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TREATABILITY STUDY WORK PLAN FOR OPERABLE UNIT 4 TASK 5 WORK PLAN JULY 1991

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TREATABILITY STUDY WORK PLAN FOR OPERABLE UNIT 4

TASK 5 WORK PLAN

FEED MATERIALS PRODUCTION CENTER
FERNALD, OHIO

REMEDIAL INVESTIGATION and FEASIBILITY STUDY

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U.S. DEPARTMENT OF ENERGY

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LIST OF ACRONYMS

AA	Atomic Absorption
ANS	American Nuclear Society
ANSI	American National Standards Institute
ARAR	Applicable or Relevant and Appropriate Requirements
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLP	Contract Laboratory Program
COD	Chemical Oxygen Demand
d	day
DOE	U.S. Department of Energy
DQO	Data Quality Objective
EDTA	ethylenediaminetetraacetic acid
EPA	U.S. Environmental Protection Agency
FFCA	Federal Facilities Compliance Agreement
FMPC	Feed Materials Production Center
FS	Feasibility Study
g	gram
GC	gas chromatography
hr	hour
HEPA	High Efficiency Particular Air
HSL	Hazardous Substance List
ICP	Inductively Coupled Plasma
ITAS	IT Analytical Services
kg	kilogram
L	liter
uci	microCuries

LIST OF ACRONYMS
(Continued)

MCL	Maximum Contaminant Level
mg	milligram
mrem	millirem
MS	mass spectrometry
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QCC	Quality Control Coordinator
pCi	picoCurie
ppm	parts per million
psi	pounds per square inch
RAO	Remedial Action Objective
RCRA	Resource Conservation and Recovery Act
RfD	Toxicity based reference doses
RI	Remedial Investigation
TCLP	Toxicity Characteristic Leaching Procedure
UCS	Unconfined Compression Strength
WL	Working Level
WMCO	Westinghouse Materials Company of Ohio
XRF	X-ray fluorescence
yr	year

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CHEMICAL ABBREVIATIONS

U	uranium
Th	thorium
Pb	lead
Ra	radium
Po	polonium
Pa	protactinium
Ac	actinium
As	arsenic
Ba	barium
Be	beryllium
Cd	cadmium
Cr	chromium
Cu	copper
Mn	manganese
Se	selenium
Tl	thallium
Va	vanadium
Zn	zinc
HNO_3	nitric acid
HCl	hydrochloric acid
KCN	potassium cyanide
NH_4OH	ammonium hydroxide
M	molar
Conc	concentration
MgO	magnesium oxide

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CHEMICAL ABBREVIATIONS
(Continued)

$Ca(OH)_2$	calcium hydroxide
$Na_2O:SiO_2$	aqueous sodium silicate
NaOH	sodium hydroxide
Na_3PO_4	trisodium phosphate
Na_2S	sodium monosulfide

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OPERABLE UNIT 4 TREATABILITY WORK PLAN

1.0 PROJECT DESCRIPTION

1.1 SITE DESCRIPTION

The Feed Materials Production Center (FMPC) is a contractor-operated federal facility for the production of purified uranium metal for the U.S. Department of Energy (DOE). The FMPC is located on 1050 acres in a rural area approximately 20 miles northwest of downtown Cincinnati, Ohio. On July 18, 1986, a Federal Facilities Compliance Agreement (FFCA) was jointly signed by the U.S. Environmental Protection Agency (EPA) and DOE to ensure that environmental impacts associated with past and present activities at the FMPC are thoroughly investigated so that appropriate remedial actions can be assessed and implemented.

A Remedial Investigation/Feasibility Study (RI/FS) has been initiated to develop these remedial actions. A part of this RI/FS is Operable Unit 4. Operable Unit 4 consists of Silos 1 and 2 (K-65 silos) and Silo 3 (metal oxide silo), the unused Silo 4, and the silo structures and surrounding berms. Operable Unit 4 is located south of the waste pit area. The FS for Operable Unit 4 is considering remedial actions for the silo structures and for waste stored in the silos and in the adjoining silo berms.

Silos 1 and 2 were used for the storage of radium-bearing residues formed as by-products of uranium ore processing. Silos 1 and 2 received residues from 1952 to 1958. Raffinates (residues resulting from uranium solvent extraction) were pumped into the silos where the solids would settle. The free liquid was decanted through a series of valves placed at various levels along the height of the silo wall. Settling and decanting continued until the silo material was approximately four feet below the top of the vertical wall.

Historic analysis of the Silos 1 and 2 residues indicates that approximately 11,200 kilograms of uranium (0.71 percent U-235) is present. Analytical results of residue samples taken in July 1988 indicated the uranium concentration was 1400 parts per million (ppm) in Silo 1 and 1800 ppm in Silo 2. In addition, approximately 0.13 to 0.21 ppm of radium was estimated to be in the silo residues.

Data from the 1989 sampling effort conducted by Westinghouse Materials Company of Ohio (WMCO) for Silos 1 and 2 indicate that the concentration of radium-226 (Ra-226) in Silo 1 ranges from 89,280 picoCuries/gram (pCi/g) to 192,600 pCi/g; in Silo 2 it ranges from 657 to 145,300 pCi/g. Thorium-230 (Th-230) concentrations in Silo 1 range from 10,569 to 43,771 pCi/g and from 8365 to 40,124

pCi/g in Silo 2. The concentration of lead-210 (Pb-210) in Silo 1 ranges from 48,490 to 181,100 pCi/g and from 77,940 to 399,200 pCi/g in Silo 2. Total uranium concentrations in Silo 1 range from 1189 to 2753 ppm and from 137 to 3717 ppm in Silo 2.

Radon and the elements resulting from its decay (daughter products, progeny, etc.) are the nuclides of concern from a health and environmental perspective. Radon is diffusing out of the silos via cracks and structural joints. Radon and its daughter products are relatively mobile and capable of migrating through air and water. Due to the probable diffusion of radon into the berms, it is believed that the berms and subsoils contain elevated levels of Pb-210 and polonium-210 (Po-210). There may have been leakage from the existing leachate collection system beneath the silos into the surrounding soils. If this has occurred, the potential for uptake of long-lived radionuclides would be a major hazard. Sampling of the berms and soil beneath the silos is scheduled and, upon completion, will confirm the nature and extent of contamination and contaminant migration, if any.

Silos 3 and 4 were constructed in 1952 in a manner similar to Silos 1 and 2; however, the silos were designed to receive dry materials only. Raffinate slurries from refinery operations were dewatered in an evaporator and spray-calcined to produce dry materials for storage in the silo. The material was blown in under pressure to fill Silo 3. Silo 4 was never used and remains empty today.

Silo 3 contains silica, uranium (738 to 4554 ppm), Th-230 (21,010 to 71,650 pCi/g), a very small amount of Ra-226 (467 to 6435 pCi/g), and other metal oxides. Silo 3 is not a significant radon source, and due to the physical characteristics of the silo contents (dry and powdery), it is not believed to be the source of any contaminant migration to the surrounding and underlying areas. It is, however, still a source of radioactivity and a potential airborne contaminant hazard due to its dry, powdery consistency.

1.2 REMEDIAL ACTION OBJECTIVES

The remedial action objectives (RAOs) are medium-specific cleanup goals for protecting human health and the environment. They address the contaminants of concern as well as exposure routes and receptors identified in the baseline risk assessment. For Operable Unit 4, it must be demonstrated that remedial alternatives meet airborne and direct radiation RAOs immediately adjacent to the silos, as well as drinking water RAOs in any perched water that might be encountered directly below the silos. RAOs for the silo material are given in Figure 1-1 and Tables 1-1 and 1-2.

Direct Radiation	For Human Health: Prevent current and future radiation doses to a member of the public from exceeding 25 mrem/year. For Environmental Protection: Prevent current and future radiation doses from causing detectable chronic effects.
Air	For Human Health: Prevent potential current and future above-background air pathway radiation doses from exceeding 2.5 mrem/yr. Prevent radon decay product concentrations from exceeding 0.02 working level (wl) and average radon release rate from exceeding 20 pCi/m^2/s. For Environmental Protection: Prevent current and future radiation emissions from causing detectable chronic effects.
Groundwater	For Human Health: Prevent release of U, Ra, Th, Pa, Po, Pb, and Ac to the groundwater in excess of concentrations shown in Table 1-1. Prevent release of As, Ba, Be, Cd, Cr, Cu, Pb, Mn, Se, Tl, Va, and Zn to the groundwater in excess of concentrations shown in Table 1-2. For Environmental Protection: None. Groundwater concentrations have not been found to represent an environmental hazard.

Figure 1-1. Remedial Action Objectives for Operable Unit 4 Silo Material

TABLE 1-1

OPERABLE UNIT 4
GROUNDWATER REMEDIAL ACTION OBJECTIVES FOR RADIONUCLIDES

Constituent	Drinking Water Concentration Corresponding to 4 mrem/yr (pCi/L)	FMPC Action Level for a Single Operable Unit[a] (pCi/L)
Uranium-234[c]	19	4.8
Uranium-235[c]	21	5.3
Uranium-238[c]	21	5.3
Thorium-228	14	3.5
Thorium-230	10	2.5
Thorium-232	2.0	0.5
Protactinium-231	0.5	0.1
Polonium-210	3.0	0.7
Lead-210	1.0	0.2
Actinium-227	0.4	0.1
Radium[b,c]	5.0	1.2

[a] Twenty-five percent of ARAR or risk-based standard.
[b] Maximum contaminant level (MCL).
[c] On June 17, 1991, EPA proposed new MCLs for uranium and radium. The proposed limits are 20 g/L for total uranium, 20 pCi/L for Ra-226, and 20 pCi/L for Ra-228.

