamine	2-Methylnaphthalene	2,4-Dinitrotoluene			
Hexachloroethane	Nitrobenzene-d_5(a)	2,6-Dinitrotoluene			
2-Fluorophenol(a)		Diethyl Phthalate			
Phenol-d_6(a)		4-Chlorophenyl phenyl ether			
		Fluorene			
		4-Nitroaniline			
		2-Fluorobiphenyl(a)			
		2,4,6-Tribromophenol(a)			

(a)The indicated compound is a surrogate standard.

Base-Neutrals	Acids
1,2,4-trichlorobenzene	pentachlorophenol
acenaphthene	phenol
2,4-dinitrotoluene	2-chlorophenol
pyrene	4-chloro-3-methylphenol
N-nitroso-di-n-propylamine	4-nitrophenol
1,4-dichlorobenzene	

Add 1.0 milliliter of the matrix spiking solution to each of two 1-liter aliquots of the water sample selected for spiking. Add 1.0 milliliter of the matrix spiking solution to each of two 30-gram aliquots of the solids sample selected for spiking. This will be equivalent to 100 and 200 µg/mℓ in the final extract volume for the base-neutral and acid matrix spike compounds, respectively.

- Decafluorotriphenyl phosphine (DFTPP) Standard
  Prepare a 25 ng/µℓ solution of DFTPP in methylene chloride.

Calibration

The following procedures summarize the GC/MS calibration process:

- Tune up GC/MS system by injecting 50 nanograms of DFTPP as outlined in Section 11.4.2.1.
- Calibrate using the five calibration standards as outlined in Section 11.4.2.2.
- Calculate response factors and evaluate calibration and system performance check compound responses as outlined in Section 11.4.2.3 for quantification.

GC/MS Quantitation of HSL Acid and Base-Neutral Compounds

Calculate the compound concentrations in the sample as determined using the following equations from the CLP methods in WA-87-K236:

- Water

$$\text{Concentration } \mu g/l = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RF)(V_o)(V_i)}$$

where

A_x = area of the characteristic ion for the compound to be measured,

A_{is} = area of the characteristic ion for the internal standard,

I_s = amount of internal standard injected in nanograms,

V_o = volume of water extracted in milliliters,

RF = response factor calculated for the compound of interest,

V_i = volume of extract injected (microliters), and

V_t = volume of total extract. (Use 2,000 microliters or a factor of this when dilutions are made. The 2,000 microliters are derived from combining half of the one milliliter base-neutral extract and half of the one milliliter acid extract.)

- <u>Sediment/Soil</u>

$$\text{Concentration } \mu g/kg \text{ (Dry Weight Basis)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RF)(V_i)(W_s)(D)}$$

where

A_x = area of the characteristic ion for the compound to be measured,

A_{is} = area of the characteristic ion for the internal standard,

I_s = amount of internal standard injected in nanograms,

V_t = Volume of low-level total extract (use 1,000 µℓ or a factor of this when dilutions are made).

RF = response factor calculated for the compound of interest,

V_i = volume of extract injected (microliters),

$D = \frac{100 - \%\ \text{moisture}}{100}$, and

W_s = weight of sample extracted (grams).

Tentative Identification of Non-HSL Acid/Base-Neutral Compounds

A library search will be executed for non-HSL sample components for the purpose of tentative identification. For this purpose, a recent version of the U.S. EPA/NIH Mass Spectral Library will be used.

Up to 20 non-HSL substances of greatest apparent concentration will be tentatively identified via a forward search of the U.S. EPA/NIH mass spectral library. (Substances with responses less than 10 percent of the internal standard are not required to be searched in this fashion.) Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Computer generated library search routines will not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. The following guidelines will be followed when making the identification:

- Relative intensities of major ions in the reference spectrum (ions greater than 10 percent of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20 percent. (Example: For an ion with an abundance of 50 percent of the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.)
- Molecular ions present in reference spectrum should be present in sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds. Data system library reduction programs can sometimes create these discrepancies.

If, in the opinion of the mass spectral specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (i.e., unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, include them. An estimated concentration for non-HSL components tentatively identified shll be quantified by the internal standard method. For quantification, the nearest internal standard free of interferences shall be used.

The formula for calculating concentrations is the same as in GC/MS Quantitation of HSL Acid and Base Neutral Compounds. Total area counts from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A response factor (RF) of one is to be assumed. The value from this quantitation shall be qualified as estimated. This estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

9.4.3.3 Pesticides and PCBs

Method Summary

Hexane extracts of water and sediment/soil are extracted as described in the Semivolatile Organics Method Summary and analyzed on a GC with an ECD. If pesticides or PCBs are tentatively identified, a second GC/ECD analysis is required using an alternate column. Quantitation must be on a packed column, whereas confirmation can be on either a packed or a capillary column.

Interferences

Method interference may be caused by sample matrix effects or contaminants in solvents, reagents, glassware, and other sample processing hardware. Materials must be routinely demonstrated to be free from interferences under the conditions of the analysis using laboratory method blanks.

Analysis Rate

Instrument analysis GC/ECD, including blanks, duplicates, and standards, allows four to six samples to be analyzed in an eight-hour period.

Instrumentation and Operating Conditions

The following instrumentation and operating conditions will be used to perform HSL pesticide and PCB analysis:

- Gas chromatographs including
  - Data system to measure peak areas/heights and recording retention times;
  - Strip chart recorder with recording integrator;
- GC Condition
  - GC Column No. 1 - 1.8 meters long x 4 millimeters inside diameter (6 millimeters outside diameter) glass column with 1.5 percent OV-17/1.95 percent OV-210 or equivalent on 100/120 mesh Gas Chrom Q;
  - GC Column No. 2 - 1.8 meters long x 2 millimeters inside diameter (6 millimeters outside diameter) glass column with 3 percent OV-1 or equivalent on 100/120 mesh Gas Chrom Q;
  - Column Temperature Program - Isothermal from 190 to 210 degrees Celsius;
  - Column Flow - 30 milliliters per minute of a 95/5 percent argon/methane mixture. Alternately, nitrogen may be used as a carrier gas;
  - Injector Temperature - 225 to 250 degrees Celsius; and
  - Detector Temperature - 300 to 310 degrees Celsius.

Materials and Reagents

All volumetric glassware will be Type A and traceable to NBS standards. All analytical standards and chemical reagents used for the analysis of HSL pesticide and PCB compounds will be ultrahigh purity or ACS reagent grade, as appropriate.

Standards

The following procedures summarize the appropriate CLP protocol for generating calibration, evaluation, surrogate, and matrix spike standards:

- Stock Standards
  Stock standard solutions [1.0 microgram per microliter (µg/µl)] can be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions by accurately weighing about 0.0100 gram of pure material. Dissolve the material in toluene; dilute to volume in a 10-milliliter volumetric flask with isooctane. Transfer the stock standard solutions into an appropriate size bottle/vial with a teflon-lined septum. Store at 4 degrees Celsius and protect from light. Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem. Commercially prepared stock standards can be used if they are traceable to U.S. EPA, Las Vegas, Environmental Monitoring and Support Laboratory supplied standards.

- Working Standards
  Prepare mixtures of standards diluted with hexane that will provide approximately half full-scale response for all the compounds of interest. (This would be approximately 0.010 ng/µl for aldrin.) All individual component standards can be in one mixture if all compounds have a 25 percent or greater resolution between peaks. Include dibutyl-chlorendate in the mixture. All multicomponent standards, i.e., PCB aroclors, chlordane, and toxaphene, must be in separate solutions with the exception of Aroclors 1016/1260.

- Evaluation Standards
  Prepare working standard mixtures diluted with hexane containing aldrin, endrin, 4,4'-DDT, and dibutylchlorendate to evaluate the GC column. Prepare three concentration levels according to the following general criteria:

  - The low-level evaluation standard will provide compound responses at approximately 20 percent of scale.

  - The middle-level evaluation standard will provide compound responses at approximately 50 percent of scale.

  - The high-level evaluation standard will provide compound responses at approximately 100 percent of scale. The dibutylchlorendate concentration in the high standard must equal 0.1 µg/ml.

- Surrogate Standard Spiking Solution
  The compound specified is dibutylchlorendate. Prepare a solution of 1.0 ug/ml dibutylchlorendate in acetone. Add 1.0 milliliter of this surrogate standard to all water samples extracted for pesticides and PCBs. This will be equivalent to 0.10 µg/ml in the final 10-milliliter hexane extract. Prepare a solution at 20 µg/ml dibutylchlorendate in methanol. Add 0.100 milliliter of this surrogate standard to all solid samples extracted for pesticides and PCBs. This will be equivalent to 0.10 ug/ml in the final extract volume.

- Matrix Standard Spiking Solution
  Prepare a spiking solution in methanol that contains the following pesticides at the specified concentrations for the corresponding matrix type:

PESTICIDE	WATER (µg/ml)	SOLIDS (µg/ml)
Lindane	0.20	2.0
Heptachlor	0.20	2.0
Aldrin	0.20	2.0
Dieldrin	0.50	5.0
Endrin	0.50	5.0
4,4'-DDT	0.50	5.0

Add 1.0 milliliter of the pesticide water matrix spiking solution to each of two 1-liter aliquots of the water sample selected for spiking. Add 0.400 milliliter of the pesticide solids matrix spiking solution to each of two 30-gram aliquots of the solids sample selected for spiking. This will be equivalent to the following pesticide concentrations in the final extracts of the corresponding sample matrices:

PESTICIDE	WATER (µg/ml)	SOLIDS (µg/ml)
Lindane	0.02	0.04
Heptachlor	0.02	0.04
Aldrin	0.02	0.04
Dieldrin	0.05	0.10
Endrin	0.05	0.10
4,4'-DDT	0.05	0.10

Calibration
The GC is calibrated using the external standard technique for all packed columns used for quantitation. The GC calibration procedures are described in Section 11.4.2.7.

Quantitation of Pesticides and PCBs

Response can be measured by the manual peak height technique or by automated peak height or peak area measurements from an integrator. Calculate the concentration in the sample using the following equation from the CLP method in WA-87-K236:

- Water

$$\text{Concentration } \mu g/l = \frac{(A_x)(I_s)(V_t)}{(A_s)(V_i)(V_s)}$$

where

A_x = response for the parameter to be measured,

A_s = response for the external standard,

V_t = volume of total extract (microliters) (take into account any dilutions),

I_s = amount of standard injected in nanograms,

V_i = volume of extract injected (microliters), and

V_s = volume of water extracted (milliliters).

- Sediment/Soil

$$\text{Concentration } \mu g/kg \text{ (Dry Weight Basis)} = \frac{(A_x)(I_s)(V_t)}{(A_s)(V_i)(W_s)(D)}$$

where

A_x = response for the parameter to be measured,

I_s = amount of standard injected in nanograms,

A_s = response for the external standard,

V_i = volume of extract injected (microliters), and

V_t = volume of low-level total extract (Use 20,000 microliters or a factor of this when dilutions are made other than those accounted for below):

- 1/20 total extract taken for pesticide analysis (derived from 0.5 milliliter of 10 milliliter extract)

- Final concentration to 1.0 milliliter for pesticide analysis

$D = \frac{100 - \%\ moisture}{100}$, and

W_s = weight of sample extracted (grams).

For multicomponent mixtures (chlordane, toxaphene, and PCBs), match retention times of peaks in the standards with peaks in the sample. Quantitate every identifiable peak (greater than 50 percent of the total area must be used) unless interference with individual peaks persists after cleanup. Add peak height or peak area of each identified peak in the chromatogram. Calculate as total response in the sample versus total response in the standard.

9.4.4 RADIOLOGICAL ANALYSES

Radiological analyses will be performed in accordance with the IT/RSL Radioanalytical Methodology and Procedures Manual.

This manual includes the following procedures pertinent to radiological analyses:

- RSL-001: Acquisition and Use of Standard Reference Material;
- RSL-002: Standardization of Carrier Solutions;
- RSL-102: Operation of Alpha Spectrometer Systems;
- RSL-103: Calibration of Liquid Scintillation Counting Systems;
- RSL-104: Operation of Low Background Alpha/Beta Counting Instruments;
- RSL-105: Calibration of Alpha Spectrometer Systems;
- RSL-106: Calibration of Alpha/Beta Counting Instruments;
- RSL-107: Operation of Liquid Scintillation Counting Instruments;

- RSL-108: Operation of Alpha Scintillation Counting Instruments;
- RSL-109: Background Determination of a Germanium Detector System;
- RSL-110: Energy Calibration of Computer-Assisted Germanium Spectroscopy Systems;
- RSL-111: Linearity Check of Germanium Gamma-ray Spectroscopy Systems;
- RSL-112: Operation of Germanium Gamma Spectroscopy System (ND 66/680);
- RSL-113: Determination of Germanium Detector Efficiency Tables;
- RSL-114: Determination of Germanium Detector Counts Reproducibility;
- RSL-201: Samples Preparation;
- RSL-202: Extraction of Iron From Soil or Metallic Samples;
- RSL-304: Determination of Selected Alpha Emitting Isotopes;
- RSL-305: Determination of Strontium-89,90 in Water Samples;
- RSL-306: Determination of Strontium-89,90 in Milk Samples;
- RSL-308: Determination of Gross Alpha and/or Beta Activity;
- RSL-309: Determination of Radium-226,228 in Aqueous Samples;
- RSL-310: Determination of Technicium-99;
- RSL-501: Data Verification;
- RSL-601: Performing Intralaboratory Quality Control Analysis;
- RSL-602: Performing Interlaboratory Quality Control;
- RSL-603: Internal Surveillances and Audits;
- RSL-703: Storage and Maintenance of Records;

- RSL-801: Instructions for Glassware Cleaning Laboratory Housekeeping;
- RSL-802: Checking Laboratory Measuring and Test Equipment;
- RSL-901: Training and Qualifications of Laboratory Technicians;
- RSL-902: Q.A. Orientation of New Employees;
- RSL-903: Material and Equipment Procurement, Receipt, Storage, and Control;
- RSL-904: Reporting of Defects and Noncompliances per 10 CFR 21;
- RSL-1001: Sample Receiving;
- RSL-1002: Radiological Screening and Classification of Mixed Waste Samples; and
- RSL-1004: Sample and Data Flow.

9.4.5 GEOTECHNICAL ANALYSES

The geotechnical laboratory will utilize industry-recognized analytical methods from source documents published by agencies such as ASTM and the U.S. EPA. Table 9-4 provides the list of test methods to be used, preservation requirements, and transportation requirements.

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TABLE 9-4

LABORATORY GEOTECHNICAL TESTING
METHOD, PRESERVATIVE, AND TRANSPORTATION REQUIREMENTS
FEED MATERIALS PRODUCTION CENTER
FERNALD, OHIO

TEST	METHOD[a]	PRESERVATION[1]	TRANSPORTATION[2]
Modified Proctor Compaction	D1557	B	I
Vertical Permeability	D2434	A	I
Unconfined Compression	D2166	D	II
Triaxial Shear	D2850	D	II
Consolidation	D2435	C,D	II
Classification of Soil, Visual Examination	D2487	B	I
Grain Size Analysis	D422	B	I
Specific Gravity	D854	A	I
Atterberg Limits	D4318	B	I
Relative Density	D4254	B	I
Water Content	D2216	B	I

[a]American Society for Testing and Materials, 1985, "Soil and Rock; Building Stones," Annual Book of ASTM Standards, Vol. 04.08.

[1]A No special requirements - plastic pails, plastic bags - unless hazardous material, then sample must be sealed.
B Sealed, moisture-proof containers: plastic bag or pails, glass or plastic jars, thin-walled tubes, liners, or rings.
C Sealed, moisture-proof containers: thin-walled tubes, liners, or rings.
D Sealed, moisture-proof, thin-walled tubes.

[2]I No special requirements if nonhazardous material, follow DOT regulations if hazardous material.
II Protection against vibration, shock, and temperature extremes.

10.0 DATA REDUCTION, VALIDATION, AND REPORTING

The following procedures summarize the practices routinely used by the laboratory and engineering groups for data reduction, validation, and reporting. Numerical analyses, including manual calculations, mapping, and computer modeling will be documented and subjected to quality control review and peer review. Records of numerical analyses will be legible and complete enough to permit reconstruction of the work by a qualified individual other than the originator. The Data Management Plan is presented in Volume IV of the Work Plan.

Laboratory-specific data reduction information is presented in Sections 3.6 and 10.2 of the IT Analytical Services QA Manual and corresponding sections of Laboratory-Specific Attachments and Standard Operating Procedures.

10.1 ANALYTICAL LABORATORY DATA

All laboratory results and QC data will be reviewed by the appropriate laboratory manager and QC coordinator. The QC data will be reported with the analytical results. The laboratory data reduction, validation, and reporting process is presented in Figure 9-1.

Laboratory calculations and data reduction are independently checked by another analyst or supervisor in accordance with Section 10.2.1 of the IT Analytical Services QA Manual and corresponding sections of Laboratory-Specific Attachments and Standard Operating Procedures. This process must be satisfactorily completed for the data to be considered valid. At least 20 percent of all data shall be checked in this manner. If data cannot be verified, the analyst will have to rerun the sample(s).

10.1.1 DATA VERIFICATION

- Calculate QC data before completing other calculations and before reporting data.
- Calculate analyses results and complete data sheets; sign and date each page.
- Request that another analyst or supervisor approve the notebook or laboratory data sheets by formal checking.
- Record data on project data summary sheets (as necessary); initial and date forms.
- Check other parameters for relative concentration values (alert supervisor, if necessary).
- File instrument charts (e.g., metals) in appropriate data files; enter required information on form.
- Enter QC data on appropriate forms and charts.

10.1.2 REPORT PREPARATION

- Review data on project data sheets and previous similar project data, if available.
- Check laboratory data sheets for comments regarding sample analyses.
- Review detection limits and report summarized data with appropriate significant figures and units.
- Submit data to project group for report preparation.
- Verify typed data for formal checking.
- Discuss results with site manager prior to submittal of report.
- Report results externally.
- Transmit appropriate records to laboratory project files.

10.1.3 LABORATORY MANAGEMENT REVIEW

The laboratory manager shall review testing results prior to distribution beyond the laboratory. The reviewer shall:

- Compare analyses performed to the proposed testing record.
- Review results for reasonableness.

10.1.4 DATA VERIFICATION OF PREVIOUS STUDIES

Data validation and verification procedures for determining the acceptability of data generated during previous studies for the FMPC will follow the same criteria described in Sections 10.0 and 11.0 of the project-specific QAPP. The criteria for validation of laboratory data described in Section 11.0 closely follows the criteria established in the U.S. EPA's Contract Laboratory Program. However, U.S. EPA-approved alternate analytical methods including, but not limited to, the following references will also be considered acceptable:

- U.S. EPA, 1985, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, U.S. EPA, Office of Solid Waste and Emergency Response, Washington, D.C.
- U.S. EPA, 1984, Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, as published in the October 26, 1984, Federal Register, Volume 49, No. 209, pages 43234 to 43442.
- U.S. EPA, 1983, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, U.S. EPA, Office of Research of Development, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

Data generated by U.S. EPA-approved alternate analytical methods will be verified and validated in accordance with the quality control criteria specified within the alternate method or its originating reference document.

10.2 FIELD DATA VALIDATION

Field data generated in accordance with the project-specific work plan will primarily consist of radiological screening data, field temperature, pH, and specific conductance data, and data associated with soil boring advancement, monitoring well installation and development, and geophysical logging and soil classification. This data will be validated by review of the project documentation to check that all forms specified in the work plan and QAPP have been completely and correctly filled out and that documentation exists for the required instrument

calibration. This documentation will be considered sufficient to provide that proper procedures have been followed during the field investigation.

10.3 ENGINEERING ANALYSIS AND CALCULATIONS

Analysis activities performed in the office shall be completely documented and the resulting documentation formally checked.

10.3.1 NUMERICAL ANALYSIS PROCEDURES

Analysis activities shall be performed in a planned and controlled manner. Performance responsibility rests with the project director. Prior to initiating the activities, the project director shall discuss the scope of the work, contractual and regulatory requirements, and applicable QA/QC procedures with assigned personnel. At the request of the project director, this will be done by the QA officer.

To provide evidence of satisfactory work performance and the basis for information transmitted externally, analyses and their results shall be completely documented. Documentation may include calculations, computer programs, logs, drawings, and tables.

10.3.1.1 Calculations

Calculations shall be legible and in a form suitable for reproduction, filing, and retrieval. Documentation shall be sufficient to permit a technically qualified individual to review and understand the calculations and verify the results.

Calculations shall be performed on standard calculation paper whenever possible. All calculation pages shall be individually identified, with the exception of large computer output. Calculation paper provides spaces for the originator's name and date of work, the checker's name and date, calculation subject, project number, and page number. All of this information shall be completed for each page. For extra pages, such as large graphs, this information shall also be included.

10.3.2.3 Drawings

Drawings shall be checked like calculations (Section 10.3.2.1) using yellow and red markers. Checkprints of the same drawing shall be marked CP1, CP2, etc., to show progression of the checking process.