TABLE 1-2

OPERABLE UNIT 4
GROUNDWATER REMEDIAL ACTION OBJECTIVES FOR CHEMICALS

Constituent	Basis for Remedial Objective	Acceptable Water Concentration (mg/L)	FMPC Action Level for a Single Operable Unit[a] (mg/L)
Arsenic	0.05 mg/L MCL[b]	0.05	0.01
Barium	5.0 mg/L proposed MCL	5.0	1.3
Beryllium	0.005 mg/kg-d RfD[c]	0.18	0.045
Cadmium	0.005 mg/L proposed MCL	0.005	0.001
Chromium	0.1 mg/L proposed MCL	0.1	0.03
Copper	1.3 mg/L[d]	1.3	0.32
Lead	0.05 mg/L[e] MCL	0.05	0.013
Manganese	0.2 mg/kg-d RfD	7.0	1.8
Selenium	0.05 proposed MCL	0.05	0.013
Thallium	0.00007 mg/kg-d RfD	0.0025	0.0005
Vanadium	0.007 mg/kg-d RfD	0.25	0.063
Zinc	0.2 mg/kg-d RfD	7.0	1.8

[a]Twenty-five percent of ARAR of risk-based standard.
[b]Maximum Contaminant Level.
[c]Toxicity-based reference doses (RfD).
[d]EPA is considering a substantially lower number.
[e]Drinking Water Health Advisory.

Ten remediation alternatives for Operable Unit 4 are listed in the DOE report "Initial Screening of Alternatives for Operable Unit 4, Task 12 Report, October 1990." Nine of these alternatives are still under consideration. Laboratory data are needed to evaluate the alternatives, eliminate alternatives that are not technically feasible, and aid in the selection of a preferred alternative. Further details of the alternatives are given in Section 2.0.

1.3 TREATABILITY STUDY

1.3.1 Justification

The justification to conduct these tests is provided by EPA in "Guide for Conducting Treatability Studies Under CERCLA." The document recommended treatability tests for those substances that do not have standard treatment methods or supporting data in the literature that prove the material of interest can be effectively treated to render it nonhazardous. More explicitly in the case of Operable Unit 4, the purpose of treatment is to render the material nonleachable so that it is not hazardous by characteristic under the Resource Conservation and Recovery Act (RCRA).

In the literature, the report "Characteristics of Fernald's Silos 1 and 2 Residue Before, During and After Vitrification," provides justification that vitrification of the Silos 1 and 2 materials is feasible and can be a viable option for the stabilization of these wastes. However, in order to be able to compare the effectiveness of vitrification in the feasibility studies and in the subsequent engineering designs, data must be available for the other alternatives so that a decision based on fact rather than conjecture can be made.

Because the Silo 3 wastes were produced at the FMPC site, and because metal reduction by solvent extraction is a proven technology for uranium oxides, these oxides are not the subject of an extraction study. Yet, because of the unique nature of the Silo 1 and 2 materials and because of the lack of process knowledge concerning their chemical rather than elemental composition, it is not obvious if an extraction process can be developed that would remove a sufficient quantity of metals in order to render the material RCRA nonhazardous. Unlike the Silo 3 material, the original Silos 1 and 2 material was processed at the Mallinckrodt Chemical Works. Production records from this facility are no longer available except for elemental analyses developed by NLO, Bettis, et al. These analyses are not sufficient in detail to support a metals extraction decision as feasible or not feasible.

Similarly, the cementation process requires a unique recipe to be formulated for each unique waste form. Because neither the Silo 3 nor the Silos 1 and 2 materials have been the basis of a cementation study, a bench-scale study must be performed to determine whether cementation is a feasible option.

These treatability studies are necessary to eliminate alternatives in the Operable Unit 4 FS. This study is currently carrying nine alternatives and two different stabilization options. The studies are needed to definitively provide information that would reduce the number of options that have to be considered.

Finally, because of the unique nature of the material in the silos, the materials deserve special consideration to ensure that the ultimate remedial action alternative selected by DOE in the Record of Decision (ROD) can be supported without the potential for criticism by the local community and environmental political action groups. The project cannot afford to arrive at the end of the process without the appropriate documentation of its decision-working process.

1.3.2 EPA Treatability Guidance

EPA's "Guide for Conducting Treatability Studies Under CERCLA" outlined a three-tiered approach to conducting treatability studies for a Superfund site. The original interpretation of the approach can be seen in Figure 1-2. The remedy evaluation phase, in accordance with the EPA Guidance, of the RI/FS may require a minimum of three tiers of treatability testing:

- Remedy screening
- Remedy selection
- Remedy design

The terminology of this approach has been revised to reflect Figure 1-3. This illustrates these three levels of treatability testing and how this treatability plan compares with these requirements.

Pre-ROD treatability studies provide the critical performance and cost data needed to (1) evaluate all potentially applicable treatment alternatives and (2) select an alternative for remedial action based on the nine RI/FS evaluation criteria.

The detailed analysis of alternatives phase of the RI/FS follows the development and screening of alternatives and precedes the actual selection of a remedy in the ROD. During the detailed analysis, all remedial alternatives are evaluated based on nine RI/FS evaluation criteria. These criteria are as follows:

- Overall protection of human health and the environment
- Compliance with applicable or relevant and appropriate requirements (ARARs)
- Long-term effectiveness and permanence
- Reduction of toxicity, mobility, and volume through treatment
- Short-term effectiveness
- Implementability

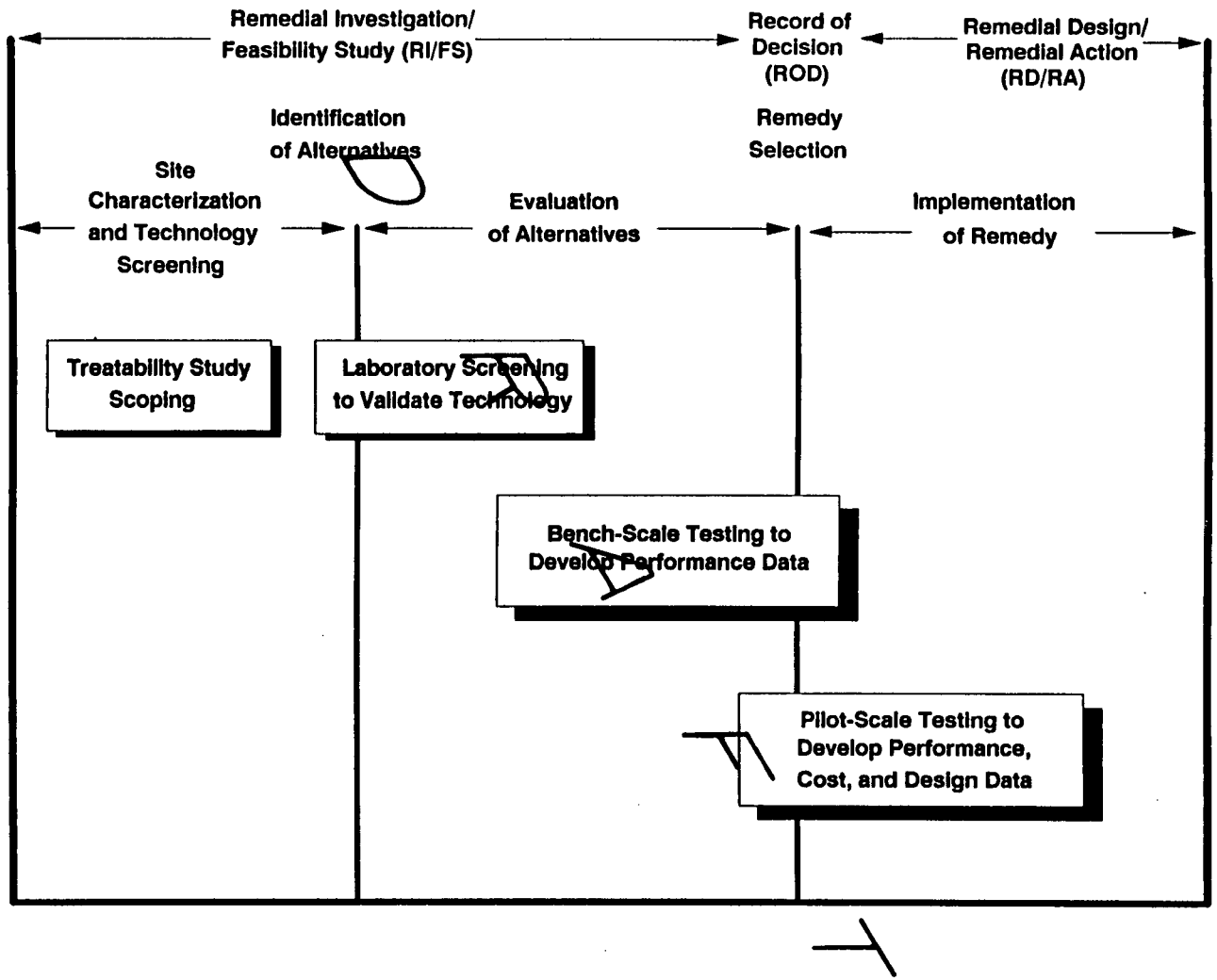

FIGURE 1-2. The Role of Treatability Studies in the RI/FS and RD/RA Process.

Source: Guide for Conducting Treatability Studies Under CERCLA. Interim Final 12/89.

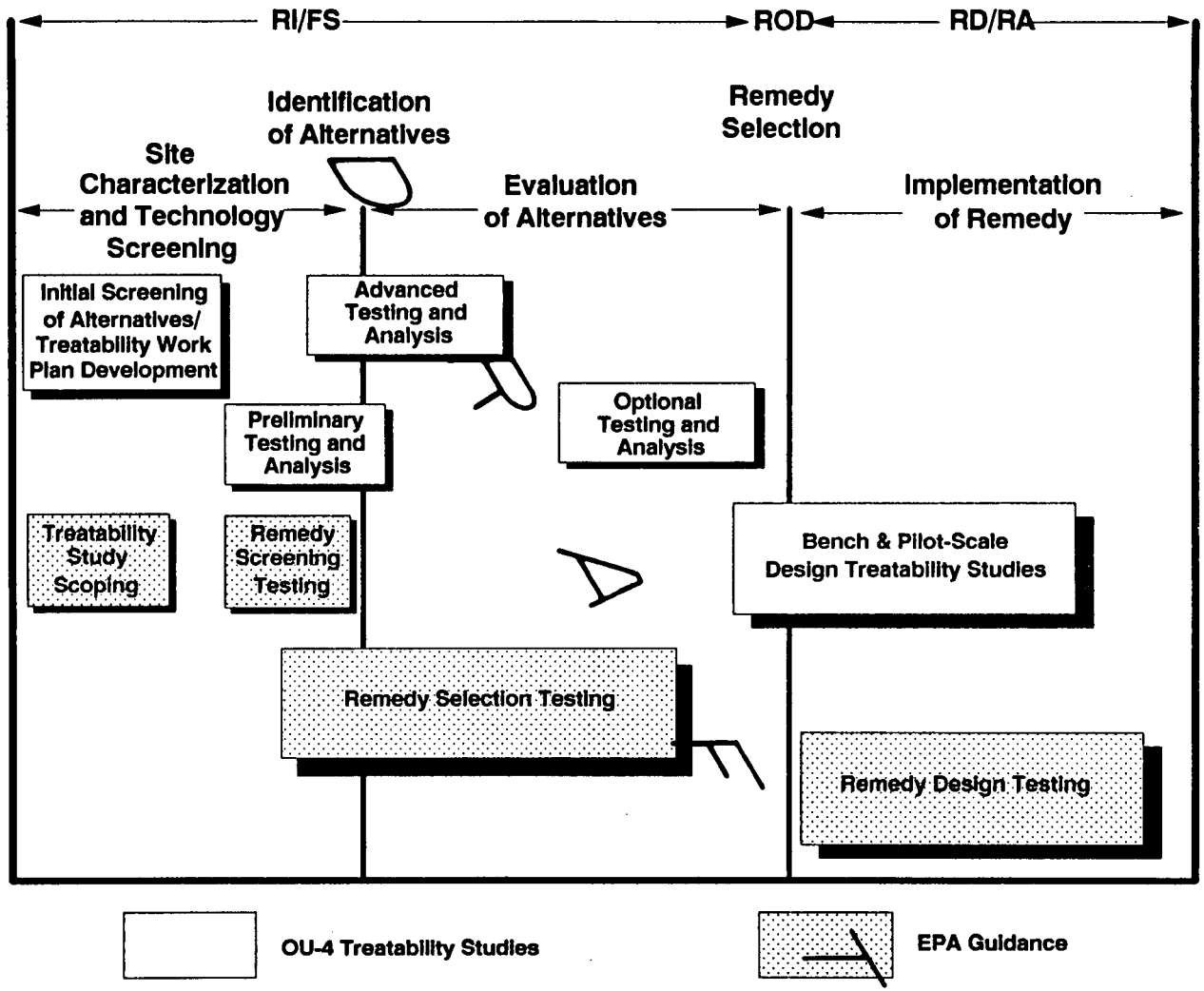

FIGURE 1-3 Relationship of the Operable Unit 4 Treatability Studies to the RI/FS Process.

Source: Guide for Conducting Treatability Studies Under CERCLA. Under Revision.

- Cost
- State acceptance
- Community acceptance

These criteria are described in detail in "Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA" (EPA/540/G-89/004).

Remedy screening is the first step in the tiered approach. Its purpose is to determine the feasibility of a treatment alternative for the contaminants/matrix or interest. These tests are typically conducted under conditions that are favorable to the technology. These small-scale studies are designed to provide a qualitative evaluation of the technology and are conducted with minimal levels of quality assurance/quality control (QA/QC). Tests conducted under this tier are generic in nature (not vendor specific). If the feasibility of the treatment cannot be demonstrated, the alternative should generally be screened out at this time.

The remedy selection tier of the treatability study program is designed to determine whether a treatment alternative can meet the operable units' cleanup criteria and at what cost. The purpose of this tier is to generate the performance and cost data necessary for remedy evaluation in the detailed analysis of the FS alternative phase. The cost data developed in this tier should support cost estimates of +50/-30 percent accuracy. The performance data will be used to determine if the technology will meet ARARs or cleanup goals. Remedy selection studies are typically small scale incorporating generic tests using bench-, or pilot-scale equipment in either the laboratory or field. The study costs are higher than those encountered in the remedy screening tier and require longer durations to complete. The levels of QA/QC are moderate to high because the data from these studies will be used to support the ROD.

In the remedy design tier treatability study, detailed scale-up, design, performance, and cost data are generated to implement and optimize the selected remedy. Remedy design studies are performed after the ROD, usually as part of the remedy implementation. These studies are performed on full-scale or near full-scale equipment with the purpose of generating detailed, scale-up design and cost data. The study should focus on optimizing process parameters. These studies require moderate to high QA/QC and are typically vendor specific.

1.3.3 Approach

Treatability studies on the silo materials will be performed as part of the remedy evaluation phase of the RI/FS. These treatability studies will aid in the selection of a remedial action alternative that is

feasible, implementable, and cost-effective. These studies will consider both the stabilization of the Silos 1 and 2, and Silo 3 material and the leaching, stabilization, and leachate purification of the Silos 1 and 2 wastes. Because of the differences in the hazardous substances found between Silos 1 and 2 and Silo 3, these materials will be handled as separate treatability study samples.

This treatability study will provide data for evaluating the performance of the stabilization and leaching/stabilization remedial alternatives. Reagent formulations for stabilization of the materials from Silos 1 and 2 and Silo 3 will be determined. Additional leaching experiments will be performed on the Silos 1 and 2 material to determine if the metals and radioactive contaminants can be extracted so that the remaining solid residue can be disposed of as nonhazardous waste, thus reducing the volume of mixed waste. Metals in the leachate from the extraction tests will be precipitated and stabilized. If necessary, the leachate will undergo additional treatment following the metal precipitation step. See Figure 1-4 for the overall flow sheet for this treatability study.

This work plan covers the remedy screening, and remedy selection tiers of the treatability studies as described in the EPA guidance. The remediation screening is performed in the preliminary screening studies and the remediation selection is performed in the advanced treatability studies. The preliminary screening studies will determine the potential reagents and conditions for stabilization and/or leaching of the silo material. Composite samples will be tested in the preliminary screening experiments to minimize total number of experiments, cost, and waste generation. The advanced screening will optimize the systems devised in the laboratory screening experiments. The effect of silo material variability will be evaluated in the advanced studies by testing the promising formulations and/or leaching on the top, middle, and bottom layers from each silo.

1.3.4 Stabilization of Untreated Silo Material

In the preliminary screening, the main effects of various stabilization reagents (i.e., portland cement Type II, Type F fly ash, sodium silicate, attapulgite, clinoptilolite, and water) will be tested. The samples from the 1989 and 1990-1991 sampling efforts will be subjected to this screening process. The data produced will be used to determine the scope of the advanced screening studies. Samples from the 1990-1991 sampling effort will be used in the advanced screening studies.

Vitrification studies of untreated silo material are not included in the scope of this work plan but are being conducted separately. It is mentioned here so that the reader is aware that all currently available stabilization technologies are being considered.