If a drawing is revised, the entire checking process shall be repeated for the revised areas only. A new checkprint shall be prepared. Under no circumstances shall revisions be made without the formal checking procedure.

10.3.2.4 Logs and Tables

Final subsurface logs shall be verified by the responsible member of the project group. The verification shall provide that changes from the original field representative's logs to the final log sheets are consistent with the results of the laboratory testing or other investigations. The final log sheets shall be checked in the same manner as all calculations or drawings, with the checker signing and dating all checkprints.

In addition, all final tables presenting information, data, or the results of analyses shall be checked using the standard process (Section 10.3.2.1). Checkprints of the same table shall be marked CP1, CP2, etc., to show progression of the checking process.

11.0 INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

Internal QC checks are performed to verify the resulting quality of the measurement tasks of the field and laboratory investigation and associated tasks.

11.1 TRAINING

The ASI and subcontractor personnel working on the FMPC investigation will be properly trained, qualified individuals. Prior to commencement of field work, the field personnel will be given instruction specific to the FMPC investigation, covering the following areas:

- Organization and lines of communication and authority;
- Description of the work areas;
- Overview of the Work Plan and the QAPP;
- Documentation requirements;
- Documentation of recurring training;
- Decontamination procedures;
- Emergency procedures; and
- Sampling methods for the procedures field personnel are authorized to perform.

Training of field personnel will be provided by the site manager or a qualified designee. Documentation of training will be recorded.

11.2 PROCUREMENT/CONTROL OF SUBCONTRACTORS

Controls identified as necessary for subcontractor procurement and furnishing quality-related items and/or services shall be based on the effect an item or service will have on project results.

The responsibility for quality compliance will be delegated to a subcontractor in the procurement documents, provided that the subcontractor has documented an acceptable QA program. If a

subcontractor does not have such a program, ASI will use the procurement documents to "pass down" the performance of specific quality requirements. The "pass down" provides that appropriate requirements can be met without asking the subcontractors to have their own individual QA programs. If necessary, prequalification audits may be performed by QA personnel to determine subcontractor acceptability.

To verify subcontractor conformance to project quality requirements, ASI (or designee) shall, as necessary, perform field inspections, review subcontractor-prepared documentation, and perform audits of subcontractor activities. Subcontractors shall provide access to their work areas and records for inspection and auditing.

For field operations, ASI (or designee) project personnel shall perform an inspection of each subcontractor prior to starting work. The intention of this check is to verify that the subcontractor has fulfilled the requirements necessary to perform their activities. The inspection shall include the type and condition of equipment, calibration of equipment, and qualifications of personnel. If requirements are not met, sufficient grounds for suspension of activities exist. Equipment which does not meet ASI and project requirements shall not be used without repair or replacement to the satisfaction of the site manager.

11.3 QUALITY ASSURANCE REVIEW OF REPORTS, PLANS, AND SPECIFICATIONS

Prior to issuance of a final report, it shall be reviewed by knowledgeable members of the project staff and the project director, or designated representative. This review shall address whether:

- The report satisfies the scope of work, client requirements, and pertinent regulatory requirements;
- Assumptions are clearly stated, justified, and documented;

- A reference is cited for any information utilized in report preparation that was originated outside the project;
- The report correctly and accurately presents the results obtained by the project work;
- The tables and figures presented in the report are prepared, checked, and approved according to requirements;
- The report figures are signed and dated by the appropriate members of the project staff and project management;
- The bases for the recommendations and conclusions presented in the report are clearly documented; and
- The typed report has been proofread, and punctuation, grammar, capitalization, and spelling are correct.

In addition to review by the project group, specific reports will undergo a peer review process by the project advisors. The peer review process is intended to complement the verification of numerical analyses and data processing. While verification provides review and confirmation of largely definitive work, peer review provides evaluations and assessment of interpretations, judgments, and decisions made. Final reports will not be issued until peer review comments are resolved.

Peer reviews shall be coordinated by the project director. Reviews will address the following, as appropriate:

- Were the work plans and procedures, as developed, sufficient to control and permit duplication of the project activities?
- Have procedures been correctly used?
- Did the procedures used result in obtaining data objectives of the project and have the objectives been properly translated from applicable contractual requirements, industry standards, and federal/state/local regulations?
- Have sufficient data of adequate quality been collected to reach conclusions which can be justified and verified?

- Are assumptions, interpretations, judgments, or decisions supported by the data, and are they defendable?
- What is the effect of variations upon results?
- Is documentation sufficient to verify the validity and reproducibility of report results?

Peer reviews will be documented. At a minimum, the review report will include the following:

- Date(s) of review;
- Participants
  - Reviewers;
  - Project personnel;
  - Any QA personnel;
- Activities reviewed including interpretations, decisions, and judgments;
- Statement of agreement or disagreement with interpretations, decisions, and judgments, including the means for resolving disagreements;
- Recommendations for changes, the extension of existing activities, or the establishment of additional studies; and
- If questions remain unresolved beyond the end of the review, the means for closing the questions.

Review reports shall be transmitted to project management and appropriate QA personnel.

11.4 QUALITY CONTROL CHECKS AND PROCEDURES

This section describes the QC operations that must be performed to satisfy the analytical requirements of the contract. All QC operations must be performed in accordance with the following procedures for inorganic and organic parameters, respectively:

- U.S. Environmental Protection Agency, 1985, "Chemical Analytical Services for Multi-Media Multi-Concentration Metals and Inorganics," WA-87-K026, Washington, DC.
- U.S. Environmental Protection Agency, 1985, "Chemical Analytical Services for Multi-Media Multi-Concentration Organics, GC/MS Techniques," WA-87-K236, Washington, DC.

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11.4.1 INORGANIC QUALITY CONTROL

QC procedures have already been described with the appropriate analytical procedures in Section 9.0. The purpose of this section is to augment those QC procedures and to summarize the minimum QC requirements for inorganic analysis.

Accordingly, the following QC operations must be performed:

- Initial calibration;
- Initial calibration verification (QC sample);
- Continuing calibration verification;
- Calibration blank;
- Preparation (method) blank analysis;
- ICP interference check sample analysis;
- ICP serial dilution analysis;
- Matrix spike analysis;
- Duplicate sample analysis;
- Graphite furnace atomic absorption spectrophotometry QC analysis (Method of Standard Additions may be required under certain conditions);
- Method of Standard Additions; and
- Laboratory QC sample analysis (performance evaluation samples).

11.4.1.1 Initial Calibration

For atomic absorption systems, calibration standards are prepared by diluting the stock metal solutions at the time of analysis. Calibration standards must be prepared fresh each time an analysis is to be made and discarded after use. Prepare a blank and at least three calibration standards in graduated amounts in the appropriate range. One atomic absorption calibration standard must be at the contract required detection limit, except for mercury. The calibration standards must be prepared using the same type of acid or combination of acids and at the same

concentration as will result in the samples following sample preparation. Beginning with the blank and working toward the highest standards, aspirate or inject the solutions and record the readings. Calibration standards for furnace procedures should be prepared as described in the individual methods for that metal.

For cyanide and mercury, follow the calibration procedures outlined in Section 9.0. One cyanide calibration standard must be at the contract required detection limit. For ICP systems, calibrate the instrument according to instrument manufacturer's recommended procedures.

11.4.1.2 Initial Calibration Verification (Quality Control Sample)

After the ICP, atomic absorption, and cyanide systems have been calibrated, the accuracy of the initial calibration shall be verified and documented for every analyte by the analysis of U.S. EPA initial calibration verification solution. The initial calibration verification solution or QC sample is a solution obtained from an outside source having known concentration values to be used to verify the calibration standards.

The initial calibration verification standard must be run at each wavelength used for analysis.

If the initial calibration verification solution is not available from U.S. EPA, or where a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for calibration, but within the calibration range. An independent standard is defined as a standard composed of the analytes from a different source than those used in the standard for the initial calibration. For ICP, the initial calibration verification solution must be run at each wavelength used in the analysis of the sample. When measurements exceed the control limits, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

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The values for the initial and subsequent continuing calibration verifications shall be recorded on Form II (Appendix A) for ICP, atomic absorption, and cyanide analyses, as indicated. Hexavalent chromium, nitrate, and fluoride analyses will follow the same requirements as cyanide.

11.4.1.3 Continuing Calibration Verification

To provide calibration accuracy during each analysis run, one of the following standards is to be used for continuing calibration verification and must be analyzed for each analyte at a frequency of 10 percent or every two hours during an analysis run, whichever is more frequent. The standard must also be analyzed for each analyte at the beginning of the run and after the last analytical sample. The analyte concentrations in the continuing calibration standard must be one of the following solutions at or near the midrange levels of the calibration curve:

- U.S. EPA solutions;
- NBS Standard Reference Material 1643a; and
- A laboratory-prepared standard solution - This solution must be from an outside source other than that used to prepare the calibration standards.

The following initial and continuing calibration verification control limits must be met before analysis can proceed:

ANALYTICAL METHOD	INORGANIC SPECIES	PERCENT OF TRUE VALUE
ICP/AA	Metals	90-110
Cold Vapor AA	Mercury	80-120
Other Parameters	i.e., Cyanide	85-115

The same calibration standard must be used throughout the analysis runs for a case of samples received.

If the deviation of the continuing calibration verification is greater than the control limits specified above, the instrument

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must be recalibrated and the preceding 10 samples are analyzed for the analytes affected. Information regarding the continuing verification of calibration shall be recorded on Form II (Appendix A) for ICP, atomic absorption, and cyanide as indicated. Hexavalent chromium, nitrate, and fluoride follow the same requirements as cyanide.

11.4.1.4 Calibration Blank

A calibration blank (zero standard) is analyzed each time the instrument is calibrated, at the beginning and the end of the run, and at a frequency of 10 percent during the run. The results for the calibration blank solution shall be recorded on Form III (Appendix A) for ICP, atomic absorption, and cyanide analyses, as indicated. Hexavalent chromium, nitrate, and fluoride analyses will follow the same requirements as cyanide. Blanks are to be reported down to the instrument detection limit. If this blank result is greater than the contract required detection limit (Table 4-1), terminate analysis, correct the problem, and recalibrate.

11.4.1.5 Preparation (Method) Blank Analysis

At least one preparation blank (or method blank) consisting of deionized distilled water processed through each sample preparation procedure (i.e., water, solids) must be prepared and analyzed with every 20 samples received or with each batch of samples digested, whichever is more frequent. Each data package must contain the results of all the preparation blank analyses associated with the samples.

This blank is to be reported for each sample set and used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner:

- If the concentration of the blank is less than or equal to the contract required detection level (Table 4-1), no correction of sample results is performed.

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- If the concentration of the blank is above the contract-required detection level: for any group of samples associated with a particular blank, the concentration of the sample with the least concentrated analyte must be 10 times the blank concentration, or all samples associated with the blank and less than 10 times the blank concentration must be redigested and reanalyzed, with the exception of an identified aqueous-soil field blank. The sample value is not to be corrected for the blank value.

  The values for the preparation blank shall be recorded in µg/l on Form III (Appendix A) for ICP, atomic absorption, and cyanide analyses, as indicated. Hexavalent chromiumm, nitrate, and fluoride analyses will follow the same requirements as cyanide.

11.4.1.6 ICP Interference Check Sample Analysis

To verify interelement and background correction factors, the IT laboratory must analyze and report the results for an ICP interference check sample at the beginning and end of each sample analysis run (or a minimum of twice per eight-hour working shift, whichever is most frequent). The ICP interference check sample must be obtained from U.S. EPA, if available. Results for the check sample analysis during the analytical runs must fall within the control limit of ±20 percent of the true value for the analytes included in the interference check sample. If not, terminate the analysis, correct the problem, recalibrate, reverify the calibration, and reanalyze the samples. If the ICP interference check sample is not available from U.S. EPA, an independent ICP check sample must be prepared with interferent and analyte concentrations at the levels specified in the previously referenced U.S. EPA CLP protocol. The mean value and standard deviation must be established by initially analyzing the check sample at least five times repetitively for each parameter on Form IV (Appendix A). Results must fall within the control limit of ±20 percent of the established mean value.

Results of all interference check sample analyses for all ICP parameters must be recorded on Form IV (Appendix A).

11.4.1.7 ICP Serial Dilution Analysis

Prior to reporting concentration data for the analyte elements, the IT laboratory must analyze and report the results of the ICP serial dilution analysis. The ICP serial dilution analysis must be performed on each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium) for each set of samples, or for each 20 samples received, whichever is more frequent. Samples identified as field blanks cannot be used for serial dilution analysis. If the analyte concentration is sufficiently high (minimally a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution must agree within 10 percent of the original determination. If the dilution analysis is not within 10 percent, a chemical or physical interference effect should be suspected, and the data should be flagged with an "E." Serial dilution results must be reported on QC Report Form IX (Appendix A).

11.4.1.8 Matrix Spike Analysis

The spiked sample analysis is designed to provide information about the effect of the sample matrix on the digestion and measurement methodology. The spike is added before the digestion and prior to any distillation steps (i.e., CN). At least one spiked sample analysis must be performed on each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium) for each set of samples or for each 20 samples received, whichever is more frequent. Samples identified as field blanks cannot be used for spiked sample analysis. The analyte spike must be added in the amount given in the U.S. EPA CLP protocol for each element analyzed. If two analytical methods are used to obtain the reported values for the same element for a set of samples (i.e., ICP, atomic absorption), spike samples must be run by each method used. If the spike recovery is not within the limits of 75 to 125 percent, the data of all samples received associated with that spiked sample must be flagged with the letter, "N." An exception to this rule is granted in situations where the sample concentration exceeds the

spike concentration by a factor of four or more. In such a case, the spike recovery should not be considered and the data shall be reported unflagged even if the percent recovery does not meet the 75 to 125 percent recovery criteria. In the instance where there is more than one spiked sample per matrix per sample set, if one spike sample recovery is not within contract criteria, flag all the samples of the same matrix in the set. Individual component percent recoveries are calculated using the following equation from the CLP methods in WA-87-K026:

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

where

SSR = spiked sample result

SR = sample result

SA = spike added.

When the sample concentration is less than the instrument detection limit, use SR = 0 for purposes of calculating the percent recovery. The spiked sample results must be reported on Form V (Appendix A) for ICP, atomic absorption, and cyanide analyses, as indicated. Hexavalent chromium, nitrate, and fluoride analyses will follow the same requirements as cyanide.

11.4.1.9 Duplicate Sample Analysis

At least one duplicate sample must be analyzed from each group of samples of a similar matrix type (i.e., water, soil) for each set of samples or for each 20 samples received, whichever is more frequent. Samples identified as field blanks cannot be used for duplicate sample analysis. If two analytical methods are used to obtain the reported values for the same element for a set of samples (i.e., ICP, atomic absorption), duplicate samples must be run by each method used. The RPD for each component is calculated using the following equation from the CLP methods in WA-87-K026:

$$RPD = \frac{|D_1 - D_2|}{(D_1+D_2)/2} \times 100$$

where

RPD = relative percent difference

D_1 = first sample value

D_2 = second sample value (duplicate)

The results of the duplicate sample analysis must be reported on Form VI (Appendix A). A control limit of ±20 percent for RPD shall be used for sample values greater than five times the contract required detection limit. A control limit of ± the contract required detection limit shall be used for sample values less than five times the contract required detection limit (Table 4-1), and this control limit (± contract required detection limit) should be entered in the "Control Limit" column on Form VI. If one result is above the 5-times contract required detection limit level and the other is below, use the ± contract required detection limit criteria. If either sample value is less than the contract required detection limit, the RPD is not calculated and is indicated as "NC" on Form VI.

If the duplicate sample results are outside the control limits, flag all the data for samples received associated with that duplicate sample with an "*" on Forms I and V. In the instance where there is more than one duplicate sample per set, if one duplicate result is not within contract criteria, flag all the samples of the same matrix in the set. Hexavalent chromium, nitrate, and fluoride will follow the same requirements as cyanide.

11.4.1.10 Graphite Furnace Atomic Absorption Spectrophotometry QC Analyses

Because of the nature of the graphite furnace atomic absorption technique, the special procedures summarized in Exhibit E of WA-87-K026 will be required for quantitation. In general, a

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single-point method of addition or post-digestion spike at twice the method detection limit must be performed on every sample for arsenic and selenium. If the post-digestion spike recovery is not between 85 and 115 percent, the sample must be reanalyzed by the method of standard addition.

All furnace analyses, except during full methods of standard addition, will require duplicate injections for which the average absorbance or "concentration" will be reported. All analyses must fall within the calibration range. The raw data package must contain absorbance or "concentration" values, the average value, and the relative standard deviation (RSD) or coefficient of variation (CV). For concentrations greater than the contract required detection limit, the duplicate injection readings must agree within 20 percent RSD or CV, or the sample must be rerun once. If the readings are still out, flag the value with an "M" on Form I (Appendix A).

11.4.1.11 Method of Standard Additions

The method of standard additions (MSA) shall be used for the analysis of EP toxicity extracts for metals and whenever a new sample matrix is being analyzed.

The following procedures will be incorporated into MSA analyses:

- Data from MSA calculations must be within the linear range as determined by the calibration curve generated at the beginning of the analytical run.

- The sample and three spikes must be analyzed consecutively for MSA quantitation (the "initial " spike run data is specifically excluded from use in the MSA quantitation). Only single injections are required for MSA quantitation.

  Spikes should be prepared such that:

  - Spike 1 is approximately 50 percent of the sample absorbance

  - Spike 2 is approximately 100 percent of the sample absorbance

- Spike 3 is approximately 150 percent of the sample absorbance.

The data for each MSA analysis should be clearly identified in the raw data documentation along with the slope, intercept, and correlation coefficient (r) for the least squares fit of the data and the results reported on Form VIII (Appendix A). Reported values obtained by MSA are flagged on the data sheet (Form I, Appendix A) with the letter "s."

If the correlation coefficient (r) for a particular analysis is less than 0.995, the MSA analyses must be repeated once. If the correlation coefficient is still <0.995, the results on Form I must be flagged with "+."

11.4.1.12 Laboratory Control Sample (LCS) Analysis (Performance Evaluation Samples)

Aqueous laboratory QC samples must be analyzed for each analyte using the same sample preparation and analytical methods employed for the samples.

The aqueous LCS solution must be obtained from U.S. EPA (if unavailable, the U.S. EPA initial calibration verification solution may be used). The aqueous LCS must be prepared and analyzed with the samples for each of the procedures applied to each set of samples received. One aqueous LCS must be analyzed for every 20 samples received or for each batch of samples digested, whichever is more frequent. Each data package must contain the results of all the LCS analyses associated with the samples on that set. For cyanide, the distilled midrange calibration standard may be used as the aqueous LCS. An aqueous LCS is not required for mercury analysis.

All aqueous LCS results will be reported on Form VII (Appendix A) in terms of true concentration and percent recovery as calculated by the following equation from the CLP method in WA-87-K026:

$$\%R = (\text{Observed}/\text{True}) \times 100$$

where "observed" is the measured concentration. If the percent recovery for the aqueous LCS falls outside the control limits of

80 to 120 percent, the analyses must be terminated, the problems corrected, and the previous samples associated with that LCS reanalyzed (i.e., previous 19 samples or the batch of samples from the set).

Once a month, a solid LCS available from EMSL-LV must be prepared and analyzed using each of the procedures applied to the solid samples received. If this U.S. EPA solid LCS is unavailable, other U.S. EPA QC check samples or other certified materials may be used.

The monthly results of the solid LCS samples should be reported on a duplicate Form VII (Appendix A) and submitted monthly to EMSL/Las Vegas and SMO on the fifteenth of every month.

If the percent recovery for the solid LCS sample is outside the control limits established by U.S. EPA or other supplier of the LCS, no further sample analyses may be done until the analytical problems are solved and satisfactory LCS results are obtained.

11.4.2 ORGANIC QUALITY CONTROL

The following sections summarize the QC requirements described in Exhibit E of WA-87-K236 for the analysis of HSL organic compounds by GC/MS and GC. Specifically, the following QC operations must be performed:

- Tuning and GC/MS mass calibration;
- Calibration of the GC/MS system;
- Continuing calibration of the GC/MS system;
- Preparation (method) blank analysis;
- Surrogate standard spike analysis;
- Matrix spike/matrix spike duplicate analysis (MS/MSD); and
- Pesticide and PCB QC requirements.

11.4.2.1 Tuning and GC/MS Mass Calibration

Prior to initiating any ongoing data collection, it is necessary to establish that a given GC/MS meets the standard mass spectral abundance criteria. This is accomplished through the analysis of DFTPP or BFB.

Decafluorotriphenylphosphine (DFTPP)

Each GC/MS system used for the analysis of semivolatile or pesticide HSL compounds must be hardware tuned to meet the abundance criteria listed in Table 11-1 for a 50 nanogram injection of DFTPP. DFTPP may be analyzed separately or as part of the calibration standard. The criteria must be demonstrated daily or for each 12-hour period, whichever is more frequent, before samples can be analyzed. DFTPP must be injected to meet this criterion. Background-subtraction should be straightforward and designed only to eliminate column bleed or instrument background ions. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting the method specifications are contrary to the objectives of QA and are unacceptable. Instrument conditions must be consistent with those used in sample analysis.

Bromofluorobenzene (BFB)

Each GC/MS system used for the analysis of volatile HSL compounds must be hardware tuned to meet the abundance criteria listed in Table 11-2 for a maximum of a 50 nanogram injection of BFB. This criterion must be demonstrated daily or for each 12-hour time period, whichever is more frequent. Background subtraction should be straightforward and designed only to eliminate column bleed or instrument background ions. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting the contract specifications are contrary to the objectives of QA and are unacceptable.

Documentation of the mass calibration must be provided in the form of a bar graph plot and as a mass listing.

TABLE 11-1

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

MASS	ION ABUNDANCE CRITERIA
51	30.0 - 60.0 percent of mass 198
68	Less than 2.0 percent of mass 69
69	Mass 69 relative abundance
70	Less than 2.0 percent of mass 69
127	40.0 - 60.0 percent of mass 198
197	Less than 1.0 percent of mass 198
198	Base peak, 100 percent relative abundance
199	5.0 - 9.0 percent of mass 198
275	10.0 - 30.0 percent of mass 198
365	Greater than 1.00 percent of mass 198
441	Present but less than mass 443
442	Greater than 40 percent of mass 198
443	17.0 - 23.0 percent of mass 442

The laboratory is required to complete a Form V (Appendix B) and mass calibration each time an analytical system is tuned.

11.4.2.2 Calibration of the GC/MS System

Prior to the analysis of samples and after tuning criteria have been met, the GC/MS system must initially be calibrated at a minimum of five concentrations to determine the linearity of response utilizing HSL compound standards. For GC/MS analysis, typical linear ranges of zero to 400 nanograms for base neutrals, zero to 1,000 nanograms for phenols, and zero to 1,000 nanograms for volatiles. Once the system has been calibrated, the calibration must be verified each 12-hour time period for each GC/MS system.

Volatile HSL Compounds

Initial calibration of volatile HSL compounds is required at 20, 50, 100, 150, and 200 µg/ℓ. Surrogate and internal standards shall be used with each of the calibration standards. Utilizing the analytical protocol specified in Exhibit D of WA-87-K236, will result in 100 to 1,000 total nanograms analyzed. If an analyte saturates at the 200 µg/l concentration level, and the GC/MS system is calibrated to achieve a detection sensitivity of no less than 5 µg/l, the laboratory must document it on Form VI (Appendix B) and proceed with a four-point initial calibration for that specific analyte. Secondary ion quantitation is only allowed when there are sample interferences with the primary ion. Analyze all method blanks and standards under the same condition as the samples.

Acid and Base-Neutral HSL Compounds

Initial calibration of semivolatile HSL compounds is required at 20, 50, 80, 120, and 160 total nonograms. If an analyte saturates at the 160 total nanogram concentration level and the GC/MS system is calibrated to achieve a detection sensitivity of no less than the contract required detection limit, the laboratory must document it on Form VI (Appendix B) and in the case narrative and proceed with a four-point initial calibration for the

TABLE 11-2

BFB KEY IONS AND ION ABUNDANCE CRITERIA

MASS	ION ABUNDANCE CRITERIA
50	15.0 to 40.0 percent of the base peak
75	30.0 to 60.0 percent of the base peak
95	Base peak, 100 percent relative abundance
96	5.0 to 9.0 percent of the base peak
173	Less than 2.00 percent of mass 174
174	Greater than 50.0 percent of the base peak
175	5.0 to 9.0 percent of mass 174
176	Greater than 95.0 percent but less than 101.0 percent of mass 174
177	5.0 to 9.0 percent of mass 176

specific analyte. The use of a secondary ion for quantification is only allowed when there are sample interferences with the primary ion. Nine compounds: benzoic acid, 2,4-dinitrophenol, 2,4,5-trichlorophenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, 4-nitrophenol, 4,6-dinitiro-2-methylphenol, and pentachlorophenol will only require a four-point initial calibration at 50, 80, 120, and 160 total nanograms since detection at less than 50 nanograms per injection is difficult.

Analyze each calibration standard and tabulate the area or the primary characteristic ion against concentration for each compound including all contract required surrogate compounds. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units. Late eluting compounds usually will have much better agreement.

Response Factor (RF) Calculations

Using Tables 9-2 and 9-3, calculate the RF for each compound at each concentration level using the following equation from the CLP method in WA-87-K236:

$$RF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

where

A_x = area of the characteristic ion for the compound to be measured

A_{is} = area of the characteristic ion for the specific internal standard

C_{is} = concentration of the internal standard (ng/µl)

C_x = concentration of the compound to be measured (ng/µl).

Using the RF from the initial calibration, calculate the percent relative standard deviation for compounds labeled as calibration check compounds on Form VI (Appendix B) using the following equation from the CLP method in WA-87-K236:

$$\%RSD = \frac{SD}{\bar{x}} \times 100$$

where

SD = standard deviation of initial five response factors (per compound)

$\bar{x}$ = mean of initial five response factors (per compound).

The percent relative standard deviations for each individual calibration check compound must be less than 30 percent. This criteria must be met for the initial calibration to be valid. Report results on Form VI (Appendix B).

A system performance check must be performed to check that minimum average response factors are met before the calibration curve is used.

For volatiles, the five system performance check compounds (SPCCs) are: chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. The minimum acceptable average response factor for these compounds is 0.300; 0.250 for bromoform. These compounds typically have RFs of 0.4 to 0.6 and are used to check compound instability and degradation caused by contaminated lines or active sites in the system.

For semivolatiles, the SPCCs are: N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol. The minimum acceptable average RF for these compounds is 0.050. These compounds (SPCCs) typically have very low RFs (0.1 to 0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. These compounds are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated. Report SPCC results on Form VI (Appendix B).

The initial calibration is valid only after both the percent relative standard deviation for calibration check compounds (CCC) and the minimum average RF for SPCC have been met. Only after both criteria are met can sample analysis begin.

11.4.2.3 Continuing Calibration of the GC/MS System

System Performance Check

A calibration standard(s) containing all volatile or semivolatile HSL compounds, including all required surrogates, must be performed each 12 hours during analysis. Compare the RF data from the standard each 12 hours with the mean RF from the initial calibration for a specific instrument. A system performance check must be made each 12 hours. If the SPCC criteria are met, a comparison of RF is made for all compounds. This is the same check that is applied during the initial calibration (Form VI, Appendix B). If the minimum SPCC RF are not met, the system must be evaluated and corrective action must be taken before sample analysis begins.

The minimum RF for volatile SPCC is 0.300 (0.250 for bromoform).

The minimum RF for semivolatile SPCC is 0.050.

Report results on Form VII (Appendix B).

Calibration Check

After the system performance check is met, calibration check compounds listed on Form VII (Appendix B) are used to check the validity of the initial calibration. Calculate the percent difference using the following equation from the CLP method in WA-87-K236:

$$\% \text{ Difference} = \frac{\overline{RF}_I - RF_C}{\overline{RF}_I} \times 100$$

where

$\overline{RF}_I$ = average RF from initial calibration

RF_c = RF from current verification check standard.

If the percent difference for any compound is greater than 20, the laboratory should consider this a warning limit. If the percent difference for each CCC is less than 25 percent, the initial calibration is assumed to be valid. If the criteria are not met (greater than 25 percent difference) for any one calibration-check compound, corrective action must be taken. Problems similar to those listed under SPCC could affect this criteria. If no source of the problem can be determined after corrective action has been taken, a new initial five-point calibration must be generated. This criteria must be met before sample analysis begins.

Concentration Levels for Continuing Calibration

The concentration for each volatile HSL compound in the continuing calibration standard(s) is 50 µg/ℓ.

The concentration for each semivolatile HSL compound in the continuing calibration standard(s) is 50 total nanograms. Nine compounds: benzoic acid, 2,4-dinitrophenol, 2,4,5-trichlorophenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, 4-nitrophenol, 4,6-dinitro-2-methylphenol, and pentachlorophenol will be analyzed at 80 total nanograms of each compound injected.

11.4.2.4 Preparation (Method) Blank Analysis

A method blank is a volume of deionized, distilled laboratory water for water samples, or a purified solid matrix (sodium sulfate) for soil/sediment samples carried through the entire analytical scheme (extraction, concentration, and analysis). The method blank volume or weight must be approximately equal to the sample volumes or sample weights being processed. The method blank analysis must be performed at the following frequency:

- For the analysis of volatile HSL compounds, a method blank analysis must be performed every 12 hours, once per sample set, or with every 20 samples of similar concentration and/or sample matrix, whichever is more frequent, on each GC/MS system used to analyze samples.

- For the analysis of semivolatile or pesticide HSL compounds, a method blank analysis must be performed once each sample set, with every 20 samples of similar concentration and/or sample matrix or whenever samples are extracted by the same procedure (separatory funnel or continuous extraction), whichever is more frequent. The method blank associated with a specific set or group of samples must be analyzed on each GC/MS or GC system used to analyze that specific group or set of samples.

- It is the laboratory's responsibility to minimize method interference caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatographs.

- For the purposes of this protocol, an acceptable laboratory method blank should meet the following criteria:

  - A method blank for volatile analysis must contain no greater than five times the contract-required detection limit (Table 4-2) of common laboratory solvents (common laboratory solvents are: methylene chloride, acetone, and toluene).

  - A method blank for semivolatile analysis must contain no greater than five times the contract required detection limit (Table 4-2) of common phthalate esters.

  - For all other HSL compounds not listed above, the method blank must contain less than the contract required detection limit of any single HSL compound.

If a laboratory method blank exceeds criteria, the laboratory must consider the analytical system out of control. The source of the contamination investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. All samples processed with a method blank that is out of control (i.e., contaminated) must be reextracted/repurged and reanalyzed. Method blank results are to be

reported on Form I, Appendix A or Form I and Form IV in Appendix B.

11.4.2.5 Surrogate Standard Spike Analysis

Surrogate standard determinations are performed on all samples and blanks. All samples and blanks are fortified with surrogate spiking compounds before purging or extraction in order to monitor preparation and analysis of samples.

Each sample (including matrix spike and matrix spike duplicate) and blank are spiked with surrogate compounds prior to purging or extraction. The surrogate spiking compounds shown on Form II (Appendix B) are used to fortify each sample or blank with the proper concentrations. Performance-based criteria are generated from laboratory results. Therefore, deviations from spiking protocol will not be permitted.

Surrogate spike recovery must be evaluated for acceptance by determining whether the concentration (measured as percent recovery) falls inside the contract required recovery limits presented on Form II (Appendix B).

Method Blank Surrogate Spike Recovery

The laboratory must take corrective action if any one of the following conditions exist:

- Recovery of any one surrogate compound in the volatile fraction is outside the required surrogate spike recovery limits; and
- Recovery of any one surrogate compound in either the base-neutral or acid fraction is outside surrogate spike recovery limits.

Corrective action will include the following:

- Check calculations so there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc.; also, check instrument performance.

- Recalculate or reinject/repurge the blank or extract if the above corrective actions fail to solve the problem.
- Reextract and reanalyze the blank.

Sample Surrogate Spike Recovery

The laboratory must take corrective action if any of the following conditions exists:

- Recovery of any one surrogate compound in volatile fraction is outside the contract surrogate spike recovery limits;
- Recovery of any one surrogate compound in either base-neutral or acid fraction is below 10 percent; or
- Recoveries of two surrogate compounds in either acid or base neutral fractions are outside surrogate spike recovery limits.

Corrective action will include the following:

- Check calculations so there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc.; and check instrument performance.
- Recalculate or reanalyze the sample or extract if the above corrective action fails to solve the problem.
- Reextract and reanalyze the sample if none of the above present a problem.

11.4.2.6 Matrix Spike/Matrix Spike Duplicate Analysis (MS/MSD)

In order to evaluate the matrix effect of the sample upon the analytical methodology, the compounds listed on Form III (Appendix B) are designated to be used for matrix spike and matrix spike duplicate analysis. These compounds are subject to change depending upon availability and suitability for use as matrix spikes.

An MS/MSD must be performed once for every sample set or with every 20 samples of similar sample matrix, whichever is greater. The analytical protocols require that a uniform amount

of matrix spiking solution be added to the sample aliquots prior to extraction. Samples requiring dilutions and chosen as the matrix spike/matrix spike duplicate samples, must be analyzed at the same dilution as the original unspiked sample.

Matrix Spike Percent Recovery

Individual component recoveries of the matrix spike are calculated using the following equation from the CLP method in WA-87-K236:

$$\text{Matrix Spike Percent Recovery} = \frac{SSR - SR}{SA} \times 100$$

where

SSR = spike sample results

SR = sample result

SA = spike added from spiking mix.

Relative Percent Difference (RPD)

The relative percent difference for each component is calculated using the following equation from the CLP method in WA-87-K236:

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

where

D_1 = first sample value

D_2 = second sample value (duplicate)

The results for the nonspiked HSL compounds in the matrix spike and matrix spike duplicate will be reported on Form I (Appendix B). The percent recovery and relative percent difference results will be reported on Form III (Appendix B).

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11.4.2.7 Pesticide and PCB QC Requirements

The following minimum QC requirements must be met for validation of pesticide and PCB analytical results:

- Preparation (method) blanks must be performed as described in Section 11.4.2.4.

- Matrix spike/matrix spike duplicate analyses must be performed as described in Section 11.4.2.6.

- Spike all standards, samples, blanks, matrix spike, and matrix spike duplicate samples with dibutylchlorendate as described in Section 11.4.2.5.

- Before performing any sample analyses, the laboratory is required to determine the retention time window for each pesticide/PCB compound listed in Table 4-2 and the surrogate spike compound dibutylchlorendate. These retention time windows are used to make tentative identification of pesticides/PCBs during sample analysis. The following procedure is used to establish retention time windows:

  - Prior to establishing retention time windows, the GC operating conditions (oven temperature and flow rate) must be adjusted such that 4,4'-DDT has a retention time of >12 minutes on packed GC columns.

  - Make three injections of all single-component pesticide mixtures, multiresponse pesticides, and PCBs throughout the course of a 24-hour period.

  - Verify the retention time shift for dibutylchlorendate in each standard. The retention time shift between the initial and subsequent standards must be less than a 2 percent difference for packed columns (less than 0.3 percent for capillary column). If this criterion is not met, continue injecting replicate standards to meet criteria.

  - Calculate the standard deviation of the three absolute retention times for each single-component pesticide. For multiresponse pesticides or PCBs, choose one major peak from the envelope and calculate the standard deviation of the three retention times for that peak.

  - Plus or minus three times the standard deviation of the absolute retention times for each pesticide/PCB will be used to define the retention time window; however, the experience of the analyst should weigh heavily on the interpretation of chromatograms. For multiresponse pesticide/PCBs, the analyst should

utilize the retention time window, but should primarily rely on pattern recognition.

- In those cases where the standard deviation for a particular pesticide/PCB is zero, the laboratory may substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

- The laboratory must calculate retention time windows for each pesticide/PCB on each GC column used at the beginning of the program and whenever a new GC column is installed. The data must be retained by the laboratory and made available during any on-site laboratory evaluation.

- Report results on Form IX (Appendix B).

- The external standard quantification procedure must be used to quantitate all pesticides and PCBs. Prior to performing any sample analyses, the laboratory must demonstrate the linear response range for each pesticide and PCB by the following procedures:

  - Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with hexane. One of the external standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

  - Using injections of 2 to 5 microliters of each calibration standard, tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound.

  - The three-point calibration should be done on each quantitation column and each instrument at the beginning of the project and each time a new column is installed. The data must be retained by the laboratory and made available for inspection during on-site evaluations.

- Prior to sample analysis, perform an evaluation check for linearity by the following procedure:

  - Prepare Evaluation Standards A, B, and C (aldrin, endrin, 4,4'-DDT, and dibutylchlorendate) at the three concentration levels described in Exhibit D of WA-87-K236.

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- Analyze the three evaluation standards sequentially at the beginning of each 72-hour period and calculate the calibration factor for each compound in evaluation standards using the following equation from the CLP method in WA-87-K236:

$$\text{Calibration Factor} = \frac{\text{Total Areas of Peak}}{\text{Mass Injected (in nanograms)}}$$

- Using the calibration factors, calculate the percent relative standard deviation for each compound at the three concentration levels using the following equation:

$$\%RSD = \frac{SD}{\bar{x}} \times 100$$

where

SD = the standard deviation

$\bar{x}$ = the mean of the three calibration factors for the compound of interest.

The percent relative standard deviation for aldrin, endrin, and dibutylchlorendate must be $\leq$ 10 percent. The 10 percent RSD linearity criteria pertain only to columns being used for pesticide/PCB quantitation. If a column is used only for surrogate quantitation, the 10 percent RSD is only required for dibutylchlorendate.

Report results on Form VIII (Appendix B).

- From the analysis of the Evaluation B standard described above, determine the percent breakdown for 4,4'-DDT and endrin by the following equations from the CLP method in WA-87-K236:

$$\text{\% breakdown for 4,4'-DDT} = \frac{\text{Total DDT degradation peak area (DDE + DDD)}}{\text{Total DDT peak area (DDT + DDE + DDD)}} \times 100$$

% breakdown for endrin =

$$\frac{\text{Total endrin degradation peak areas (endrin aldehyde + endrin ketone)}}{\text{Total endrin peak area (endrin + endrin aldehyde + endrin ketone)}} \times 10$$

The percent breakdown for endrin or 4,4'-DDT must not exceed 20 percent, or corrective action must be taken

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before analysis continues. Evaluate endrin and 4,4'-DDT breakdown each time the Evaluation B standard is analyzed (Table 11-3). Report the percent endrin and 4,4-'DDT breakdown results on Form VIII (Appendix B).

- Prepare individual standard mixtures A and B containing all the single-component pesticides. The single-component pesticides are divided into two groups to prevent overlap of compounds during packed GC column analysis.

  Analyze individual standard mixture A and B and all multiresponse pesticide/PCBs at the beginning of each 72-hour period (Table 11-3) and analyze individual standard mixture A or B intermittently throughout the analysis and whenever sample analysis is completed.

  Calculate the calibration factor for each single component compound in individual standard mixtures A and B. Calculate the percent difference by the following equation from the CLP method in WA-87-K236:

$$\text{Percent Difference} = \frac{R_1 - R_2}{R_1} \times 100$$

where

R_1 = calibration factor from first analysis

R_2 = calibration factor from second analysis.

The percent difference between calibration factors for each single-component compound must not exceed 15 percent for a quantitation run nor exceed 20 percent for a confirmation run during the 12-hour period. If the above criteria are exceeded, the affected samples have to be reanalyzed. Report results on Form IX (Appendix B).

- The retention time shift for dibutylchlorendate must be evaluated after the analysis of each sample. The retention time shift may not exceed a 2 percent difference for packed GC columns between the initial standard analysis and any sample analyzed during the 72-hour period.

  Calculate the retention time shift using the following equation from the CLP method in WA-87-K236:

$$\text{Percent Difference} = \frac{RT_I - RT_S}{RT_I} \times 100$$

TABLE 11-3

PESTICIDE AND PCB
72-HOUR ANALYTICAL SEQUENCE

1. Evaluation Standard Mix A
2. Evaluation Standard Mix B
3. Evaluation Standard Mix C
4. Individual Standard Mix A(a)
5. Individual Standard Mix A(a)

6. Toxaphene
7. Aroclors 1016/1260
8. Aroclor 1221(b)
9. Aroclor 1232(b)
10. Aroclor 1242

11. Aroclor 1248
12. Aroclor 1254
13. 5 samples
14. Evaluation Standard Mix B
15. 5 samples
16. Individual Standard Mix A or B
17. 5 samples

18. Evaluation Standard Mix B
19. 5 samples
20. Individual Standard Mix A and B (whichever not run in Step 16)
21. 5 samples
22. Repeat the above sequence starting with Evaluation Standard Mix B (Step 14 above)
23. Pesticide/PCB analysis sequence must end with Individual Standard Mix A or B regardless of the number of samples analyzed

(a)These standards may be combined into one mixture.

(b)Aroclors 1221 and 1232 must be analyzed once every 30 days on each instrument and each column. Copies of these chromatograms must be submitted with the results for each sample set for all instruments and columns used to quantitate samples in that set.

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where

RT_I = absolute retention time of dibutylchlorendate in the initial standard (Evaluation Standard Mix A)

RT_S = absolute retention time of dibutylchlorendate in the sample.

Report the retention time shift for all standards, blanks, and samples on Form VIII (Appendix B).

- All pesticides or PCBs tentatively identified during the primary analysis must be confirmed by analysis on the second GC column. All the QC requirements described above apply except that only standards for the compounds to be confirmed are analyzed.

11.4.3 RADIOLOGICAL LABORATORY ANALYSIS QUALITY CONTROL

The radiological analysis laboratory QC will provide that the required analysis, QC sample checks, and verification of the results are performed and that the acceptability of the results is known and verfiable. Any deficiencies in the testing program will be identified so proper corrective action can be taken. A more detailed discussion of radiological QC requirements is included in the IT/RSL Radioanalytical Methodology and Procedures Manual and the IT/RSL QA Manual.