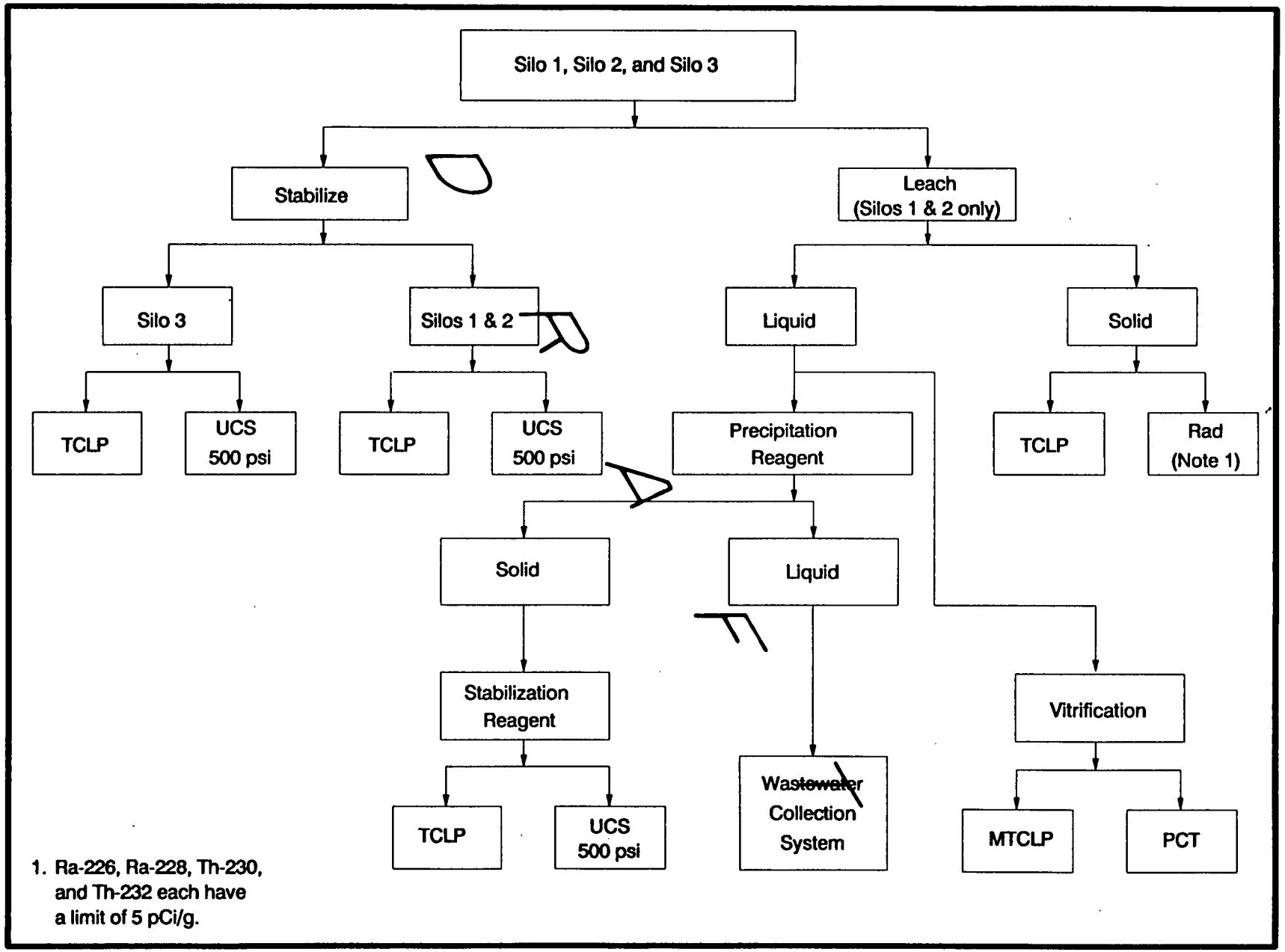

FIGURE 1-4 TREATABILITY FLOWSHEET

1.3.5 Silos 1 and 2 Metals Extraction/Precipitation/Stabilization/Vitrification

Only preliminary or remedy screening will be performed. The screening will test various chemical leaching techniques on residues from the Silos 1 and 2. The samples will be subjected to this screening process to determine the responsiveness of the silo material to various acid and ethylenediaminetetraacetic acid (EDTA) leaching schemes. The most promising leaching methods, as determined by the laboratory screening, will be applied in the advanced phase analysis. The treatability study will also study leaching kinetics, solids washing, solid/liquid separation, precipitation of remaining metals in the leachate solution, and vitrification of the leachate and stabilization of the material precipitated from the leachate. The most effective stabilization reagents determined from the screening described in Section 1.3.2 will be used to stabilize the precipitated material (Figure 1-4). Samples from the 1990-1991 sampling effort will be used in the advanced phase operations.

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2.0 REMEDIAL TECHNOLOGY DESCRIPTION

Several remediation technologies are being considered for Operable Unit 4. These alternatives have been described in detail in the DOE report, "Initial Screening of Alternatives for Operable Unit 4, Task 12 Report, October 1990." In the Task 12 report, Silos 1 and 2 are treated by the same alternatives because the materials in the structures are similar. Silo 3 is treated in separate alternatives.

The stabilization technology considered in the following alternatives consists of making a concrete-like material out of the waste with the addition of cement, fly ash, and some other compounds. The leaching technology consists of dissolving the radioactive and hazardous components with a solvent, followed by precipitation and stabilization or vitrification of the metals in the leachate. The leaching procedure would greatly reduce the volume of material to be stabilized and disposed of as low-level radioactive waste. The reduction in volume of radioactive and hazardous waste material would greatly reduce the final disposal and transportation costs, which represents the major costs associated with all the viable remedial action alternatives. Solids remaining from the metals extraction would meet limits for residual radioactive and hazardous material in soil. This treated material could be used as on-property fill.

2.1 SUMMARY OF ALTERNATIVES - SILOS 1 AND 2

Alternative 0A - No Action

This alternative calls for no action and provides a baseline against which the other alternatives can be compared. It provides for the silos and its contents to remain unchanged without the implementation of any removal, treatment, containment, or mitigation technologies. However, it does include the installation of long-term monitoring equipment as well as the cost of the monitoring program.

Alternative 1A - Nonremoval, Silo 1 Isolation

This nonremoval alternative for Silos 1 and 2 consists of enhancing the containment integrity of the silos and utilizing them as permanent disposal facilities. An impermeable clay cap and slurry wall are among the technologies considered for this alternative.

Alternative 2A - Nonremoval, In Situ Stabilization, and Cap

This nonremoval alternative for Silos 1 and 2 consists of in situ stabilization and capping. Conventional physical stabilization and vitrification were considered as options. However, in situ vitrification was screened out as a process option due to concerns about the difficulty of implementability. The capping and isolation technologies, with the exception of the slurry wall, are identical to those described for Alternative 1A.

Alternative 6 - Removal, Treatment, and On-Property Disposal

This alternative for Silos 1 and 2 calls for the removal and conventional stabilization or vitrification of the silo contents before on-property disposal in an engineered disposal facility. This alternative includes silo demolition and disposal of the debris. See Figure 2-1 for a flow diagram of Alternative 6.

Alternative 7 - Removal, Treatment, and Off-Site Disposal

This alternative for removal of the Silos 1 and 2 material is identical to Alternative 6 except that the material would be packaged for shipment to an approved off-site disposal facility. The flow diagram for Alternative 7 is in Figure 2-2.

Alternative 8 - Removal, Contaminant Separation, and On-Property Disposal

This removal alternative for the Silos 1 and 2 material is similar to Alternative 6 but adds an additional step of contaminant separation to remove various radionuclides and metals before stabilization or vitrification and on-property disposal. This would result in significant volume reduction of material to be disposed of as radioactive waste. The waste materials will be subjected to acid and EDTA leaching processes to dissolve the radioactive and hazardous metals, including lead, uranium, thorium, and radium. This leaching process is based on data from Seely (1977), Mound Laboratories (1951), and Battelle (1981). Lead, barium, copper, and other metals will also be dissolved in the extraction fluid. Following this leaching stage, the remaining solids will enter a solid/liquid separation stage, and the leachate containing the radioactive and hazardous materials will be sent to a precipitation stage. This precipitation stage will add selected anions to yield a radioactive/hazardous precipitate to be vitrified or stabilized for disposal. With the successful leaching process, the raffinate residues remaining after the acid or EDTA leaching processes will be disposed of as a nonhazardous waste. See Figure 2-3 for the flow diagram of this alternative.

Alternative 9 - Removal, Contaminant Separation, and Off-Site Disposal

This alternative is identical to Alternative 8, except that the material would be packaged and shipped to an approved off-site disposal facility and the nonhazardous portion is sent to a landfill or is used as backfill on property. See Figure 2-4 for the flow diagram.

2.2 SUMMARY OF ALTERNATIVES - SILO 3

Alternative 0B - No Action

The no-action alternative for Silo 3, as was the case for Silos 1 and 2, provides a baseline but no remedial action. Only installation of long-term monitoring equipment and the cost of the monitoring program are included.

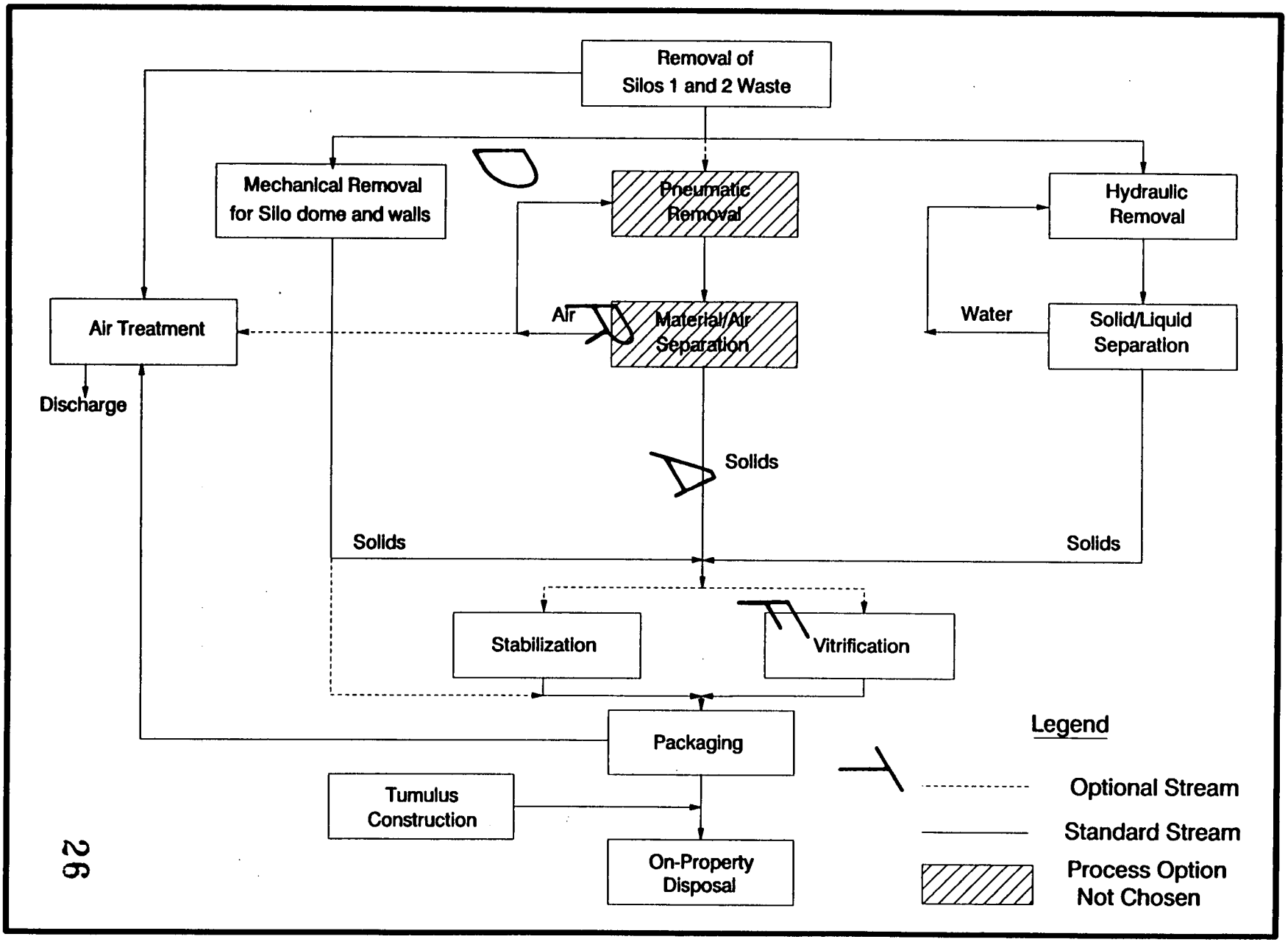

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FIGURE 2-1 ALTERNATIVE 6 - REMOVAL, TREATMENT, ON-PROPERTY DISPOSAL - SILOS 1 AND 2