11.4.3.1 Duplicate Sample Analysis

One duplicate sample will be run for every 10 to 15 samples. The results of these analyses will be used to determine the percent relative standard deviation (percent RSD), which will be recorded on radiological analysis quality control forms (e.g., control charts) for the parameters being tested. If the results of the percent relative standard deviation are excessive (i.e., outside the control limits) for the materials analyzed and method used, the samples will be reanalyzed.

11.4.3.2 Matrix Spike Analysis

One sample out of every 10 to 15 will be spiked prior to analysis with the parameters of interest to determine the percent bias.

The percent bias will be recorded on radiological analysis QC forms (e.g., control charts). If the results of the percent bias determinations fall outside appropriate values (i.e., control limits) for the material analyzed and method used, the samples will be reanalyzed and the valves recorded.

11.4.3.3 Method and Field Blank Analyses

At least one method blank for each group of samples will be analyzed for the pertinent radiological parameters. The method blanks will consist of a distilled or deionized water sample.

Field blanks will be analyzed to monitor for possible sample contamination during storage and shipment. Field blanks will be prepared by filling two sample containers with distilled water and shipping the blanks to the lab with the sample bottles. The field blanks accompany the sample bottles through collection and shipment to the laboratory and are stored with the samples. If the field blanks indicate possible contamination of the samples, the laboratory manager and site manager will be notified immediately and, depending upon the nature and extent of the contamination, the sample results may be corrected for the field blank concentration or the sources resampled. Results of method and field blank analyses will be filed with the corresponding sample analytical data.

11.5 FIELD QUALITY CONTROL

Field activities are the responsibility of the site manager. Prior to initiating field work, the site manager shall discuss the scope of work, contractual and regulatory requirements, and applicable QA/QC procedures with assigned personnel. At the request of the site manager, this may be done by the QA officer or senior members of the project staff.

Once in the field, field personnel are responsible for all daily QC activities. Included in this responsibility shall be the supervision of subcontractors.

11.5.1 PERFORMANCE DOCUMENTATION

To provide evidence of satisfactory work performance and the basis for subsequent activities and information transmitted external to ASI, the results of field investigations and sampling shall be completely documented. Whenever possible, information shall be recorded on a standardized form. Documentation shall include a daily log of project activities and the appropriate subsurface logs, test and survey data forms, monitoring/sampling equipment installation records, photographs, and field collection and sampling custody forms.

Members of the project staff working in field operations shall keep a daily log of project activities. Each page of the Field Activity Daily Log (Figure 5-1) shall be signed by the person preparing the log. Items to be included in the daily log, as appropriate, are:

- Project identification;
- Field activity subject;
- General work activity;
- Unusual events;
- Changes to plans and specifications;
- Visitors on site;
- Subcontractor progress or problems;
- Communication with the client or others;
- Weather conditions; and
- Personnel on site.

Copies of the Field Activity Daily Logs should be sent to the site manager and the on-site technical manager, approximately on a weekly basis. After review of the logs, they should be routed to other members of the project staff as needed. If the logs are not submitted as required, it is the responsibility of the site manager, or designee, to contact the field personnel.

As part of field activities, a photographic record should be prepared. Photographs should be in color. As examples, photographs should be taken of the general site layout, geologic features, field equipment and installations, sampling stations, and field testing. When test pits or trenches are dug, photographs should be taken of the subsurface profiles, if possible.

Photographs are to be identified by the project number, date taken, and a brief description. This may be done individually on the back of each photograph or in an album in which the photographs are mounted. Album photographs must be labeled with individual descriptions and dates taken.

Before photographs are taken, the client should be contacted to determine their policy concerning photographs.

Appropriate test survey and equipment installation data forms shall also be prepared. All requested information shall be addressed. If not applicable, requested information should be designated as such.

During the performance of the field program, a copy of the field records will be sent to the site manager and on-site technical manager with the original being placed in the central file at the ASI/Fernald office. These copies can provide adequate documentation of work activities should the originals be destroyed, lost, or stolen.

Variances from standard approved field operational procedures and plans shall be documented in a Variance Log (Figure 16-2) or equivalent form. It is recognized that procedures such as work plans cannot be prepared which properly foresee all conditions encountered during a field program. The project director, or designee, shall initiate and chronologically maintain the Variance Log. All items recorded in the Variance Log require the approval of the project director and the QA officer. Approval by the project director or QA officer can be initiated on a verbal basis via telephone. The Variance Log shall contain: date and nature of the variance, applicable document, and personnel initiating the variance. In no case shall a subcontractor initiate a variance. If a variance is proposed by the client, it shall be so recorded.

Formal approval of the Variance Log shall be in writing. The project director shall be provided with a copy of all entries made in the log. Upon receipt, the project director shall review copies of the logs and, when in agreement, indicate approval by signing and dating each variance. The copy shall be forwarded to the QA officer for review, signing, and dating, then returned to the project director for inclusion in the project files. Originals of the Variance Log shall be kept on site until the field work is complete.

11.5.2 VERIFICATION OF DATA REDUCTION

The numerical reduction of field data shall be formally checked using the standard process outlined in Section 10.0. Checking shall be performed prior to the presentation of results. If it becomes necessary to present or use unchecked results, transmittals or subsequent calculations shall be marked "preliminary" until such time that the results are checked and determined to be correct.

All data reduction and resulting tables and graphs shall be checked. This includes computer input, if reduction is performed by computer. Data sheets shall be complete, with all requested information addressed.

The verification of field data reduction is the responsibility of project personnel. The assignment of a checker shall be made or approved by the technical manager or the project director.

12.0 QUALITY ASSURANCE AUDITS

To verify compliance with QAPP requirements, the QA officer and other technically qualified personnel (if required) will perform planned and documented audits of project activities. These audits will consist, as appropriate, of an evaluation of QA procedures and the effectiveness of their implementation, an evaluation of work areas and activities, and a review of project documentation. Audits will be performed in accordance with written checklists and, as appropriate, technical specialists. Audit results will be formally documented and sent to project director and supramanagement.

Audits may include, but not be limited to, the following areas:

- Field operation work procedures and records;
- Laboratory testing and records;
- Equipment calibration and records;
- Identification and control of samples;
- Numerical analyses;
- Computer program documentation and verification;
- Transmittal of information; and
- Record control and retention.

Audits for this project will, as appropriate, cover laboratory activities, field operations and documentation, and final reports. Auditing will be performed in accordance with the applicable section(s) of the Advanced Sciences, Inc. (ASI) Quality Assurance Manual. Internal laboratory audits are also discussed in Laboratory-Specific Attachments.

An individual audit plan shall be developed to provide a basis for each audit. This plan shall identify the audit scope, activities to be audited, audit personnel, any applicable documents, and the schedule. The plan shall be consistent with the project scope of work schedule, and requirements.

12.0 QUALITY ASSURANCE AUDITS

To verify compliance with QAPP requirements, the QA officer and other technically qualified personnel (if required) will perform planned and documented audits of project activities. These audits will consist, as appropriate, of an evaluation of QA procedures and the effectiveness of their implementation, an evaluation of work areas and activities, and a review of project documentation. Audits will be performed in accordance with written checklists and, as appropriate, technical specialists. Audit results will be formally documented and sent to project director and supramanagement.

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- Field operation work procedures and records;
- Laboratory testing and records;
- Equipment calibration and records;
- Identification and control of samples;
- Numerical analyses;
- Computer program documentation and verification;
- Transmittal of information; and
- Record control and retention.

Audits for this project will, as appropriate, cover laboratory activities, field operations and documentation, and final reports. Auditing will be performed in accordance with the applicable section(s) of the Advanced Sciences, Inc. (ASI) Quality Assurance Manual. Internal laboratory audits are also discussed in Laboratory-Specific Attachments.

An individual audit plan shall be developed to provide a basis for each audit. This plan shall identify the audit scope, activities to be audited, audit personnel, any applicable documents, and the schedule. The plan shall be consistent with the project scope of work schedule, and requirements.

A report audit may examine, as appropriate, the documentation and verification of field and laboratory data and results; performance, documentation, and verification of analyses; documentation and verification of computer programs; preparation and verification of drawings, logs, and tables; content, consistency, and conclusions of the report; compliance with regulatory and project requirements; and maintenance and filing of project records.

The records of field operations shall be reviewed to verify that field-related activities were performed in accordance with appropriate project procedures. Items reviewed shall include, but not be limited to, the calibration records of field equipment; daily field activity logs; photographs; and data, logs, and checkprints resulting from the field operations.

The auditing of laboratory testing records shall include, but not be limited to, the originals and checkprints of laboratory data sheets, originals and checkprints of data presentations prepared by the laboratory staff, and laboratory test scheduling records for the project.

Auditing of analyses shall include a complete review of calculations, computer input, sketches, charts, tables, and their associated checkprints that were prepared by the project group. These items shall be reviewed to verify conformance to project requirements.

The report preparation process shall be reviewed to verify that:

- The report correctly and accurately presents the results obtained by the project work;
- All information presented in the report is substantiated by project work;
- The logs, tables, and figures presented in the report are prepared and checked according to applicable requirements; and

- The report satisfies the scope of work, applicable requirements, and any pertinent regulatory requirements.

A report audit shall also, as appropriate, review the maintenance and control of project documents to verify that applicable procedures have been implemented.

A report audit may be performed prior to issuance of a final report. The issuance of the final submittal shall be postponed if the QA officer determines that the work does not meet requirements. If the project schedule demands issuance of a report prior to audit, it may be issued as "preliminary" or "draft," or WMCO may be formally notified that an internal audit is in progress and that the submittal will be considered final only after completion of the audit.

Checklists shall be prepared by the auditors and used to conduct all audits. They shall be developed to accomplish the review of necessary items and to document the results of the audit.

During an audit and upon its completion, the auditors will discuss the findings with the individuals audited and cite corrective actions to be initiated. Minor administrative findings which can be resolved to the satisfaction of the auditors during an audit are not required to be cited as items requiring corrective action. All findings that are not resolved during the course of the audit and findings affecting the overall quality of the project, regardless of when they are resolved, shall be noted on the audit checklists.

Following completion of an audit, the auditors shall prepare and submit an audit report to the project director. This report shall serve to notify management of audit results. The report may also be sent to individuals contacted during the audit and the management of any affected subcontractor.

The report shall be prepared as soon as possible (within 30 days) after the audit and contain, as appropriate:

- Date(s) of the audit;
- Identification of audit participants;
- Identification of activities audited;
- Audit results;
- Description of items requiring corrective action and, if possible, the means of correction;
- Due date for completion of corrective actions and/or audit response; and
- Means for audit response (in writing).

If corrective action is required by the audit report, the corrective action shall be undertaken and completed on schedule. If required, the QA officer is empowered to stop work.

The individuals audited shall respond in writing to the audit report. The response shall clearly state the corrective action taken or planned. If all corrective actions have not been completed prior to issuance of the audit reponse, a scheduled date for completion shall be provided. It is noted that all requests for corrective action must be addressed to the satisfaction of the QA officer.

Completion of corrective action shall be verified by the auditors through written communication, reaudit, or other appropriate means. After acceptance and verification of corrective actions, an audit closure report shall be issued to the same individuals receiving the audit report.

After acceptance and verification of all corrective actions, an audit closure report is issued by the QA officer.

13.0 PREVENTIVE MAINTENANCE

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; and
- Frequency that maintenance is required.

The implementation of a preventive maintenance program is dependent upon the specific instruments and equipment used within a laboratory or a field program; therefore, this section does not designate specific practices for instruments and equipment. The laboratories and field program will maintain a preventive maintenance program which meets the guidelines presented in this section.

Within a laboratory, the laboratory 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. The appropriate task leader, as part of the field program, is responsible for preparation, implementation, and documentation of the program within his task. The QA officer will review implementation to verify compliance.

The 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. Frequency should be stated in terms of monthly, quarterly, etc.;

- For each instrument in the program, provide

  - A list of spare parts maintained by the laboratory or field program;

  - External service contracts; and

  - 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 the IT Analytical Services QA Manual. The master schedule 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; and

- Record of periodic maintenance.

The record of maintenance is documented in ink in bound notebooks which are kept with the corresponding laboratory instrument or on site with the field program. Tables 8-1 through 8-3 list laboratory and field equipment and their related preventive maintenance programs.

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

As part of the analytical QC program, the laboratory will apply the precision and accuracy criteria specified in the U.S. EPA CLP protocols previously cited for each parameter that is analyzed. When the analysis of a sample set is completed, the QC data generated are reviewed and evaluated to validate the data set. The review is based on the following criteria:

- Reagent/Method 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 of future samples halted until the system can be brought under control. A high background is defined as a background value sufficient to result in a difference in the sample values, if not corrected, greater than or equal to the smallest significant digit known to be true. A method blank should contain no greater than two times the parameter detection limit for most parameters (1,2,3,4,5)*.

- Field and Trip Blank Evaluation
  Field and trip blank results are evaluated for high readings similar to the reagent and/or method blanks described above. If high field blank readings are encountered (i.e., a value sufficient to result in a difference in the sample values, if not corrected, greater than or equal to the smallest significant digit known to be true), the procedure for sample collection, shipment, and laboratory analysis should be reviewed. If both the reagent and/or method blanks and the field blanks exhibit significant background contamination, the source of contamination is probably within the laboratory. Ambient air in the laboratory and reagents should be checked as possible sources of contamination. High field blank readings may also be due to contaminated sample bottles or cross contamination due to sample leakage and poorly sealed sample containers (1,5).

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*Criteria are referenced as applicable to:

1 - Volatile Organic Compounds
2 - Base-Neutral and acid extractable organic compounds (including pesticides and PCBs).

- Standard Calibration Curve and Response Factor Evaluation
  The calibration curve or midpoint calibration standard (check standard) is evaluated daily to determine curve 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, sample values must be corrected for nonlinearity by deriving sample concentrations from a graph or by using an appropriate algorithm to fit a nonlinear curve to the standards. In addition, if average response factors are used to calculate sample concentrations, these factors will be verified on a daily basis. Verification of calibration curves and response factors is accomplished when the evaluated response for any parameter varies from the calibrated response by less than ranges given in the applicable test methods listed in the U.S. EPA CLP protocols (1,2,3,4).

- Duplicate Sample Evaluation
  Duplicate sample analysis 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 RPD. If the precision value exceeds the control limit, the sample set must be reanalyzed for the parameter in question. Precision limits will be updated periodically following review of data. Typical values of precision are given in Tables 4-1 through 4-3.

- QC Check Standard Evaluation
  The results of QC check standard analysis are compared with the true values, and the percent recovery of the check standard is calculated. If correction is required (excessive or inadequate percent recovery), the check standard should be reanalyzed to demonstrate that the corrective action has been successful (1,2,3,4).

- 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 U.S. EPA CLP acceptance criteria as presented in Table 4-2. If acceptance criteria are not met, corrective action is taken to correct the problem and the affected sample is reanalyzed (1,2).

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3 - Metals
4 - Wet chemistry (e.g., cyanide, fluoride, and nitrate)
5 - Radiological

- Matrix Spike/Matrix Spike Duplicate Evaluation
  The observed recovery of the spike versus the theoretical spike recovery is used to calculate accuracy as defined by the percent recovery. If the accuracy value exceeds the control limit for the given parameter (Tables 4-1 through 4-3), the appropriate laboratory personnel are notified and corrective action is taken before the sample set is reanalyzed for the parameter in question. The RPD between duplicate matrix spikes is also assessed to evaluate the precision of volatile, acid/base-neutral extractable, and pesticide/PCB analyses. The acceptance criteria in Table 4-2 must be achieved or corrective action must be taken before the affected samples are reanalyzed (1,2,3,4).

15.0 DOCUMENT CONTROL

Project specific documents and drawings must be reviewed, approved, distributed, and revised as necessary.

15.1 Review and Approval of Documents and Drawings

Prior to use, the following documents and drawings must be reviewed and approved by the personnel identified:

- Quality Assurance Project Plan
  - Project QA Officer
  - ASI Project Director
  - WMCO QA Officer
  - DOE Contracting Officer's Technical Representative
  - IT Technical Manager (Review Only)

- Other Project Specific Documents (such as the Work Plan, Sampling Plan, etc.)
  - Project QA Officer
  - IT Technical Manager
  - ASI Project Director
  - DOE Contracting Officer's Technical Representative
  - WMCO QA Officer (Review Only)

- Drawings, including computer graphics and maps
  - Draftperson/Preparer
  - Checker
  - IT Technical Manager
  - ASI Project Director

Approval of these documents and drawings will be denoted by a signature and date. All documents and drawings will be reviewed and approved by the designated ASI and IT personnel before they are submitted to DOE for their review and approval. Requests for a copy of a document or a design drawing before it has gone through the complete review and approval process will merit the document or drawing to be marked "Preliminary."

15.2 Distribution

Documents and drawings will be distributed as requested to ASI, IT, WMCO, and DOE personnel. The Project QA Officer will control distribution of all quality-related documents, including the QAPP, Work Plan, and Sampling Plan. The Project Director will control distribution of all other documents and drawings. When a document or drawing is no longer needed, it will be destroyed or returned to the issuing group. Each copy of the QAPP, Work Plan, Sampling Plan, Health and Safety Plan, and Community Relations Plan will be identified with a document control number. The Project QA Officer will maintain a status log showing the name, control number, and mailing address of each controlled copyholder. The Project Director will also maintain a log for all other documents and drawings.

15.3 Revision of Documents and Drawings

Whenever a document or drawing cited in Section 15.1 is revised, a new review and approval of the revision will be required in accordance with the requirements of the original document or drawing.

Revisions will be issued to all holders of the original document or drawing. For the QAPP, Work Plan, Sampling Plan, Health and Safety Plan, and Community Relations Plan, 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 Project QA Officer. The Project Director, will control revisions of all other documents or drawings. Revision receipts will be returned to the Project Director.

Revisions to documents will be denoted by adding the revision number and revision date to the document title page (if reissued), revised signature page (if reissued), and each page that has been revised. Revisions to design drawings will be denoted by including the consecutive revision number and revision date in the appropriate block on the drawing and revised signature block.

Revisions to the QAPP, Work Plan, Sampling Plan, Health and Safety Plan, and Community Relations Plan will be accomplished by either a general revision in which all pages are replaced, or a limited revision in which only certain pages are replaced. For a general revision, all pages will be identified with the general revision number (e.g., 4, 5, 6, etc.) and the revision date. For a limited revision, each revised page will be pink in color and identified with the current general revision number and sequential suffix (e.g., Rev. 4.1, 4.2, 5.1, etc.) and the revision date. When necessitated by the change, additional pages will retain the original page number with a sequential suffix (e.g., page 4.1 of 6). Each limited revision will be transmitted by a revision log sheet that lists all revised pages for that

revision. The log sheet is to be filed in the front of the revised document. Each revised page in both general and limited revisions will have a vertical line in the right hand margin adjacent to the revised material. Document title pages and signature pages are not required for limited revisions.

15.4 Initiating Changes to Documents

Changes to approved plans and procedures are likely to be necessary during the course of project performance as a result of new information or events that occur during performance. Changes to previously approved plans and procedures must be approved before the change is implemented.

15.4.1 Variances

A variance is an approved variation from a previously approved project specific procedure, such as the QAPP, Work Plan, Sampling Plan, etc and does not impact the quality of the work. Variances are recorded on the VR form shown in Figure 15-1 and must be approved by the Project Director and the Project QA Officer before they are implemented. The approval can be oral, when necessary, provided that written approval is obtained within 5 working days.

Variances do not normally result in revisions to project-specific documents. They are a means of accomplishing on-the-spot changes to project specific procedures when the change is necessary for work to proceed. The change is a one-time change that is valid only for the specific activity described in the VR.

Variance Request forms are initiated as follows:

- The person identifying the need for the variance (the requestor) completes the VR form shown in Figure 15-1 through the REQUESTED BY entry, except for the variance number, which is supplied by the Project QA Officer.

- The requestor obtains the signatures of the Project Director and the Project QA Officer. When necessary, such as when communicating by telephone, the Project Director or the Project QA Officer can orally give their approval to the requestor. In this instance, the requestor notes on the VR, in an area other than in the approval signature spaces, that oral approval has been granted, along with the approvers names and the date and time that approval was granted. Written approval must be obtained by the requestor within 5 working days. When obtaining the approval of the Project QA Officer, the requestor also obtains the VR number and enters it on the VR.

- When approvals have been obtained, the change described on the VR can be implemented.

- The original of the VR is provided to the Project QA Officer for appropriate distribution and forwarding to the project files. The Project QA Officer also evaluates the VR to determine whether or not a revision to the project-specific document is required, and, if appropriate, issues a DCR within 10 working days.

FMPC RI/FS

VR No. _____

VARIANCE REQUEST

Page ____ of ____

Date: _____

VARIANCE (INCLUDE JUSTIFICATION)

APPLICABLE DOCUMENT(S) AND SECTION NO.(S)

CC:

REQUESTED BY: _____ Date: _____

Approved By: _____ Date: _____
Project Director

_____ Date: _____
Project Quality Assurance Officer

FORM005

The Project QA Officer is responsible for maintaining a log that shows for each issued VR, the number, date of issue, requestor, effected document and section, and subject matter.

15.4.2 Document Change Requests

A DCR is a means of initiating a revision to a previously approved project-specific procedure, such as the QAPP, Work Plan, Sampling Plan, etc. DCRs are recorded on the DCR form shown in Figure 15-2. Review and approval of the DCR's shall be in accordance with the requirements of the original document before they are implemented. For DCRs that involve changes to analytical laboratory activities, review by the responsible laboratory director is also required. The Project Director or the Project QA Officer may request oral approval from the other signatories when necessitated by circumstances. If the other signatories orally approve and consent for the DCR to be signed for them, the Project Director or Project QA Officer may sign their own name in the other person's signature space and write "for" before the person's printed title below the signature space.

DCRs always result in revisions to project-specific documents, as opposed to VRs, which normally do not result in revisions.

DCRs are initiated as follows:

- The requestor (normally the person who identifies the need for the change) completes the DCR form up to the EFFECTIVE DATE OF CHANGE.

- The requestor forwards the DCR to the Project QA Officer for evaluation.

- If he concurs with the DCR, the Project QA Officer enters a number in the REQUEST NO. space and also signs and dates the DCR in the appropriate space. If he does not concur, he resolves the disagreement with the requestor.

- The Project QA Officer enters the pertinent information in a DCR status and tracking log that shows the DCR number, requestor, request date, subject matter, affected document and section number(s), transmittal and return date from each signatory, distribution date to each document holder, and issue date of revised pages to the document.

- The Project QA Officer makes a copy of the DCR and forwards the original to the Project Director with a request to review, approve and return to the Project QA Officer. The date is recorded in the DCR status and tracking log.

- When the signed DCR is returned from the Project Director, the Project QA Officer makes the appropriate entry in the DCR status and tracking log, and repeats the forwarding process until all required signatures have been obtained. If any signatory refuses to sign the DCR, that signatory is responsible for communicating to the Project QA Officer the reasons for not signing. The Project QA Officer coordinates the resolution of the disagreement of the DCR, consulting with the Project Director as necessary. In the event that a

Document Change Request No. 28

DOCUMENT CHANGE REQUEST

This form is used to initiate permanent changes to controlled distribution project-specific procedures, such as the QAPP, Work Plan, and Sampling Plan.

REQUEST NO.
Issue Date:
Page ____ of ____
Do Not Write In This Block

REQUESTOR: _____ PHONE NO.: _____ DATE: _____

DOCUMENT TITLE: _____

SECTION/PARAGRAPH/PAGE NO.: _____ DOCUMENT NUMBER: _____

ISSUE DATE: _____ LATEST REVISION DATE: _____

JUSTIFICATION:

CONTENT OF CHANGE:

EFFECTIVE DATE OF CHANGE:

☐ When all approvals have been obtained: _____ Effective Date

☐ Other (Specify): _____

REQUIRED APPROVALS:

Project Director _____ Date _____

Project QA Officer _____ Date _____ WMCO QA Officer _____ Date _____

Technical Manager _____ Date _____ DOE COTR _____ Date _____

TO BE COMPLETED BY DOE

A. Prior EPA notification required? ☐ Yes ☐ No

B. Prior EPA approval required? ☐ Yes ☐ No

C. Immediate implementation? ☐ Yes ☐ No

decision is made to not proceed with the issue of the DCR, the Project QA Officer notifies the requestor and all signatories who have previously signed the DCR of this decision. An appropriate entry to this effect is made in the DCR status and tracking log.

- When all required signatures have been obtained, the project QA Officer issues the DCR to all holders of controlled copies of the affected document(s). The change described by the DCR is to be implemented on the date specified in the EFFECTIVE DATE OF CHANGE space on the DCR.

- The effective date of change and issuance of the DCR is dependent on DOE addressing the section at the bottom of the DCR for EPA notification, EPA approval, or immediate implementation.

Each controlled document copyholder who receives an approved DCR is responsible for inserting a copy of the DCR in the front (or other appropriate location) of the affected document(s). Until revised document pages are issued, the DCR serves as the controlled document copy holder's official notification that the document has been changed as described in the DCR.

Subsequent to issuing a DCR, the Project QA Officer will, as soon as feasible, issue revised document pages incorporating the change described in the DCR. Upon receipt of the revised pages, at the controlled document copyholder's option, the DCR may be discarded or retained. If retained, a notation will be made on the DCR that the change has been incorporated in a revision.

15.5 Computer Graphics Development

The following apply to all computer generated graphics produced from the Fernald database for use in the Remedial Investigation/Feasibility Study. This specifically includes visual displays generated using SAS, SAS Graph, CPS-PC, Autocad, or Freelance software.

15.5.1 Distributed Data

File Transfers - All data obtained from the database for display in graphic form is to be downloaded to a local microcomputer using Kermit File Transfer Protocol. If a location identification is to be used, the coordinates must be transferred simultaneously with the other data and accordance with 15.5.2.

Local Files - Neither the data read to a local computer from the database or the date of data file creation may be altered in any way. This includes data read into a flat file, SAS dataset, an Autocad file, or a Lotus 1-2-3 file. Each individual graphics producer is responsible for maintaining a record of the data filename and the graphic filename used for computer generated graphics and maps.

15.5.2 Location Coordinates/Elevations

Location identification in all graphics will be by State Planar Coordinates. This applies to all sample collection locations and all well locations. Elevation data will be reported relative to Mean Sea Level.

All elevation or coordinates data will be derived from the survey data maintained as part of the Project record.

15.5.3 Labeling

15.5.3.1 General

Graphics shall be titled so as to clearly identify the parameter or parameters displayed. Reporting units must be included on each. Standard two-dimensional (X-Y) or three-dimensional plots must have a scale showing the range of values being displayed.

All graphics which contain data that has not been completely verified shall include the label,

"Preliminary Data: Not for Distribution or Publication"

also labels for,

"Validation Level I" and "Validation Level II"

Validation Level I	All data transferred electronically will be manually verified against signed, hard copy reports.
Validation Level II	Result of technical analysis of all data to date.

Graphics produced during the development of software programs to be used for demonstration purposes should be labeled according to Section 15.5.3.1 above and should also include the label "developmental".

15.5.3.2 Three-Dimensional Plots

In addition to the preceding requirements, these plots must include a north arrow showing two-dimensional rotation. They must also indicate degrees of rotation from the horizontal.

15.5.3.3 Maps

Maps must also show the following:

a. File name;
b. Last date of generation and dates of preceding versions;
c. Preparer and checker initials by each generation/revision date;
d. North arrow and scaler bar.

Maps approved in accordance with 15.1 will be read into plot files with the file extension ".plt". These maps can be made available for use in the on-site map production center.

The map production center display case will contain a hard copy version of each computer generated map.

Example Format:

a. File name;
b. Paper size;
c. File size;
d. Layers included;
e. Description, including any parameters overlaid;
f. All revisions and reasons for change, initialed by preparer and checker;
g. Sign-off by the technical Manager and Project Director for all versions/revisions.

The map production center will also contain a hard copy record for all revisions to the basic map file used with overlays. All revisions or updates to the basic file will be documented, giving date of revision, nature of revision, reason for revision, and person incorporating the revision. An example of a "revision" in this case would be the extension of the area shown in the basic map incorporating the addition of digitized data. The movement of an object relative to the original digitized data file is an example of a change which would not qualify as a revision.

Maps employing data not obtained directly from the database will be labeled with the source of the data.

15.5.4 Trial Runs To Determine Data Placement

Draft graphics initially produced to ascertain data placement, such as where the software employed might cause values to be printed illegibly and placement adjustment may be required to allow readability are exempted from the requirements of 15.5.1, 15.5.2, and 15.5.3. Under no circumstances are such materials to leave the site of generation. In addition, they must be marked "Data Placement Trial Run."

3842

DRAFT

RI/FS
DOCUMENT CHANGE REQUEST

This form is used to initiate and update RI/FS plans and procedures, only.

SAFETY INVOLVED	
YES	NO
☐	☒

REQUEST NO: 24

Completed by: _____
Revision/ Issue Date: _____

DO NOT WRITE IN THIS BLOCK

REQUESTOR David Ponke PHONE NO.: 513-738-3100 DATE 12-19-88
DOCUMENT TITLE QAPP Rev. 3 DOCUMENT NUMBER NA
SECTION/PARAGRAPH/PAGE NO.: Section 16
ISSUE DATE January 1987 LATEST REVISION DATE March 1988

JUSTIFICATION: Clarification of Section 16 Due to Personnel Changes and Responsibilities.

CONTENT OF CHANGE

Per Attachment Sheets

CANCELLATION INSTRUCTIONS

CANCEL DOCUMENT NO.: NA
Reason for cancellation:

REQUIRED APPROVALS

RT Wade 1-24-89
ASI PROJECT DIRECTOR/DATE

David Ponke 12/28/88
ASI QA OFFICER/DATE

Robert M Gallimore 1/5/89
IT PROJECT DIR./DATE

NA 12/28/88
IT QA OFFICER/DATE

MMCS PROJECT MGR./DATE

MMCS IMPACT ASSESS. MGR./DATE

MMCS QA OFFICER/DATE

DOE COTR/DATE

TO BE COMPLETED BY DOE

A. PRIOR EPA NOTIFICATION REQUIRED ? YES ____ NO ____
B. PRIOR EPA APPROVAL REQUIRED ? YES ____ NO ____
C. IMMEDIATE IMPLEMENTATION. YES ____ NO ____

16.0 NONCONFORMANCE/CORRECTIVE ACTION

Nonconforming items and activities are those which do not meet project requirements, procurement document criteria, or approved work procedures. Nonconformances include malfunctions, failures, deficiencies, and deviations. Nonconformances may be detected and identified by:

- Project Staff
  During the performance of project activities, including field investigation and testing, supervision of subcontractors, performance of field inspection, and preparation and verification of numerical analyses;

- Laboratory Staffs
  During the preparation for and performance of laboratory testing, calibration of equipment, and QC activities; or

- Project QA Officer
  During the performance of audits and other quality assurance activities.

Nonconformances are not variances (Section 15.3). Variances are approved and controlled changes to an approved plan or procedure. Variances are approved prior to implementing a change. Nonconformances are uncontrolled and unapproved deviations from approved plans and procedures.

Each nonconformance affecting quality will be documented by the personnel that identifies or creates it. For this purpose, a Nonconformance Report form (Figure 16-1), ITAS laboratory nonconformance memo form (examples are shown on Figures 13-1, 13-2, and 13-3 of the ITAS Quality Assurance Manual), or audit report will be used as appropriate.

16.1 IT ANALYTICAL SERVICES NONCONFORMANCES

16.1.1 ITAS LABORATORY NONCONFORMANCES

Nonconformances identified within ITAS analytical laboratories will be reported in accordance with Section 13.0, "Nonconformances and Corrective Action", of the ITAS Quality Assurance Manual, except that laboratory nonconformances identified during ITAS-performed audits will be reported in accordance with Section 14.0, "Quality Assurance/Quality Control Audits", of the ITAS

Quality Assurance Manual. The project QA officer will be provided with copies of ITAS nonconformance reports and audit reports which identify nonconformances adversely affecting the quality of Fernald RI/FS project data.

16.1.2 FIELD NONCONFORMANCES

Nonconformances identified by personnel during the performance of field sampling or field data management activities will be reported in accordance with Section 16.3 of this QAPP.

16.2 NONCONFORMANCES IDENTIFIED DURING INTERNAL PROJECT AUDITS

Nonconformances identified during audits conducted by, or under the direction of, the project QA officer will be reported in accordance with Section 12.0 of this QAPP.

16.3 REPORTING PROJECT NONCONFORMANCES

All project nonconformances, except for those described in Sections 16.1.1 and 16.2, are to be reported on the Nonconformance Report (NR) form shown in Figure 16-1. The person(s) identifying or creating a nonconformance is responsible for reporting it, unless assurance is provided that it will be reported by someone else and the issuance of the report is verified.

Nonconformances are reported as follows:

- The person responsible for reporting the nonconformance (the initiator) completes Part 1 of the NR by describing the nonconformance including, as appropriate, the date(s) and time(s) of day of occurrence and identification, persons involved, project activity being performed, equipment involved, source of the requirement that was not complied with, and probable adverse impact, if any, of the nonconformance on the quality or completeness of project data.

NOTE: Use continuation pages as necessary to complete Part 1 and subsequent parts of the NCR. Identify all material on continuation pages with the part number and title (i.e., for Part 1 continuation "Part 1, Nonconformance Description-Continued"). Number each continuation page in the upper right corner of Page __ of __.

FERNALD
RI/FS

NONCONFORMANCE REPORT

NR No. _____

Page ____ of ____

Date: _____

1. NONCONFORMANCE DESCRIPTION

Identified By: _____ Date: _____

2. PROPOSED CORRECTIVE ACTION, INCLUDING INITIATION AND COMPLETION DATES

To Be Performed By: _____

3. APPROVAL FOR PROPOSED CORRECTIVE ACTION

_____ Date: _____
Project Director

_____ Date: _____
Project QA Officer

4. CORRECTIVE ACTION TAKEN (IF DIFFERENT FROM THAT PROPOSED)

5. CORRECTIVE ACTION COMPLETE

Performed By: _____ Date: _____

Verified By: _____ Date: _____

FORM008

CC:

- When the nonconformance description is completed, the initiator signs in the IDENTIFIED BY space if the initiator identified or created the nonconformance. If other than the initiator identified or created the nonconformance, the initiator lines through IDENTIFIED BY and adds "Reported By" and signs. The date that the initiator prepared the NR is recorded in the DATE space of Part 1.

- The initiator completes Part 2 of the NR by describing the proposed corrective action for resolving the nonconformance, after consulting with the initiator's supervisor, the Project Director, or the Project QA officer, if appropriate. Initiation and completion dates for the proposed corrective action are included whenever appropriate. The names of the person(s) and/or organizations proposed to perform the corrective action are entered in the TO BE PERFORMED BY space.

- The initiator forwards the NR to the Project QA officer.

- The Project QA officer evaluates Part 1 to determine that the condition or occurrence described represents a nonconformance. If he concurs that it is a valid nonconformance, he enters an NR number in the space provided near the top of the NR form and evaluates Parts 1 and 2 for adequacy and completeness. Any disagreements are resolved with the initiator, and as appropriate, the Project Director and/or the person(s) or organization(s) proposed to perform the corrective action.

- When the Project QA Officer is in agreement with Parts 1 and 2, he signs and dates Part 3.

- The Project QA Officer enters the pertinent information in an NR status log that shows, as a minimum, the NR number, corrective action scheduled completion date, transmittal and return date to and from the Project Director, issue date, corrective action actual completion date, and corrective action verification date.

- The Project QA officer makes a copy of the NR and transmits the original to the Project Director with a request to review and approve and return to the Project QA officer as soon as feasible. The transmittal date is recorded in the NR status log.

- When the signed NR is returned from the Project Director,

the Project QA officer records the current date in the space provided near the top of the NR form and distributes copies of the NR to all affected parties, including the initiator, the person(s) or organization(s) responsible for performing corrective action, the Project Director, and the DOE COTR. The NR original is placed in the project files. Appropriate entries are made in the NR status log.

16.4 CORRECTIVE ACTION PERFORMANCE

Corrective action for nonconformances is performed and completed as follows:

- When the person(s) or organization(s) responsible for performing corrective action receive a copy of the "issued" NR, appropriate action is taken to initiate and complete the corrective by no later than the scheduled dates. If the scheduled dates are not met, the Project Director and Project QA officer are notified prior to those dates.

- When the corrective action is complete, the performing person(s) or organization(s) complete Part 4 of the NR by describing any corrective actions different from that proposed in Part 2. If the corrective action taken is the same as that proposed, "Same as proposed" is entered in Part 4.

- The person performing or responsible for the corrective action sign in the PERFORMED BY space of Part 5 on the original of the NR (if available) or on a copy, enters the date that Part 5 was signed, and forwards the signed NR to the Project QA officer.

- The Project QA officer will make appropriate entries in the NR status log when the signed NR is received.

16.5 CORRECTIVE ACTION VERIFICATION

Completion of corrective actions for nonconformances will be verified by the Project QA officer. Following verification, the Project QA officer will sign the original of the NR in Part 5 and make the appropriate entry in the NR status log.

16.6 EVALUATION OF RECURRING NONCONFORMANCES

As each NR is received, the Project QA officer will determine if it describes a recurring nonconformance. For each recurring nonconformance, the Project QA officer will coordinate an evaluation to determine the cause and will initiate any action required for changes in project requirements or procedures to prevent further recurrences. The Project QA officer, with the assistance of the Project Director, as appropriate, will ensure that the evaluation and results are properly documented.

17.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

There are many forms of quality reporting to various levels of management. Adequate provisions have been made within the QAPP for the reporting of quality-related considerations. Quality-related reports submitted to management include:

- The reporting by project personnel of nonconformances to project management;
- Reporting of the QC Coordinator to laboratory management concerning data accuracy and precision achieved through QC sample analyses and analyses of performance evaluation samples;
- Reporting of the QC Coordinator to laboratory management of nonconformances observed during internal laboratory system audits; and
- Audits conducted by the QA officer (Section 12.0).

A final report for each phase of the project will include a separate QA section which summarizes data quality information contained in the report.

Following completion of an audit, the auditors shall prepare and submit an audit report to the project director. This report shall serve to notify management of audit results. The report may also be sent to individuals contacted during the audit and the management of any affected subcontractor.

The report shall be prepared as soon as possible (within 30 days) after the audit and contain, as appropriate:

- Date(s) of the audit;
- Identification of audit participants;
- Identification of activities audited;
- Audit results;
- Description of items requiring corrective action and, if possible, the means of correction;

- Due date for completion of corrective actions and/or audit response; and
- Means for audit response (in writing).

If corrective action is required in the audit report, the corrective action shall be undertaken and completed on schedule. If required, the QA officer is empowered to stop work on the project pending resolution.

The individuals audited shall respond in writing to the audit report. The response shall clearly state the corrective action taken or planned. If all corrective actions have not been completed prior to issuance of the audit reponse, a scheduled date for completion shall be provided. It is noted that all requests for corrective action must be addressed to the satisfaction of the QA officer.

Completion of corrective action shall be verified by the auditors through written communication, reaudit, or other appropriate means. After acceptance and verification of corrective actions, an audit closure report shall be issued to the same individuals who received the audit report.

18.0 RECORDS ADMINISTRATION

This project will require the administration of on-site, laboratory, and off-site office project record files. The records systems shall provide adequate control and retention for project-related information. Record control shall include receipt from external sources, transmittal, transfer to storage, and indication of record status. Retention shall include receipt at storage areas, indexing and filing, storage and maintenance, and retrieval.

18.1 ON-SITE PROJECT FILES

18.1.1 RECORD CONTROL

The control of records provides for the flow of information both internal and external. Receipt of records from outside sources and transmittal of records external are discussed in Section 3.3 of the QAPP.

Following receipt of information from external sources, completion of the field phases of the project, completion of analyses, and issuance of reports or other transmittals; associated records shall be submitted to the on-site files at the ASI/Fernald office. They shall include records generated by subcontractors. Records shall be legible and easily identifiable. In addition, field records and records transmitted between ASI and subcontractor offices shall be adequately protected from damage and loss during transfer (e.g., hand carrying, making copies prior to shipment).

Field records and checkprints; laboratory data summaries; numerical calculations and checkprints; reports and other data transmittals; copies of proposals, purchase orders for project services and contracts; correspondence including incoming and outgoing letters, memorandums, and telephone records; photographs; reference material; and drawing checkprints shall be

transferred to the on-site file. Documentation and verification of computer programs shall be submitted to the on-site file (for project-specific programs).

Records submitted to the on-site file, with the exception of correspondence, should be bound, placed in folders or binders, or otherwise secured for filing.

18.1.2 RECORD STATUS

All individuals on the project staff are responsible for reporting obsolete or superseded project-related information to the project director. In turn, the project director shall notify the project and laboratory staffs and the QA officer of the resulting status change in project documents, such as drawings and project procedures. The source of such documents may be ASI, any of ASI's subcontractors, or WMCO. Notification of personnel of status changes in QA procedures shall be the responsibility of the QA officer.

In general, outdated drawings and other documents shall be marked "void." However, the project director may request the copies be destroyed. It is recommended that one copy of void documents be maintained for the on-site files with the reasons for and date of voiding clearly indicated.

To denote calculations, drawings, and other material which have not been formally checked, or are based on information which has not been checked, or do not contribute to final project information; these documents shall be marked "preliminary."

18.1.3 RECORD RETENTION

Information associated with the project shall be retained in the files.

These files will include the following:

- Project file (project material except drawing originals, records related to laboratory analysis [Section 18.3]);
- Original drawing file; and
- Quality assurance file.

Project records shall be received at the various storage areas by designated personnel. Designated personnel shall check that incoming records have proper identification for filing, are legible, and are in suitable condition for storage. Indexing and filing of records shall be performed only by the designated personnel.