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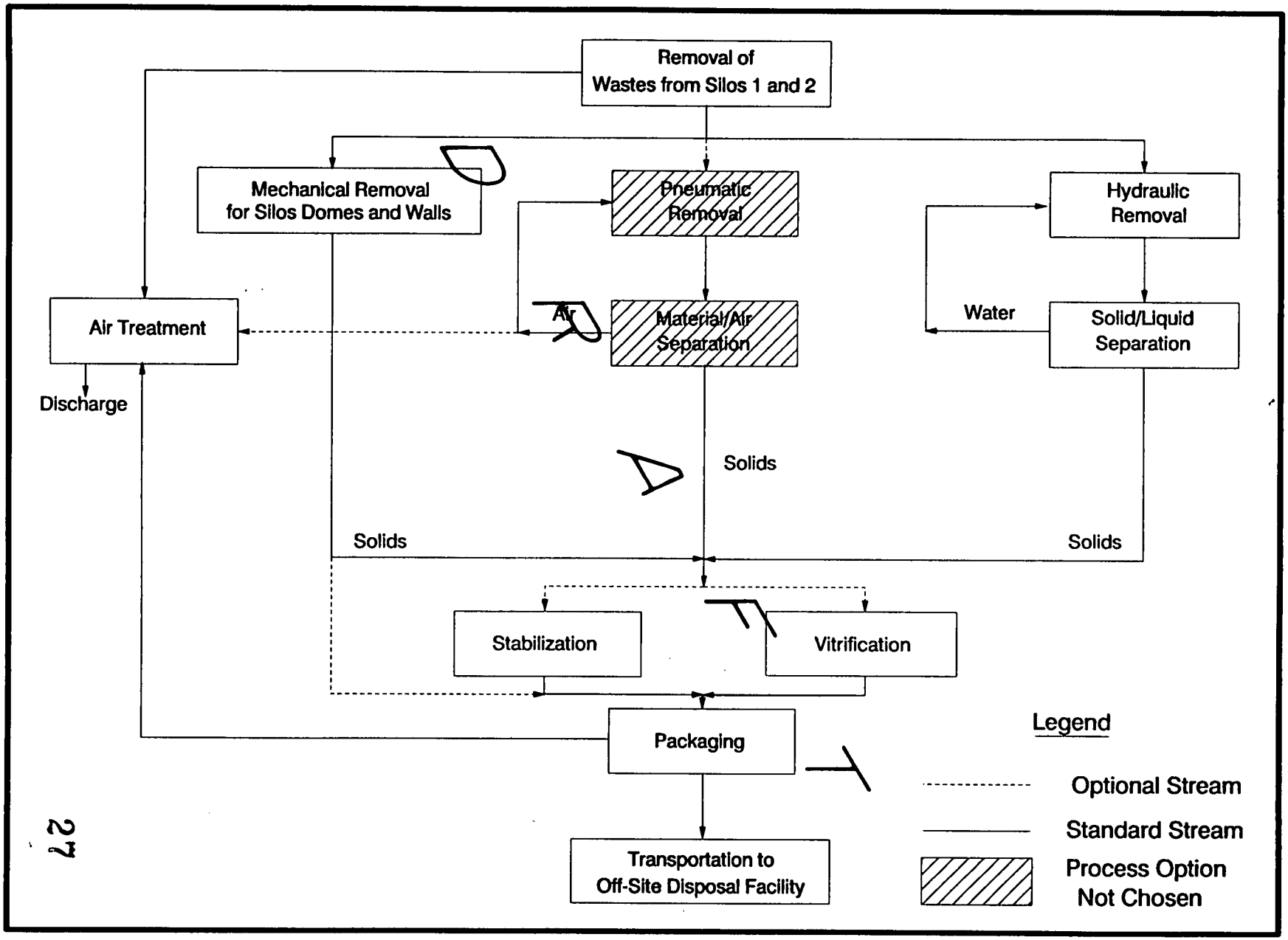

FIGURE 2-2 ALTERNATIVE 7 - REMOVAL,TREATMENT,OFF-SITE DISPOSAL - SILOS 1 AND 2

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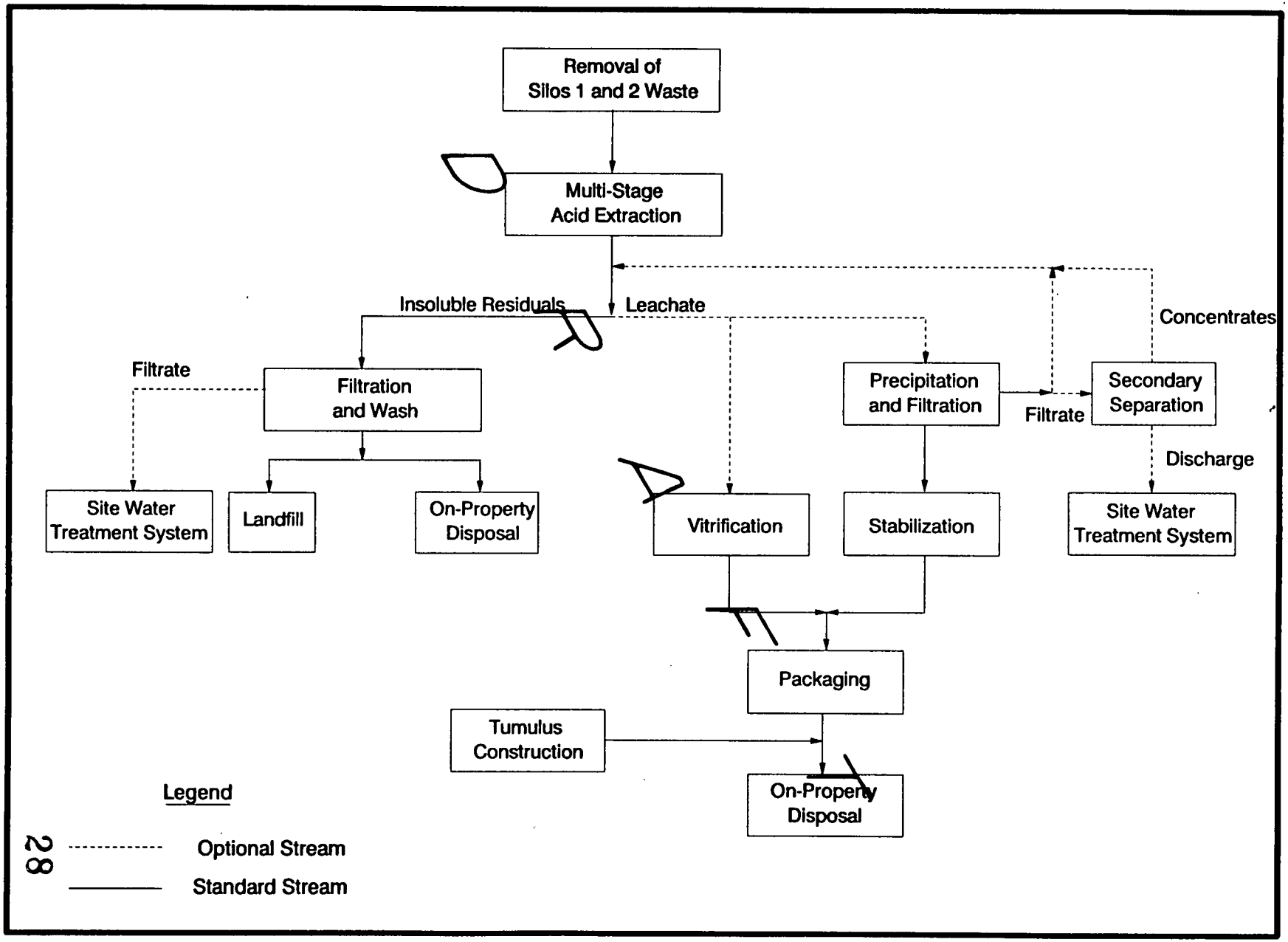

FIGURE 2-3 ALTERNATIVE 8 - REMOVAL, CONTAMINANT SEPARATION, ON-PROPERTY DISPOSAL - SILOS 1 AND 2

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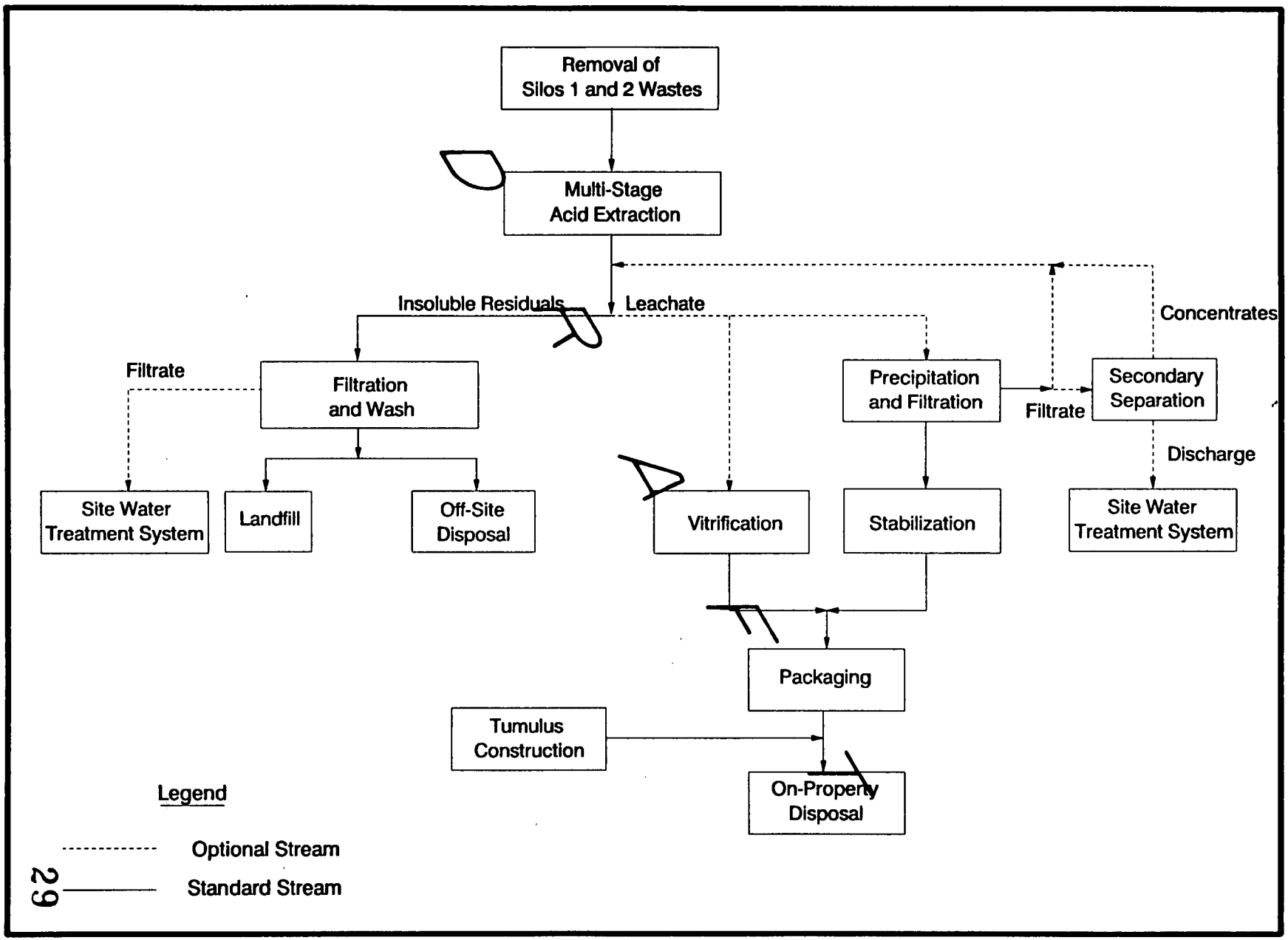

FIGURE 2-4 ALTERNATIVE 9 - REMOVAL, CONTAMINANT SEPARATION, OFF-SITE DISPOSAL - SILOS 1 AND 2

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Alternative 1B - Nonremoval, Silo Isolation

This nonremoval alternative for Silo 3 consists of enhancing the containment integrity of the silo and utilizing it as a permanent disposal facility. An impermeable clay cap and slurry wall are among the technologies considered for this alternative.

Alternative 2B - Nonremoval, In Situ Stabilization, and Cap

This nonremoval alternative for Silo 3 consists of in situ stabilization and capping. The capping and isolation technologies, with the exception of the slurry wall, are identical to those described in Alternative 1B.

Alternative 3 - Removal and On-Property Disposal

This alternative for Silo 3 calls for removal and conventional stabilization or vitrification before disposal in an engineered on-property disposal facility. This alternative includes silo demolition and disposal of the debris. The flow diagram for Alternative 3 for Silo 3 is identical to Alternative 6 for Silos 1 and 2 except that the feed for the process is from Silo 3.

Alternative 4 - Removal of Metal Oxides and Off-Site Disposal

This alternative for Silo 3 is identical to Alternative 3, except that the material would be packaged for shipment to an approved off-site disposal facility. The flow diagram for Alternative 4 is analogous to that for Alternative 7.

Alternative 5 - Removal and Replacement in Rehabilitated Silos

This alternative for Silo 3 provides for the removal of the metal oxides and their return to a rehabilitated Silo 3 or Silo 4 reconstructed as a permanent disposal facility. This alternative was not carried through to detailed analysis because of its inadequate effectiveness and implementability.

Three alternatives for the three silos are considered nonviable. These alternatives are the "No Action" alternatives, 0A (Silos 1 and 2) and 0B (Silo 3); and Alternative 5, "Removal and Replacement in Rehabilitated Silo 3."

For Silos 1 and 2, the data from this treatability study will be used to help evaluate the stabilization Alternatives 2A, 6, and 7 and the leaching/stabilization Alternatives 8 and 9. The data will be used in the evaluation of the Silo 3 stabilization Alternatives 2B, 3, and 4 (see Figure 1-4).

As currently planned, vitrification studies for untreated silo material will be conducted separately.

3.0 TEST AND DATA QUALITY OBJECTIVES

The purpose of this treatability testing program for this laboratory is to assess the performance of various stabilization/leaching technologies on the Operable Unit 4 wastes in support of the RI/FS. To select a preferred alternative for the Operable Unit 4 RI/FS, a waste treatment technology must be screened, data for risk assessment studies and ARARs determination must be generated, and the foundation for the subsequent treatability studies must be set. In addition, the level of QA applied during experimentation and analysis must be established.

This section will establish the performance objectives for the treatment technologies, the additional data desired for use in subsequent stages of the RI/FS, and the data quality objectives (DQOs).

3.1 PERFORMANCE OBJECTIVES AND DESIRED DATA - STABILIZATION

Specific test objectives have been established so that the performance of the various stabilization mixtures can be evaluated in the areas of leachability, unconfined compressive strength (UCS), and final waste form volume. These performance objectives will be used to determine if a particular reagent mixture produces an acceptable waste form. The specific objectives of the laboratory treatability testing program are as follows:

- To determine the cement stabilization reagents and relative quantities required so that the final waste form meets TCLP criteria
- To determine the cement stabilization reagents and relative quantities required so that the final waste form achieves a compressive strength of at least 500 pounds per square inch (psi)
- To minimize the final volume of treated waste
- To estimate the volumes of waste that will be generated by each process
- To provide preliminary cost and design data for the RI/FS
- To provide leaching characteristics for use in fate and transport modeling
- To develop preliminary reagent mixtures for use in later treatability studies
- To develop process parameters for use in later treatability studies:
  - For cement stabilization: shear strength, waste form temperature rise with reagent addition, general description of waste before and after reagent addition, permeability of raw sample, and percent water in the waste
- To provide data for evaluation of Silos 1 and 2 Alternatives:

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- 2A - Nonremoval, In Situ Stabilization, and Cap
- 6 - Removal, Treatment, and On-Property Disposal
- 7 - Removal, Treatment, and Off-Site Disposal

and Silo 3 Alternatives:

- 2B - Nonremoval, In Situ Stabilization, and Cap
- 3 - Removal and On-Property Disposal
- 4 - Removal and Off-Site Disposal

3.2 DATA QUALITY OBJECTIVES - STABILIZATION

The establishment of DQOs is the part of the process that defines the data quality needs of the project. The implementation of an appropriate QA/QC program is required to ensure that data of known and documented quality are generated. The DQOs will define the level of QA/QC for the treatability testing and analysis. The DQOs for this testing program are quantitative in nature because the stabilized waste must meet specific performance criteria, namely a UCS greater than 500 psi and Toxicity Characteristic Leaching Procedure (TCLP) leachability criteria.

DQO analytical levels are defined in EPA's "Guide for Conducting Treatability Studies Under CERCLA." This guide states that the requisite analytical levels are dictated by the types and magnitudes of decisions to be made based on the data and the objective of the screening. A description of the analytical levels is presented in Table 3-1, an excerpt from EPA's guide. A discussion of the DQOs for each stage of the screening for cement stabilization follows. A list of tests and associated DQOs for stabilization are listed in Table 3-2.

Composite samples will be used in the initial stage(s) to minimize the total number of experiments, cost, and waste generation. These experiments will aid in the resolution of general ranges of reagent formulations needed to stabilize and vitrify the waste and to elucidate on potential problems with different stabilization schemes. Experiments with strata samples will be conducted to determine the effects of waste material variability on the stabilization processes. Pocket penetrometer compressive strength and Modified Toxicity Characteristic Leaching Procedure (MTCLP) screening data will be acquired in the initial stage(s) to minimize costs and waste generation.

3.2.1 Preliminary Screening (Composite Samples)

The preliminary screening will be an initial run in two or three stages on composite samples and will not require a high statistical confidence level. The tests performed on the stabilized waste samples will be waste form temperature after reagent addition, an unconfined compressive strength test with a

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Table 3-1. Summary of Analytical Levels

Level I	
Type of analysis	Field Screening or analysis with portable instruments.
Limitations	Usually not compound-specific, but results are available in real time. Not quantifiable.
Data Quality	Can provide an indication of contamination presence. Few QA/QC requirements.
Level II	
Type of analysis	Field analysis with more sophisticated portable instruments or mobile laboratory. Organics by GC; inorganics by AA, ICP, or XRF.
Limitations	Detection limits vary from low parts per million to low parts per billion. Tentative identification of compounds. Techniques/instruments limited mostly to volatile organics and metals.
Data quality	Depends on QA/QC steps employed. Data typically reported in concentration ranges.
Level III	
Type of analysis	Organics/inorganics performed in an off-site analytical laboratory. May or may not use CLP procedures. Laboratory may or may not be a CLP laboratory.
Limitations	Tentative compound identification in some cases.
Data quality	Detection limits similar to CLP. Rigorous QA/QC.
Level IV	
Type of analysis	Hazardous Substances List (HSL) organics/inorganics by GC/MS, AA, ICP. Low parts-per-billion detection limits.
Limitations	Tentative identification of non-HSL parameters. Validation of laboratory results may take several weeks.
Data quality	Goal is data of known quality. Rigorous QA/QC.
Level V	
Type of analysis	Analysis by nonstandard methods.
Limitations	May require method development or modification. Method-specific detection limits. Will probably require special lead time.
Data quality	Method-specific

Source: EPA, "Guide for Conducting Treatability Studies under CERCLA, "December 1989.