For the on-site file, the individual file folders shall be divided into appropriate categories based on content, and numbered and filed sequentially within each category.

A numbered index for the project on-site file shall be prepared and maintained. The index shall list the individual file folders and identify the records therein to facilitate locating the records. The index shall be kept in a separate folder at the front of the on-site file. If appropriate, information on project material not stored in the on-site file should be included with the index.

Record storage in the on-site files shall utilize facilities that provide a suitable environment to minimize deterioration or damage and that prevent loss. The facilities shall, when possible, have controlled access and shall provide protection from excess moisture and temperature extremes. Records shall be secured in binders, placed in folders or envelopes, or otherwise secured for storage in containers (e.g., steel file cabinets).

Storage systems shall provide for the prompt retrieval of information for reference or use outside the storage areas. For the on-site file, sign-out sheets shall be maintained so that a record of files removed is available. Project records shall be accessible by WMCO.

18.2 OFF-SITE OFFICE PROJECT FILES

Appropriate requirements for off-site office control and retention of records generated as a result of this project shall be followed. A file, similar to the on-site file (Sections 18.1.1 and 18.1.3), will be established and maintained by the office personnel under the direction of the appropriate office manager.

Upon completion of the project or project phase, the off-site office project file will be transferred to, and integrated with, the on-site files.

18.3 LABORATORY FILES

Laboratories will maintain record systems for documents pertinent to testing performance. These systems will provide record control and retention similar to that outlined in Sections 18.1.1 through 18.1.3 for the on-site office files (i.e., submittal to the file, indexing and filing by designated personnel, filing by categories or subjects, storage and maintenance to minimize damage and prevent loss, record retrieval).

Upon completion of the project, the originals of all laboratory data and records will be turned over to WMCO. Contingent upon approval by WMCO, laboratory data files and records will be microfilmed for archival storage. Project laboratories will be allowed to retain copies of their project data and records for their file.

APPENDIX A

U.S. ENVIRONMENTAL PROTECTION AGENCY
CONTRACT LABORATORY PROGRAM
REPORTING FORMS FOR INORGANICS ANALYSIS

Form I

U.S. EPA Contract Laboratory Program
Sample Management Office
P.O. Box 818 - Alexandria, VA 22313
703/557-2490 FTS: 8-557-2490

EPA Sample No. _____

Date _____

INORGANIC ANALYSIS DATA SHEET

LAB NAME _____ CASE NO. _____

SOW NO. _____

LAB SAMPLE ID. NO. _____ QC REPORT NO. _____

Elements Identified and Measured

Concentration: Low _____ Medium _____

Matrix: Water _____ Soil _____ Sludge _____ Other _____

ug/L or mg/kg dry weight (Circle One)

1.	Aluminum	13.	Magnesium
2.	Antimony	14.	Manganese
3.	Arsenic	15.	Mercury
4.	Barium	16.	Nickel
5.	Beryllium	17.	Potassium
6.	Cadmium	18.	Selenium
7.	Calcium	19.	Silver
8.	Chromium	20.	Sodium
9.	Cobalt	21.	Thallium
10.	Copper	22.	Vanadium
11.	Iron	23.	Zinc
12.	Lead		Precent Solids (%)

Cyanide _____

Footnotes: For reporting results to EPA, standard result qualifiers are used as defined on Cover Page. Additional flags or footnotes explaining results are encouraged. Definition of such flags must be explicit and contained on Cover Page, however.

Comments: _____

Lab Manager _____

Form II

Q. C. Report No. _____

INITIAL AND CONTINUING CALIBRATION VERIFICATION[3]

LAB NAME _____ CASE NO. _____

SOW NO. _____

DATE _____ UNITS _____

Compound	Initial Calib.[1]			Continuing Calibration[2]					
Metals:	True Value	Found	%R	True Value	Found	%R	Found	%R	Method[4]
1. Aluminum									
2. Antimony									
3. Arsenic									
4. Barium									
5. Beryllium									
6. Cadmium									
7. Calcium									
8. Chromium									
9. Cobalt									
10. Copper									
11. Iron									
12. Lead									
13. Magnesium									
14. Manganese									
15. Mercury									
16. Nickel									
17. Potassium									
18. Selenium									
19. Silver									
20. Sodium									
21. Thallium									
22. Vanadium									
23. Zinc									
Other: _____									
Cyanide									

[1] Initial Calibration Source _____ [2] Continuing Calibration Source _____

[3] Control Limits: Mercury and Tin 80-120; Other Metals 90-110; Cyanide 85-115

[4] Indicate Analytical Method Used: P - ICP; A - Flame AA; F - Furnace AA

Form III

Q. C. Report No. _____

BLANKS

LAB NAME _____ CASE NO. _____

DATE _____ UNITS _____

Matrix _____

Compound	Initial Calibration Blank Value	Continuing Calibration Blank Value 1	2	3	4	Preparation Blank Matrix: _____ 1	Matrix: _____ 2
Metals:							
1. Aluminum							
2. Antimony							
3. Arsenic							
4. Barium							
5. Beryllium							
6. Cadmium							
7. Calcium							
8. Chromium							
9. Cobalt							
10. Copper							
11. Iron							
12. Lead							
13. Magnesium							
14. Manganese							
15. Mercury							
16. Nickel							
17. Potassium							
18. Selenium							
19. Silver							
20. Sodium							
21. Thallium							
22. Vanadium							
23. Zinc							
Other: _____							
Cyanide							

Form IV

Q. C. Report No. _____

ICP INTERFERENCE CHECK SAMPLE

LAB NAME _____ CASE NO. _____

Check Sample I.D. _____

DATE _____ Check Sample Source _____

Units _____

Compound	Control Limits[1] Mean	Control Limits[1] Std. Dev.	True[2]	Initial Observed	Initial %R	Final Observed	Final %R
Metals:							
1. Aluminum							
2. Antimony							
3. Arsenic							
4. Barium							
5. Beryllium							
6. Cadmium							
7. Calcium							
8. Chromium							
9. Cobalt							
10. Copper							
11. Iron							
12. Lead							
13. Magnesium							
14. Manganese							
15. Mercury							
16. Nickel							
17. Potassium							
18. Selenium							
19. Silver							
20. Sodium							
21. Thallium							
22. Vanadium							
23. Zinc							
Other:							

[1] Mean value based on n = _____.

[2] True value of EPA ICP Interference Check Sample or contractor standard.

Form V

Q. C. Report No. _____

3842

SPIKE SAMPLE RECOVERY

LAB NAME _____

DATE _____

CASE NO. _____

EPA Sample No. _____

Lab Sample ID No. _____

Units _____

Matrix _____

Compound	Control Limit %R	Spiked Sample Result (SSR)	Sample Result (SR)	Spiked Added (SA)	%R[1]
Metals:					
1. Aluminum	75-125				
2. Antimony	-				
3. Arsenic	-				
4. Barium	-				
5. Beryllium	-				
6. Cadmium	-				
7. Calcium	-				
8. Chromium	-				
9. Cobalt	-				
10. Copper	-				
11. Iron	-				
12. Lead	-				
13. Magnesium	-				
14. Manganese	-				
15. Mercury	-				
16. Nickel	-				
17. Potassium	-				
18. Selenium	-				
19. Silver	-				
20. Sodium	-				
21. Thallium	-				
22. Vanadium	-				
23. Zinc	-				
Other:					
Cyanide	-				

[1] %R = [(SSR - SR)/SA] x 100

"N" - out of control

"NR" - Not required

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Comments: _____

Form VI

Q. C. Report No. _____

DUPLICATES

LAB NAME _____

DATE _____

CASE NO. _____

EPA Sample No. _____

Lab Sample ID No. _____

Units _____

Matrix _____

Compound	Control Limit[1]	Sample(S)	Duplicate(D)	RPD[2]
Metals:				
1. Aluminum				
2. Antimony				
3. Arsenic				
4. Barium				
5. Beryllium				
6. Cadmium				
7. Calcium				
8. Chromium				
9. Cobalt				
10. Copper				
11. Iron				
12. Lead				
13. Magnesium				
14. Manganese				
15. Mercury				
16. Nickel				
17. Potassium				
18. Selenium				
19. Silver				
20. Sodium				
21. Thallium				
22. Vanadium				
23. Zinc				
Other:				
Cyanide				

* Out of Control

[1] To be added at a later date.

[2] RPD = [|S - D|/((S + D)/2)] x 100

NC - Non calculable RPD due to value(s) less than CRDL

Form VII

Q.C. Report No. _____

INSTRUMENT DETECTION LIMITS AND

LABORATORY CONTROL SAMPLE

LAB NAME _____ CASE NO. _____ DATE _____

LCS NO. _____

Compound	Required Detection Limits (CRDL)-ug/l	Instrument Detection Limits (IDL)-ug/l ICP/AA ID# _____	Instrument Detection Limits (IDL)-ug/l Furnace ID# _____	Lab Control Sample ug/L mg/kg (circle one) True	Found	%R
Metals:						
1. Aluminum	200					
2. Antimony	60					
3. Arsenic	10					
4. Barium	200					
5. Beryllium	5					
6. Cadmium	5					
7. Calcium	5000					
8. Chromium	10					
9. Cobalt	50					
10. Copper	25					
11. Iron	100					
12. Lead	5					
13. Magnesium	5000					
14. Manganese	15					
15. Mercury	0.2					
16. Nickel	40					
17. Potassium	5000					
18. Selenium	5					
19. Silver	10					
20. Sodium	5000					
21. Thallium	10					
22. Vanadium	50					
23. Zinc	20					
Other:						
Cyanide	10	NR	NR			

NR - Not required

Form VIII

Q.C. Report No. _____

STANDARD ADDITION RESULTS

LAB NAME _____ CASE NO. _____

DATE _____ UNITS _____

EPA Sample #	Element	0 ADD ABS.	1 ADD		2 ADD		3 ADD		FINAL CON.[2]	r*
			CON.	ABS[1]	CON.	ABS.[1]	CON.	ABS.[1]		

[1] CON is the concentration added, ABS. is the instrument readout in absorbance or concentration.

[2] Concentration as determined by MSA

*"r" is the correlation coefficient.

+ - correlation coefficient is outside of control window of 0.995.

Form IX

Q. C. Report No. _____

ICP SERIAL DILUTIONS

LAB NAME _____ CASE NO. _____

EPA Sample No. _____

DATE _____ Lab Sample ID No. _____

Units _____

Matrix _____

Compound	Initial Sample Concentration(I)	Serial Dilution[1] Result(S)	% Difference[2]
Metals:			
1. Aluminum			
2. Antimony			
3. Arsenic			
4. Barium			
5. Beryllium			
6. Cadmium			
7. Calcium			
8. Chromium			
9. Cobalt			
10. Copper			
11. Iron			
12. Lead			
13. Magnesium			
14. Manganese			
15. Nickel			
16. Potassium			
17. Selenium			
18. Silver			
19. Sodium			
20. Thallium			
21. Vanadium			
22. Zinc			
Other: _____			

[1] Diluted sample concentration corrected for 1:4 dilution (see Exhibit D)

[2] Percent Difference = $\frac{|I - S|}{I} \times 100$

NR - Not Required, initial sample concentration less than 10 times IDL

NA - Not Applicable, analyte not determined by ICP

Form X

QC Report No. _____

HOLDING TIMES

LAB NAME _____

DATE _____ CASE NO. _____

EPA Sample No.	Matrix	Date Received	Mercury Prep Date	Mercury Holding Time (Days)	CN Prep Date	CN Holding Time (Days)

GC/MS TUNING AND MASS CALIBRATION
Decafluorotriphenylphosphine (DFTPP)

Case No _____ Contractor _____ Contract No _____

Instrument ID _____ Date _____ Time _____

Lab ID _____ Data Release Authorized By _____

m/e	ION ABUNDANCE CRITERIA	%RELATIVE ABUNDANCE
51	30.0 - 60.0% of mass 198	
68	less than 2.0% of mass 69	( )[1]
69	mass 69 relative abundance	
70	less than 2.0% of mass 69	( )[1]
127	40.0 - 60.0% of mass 198	
197	less than 1.0% of mass 198	
198	base peak, 100% relative abundance	
199	5.0 - 9.0% of mass 198	
275	10.0 - 30.0% of mass 198	
365	greater than 1.00% of mass 198	
441	present, but less than mass 443	
442	greater than 40.0% of mass 198	
443	17.0 - 23.0% of mass 442	( )[2]

THIS PERFORMANCE TUNE APPLIES TO THE FOLLOWING SAMPLES, BLANKS AND STANDARDS.

[1] Value in parentheses is % mass 69
[2] Value in parentheses is % mass 442

SAMPLE ID	LAB ID	DATE OF ANALYSIS	TIME OF ANALYSIS

FORM V 7/85

Form V. DFTPP Tuning and Mass Calibration.

GC/MS TUNING AND MASS CALIBRATION

Bromofluorobenzene (BFB)

Case No _____ Contractor _____ Contract No _____

Instrument ID _____ Date _____ Time _____

Lab ID _____ Data Release Authorized By _____

m/e	ION ABUNDANCE CRITERIA	%RELATIVE ABUNDANCE
50	15.0 - 40.0% of the base peak	
75	30.0 - 60.0% of the base peak	
95	Base peak, 100% relative abundance	
96	5.0 - 9.0% of the base peak	
173	Less than 1.0% of the base peak	
174	Greater than 50.0% of the base peak	
175	5.0 - 9.0% of mass 174	( )[1]
176	Greater than 95.0% but less than 101.0% of mass 174	( )[1]
177	5.0 - 9.0% of mass 176	( )[2]

THIS PERFORMANCE TUNE APPLIES TO THE FOLLOWING SAMPLES, BLANKS AND STANDARDS.

[1]Value in parenthesis is % mass 174
[2]Value in parenthesis is % mass 176.

SAMPLE ID	LAB ID	DATE OF ANALYSIS	TIME OF ANALYSIS

FORM V 7/85

Form V. BFB Tuning and Mass Calibration.

Initial Calibration Data
Volatile HSL Compounds

Case No. _____ Region _____ Instrument I.D. _____
Contractor _____ Calibration Date _____
Contract No. _____

Minimum $\overline{RF}$ for SPCC is 0.300 (0.25 for Bromoform) Maximum % RSD for CCC is 30%

Laboratory ID								
Compound	RF_{20}	RF_{50}	RF_{100}	RF_{150}	RF_{200}	$\overline{RF}$	% RSD	CCC* SPCC**
Chloromethane								**
Bromomethane								
Vinyl Chloride								*
Chloroethane								
Methylene Chloride								
Acetone								
Carbon Disulfide								
1,1-Dichloroethene								*
1,1-Dichloroethane								**
Trans-1,2-Dichloroethene								
Chloroform								*
1,2-Dichloroethane								
2-Butanone								
1,1,1-Trichloroethane								
Carbon Tetrachloride								
Vinyl Acetate								
Bromodichloromethane								
1,2-Dichloropropane								*
Trans-1,3-Dichloropropene								
Trichloroethene								
Dibromochloromethane								
1,1,2-Trichloroethane								
Benzene								
cis-1,3-Dichloropropene								
2-Chloroethylvinylether								
Bromoform								**
4-Methyl-2-Pentanone								
2-Hexanone								
Tetrachloroethene								
1,1,2,2-Tetrachloroethane								**
Toluene								*
Chlorobenzene								**
Ethylbenzene								*
Styrene								
Total Xylenes								

RF - Response Factor (submitted as the amount of ug/L)
$\overline{RF}$ - Average Response Factor
%RSD - Percent Relative Standard Deviation

CCC - Calibration Check Compounds (*)
SPCC - System Performance Check Compounds (**)

Form VI

Form VI. Initial Calibration - Volatile HSL Compounds.

Initial Calibration Data
Semivolatile HSL Compounds
(Page 1)

Case No: _____ Region: _____ Instrument ID: _____

Contractor _____ Calibration Date _____

Contract No. _____

Minimum $\overline{RF}$ for SPCC is 0.050 Maximum % RSD for CCC is 30%

Laboratory ID								
Compound	RF_{20}	RF_{50}	RF_{80}	RF_{120}	RF_{160}	$\overline{RF}$	% RSD	CCC* SPCC**
Phenol								
Bis(2-Chloroethyl)Ether								
2-Chlorophenol								
1,3-Dichlorobenzene								
1,4-Dichlorobenzene								*
Benzyl Alcohol								
1,2-Dichlorobenzene								
2-Methylphenol								
Bis(2-Chloroisopropyl)Ether								
4-Methylphenol								
N-Nitroso-Di-n-Propylamine								**
Hexachloroethane								
Nitrobenzene								
Isophorone								
2-Nitrophenol								*
2,4-Dimethylphenol								
Benzoic Acid	†							
Bis(2-Chloroethoxy)Methane								
2,4-Dichlorophenol								
1,2,4-Trichlorobenzene								
Naphthalene								
4-Chloroaniline								
Hexachlorobutadiene								*
4-Chloro-3-Methylphenol								*
2-Methylnaphthalene								
Hexachlorocyclopentadiene								**
2,4,6-Trichlorophenol								*
2,4,5-Trichlorophenol	†							
2-Chloronaphthalene								
2-Nitroaniline	†							
Dimethyl Phthalate								
Acenaphthylene								
3-Nitroaniline	†							
Acenaphthene								*
2,4-Dinitrophenol	†							**
4-Nitrophenol	†							**
Dibenzofuran								

Response Factor (subscript is the amount of nanograms)
$\overline{RF}$ - Average Response Factor
%RSD - Percent Relative Standard Deviation
CCC - Calibration Check Compounds (*)

SPCC - System Performance Check Compounds (**)
† Not detectable at 20 ng

Form VI

Form VI. Initial Calibration - Semi-Volatile HSL Compounds.

Initial Calibration Data
Semivolatile HSL Compounds
(Page 2)

Case No _____ Region _____ Instrument ID _____
Contractor _____ Calibration Date _____
Contract No _____

Minimum $\overline{RF}$ for SPCC is 0.050 Maximum % RSD for CCC is 30%

Laboratory ID								
Compound	RF_{20}	RF_{50}	RF_{80}	RF_{120}	RF_{160}	$\overline{RF}$	% RSD	CCC* SPCC**
2,4-Dinitrotoluene								
2,6-Dinitrotoluene								
Diethylphthalate								
4-Chlorophenyl-phenylether								
Fluorene								
4-Nitroaniline	†							
4,6-Dinitro-2-Methylphenol	†							
N-Nitrosodiphenylamine (1)								•
4-Bromophenyl-phenylether								
Hexachlorobenzene								
Pentachlorophenol	†							•
Phenanthrene								
Anthracene								
Di-N-Butylphthalate								
Fluoranthene								•
Pyrene								
Butylbenzylphthalate								
3,3'-Dichlorobenzidine								
Benzo(a)Anthracene								
bis(2-Ethylhexyl)Phthalate								
Chrysene								
Di-n-Octyl Phthalate								•
Benzo(b)Fluoranthene								
Benzo(k)Fluoranthene								
Benzo(a)Pyrene								•
Indeno(1,2,3-cd)Pyrene								
Dibenz(a,h)Anthracene								
Benzo(g,h,i)Perylene								

Response Factor (subscript denotes amount of nanograms)
$\overline{RF}$ - Average Response Factor
%RSD - Percent Relative Standard Deviation
CCC - Calibration Check Compounds (*)

SPCC - System Performance Check Compounds (**)
† - Not detectable at 20 ng
(1) - Cannot be separated from diphenylamine

Form VI

Form VI. Initial Calibration - Semi-Volatile HSL Compounds.

Continuing Calibration Check
Volatile HSL Compounds

Case No _____ Region _____ Calibration Date _____

Contractor _____ Time _____

Contract No _____ Laboratory ID _____

Instrument ID _____ Initial Calibration Date _____

Minimum RF for SPCC is 0.300 (0.25 for Bromoform); Maximum %D for CCC is 25%

Compound	RF	RF_{50}	%D	CCC	SPCC
Chloromethane					• •
Bromomethane					
Vinyl Chloride				•	
Chloroethane					
Methylene Chloride					
Acetone					
Carbon Disulfide					
1, 1-Dichloroethene				•	
1, 1-Dichloroethane					• •
Trans-1, 2-Dichloroethene					
Chloroform				•	
1, 2-Dichloroethane					
2-Butanone					
1, 1, 1-Trichloroethane					
Carbon Tetrachloride					
Vinyl Acetate					
Bromodichloromethane					
1, 2-Dichloropropane				•	
Trans-1, 3-Dichloropropene					
Trichloroethene					
Dibromochloromethane					
1, 1, 2-Trichloroethane					
Benzene			•		
cis-1, 3-Dichloropropene					
2-Chloroethylvinylether					
Bromoform					• •
4-Methyl-2-Pentanone					
2-Hexanone					
Tetrachloroethene					
1, 1, 2, 2-Tetrachloroethane					• •
Toluene				•	
Chlorobenzene					• •
Ethylbenzene				•	
Styrene					
Total Xylenes					

RF_{50} Response Factor from daily standard file at 50 ug/l
RF Average Response Factor from initial calibration Form VI

%D Percent Difference
CCC Calibration Check Compounds (•)
SPCC System Performance Check Compounds (••)

Form VII

Form VII. Continuing Calibration Data - Volatiles.