TABLE 3-2 STABILIZATION TEST DQOS

TEST	DQO/COMMENT	DQO LEVEL
PRELIMINARY (REMEDY SCREENING)		
Bulking Factor by Solvent Displacement	Minimize waste volume increase. To estimate the volume of waste that will be generated.	V
Pocket Penetrometer	During screening phases, to determine stabilization reagent formulation so that the final waste form achieves a unconfined compressible strength of at least 500 psi.	I
Modified Toxicity Characteristic Leaching Procedure (MTCLP)	During the screening phases, to determine stabilization reagent formulations so that the final waste form meets the TCLP metal leaching criteria. In addition, the test data will indicate the relative leachability of radionuclides with the various reagent formulations.	V
Waste Form Temperature	Preliminary Process Parameters	I
Shear Strength	Preliminary Process Parameters	I
ADVANCED (REMEDY SELECTION)		
Bulking Factor by Bulk Density Calculation	Minimize waste volume increase. To estimate the volume of waste waste that will be generated.	I
Unconfined Compressive Strength (ASTM D2166)	To determine the stabilization reagent formulation so that the final waste form achieves a unconfined compressive strength of at least 500 psi. In addition to provide preliminary cost and design data for the RI/FS.	III
Full TCLP	To determine stabilization reagent formulations so that the final waste form meets the TCLP criteria. To provide data for the FS risk assessment calculations.	III

PRELIMINARY (REMEDY SCREENING)		
TEST	**DQO/COMMENT**	**DQO LEVEL**
5-Day Static Leach Test	To provide data for the FS risk assessment calculations	V
Permeability	To provide data for the FS risk assessment calculations	III
Waste Form Temperature Rise	To provide preliminary process parameters	I
Shear Strength	To provide preliminary process parameters	I

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pocket penetrometer (Soiltest CT-421), shear test with a Soiltest Torvane, MTCLP, and bulking factor. Data from the CT-421 pocket penetrometer will be DQO analytical Level I. The MTCLP differs from the standard TCLP as follows: the MTCLP uses only 2.5 grams of material rather than 100 grams; the MTCLP generates only 50 milliliters of leachate rather than 2 liters; and the leachate from the MTCLP is analyzed for metals only rather than metals and organics. The data from the MTCLP will be DQO analytical Level V because the method is non-standard. The bulking factor is the measured percent volume increase/decrease of the treated waste relative to the original waste volume. In the preliminary screening, the bulking factor will be determined according to the SOP in Appendix B and will be DQO analytical Level V.

3.2.2 Advanced Screening - Silos 1 and 2

All previous stages of this screening have tested composite samples. This stage will take the one or two of the most promising reagent mixes, as determined in the previous stages, and apply them to the six strata samples (three samples from each of the two silos). This will allow for the determination of the effect of the mixes on the individual strata. This stage of the screening will also repeat the best strata sample mixes (those with the lowest reagent loading and the lowest bulking factor) to confirm their performance. For those strata samples that do not produce an adequate waste form, additional reagent formulations will be attempted.

Shear strength and temperature rise will be measured on all samples within 10 minutes after mixing the waste and reagents. The UCS will be determined with a Soiltest U-610 instrument according to the SOP given in Appendix F (ASTM D2166). TCLP, bulking factor, 5-day leach test, and permeability will be performed on samples with a 28-day UCS greater than 500 psi.

The bulking factor will be determined using the following equation:

$$BF = \frac{100 * (100+A)/P_t - 100/P_r}{100/P_r}$$

where

BF = percent bulking factor
A = percent additives
P_t = density of treated waste
P_r = density of raw waste

The density of the raw waste will be measured as in the preliminary phase. The density of the treated waste will be calculated by dividing the volume of the UCS solid cylinder (2 by 4 inches) by its weight. (See EPA document EPA/625/6-89/022 Section 4.2.4 for description of bulk density measurement of stabilized waste.)

The DQO analytical levels for UCS, permeability, and TCLP will be DQO Level III. The DQO level for the bulking factor and 5-day static leach tests will be Level V because these are nonstandard methods.

3.2.3 Advance Screening - Silo 3

Composite samples will be used in this phase of the testing program. The one or two most promising formulations for Silo 3 will be repeated on a larger scale. The UCS will be measured with a Soiltest U-610 instrument (ASTM D2166). Shear strength and temperature rise will be measured on all samples. TCLP, bulking factor, permeability, and static 5-day leach test will be performed on samples with a 28-day UCS greater than 500 psi.

The DQO analytical levels for Silo 3 advanced screening are the same as for Silos 1 and 2 advanced screening.

3.3 PERFORMANCE OBJECTIVES AND DESIRED DATA - METAL EXTRACTION/ PRECIPITATION/STABILIZATION/VITRIFICATION

Specific test objectives have been established so that the performance of various acids, precipitation agents, and stabilizing reagents can be evaluated. These performance objectives will be used to determine if metal extraction/precipitation/stabilization/vitrification merits further testing or consideration. The objectives are as follows:

- To extract RCRA metals so that the insoluble residue will pass TCLP i.e., produce a non-RCRA residue
- To reduce the level of radioactive components in the insoluble residue to below limits, i.e., 5 pCi/g each of Ra-226, Ra-228, Th-230, and Th-232 (DOE Order 5400.5)
- To determine the reagents and conditions necessary to precipitate the metals in the leachate solution
- To determine the cement stabilization and vitrification reagents and relative quantities required so that the precipitated metals in their final waste form meet TCLP
- To determine the cement stabilization and vitrification reagents and relative quantities required so that the final waste form achieves a compressive strength of 500 psi

- To minimize the final volume of treated waste
- To estimate the volumes of wastes that will be generated by each process
- To provide preliminary cost and design data for the RI/FS
- To provide leaching characteristics for use in fate and transport modeling
- To develop preliminary reagent mixture and process parameter data for use in the bench- and pilot-scale studies as follows:
  - For cement stabilization: shear strength, waste form temperature rise with reagent addition, general description of waste before and after reagent addition, permeability of treated sample, and percent water in the waste
  - For vitrification: percent water in the waste, and types and percent additives required
- To provide data for the evaluation of Alternative 8 - Removal, Contaminant Separation, and On-Property Disposal; and Alternative 9 - Removal, Contaminant Separation, and Off-Site Disposal

3.4 DQOs - METAL EXTRACTION/PRECIPITATION/STABILIZATION/VITRIFICATION

A list of tests and associated DQOs for metal extraction/precipitation/stabilization/vitrification are in Table 3-3. All screening will be done using composite samples. Qualitative tests for lead and uranium in the leachate will be used to screen out the least effective solvents. These tests are described in Appendices C and D, and will be DQO analytical Level I.

The confirmatory leaching tests will include analyses of the insoluble residue remaining after the metals have been extracted. These tests include TCLP for RCRA metals, organics, and radiological analysis for uranium, radium, thorium, and lead. These tests will identify the most effective solvents. These tests will be DQO analytical Level III.

If the leaching process is successful, the leachate from the successful runs will be used in the precipitation screening. Various precipitation reagents will be used to precipitate metals from the leachate. The relative effectiveness of the various reagents will be determined with the same qualitative tests for lead and uranium that were used in the leaching experiments. These tests will be DQO analytical Level I.

The precipitated material from the most effective precipitation reagents will be subjected to stabilization tests. The stabilized material will be tested for waste from temperature rise, shear strength, and compressive strength (Soiltest CT-421). Waste form temperature rise will be measured within ten minutes after reagents and waste are mixed. These tests are DQO analytical Level I. Samples with a

TABLE 3-3 METALS EXTRACTIONS TEST DQOs

PRELIMINARY (REMEDY SCREENING)		
TEST	**DQO/COMMENT**	**DQO LEVEL**
Bulking Factor By Solvent Displacement	Minimize waste volume increase during stabilization and vitrification. Estimate the volume of waste that will be generated.	V
Pocket Penetrometer	During screening phases, to determine stabilization reagent formulation so that the final waste form achieves an unconfined compressible strength of at least 500 psi.	I
Modified Toxicity Characteristic Leaching Procedure (MTCLP)	During the screening phases, to determine stabilization and vitrification reagent formulations so that the final waste form meets the TCLP metal leaching criteria. In addition, the test data will indicate the relative leachability of radionuclides with the various reagent formulations.	V
Waste Form Temperature Rise	Preliminary Process Parameters (Cement Stabilization)	I
Shear Strength	Preliminary Process Parameters (Cement Stabilization)	I
PCT	To provide data on vitrification for the FS ~~risk~~ assessment calculations. To provide data on the relative leachability of radionuclides and glass components with the various reagent formulations.	V
Uranium and Lead Spot Tests	To qualitatively compare the effectiveness of various solvents and reagents during leaching and precipitation experiments.	I
Radiological	To quantify the residual radionuclide (Ra, Th, U) concentrations in the insoluble residue resulting from tests with the most effective solvents. This will be used to grade solvents pass/fail.	III

PRELIMINARY (REMEDY SCREENING)		
TEST	**DQO/COMMENT**	**DQO LEVEL**
TCLP	To determine if the insoluble residue resulting from tests with the most effective solvents can be classified as non-RCRA material. This will be used to grade solvents pass/fail.	III

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28-day penetration resistance greater than 500 psi will be measured for bulking factor, MTCLP, and 5-day leach test. These tests are DQO analytical Level V.