Continuing Calibration Check
Semivolatile HSL Compounds
(Page 1)

Case No. _____ Region _____ Calibration Date _____
Contractor _____ Time _____
Contract No _____ Laboratory ID _____
Instrument ID _____ Initial Calibration Date _____

Minimum RF for SPCC is 0.050 Maximum %D for CCC is 25%

Compound	$\overline{RF}$	RF_{50}	%D	CCC	SPCC
Phenol				•	
Bis(2-Chloroethyl)Ether					
2-Chlorophenol					
1,3-Dichlorobenzene					
1,4-Dichlorobenzene				•	
Benzyl Alcohol					
1,2-Dichlorobenzene					
2-Methylphenol					
Bis(2-Chloroisopropyl)Ether					
4-Methylphenol					
N-Nitroso-Di-n-Propylamine					• •
Hexachloroethane					
Nitrobenzene					
Isophorone					
2-Nitrophenol				•	
2,4-Dimethylphenol					
Benzoic Acid †					
Bis(2-Chloroethoxy)Methane					
2,4-Dichlorophenol				•	
1,2,4-Trichlorobenzene					
Naphthalene				•	
4-Chloroaniline					
Hexachlorobutadiene				•	
4-Chloro-3-Methylphenol				•	
2-Methylnaphthalene					
Hexachlorocyclopentadiene					• •
2,4,6-Trichlorophenol				•	
2,4,5-Trichlorophenol †					
2-Chloronaphthalene					
2-Nitroaniline †					
Dimethyl Phthalate					
Acenaphthylene					
3-Nitroaniline †					
Acenaphthene				•	
2,4-Dinitrophenol †					• •
4-Nitrophenol †					• •
Dibenzofuran					

RF_{50} = Response factor from daily standard (at 50 ng concentration indicated (50 total nanograms)
$\overline{RF}$ = Average Response Factor from initial calibration Form VI
† = Due to low response, analyze at 80 total nanograms
%D = Percent Difference
CCC = Calibration Check Compounds
SPCC = System Performance Check Compounds

Form VII

Form VII. Continuing Calibration Data - Semi-Volatiles.

Continuing Calibration Check
Semivolatile HSL Compounds
(Page 2)

Case No _____ Region _____ Calibration Date _____

Contractor _____ Time _____

Contract No _____ Laboratory ID _____

Instrument ID _____ Initial Calibration Date _____

Minimum RF for SPCC is 0.050 Maximum %D for CCC is 25%

Compound	RF	RF_{50}	% D	CCC	SPCC
2,4-Dinitrotoluene					
2,6-Dinitrotoluene					
Diethylphthalate					
4-Chlorophenyl-phenylether					
Fluorene					
4-Nitroaniline †					
4,6-Dinitro-2-Methylphenol †					
N-Nitrosodiphenylamine (1)				*	
4-Bromophenyl-phenylether					
Hexachlorobenzene					
Pentachlorophenol †				*	
Phenanthrene					
Anthracene					
Di-N-Butylphthalate					
Fluoranthene				*	
Pyrene					
Butylbenzylphthalate					
3,3'-Dichlorobenzidine					
Benzo(a)Anthracene					
bis(2-Ethylhexyl)Phthalate					
Chrysene					
Di-n-Octyl Phthalate				*	
Benzo(b)Fluoranthene					
Benzo(k)Fluoranthene					
Benzo(a)Pyrene				*	
Indeno(1,2,3-cd)Pyrene					
Dibenz(a,h)Anthracene					
Benzo(g,h,i)Perylene					

RF_{50} Response Factor from daily standard file at concentration indicated (50 total nanograms)

RF Average Response Factor from initial calibration Form VI

%D Percent Difference

† - Due to low response analyze at 80 total nanograms

CCC Calibration Check Compounds (*)

SPCC System Performance Check Compounds (**)

(1) Cannot be separated from diphenylamine.

Form VII

Form VII. Continuing Calibration Data - Semi-Volatiles.

Pesticide Evaluation Standards Summary
(Page 1)

Case No: _____ Region _____ Laboratory _____
Contract No _____ GC Column _____
Date of Analysis _____ Instrument ID _____

Evaluation Check for Linearity

Laboratory ID				
Pesticide	Calibration Factor Eval Mix A	Calibration Factor Eval Mix B	Calibration Factor Eval Mix C	% RSD (≤10%)
Aldrin				
Endrin				
4 4'- DDT[1]				
Dibutyl Chlorendate				

Evaluation Check for 4.4'- DDT/Endrin Breakdown
(percent breakdown expressed as total degradation)

	Laboratory I.D	Time of Analysis	Endrin	4,4'- DDT	Combined[2]
Eval Mix B 72 Hour					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					

(1) See Exhibit E, Section 7 5 4
(2) See Exhibit E, Section 7 3 1 2 2 1

Form VIII

Form VIII. Pesticide Evaluation Standards Summary.

Pesticide Evaluation Standards Summary
(Page 2)
Evaluation of Retention Time Shift for Dibutyl Chlorendate
Report all standards, blanks and samples

SMO Sample No	Lab I.D	Time of Analysis	Percent Diff	SMO Sample No	Lab I.D	Time of Analysis	Percent Diff

Form VIII (Continued)

Form VIII. (continued).

PESTICIDE/PCB STANDARDS SUMMARY

Case No _____ Laboratory _____
Contract No. _____ GC Column _____ GC Instrument ID _____

	DATE OF ANALYSIS _____ TIME OF ANALYSIS _____ LABORATORY ID _____				DATE OF ANALYSIS _____ TIME OF ANALYSIS _____ LABORATORY ID _____			
COMPOUND	RT	RETENTION TIME WINDOW	CALIBRATION FACTOR	CONF. OR QUANT.	RT	CALIBRATION FACTOR	CONF. OR QUANT.	PERCENT DIFF **
alpha-BHC								
beta-BHC								
delta-BHC								
gamma-BHC								
Heptachlor								
Aldrin								
Heptachlor Epoxide								
Endosulfan I								
Dieldrin								
4,4'-DDE								
Endrin								
Endosulfan II								
4,4'-DDD								
Endosulfan Sulfate								
4,4'-DDT								
Methoxychlor								
Endrin Ketone								
Tech. Chlordane								
alpha-Chlordane*								
gamma-Chlordane*								
Toxaphene								
Aroclor - 1016								
Aroclor - 1221								
Aroclor - 1232								
Aroclor - 1242								
Aroclor - 1248								
Aroclor - 1254								
Aroclor - 1260								

* SEE EXHIBIT E, PART 7

** CONF. = CONFIRMATION (<20% DIFFERENCE)
QUANT. = QUANTITATION (<15% DIFFERENCE)

FORM IX

Form IX. Pesticide/PCB Standards Summary.

Pesticide/PCB Identification

Case No. _____ Laboratory _____

Contract No. _____

SAMPLE ID	PRIMARY COLUMN	PESTICIDE/ PCB	RT OF TENTATIVE ID	RT WINDOW OF APPROPRIATE STANDARD	CONFIRMATION COLUMN	RT ON CONFIRMATORY COLUMN	RT WINDOW OF APPROPRIATE STANDARD	GC/MS CONFIRMED (Y or N)

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FORM X

Form X. Pesticide/PCB Identification.

U.S. EPA Contract Laboratory Program
Sample Management Office
P.O. Box 818 - Alexandria, VA 22313
703/557-2490 FTS: 8-557-2490

Date _____

COVER PAGE
INORGANIC ANALYSES DATA PACKAGE

Lab Name _____ Case No. _____

SOW No. _____ Q.C. Report No. _____

Lab Receipt Date _____

Sample Numbers

EPA No.	Lab ID No.	EPA No.	Lab ID No.

Comments: _____

ICP interelement and background corrections applied? Yes ____ No ____.

If yes, corrections applied before _____ or after _____ generation of raw data.

Footnotes:

NR - Not required by contract at this time

Form I:

Value - If the result is a value greater than or equal to the instrument detecion limit but less than the contract-required detection limit, report the value in brackets (i.e., [10]). Indicate the analytical method used with P (for ICP), A (for Flame AA) or F (for Furnace AA).

U - Indicates element was analyzed for but not detected. Report with the instrument detection limit value (e.g., 10U).

E - Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page.

s - Indicates value determined by Method of Standard Addition.

N - Indicates spike sample recovery is not within control limits.

* - Indicates duplicate analysis is not within control limits.

+ - Indicates the correlation coefficient for method of standard addition is less than 0.995

M - Indicates duplicate injection results exceeded control limits.

Indicate method used: P for ICP; A for Flame AA and F for Furnace.

APPENDIX B

U.S. ENVIRONMENTAL PROTECTION AGENCY
CONTRACT LABORATORY PROGRAM
REPORTING FORMS FOR ORGANICS ANALYSIS

1A
VOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Lab Sample ID: _____

Sample wt/vol: _____ (g/mL) ____ Lab File ID: _____

Level: (low/med) _____ Date Received: _____

% Moisture: not dec. _____ Date Analyzed: _____

Column: (pack/cap) _____ Dilution Factor: _____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) _____	Q
74-87-3	Chloromethane		
74-83-9	Bromomethane		
75-01-4	Vinyl Chloride		
75-00-3	Chloroethane		
75-09-2	Methylene Chloride		
67-64-1	Acetone		
75-15-0	Carbon Disulfide		
75-35-4	1,1-Dichloroethene		
75-34-3	1,1-Dichloroethane		
540-59-0	1,2-Dichloroethene (total)		
67-66-3	Chloroform		
107-06-2	1,2-Dichloroethane		
78-93-3	2-Butanone		
71-55-6	1,1,1-Trichloroethane		
56-23-5	Carbon Tetrachloride		
108-05-4	Vinyl Acetate		
75-27-4	Bromodichloromethane		
78-87-5	1,2-Dichloropropane		
10061-01-5	cis-1,3-Dichloropropene		
79-01-6	Trichloroethene		
124-48-1	Dibromochloromethane		
79-00-5	1,1,2-Trichloroethane		
71-43-2	Benzene		
10061-02-6	trans-1,3-Dichloropropene		
75-25-2	Bromoform		
108-10-1	4-Methyl-2-Pentanone		
591-78-6	2-Hexanone		
127-18-4	Tetrachloroethene		
79-34-5	1,1,2,2-Tetrachloroethane		
108-88-3	Toluene		
108-90-7	Chlorobenzene		
100-41-4	Ethylbenzene		
100-42-5	Styrene		
1330-20-7	Xylene (total)		

1B
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Lab Sample ID: _____

Sample wt/vol: _____ (g/mL) ____ Lab File ID: _____

Level: (low/med) _____ Date Received: _____

% Moisture: not dec. _____ dec. _____ Date Extracted: _____

Extraction: (SepF/Cont/Sonc) _____ Date Analyzed: _____

GPC Cleanup: (Y/N) ___ pH: ____ Dilution Factor: _____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) _____	Q
108-95-2	Phenol		
111-44-4	bis(2-Chloroethyl)ether		
95-57-8	2-Chlorophenol		
541-73-1	1,3-Dichlorobenzene		
106-46-7	1,4-Dichlorobenzene		
100-51-6	Benzyl alcohol		
95-50-1	1,2-Dichlorobenzene		
95-48-7	2-Methylphenol		
108-60-1	bis(2-Chloroisopropyl)ether		
106-44-5	4-Methylphenol		
621-64-7	N-Nitroso-di-n-propylamine		
67-72-1	Hexachloroethane		
98-95-3	Nitrobenzene		
78-59-1	Isophorone		
88-75-5	2-Nitrophenol		
105-67-9	2,4-Dimethylphenol		
65-85-0	Benzoic acid		
111-91-1	bis(2-Chloroethoxy)methane		
120-83-2	2,4-Dichlorophenol		
120-82-1	1,2,4-Trichlorobenzene		
91-20-3	Naphthalene		
106-47-8	4-Chloroaniline		
87-68-3	Hexachlorobutadiene		
59-50-7	4-Chloro-3-methylphenol		
91-57-6	2-Methylnaphthalene		
77-47-4	Hexachlorocyclopentadiene		
88-06-2	2,4,6-Trichlorophenol		
95-95-4	2,4,5-Trichlorophenol		
91-58-7	2-Chloronaphthalene		
88-74-4	2-Nitroaniline		
131-11-3	Dimethylphthalate		
208-96-8	Acenaphthylene		
606-20-2	2,6-Dinitrotoluene		

1C
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Lab Sample ID: _____

Sample wt/vol: _____(g/mL)____ Lab File ID: _____

Level: (low/med) _____ Date Received: _____

% Moisture: not dec._____ dec._____ Date Extracted: _____

Extraction: (SepF/Cont/Sonc) _____ Date Analyzed: _____

GPC Cleanup: (Y/N)___ pH:____ Dilution Factor: _____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg)_____	Q
99-09-2	3-Nitroaniline		
83-32-9	Acenaphthene		
51-28-5	2,4-Dinitrophenol		
100-02-7	4-Nitrophenol		
132-64-9	Dibenzofuran		
121-14-2	2,4-Dinitrotoluene		
84-66-2	Diethylphthalate		
7005-72-3	4-Chlorophenyl-phenylether		
86-73-7	Fluorene		
100-01-6	4-Nitroaniline		
534-52-1	4,6-Dinitro-2-methylphenol		
86-30-6	N-Nitrosodiphenylamine (1)		
101-55-3	4-Bromophenyl-phenylether		
118-74-1	Hexachlorobenzene		
87-86-5	Pentachlorophenol		
85-01-8	Phenanthrene		
120-12-7	Anthracene		
84-74-2	Di-n-butylphthalate		
206-44-0	Fluoranthene		
129-00-0	Pyrene		
85-68-7	Butylbenzylphthalate		
91-94-1	3,3'-Dichlorobenzidine		
56-55-3	Benzo(a)anthracene		
218-01-9	Chrysene		
117-81-7	bis(2-Ethylhexyl)phthalate		
117-84-0	Di-n-octylphthalate		
205-99-2	Benzo(b)fluoranthene		
207-08-9	Benzo(k)fluoranthene		
50-32-8	Benzo(a)pyrene		
193-39-5	Indeno(1,2,3-cd)pyrene		
53-70-3	Dibenz(a,h)anthracene		
191-24-2	Benzo(g,h,i)perylene		

(1) - Cannot be separated from Diphenylamine

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EPA SAMPLE NO.

1E
VOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Lab Sample ID: _____

Sample wt/vol: _____(g/mL)____ Lab File ID: _____

Level: (low/med) _____ Date Received: _____

% Moisture: not dec. _____ Date Analyzed: _____

Column: (pack/cap) _____ Dilution Factor: _____

Number TICs found: _____ CONCENTRATION UNITS: (ug/L or ug/Kg) _____

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
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.				
26.				
27.				
28.				
29.				
30.				

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EPA SAMPLE NO.

1F

SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Lab Sample ID: _____

Sample wt/vol: _____ (g/mL) ____ Lab File ID: _____

Level: (low/med) _____ Date Received: _____

% Moisture: not dec. _____ dec. _____ Date Extracted: _____

Extraction: (SepF/Cont/Sonc) _____ Date Analyzed: _____

GPC Cleanup: (Y/N) ___ pH: ____ Dilution Factor: _____

Number TICs found: _____

CONCENTRATION UNITS:
(ug/L or ug/Kg) _____

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
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20.				
21.				
22.				
23.				
24.				
25.				
26.				
27.				
28.				
29.				
30.				

2A
WATER VOLATILE SURROGATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

	EPA SAMPLE NO.	S1 (TOL) #	S2 (BFB) #	S3 (DCE) #	OTHER	TOT OUT
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						

		QC LIMITS
S1 (TOL)	= Toluene-d8	(88-110)
S2 (BFB)	= Bromofluorobenzene	(86-115)
S3 (DCE)	= 1,2-Dichloroethane-d4	(76-114)

Column to be used to flag recovery values

* Values outside of contract required QC limits

D Surrogates diluted out

page __ of __

2B
SOIL VOLATILE SURROGATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Level: (low/med) _____

	EPA SAMPLE NO.	S1 (TOL) #	S2 (BFB) #	S3 (DCE) #	OTHER	TOT OUT
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						

		QC LIMITS
S1 (TOL)	= Toluene-d8	(81-117)
S2 (BFB)	= Bromofluorobenzene	(74-121)
S3 (DCE)	= 1,2-Dichloroethane-d4	(70-121)

Column to be used to flag recovery values

• Values outside of contract required QC limits

D Surrogates diluted out

page __ of __

2C
WATER SEMIVOLATILE SURROGATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

	EPA SAMPLE NO.	S1 (NBZ) #	S2 (FBP) #	S3 (TPH) #	S4 (PHL) #	S5 (2FP) #	S6 (TBP) #	OTHER	TOT OUT
01									
02									
03									
04									
05									
06									
07									
08									
09									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									

		QC LIMITS
S1 (NBZ)	= Nitrobenzene-d5	(35-114)
S2 (FBP)	= 2-Fluorobiphenyl	(43-116)
S3 (TPH)	= Terphenyl-d14	(33-141)
S4 (PHL)	= Phenol-d5	(10-94)
S5 (2FP)	= 2-Fluorophenol	(21-100)
S6 (TBP)	= 2,4,6-Tribromophenol	(10-123)

Column to be used to flag recovery values
* Values outside of contract required QC limits
D Surrogates diluted out

page __ of __

2D
SOIL SEMIVOLATILE SURROGATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Level: (low/med) _____

	EPA SAMPLE NO.	S1 (NBZ) #	S2 (FBP) #	S3 (TPH) #	S4 (PHL) #	S5 (2FP) #	S6 (TBP) #	OTHER	TOT OUT
01									
02									
03									
04									
05									
06									
07									
08									
09									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									

		QC LIMITS
S1 (NBZ)	= Nitrobenzene-d5	(23-120)
S2 (FBP)	= 2-Fluorobiphenyl	(30-115)
S3 (TPH)	= Terphenyl-d14	(18-137)
S4 (PHL)	= Phenol-d5	(24-113)
S5 (2FP)	= 2-Fluorophenol	(25-121)
S6 (TBP)	= 2,4,6-Tribromophenol	(19-122)

Column to be used to flag recovery values
* Values outside of contract required QC limits
D Surrogates diluted out

page __ of __

3A

WATER VOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS % REC #	QC LIMI REC
1,1-Dichloroethene					61-1
Trichloroethene					71-1
Benzene					76-1
Toluene					76-1
Chlorobenzene					75-1

COMPOUND	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC
1,1-Dichloroethene					14	61-1
Trichloroethene					14	71-1
Benzene					11	76-1
Toluene					13	76-1
Chlorobenzene					13	75-1

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

RPD:_____ out of _____ outside limits
Spike Recovery:_____ out of _____ outside limits

COMMENTS: _____

3B

SOIL VOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____ Level:(low/med) _____

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC LIMIT REC.
1,1-Dichloroethene					59-17
Trichloroethene					62-13
Benzene					66-14
Toluene					59-13
Chlorobenzene					60-13

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC.
1,1-Dichloroethene					22	59-17
Trichloroethene					24	62-13
Benzene					21	66-14
Toluene					21	59-13
Chlorobenzene					21	60-13

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

RPD:_____ out of _____ outside limits
Spike Recovery:_____ out of _____ outside limits

COMMENTS: _____

3C

WATER SEMIVOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS % REC #	QC LIMI REC
Phenol					12-
2-Chlorophenol					27-1
1,4-Dichlorobenzene					36-
N-Nitroso-di-n-prop.(1)					41-1
1,2,4-Trichlorobenzene					39-
4-Chloro-3-methylphenol					23-
Acenaphthene					46-1
4-Nitrophenol					10-
2,4-Dinitrotoluene					24-
Pentachlorophenol					9-1
Pyrene					26-1

COMPOUND	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD % REC #	% RPD #	QC LIMITS RPD	REC
Phenol					42	12-
2-Chlorophenol					40	27-1
1,4-Dichlorobenzene					28	36-
N-Nitroso-di-n-prop.(1)					38	41-1
1,2,4-Trichlorobenzene					28	39-
4-Chloro-3-methylphenol					42	23-
Acenaphthene					31	46-1
4-Nitrophenol					50	10-
2,4-Dinitrotoluene					38	24-
Pentachlorophenol					50	9-1
Pyrene					31	26-1

(1) N-Nitroso-di-n-propylamine

Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits

RPD: _____ out of _____ outside limits
Spike Recovery: _____ out of _____ outside limits

COMMENTS: _____

3D

SOIL SEMIVOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____ Level: (low/med) _____

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC LIMI REC.
Phenol					26-
2-Chlorophenol					25-1
1,4-Dichlorobenzene					28-1
N-Nitroso-di-n-prop.(1)					41-1
1,2,4-Trichlorobenzene					38-1
4-Chloro-3-methylphenol					26-1
Acenaphthene					31-1
4-Nitrophenol					11-1
2,4-Dinitrotoluene					28-
Pentachlorophenol					17-1
Pyrene					35-1

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC.
Phenol					35	26-
2-Chlorophenol					50	25-1
1,4-Dichlorobenzene					27	28-1
N-Nitroso-di-n-prop.(1)					38	41-1
1,2,4-Trichlorobenzene					23	38-1
4-Chloro-3-methylphenol					33	26-1
Acenaphthene					19	31-1
4-Nitrophenol					50	11-1
2,4-Dinitrotoluene					47	28-
Pentachlorophenol					47	17-1
Pyrene					36	35-1

(1) N-Nitroso-di-n-propylamine

Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits

RPD: _____ out of _____ outside limits
Spike Recovery: _____ out of _____ outside limits

COMMENTS: _____

4A
VOLATILE METHOD BLANK SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Lab File ID: _____ Lab Sample ID: _____

Date Analyzed: _____ Time Analyzed: _____

Matrix: (soil/water) _____ Level: (low/med) _____

Instrument ID: _____

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS AND MSD:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	TIME ANALYZED
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				

COMMENTS: _____

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4B
SEMIVOLATILE METHOD BLANK SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Lab File ID: _____ Lab Sample ID: _____

Date Extracted: _____ Extraction:(SepF/Cont/Sonc) _____

Date Analyzed: _____ Time Analyzed: _____

Matrix: (soil/water) _____ Level:(low/med) _____

Instrument ID: _____

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS AND MSD:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
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23				
24				
25				
26				
27				
28				
29				
30				

COMMENTS: _____

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5A
VOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION - BROMOFLUOROBENZENE (BFB)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Lab File ID: _____ BFB Injection Date: _____

Instrument ID: _____ BFB Injection Time: _____

Matrix: (soil/water) _____ Level: (low/med) _____ Column: (pack/cap) _____

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
50	15.0 - 40.0% of mass 95	
75	30.0 - 60.0% of mass 95	
95	Base peak, 100% relative abundance	
96	5.0 - 9.0% of mass 95	
173	Less than 2.0% of mass 174	( )1
174	Greater than 50.0% of mass 95	
175	5.0 - 9.0% of mass 174	( )1
176	Greater than 95.0%, but less than 101.0% of mass 174	( )1
177	5.0 - 9.0% of mass 176	( )2

1-Value is % mass 174 2-Value is % mass 176

THIS TUNE APPLIES TO THE FOLLOWING SAMPLES, MS, MSD, BLANKS, AND STANDARDS:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

page __ of __

5B

SEMIVOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION - DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Lab File ID: _____ DFTPP Injection Date: _____

Instrument ID: _____ DFTPP Injection Time: _____

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
51	30.0 - 60.0% of mass 198	
68	Less than 2.0% of mass 69	( )1
69	Mass 69 relative abundance	
70	Less than 2.0% of mass 69	( )1
127	40.0 - 60.0% of mass 198	
197	Less than 1.0% of mass 198	
198	Base Peak, 100% relative abundance	
199	5.0 to 9.0% of mass 198	
275	10.0 - 30.0% of mass 198	
365	Greater than 1.00% of mass 198	
441	Present, but less than mass 443	
442	Greater than 40.0% of mass 198	
443	17.0 - 23.0% of mass 442	( )2

1-Value is % mass 69 2-Value is % mass 442

THIS TUNE APPLIES TO THE FOLLOWING SAMPLES, MS, MSD, BLANKS, AND STANDARDS:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

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6A

VOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Matrix: (soil/water) _____ Level: (low/med) _____ Column: (pack/cap) _____

Min $\overline{RRF}$ for SPCC(#) = 0.300 (0.250 for Bromoform) Max %RSD for CCC(*) = 30.0

LAB FILE ID: RRF20 = _____ RRF50 = _____
RRF100= _____ RRF150= _____ RRF200= _____

COMPOUND		RRF20	RRF50	RRF100	RRF150	RRF200	$\overline{RRF}$	% RSD
Chloromethane	#							
Bromomethane								
Vinyl Chloride	*							
Chloroethane								
Methylene Chloride								
Acetone								
Carbon Disulfide								
1,1-Dichloroethene	*							
1,1-Dichloroethane	#							
1,2-Dichloroethene (total)								
Chloroform	●							
1,2-Dichloroethane								
2-Butanone								
1,1,1-Trichloroethane								
Carbon Tetrachloride								
Vinyl Acetate								
Bromodichloromethane								
1,2-Dichloropropane	●							
cis-1,3-Dichloropropene								
Trichloroethene								
Dibromochloromethane								
1,1,2-Trichloroethane								
Benzene								
trans-1,3-Dichloropropene								
Bromoform	#							
4-Methyl-2-Pentanone								
2-Hexanone								
Tetrachloroethene								
1,1,2,2-Tetrachloroethane	#							
Toluene	●							
Chlorobenzene	#							
Ethylbenzene	●							
Styrene								
Xylene (total)								
Toluene-d8								
Bromofluorobenzene								
1,2-Dichloroethane-d4								

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6B

SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Min $\overline{RRF}$ for SPCC(#) = 0.050 Max %RSD for CCC(*) = 30.0

LAB FILE ID: RRF20 = _____ RRF50 = _____
RRF80 = _____ RRF120= _____ RRF160= _____

COMPOUND		RRF20	RRF50	RRF80	RRF120	RRF160	$\overline{RRF}$	% RSD
Phenol	*							
bis(2-Chloroethyl)ether								
2-Chlorophenol								
1,3-Dichlorobenzene								
1,4-Dichlorobenzene	*							
Benzyl alcohol								
1,2-Dichlorobenzene								
2-Methylphenol								
bis(2-Chloroisopropyl)ether								
4-Methylphenol								
N-Nitroso-di-n-propylamine	#							
Hexachloroethane								
Nitrobenzene								
Isophorone								
2-Nitrophenol	*							
2,4-Dimethylphenol								
Benzoic acid								
bis(2-Chloroethoxy)methane								
2,4-Dichlorophenol	*							
1,2,4-Trichlorobenzene								
Naphthalene								
4-Chloroaniline								
Hexachlorobutadiene	*							
4-Chloro-3-methylphenol	*							
2-Methylnaphthalene								
Hexachlorocyclopentadiene	#							
2,4,6-Trichlorophenol	*							
2,4,5-Trichlorophenol								
2-Chloronaphthalene								
2-Nitroaniline								
Dimethylphthalate								
Acenaphthylene								
2,6-Dinitrotoluene								
3-Nitroaniline								
Acenaphthene	*							
2,4-Dinitrophenol	#							
4-Nitrophenol	#							

6C

SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Min $\overline{RRF}$ for SPCC(#) = 0.050 Max %RSD for CCC(*) = 30.

LAB FILE ID: RRF20 = _____ RRF50 = _____
RRF80 = _____ RRF120= _____ RRF160= _____

COMPOUND	RRF20	RRF50	RRF80	RRF120	RRF160	$\overline{RRF}$	% RSD
Dibenzofuran							
2,4-Dinitrotoluene							
Diethylphthalate							
4-Chlorophenyl-phenylether							
Fluorene							
4-Nitroaniline							
4,6-Dinitro-2-methylphenol							
N-Nitrosodiphenylamine (1) *							
4-Bromophenyl-phenylether							
Hexachlorobenzene							
Pentachlorophenol *							
Phenanthrene							
Anthracene							
Di-n-butylphthalate							
Fluoranthene *							
Pyrene							
Butylbenzylphthalate							
3,3'-Dichlorobenzidine							
Benzo(a)anthracene							
Chrysene							
bis(2-Ethylhexyl)phthalate							
Di-n-octylphthalate *							
Benzo(b)fluoranthene							
Benzo(k)fluoranthene							
Benzo(a)pyrene *							
Indeno(1,2,3-cd)pyrene							
Dibenz(a,h)anthracene							
Benzo(g,h,i)perylene							
Nitrobenzene-d5							
2-Fluorobiphenyl							
Terphenyl-d14							
Phenol-d5							
2-Fluorophenol							
2,4,6-Tribromophenol							

(1) Cannot be separated from Diphenylamine

7A
VOLATILE CONTINUING CALIBRATION CHECK

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date:_____ Time:_____

Lab File ID: _____ Init. Calib. Date(s):_____ _____

Matrix:(soil/water) _____ Level:(low/med) _____ Column:(pack/cap) _____

Min RRF50 for SPCC(#) = 0.300 (0.250 for Bromoform) Max %D for CCC(*) = 25.0

COMPOUND	$\overline{RRF}$	RRF50	%D
Chloromethane	#		#
Bromomethane			
Vinyl Chloride	*		*
Chloroethane			
Methylene Chloride			
Acetone			
Carbon Disulfide			
1,1-Dichloroethene	*		*
1,1-Dichloroethane	#		#
1,2-Dichloroethene (total)			
Chloroform	*		*
1,2-Dichloroethane			
2-Butanone			
1,1,1-Trichloroethane			
Carbon Tetrachloride			
Vinyl Acetate			
Bromodichloromethane			
1,2-Dichloropropane	*		*
cis-1,3-Dichloropropene			
Trichloroethene			
Dibromochloromethane			
1,1,2-Trichloroethane			
Benzene			
trans-1,3-Dichloropropene			
Bromoform	#		#
4-Methyl-2-Pentanone			
2-Hexanone			
Tetrachloroethene			
1,1,2,2-Tetrachloroethane	#		#
Toluene	*		*
Chlorobenzene	#		#
Ethylbenzene	*		*
Styrene			
Xylene (total)			
Toluene-d8			
Bromofluorobenzene			
1,2-Dichloroethane-d4			

7B
SEMIVOLATILE CONTINUING CALIBRATION CHECK

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date:_____ Time:_____

Lab File ID: _____ Init. Calib. Date(s):_____ _____

Min RRF50 for SPCC(#) = 0.050 Max %D for CCC(*) = 25.

COMPOUND	$\overline{RRF}$	RRF50	%D
Phenol	*		*
bis(2-Chloroethyl)ether			
2-Chlorophenol			
1,3-Dichlorobenzene			
1,4-Dichlorobenzene	*		*
Benzyl alcohol			
1,2-Dichlorobenzene			
2-Methylphenol			
bis(2-Chloroisopropyl)ether			
4-Methylphenol			
N-Nitroso-di-n-propylamine	#		#
Hexachloroethane			
Nitrobenzene			
Isophorone			
2-Nitrophenol	*		*
2,4-Dimethylphenol			
Benzoic acid			
bis(2-Chloroethoxy)methane			
2,4-Dichlorophenol	*		*
1,2,4-Trichlorobenzene			
Naphthalene			
4-Chloroaniline			
Hexachlorobutadiene	*		*
4-Chloro-3-methylphenol	*		*
2-Methylnaphthalene			
Hexachlorocyclopentadiene	#		#
2,4,6-Trichlorophenol	*		*
2,4,5-Trichlorophenol			
2-Chloronaphthalene			
2-Nitroaniline			
Dimethylphthalate			
Acenaphthylene			
2,6-Dinitrotoluene			
3-Nitroaniline			
Acenaphthene	*		*
2,4-Dinitrophenol	#		#
4-Nitrophenol	#		#

SEMIVOLATILE CONTINUING CALIBRATION CHECK

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date: _____ Time: _____

Lab File ID: _____ Init. Calib. Date(s): _____ _____

Min RRF50 for SPCC(#) = 0.050 Max %D for CCC(*) = 25.

COMPOUND	$\overline{RRF}$	RRF50	%D
Dibenzofuran			
2,4-Dinitrotoluene			
Diethylphthalate			
4-Chlorophenyl-phenylether			
Fluorene			
4-Nitroaniline			
4,6-Dinitro-2-methylphenol			
N-Nitrosodiphenylamine (1)	#		#
4-Bromophenyl-phenylether			
Hexachlorobenzene			
Pentachlorophenol	#		#
Phenanthrene			
Anthracene			
Di-n-butylphthalate			
Fluoranthene	#		#
Pyrene			
Butylbenzylphthalate			
3,3'-Dichlorobenzidine			
Benzo(a)anthracene			
Chrysene			
bis(2-Ethylhexyl)phthalate			
Di-n-octylphthalate	#		#
Benzo(b)fluoranthene			
Benzo(k)fluoranthene			
Benzo(a)pyrene	*		*
Indeno(1,2,3-cd)pyrene			
Dibenz(a,h)anthracene			
Benzo(g,h,i)perylene			
Nitrobenzene-d5			
2-Fluorobiphenyl			
Terphenyl-d14			
Phenol-d5			
2-Fluorophenol			
2,4,6-Tribromophenol			

(1) Cannot be separated from Diphenylamine

8A

VOLATILE INTERNAL STANDARD AREA SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Lab File ID (Standard): _____ Date Analyzed: _____

Instrument ID: _____ Time Analyzed: _____

Matrix: (soil/water) _____ Level: (low/med) _____ Column: (pack/cap) _____

		IS1(BCM) AREA #	RT	IS2(DFB) AREA #	RT	IS3(CBZ) AREA #	RT
	12 HOUR STD						
	UPPER LIMIT						
	LOWER LIMIT						
	EPA SAMPLE NO.						
01							
02							
03							
04							
05							
06							
07							
08							
09							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							

IS1 (BCM) = Bromochloromethane
IS2 (DFB) = 1,4-Difluorobenzene
IS3 (CBZ) = Chlorobenzene

UPPER LIMIT = + 100% of internal standard area.
LOWER LIMIT = - 50% of internal standard area.

Column used to flag internal standard area values with an asterisk

page __ of __

8B

SEMIVOLATILE INTERNAL STANDARD AREA SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Lab File ID (Standard): _____ Date Analyzed: _____

Instrument ID: _____ Time Analyzed: _____

		IS1(DCB) AREA #	RT	IS2(NPT) AREA #	RT	IS3(ANT) AREA #	RT
	12 HOUR STD						
	UPPER LIMIT						
	LOWER LIMIT						
	EPA SAMPLE NO.						
01							
02							
03							
04							
05							
06							
07							
08							
09							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							

IS1 (DCB) = 1,4-Dichlorobenzene-d4
IS2 (NPT) = Naphthalene-d8
IS3 (ANT) = Acenaphthene-d8

UPPER LIMIT = + 100% of internal standard area.
LOWER LIMIT = - 50% of internal standard area.

Column used to flag internal standard area values with an asterisk

page __ of __

8C
SEMIVOLATILE INTERNAL STANDARD AREA SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Lab File ID (Standard): _____ Date Analyzed: _____

Instrument ID: _____ Time Analyzed: _____

		IS4(PHN) AREA #	RT	IS5(CRY) AREA #	RT	IS6(PRY) AREA #	RT
	12 HOUR STD						
	UPPER LIMIT						
	LOWER LIMIT						
	EPA SAMPLE NO.						
01							
02							
03							
04							
05							
06							
07							
08							
09							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							

IS4 (PHN) = Phenanthrene-d10
IS5 (CRY) = Chrysene-d12
IS6 (PRY) = Perylene-d12

UPPER LIMIT = + 100% of internal standard area.
LOWER LIMIT = - 50% of internal standard area.

Column used to flag internal standard area values with an asterisk

page __ of __

FORM VIII SV-2

1D
PESTICIDE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Lab Sample ID: _____

Sample wt/vol: _____ (g/mL) ____ Lab File ID: _____

Level: (low/med) _____ Date Received: _____

% Moisture: not dec. _____ dec. _____ Date Extracted: _____

Extraction: (SepF/Cont/Sonc) _____ Date Analyzed: _____

GPC Cleanup: (Y/N) ___ pH: ____ Dilution Factor: _____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) _____	Q
319-84-6	alpha-BHC		
319-85-7	beta-BHC		
319-86-8	delta-BHC		
58-89-9	gamma-BHC (Lindane)		
76-44-8	Heptachlor		
309-00-2	Aldrin		
1024-57-3	Heptachlor epoxide		
959-98-8	Endosulfan I		
60-57-1	Dieldrin		
72-55-9	4,4'-DDE		
72-20-8	Endrin		
33213-65-9	Endosulfan II		
72-54-8	4,4'-DDD		
1031-07-8	Endosulfan sulfate		
50-29-3	4,4'-DDT		
72-43-5	Methoxychlor		
53494-70-5	Endrin ketone		
5103-71-9	alpha-Chlordane		
5103-74-2	gamma-Chlordane		
8001-35-2	Toxaphene		
12674-11-2	Aroclor-1016		
11104-28-2	Aroclor-1221		
11141-16-5	Aroclor-1232		
53469-21-9	Aroclor-1242		
12672-29-6	Aroclor-1248		
11097-69-1	Aroclor-1254		
11096-82-5	Aroclor-1260		

2E
WATER PESTICIDE SURROGATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

	EPA SAMPLE NO.	S1 (DBC) #	OTHER
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			
27			
28			
29			
30			

ADVISORY QC LIMITS (24-154)

S1 (DBC) = Dibutylchlorendate

Column used to flag recovery values

• Values outside of QC limits

D Surrogates diluted out

page __ of __

FORM II PEST-1

1/87 Re

2F
SOIL PESTICIDE SURROGATE RECOVERY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Level:(low/med)_____

	EPA SAMPLE NO.	S1 (DBC)#	OTHER
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			
27			
28			
29			
30			

S1 (DBC) = Dibutylchlorendate — ADVISORY QC LIMITS (20-150)

Column to be used to flag recovery values

* Values outside of QC limits

D Surrogates diluted out

3E

WATER PESTICIDE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS % REC #	QC LIMIT REC.
Lindane					56-1
Heptachlor					40-1
Aldrin					40-1
Dieldrin					52-1
Endrin					56-1
4,4' DDT					38-1

COMPOUND	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC
Lindane					15	56-1
Heptachlor					20	40-1
Aldrin					22	40-1
Dieldrin					18	52-1
Endrin					21	56-1
4,4' DDT					27	38-1

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

RPD:_____ out of _____ outside limits
Spike Recovery:_____ out of _____ outside limits

COMMENTS: _____

3F

SOIL PESTICIDE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____ Level:(low/med) _____

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC LIMI REC
Lindane					46-1
Heptachlor					35-1
Aldrin					34-1
Dieldrin					31-1
Endrin					42-1
4,4' DDT					23-1

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC
Lindane					50	46-1
Heptachlor					31	35-1
Aldrin					43	34-1
Dieldrin					38	31-1
Endrin					45	42-1
4,4' DDT					50	23-1

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

RPD:_____ out of _____ outside limits
Spike Recovery:_____ out of _____ outside limits

COMMENTS: _____

8D
PESTICIDE EVALUATION STANDARDS SUMMARY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Instrument ID:_____ GC Column ID: _____

Dates of Analyses:_____ to _____

Evaluation Check for Linearity

PESTICIDE	CALIBRATION FACTOR EVAL MIX A	CALIBRATION FACTOR EVAL MIX B	CALIBRATION FACTOR EVAL MIX C	%RSD (</= 10.0%)
ALDRIN				
ENDRIN				
4,4'-DDT				(1)
DBC				

(1) If > 10.0% RSD, plot a standard curve and determine the ng for each sample in that set from the curve.

Evaluation Check for 4,4'-DDT/Endrin Breakdown
(percent breakdown expressed as total degradation)

		DATE ANALYZED	TIME ANALYZED	ENDRIN	4,4'-DDT	COMBINED (2)
	INITIAL					
01	EVAL MIX B					
02	EVAL MIX B					
03	EVAL MIX B					
04	EVAL MIX B					
05	EVAL MIX B					
06	EVAL MIX B					
07	EVAL MIX B					
08	EVAL MIX B					
09	EVAL MIX B					
10	EVAL MIX B					
11	EVAL MIX B					
12	EVAL MIX B					
13	EVAL MIX B					
14	EVAL MIX B					

(2) See Form instructions.

8E
PESTICIDE EVALUATION STANDARDS SUMMARY
Evaluation of Retention Time Shift for Dibutylchlorendate

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Instrument ID:_____ GC Column ID: _____

Dates of Analyses:_____ to _____

	EPA SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED	TIME ANALYZED	% D	*
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						
32						
33						
34						
35						
36						
37						
38						

* Values outside of QC limits (2.0% for packed columns, 0.3% for capillary columns)

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10

PESTICIDE/PCB IDENTIFICATION

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

GC Column ID (1): _____ GC Column ID (2): _____

Instrument ID (1): _____ Instrument ID (2): _____

Lab Sample ID: _____

Lab File ID: _____ (only if confirmed by GC/MS)

PESTICIDE/PCB	RETENTION TIME	RT WINDOW OF STANDARD FROM	TO	QUANT? (Y/N)	GC/MS? (Y/N)
01 _____	Column 1 _____	_____	_____	_	_
02	Column 2 _____	_____	_____	_	_
03 _____	Column 1 _____	_____	_____	_	_
04	Column 2 _____	_____	_____	_	_
05 _____	Column 1 _____	_____	_____	_	_
06	Column 2 _____	_____	_____	_	_
07 _____	Column 1 _____	_____	_____	_	_
08	Column 2 _____	_____	_____	_	_
09 _____	Column 1 _____	_____	_____	_	_
10	Column 2 _____	_____	_____	_	_
11 _____	Column 1 _____	_____	_____	_	_
12	Column 2 _____	_____	_____	_	_

Comments: _____