The vitrified leachate will be subjected to MTCLP, bulking factor, and Nuclear Waste Glass Product Consistency Test (PCT). These tests are DQO Level V.

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4.0 EXPERIMENTAL DESIGN AND PROCEDURES

4.1 TESTING PROCEDURES

4.1.1 Stabilization

The waste will be sieved through a 3/8-inch mesh screen before testing. Obvious debris will be removed. In the preliminary phase, approximately 100 grams of waste and correct amounts of reagents will be mixed in a plastic container, slightly compacted by tapping with a bolt, and the container sealed with a lid. Mixing will be done by hand with a spatula until the mixture has an even consistency without any lumps.

In the advanced phase, approximately 300 grams of waste per mold will be mixed with the correct amount of reagents, in an American Society for Testing and Materials (ASTM) grade Hobart Planetary mixer. The mixture will be added to a two- by four-inch Jatco plastic cylinder in three to six aliquots. The mixture will be compacted using a vibratory table. After the molds are loaded, they will be capped and sealed with tape until the sample is tested on Day 28.

4.1.2 Metal Extractions

4.1.2.1 Acid Extractions

Approximately 1-gram aliquots of each sample will be weighed in the hood in HACH COD Digester Vials (rated pressure 10 atm). Room temperature vials will be shaken with acid for two hours. Room temperature will be the actual temperature inside the hood and this value will be recorded in a standard laboratory notebook. The digestions will be carried out in a HACH Micro COD Digester (Appendix E). The extractions will be heated at 100°C and digested for two hours in the HACH Digester within the hood. After digestion, the samples will be separated by decanting into a 20-mL scintillation vial. Solids will be retained in the COD vial until the decision can be made whether to carry them into the next phase or not. This will be based on the lead and uranium content of the extract. If one of the digestions is clearly superior to the others, further treatment of the others will be aborted. A superior digestion will be one that extracts the greatest amount of lead and uranium. If not processed further, solids will be transferred to a one-pint container for disposal. Liquids will be syringe-filtered (0.45μ) into 8-mL scintillation vials. The filtered samples will be diluted (ca 1:1000 to 1:10,000) into 20-mL scintillation vials and analyzed for lead (Jungreis, "Spot Test Analysis," Appendix C. The carbon tetrachloride in the original procedure has been replaced by 1,1,1-trichloroethane. The solutions will be separated by removing the bottom layer with a pasteur pipet rather than a separatory funnel. Samples diluted 1:1000 to 1:10000 with deionized water will then be transferred to a COD vial containing 5 mL of 0.1 percent potassium cyanide, sealed, shaken, and allowed to

settle. Quantification of the lead will be by HACH DRL-3. The HACH DRL-3 is a spectrophotometer used to measure the absorbance of the lead solution. As an extra precaution, the COD vials containing cyanide buffer have been preloaded with reagent so that the maximum amount of reagent handled at any one time will be 5 mL. Uranium analysis will be performed on the organic layer after the lead content has been determined. The uranium content will be determined as described in Appendix D (Spot Tests in Inorganic Analysis, F. Feigl).

4.1.2.2 EDTA Extractions

Literature results using EDTA as leachate are contradictory. It appears that EDTA might have some benefit as an extractant. Because of this, a range finding test using 0.2 molar EDTA will also be run.

Samples will be analyzed for lead as before (EDTA samples may require pretreatment nitric acid digestion) and for uranium using a modified Feigl test (F. Feigl, "Spot Tests in Inorganic Analysis," Appendix E).

Criteria for success will be the magnitude of lead and uranium leached compared to the other processes.

4.1.2.3 Precipitation

Leachate from the acid or EDTA extractions will be placed in a beaker. Measured quantities of precipitation reagents will be added and stirred in by hand until completely dissolved. The mixture will be centrifuged to settle the solids so that the liquid can be decanted.

4.1.2.4 Stabilization

The procedure for the stabilization of precipitated material will be the same as described in Section 4.1.1, except that the precipitate will not be sieved.

4.1.2.5 Vitrification

The leachate will be analyzed to determine the metals concentration. This will be used to estimate the quantities of glass-making reagents required. The leachate will be evaporated to a dry solid; reagents will be mixed in by hand, and placed in a crucible. The mixture will be melted in the muffle furnace at approximately 1250°C.

4.2 PRELIMINARY CHARACTERIZATION

During geotechnical testing, the Silos 1 and 2 material of the 1989 samples was composited according to physical appearance, moisture content, and texture of the material. The material from Silo 1 was composited into groups of brown, sandy brown, and light brown. The material from Silo 2 was com-

posited into groups of wet muddy, white, and sandy brown. The material weights listed in Table 4-1 are the actual weights of the geotechnical samples collected during the 1989 sampling effort. The material from each silo will be composited to form a single composited sample for each silo on the weight percent basis shown in Table 4-1. This composited material will be used in the initial studies of the stabilization and leaching procedures.

For a physical characterization of the Silos 1 and 2 material from the 1989 sampling program, see Certificate of Analysis, IT Geotechnical Services Project No. 482331, dated March 22, 1989.

Chemical characterization of the composite samples will be analyzed before the laboratory screening of the treatability testing is performed. A list of the analyses is given in Table 4-2.

4.3 STABILIZATION OF UNTREATED MATERIAL

4.3.1 Preliminary Screening

In the preliminary screening, the main effects of various stabilization reagents (i.e., portland cement Type II, Type F fly ash, sodium silicate, attapulgite, clinoptilolite, and water) will be tested. Composite samples from the 1989 and 1990-1991 silo sampling programs will be tested. The data produced will be used to define the scope of the advanced screening better. A stabilization flow sheet is given in Figure 4-1. The preliminary screening data will also help to define the best reagents to stabilize the metals and radioactive materials precipitated from the leaching processes (Alternatives 8 and 9).

The preliminary screening phase consists of up to three separate stages, Stage 1, Stage 2, and Stage 3. The experimental matrices for the Stages 1 and 2 are in Table 4-3. The formulations for Stage 3, if required, will be developed after analyzing the results from the initial screening test.

There are two sets of tests in Table 4-3, a statistically based screening test matrix (Group I) and two single variable matrices (Groups II and III).

In the statistical screening matrix, composite samples will be treated with a combination portland Type II cement, PQ Corp Type N sodium silicate, and Type F commercial fly ash (Table 4-3, Group I). The stabilization matrix is based on the extreme vertices design for mixtures that have constraints on the values of each factor (McClean and Anderson 1966; Diamond 1981). Because this is a screening study, all two-dimensional face centroids have been omitted from the study.

TABLE 4-1

SAMPLE COMPOSITING

Silo No.		Soil Type	
Silo 1	Brown	Sandy Brown	Light Brown
Total kg % w/w (weight soil type/weight composite sample)	2.3 kg 9.1 kg 12.0 kg 4.2 kg 27.6 kg 41.0	4.7 kg 4.7 kg 7.0	35.0 kg 35.0 kg 52.0
Silo 2	Wet Muddy	White	Sandy Brown
Total kg % w/w (weight soil type/weight composite sample)	12.0 kg 12.0 kg 33.2	9.4 kg 4.0 kg 2.8 kg 2.3 kg 18.5 kg 51.2	2.8 kg 2.8 kg 5.6 kg 15.5

TABLE 4-2

CHEMICAL CHARACTERIZATION OF SILO WASTE

<u>Inorganic, Nonmetallics</u>
Alumina
Boron
Bromide
Calcium
Carbonate
Chloride
Cyanides
Fluoride
Magnesium
Nitrogen and ammonia
Nitrate
Nitrite
Total phosphorous
Alkyl phosphorous and alkyl phosphorous oxide compounds
Silica
Sulfate
Sulfide
Sulfite
Zinc
<u>Organics</u>
Total organic carbon
Oil and grease
Alcohols
Carboxylic acids
<u>Other Properties</u>
Acidity
pH

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TABLE 4-3
STABILIZATION MATRICES

GROUP I EXPERIMENTS STATISTICAL MATRIX						
Run Number	Waste (g)	Portland Cement Type II (g)	Fly Ash Type F (g)	Sodium Silicate Type N (g)	Attapulgite and Clinoptilolite Each (g)	Potential Range of Water Needed (g)
1	100	64	64	0.00	6	9 - 6.5
2	100	68	68	0.07	6	11 - 71
3	100	51	31	0.00	6	0 - 35
4	100	54	33	0.07	6	0 - 38
5	100	31	51	0.00	6	0 - 35
6	100	33	54	0.07	6	0 - 38
7	100	26	26	0.00	6	0 - 15
8	100	27	27	0.07	6	0 - 16
9	100	43	43	0.04	6	0 - 37

GROUP II EXPERIMENTS EFFECT OF SITE FLY ASH						
Run Number	Waste (g)	Portland Cement Type II (g)	Site Fly Ash Type F (g)	Sodium Silicate ~~Type N~~ (g)	Attapulgite and Clinoptilolite Each (g)	Potential Range of Water Needed (g)
10	100	43	43	0.00	6	0 - 37
11	100	43	43	0.04	6	0 - 37

GROUP III EXPERIMENTS EFFECT OF ADSORBANT						
Run Number	Waste (g)	Portland Cement Type II (g)	Site Fly Ash Type F (g)	Sodium Silicate Type N (g)	Attapulgite and Clinoptilolite Each (g)	Potential Range of Water Needed (g)
12	100	43	43	0.04	12A	0 - 37
13	100	43	43	0.04	12C	0 - 37

*12A and 12C: Add 12 grams of attapulgite and clinoptilolite, respectively.

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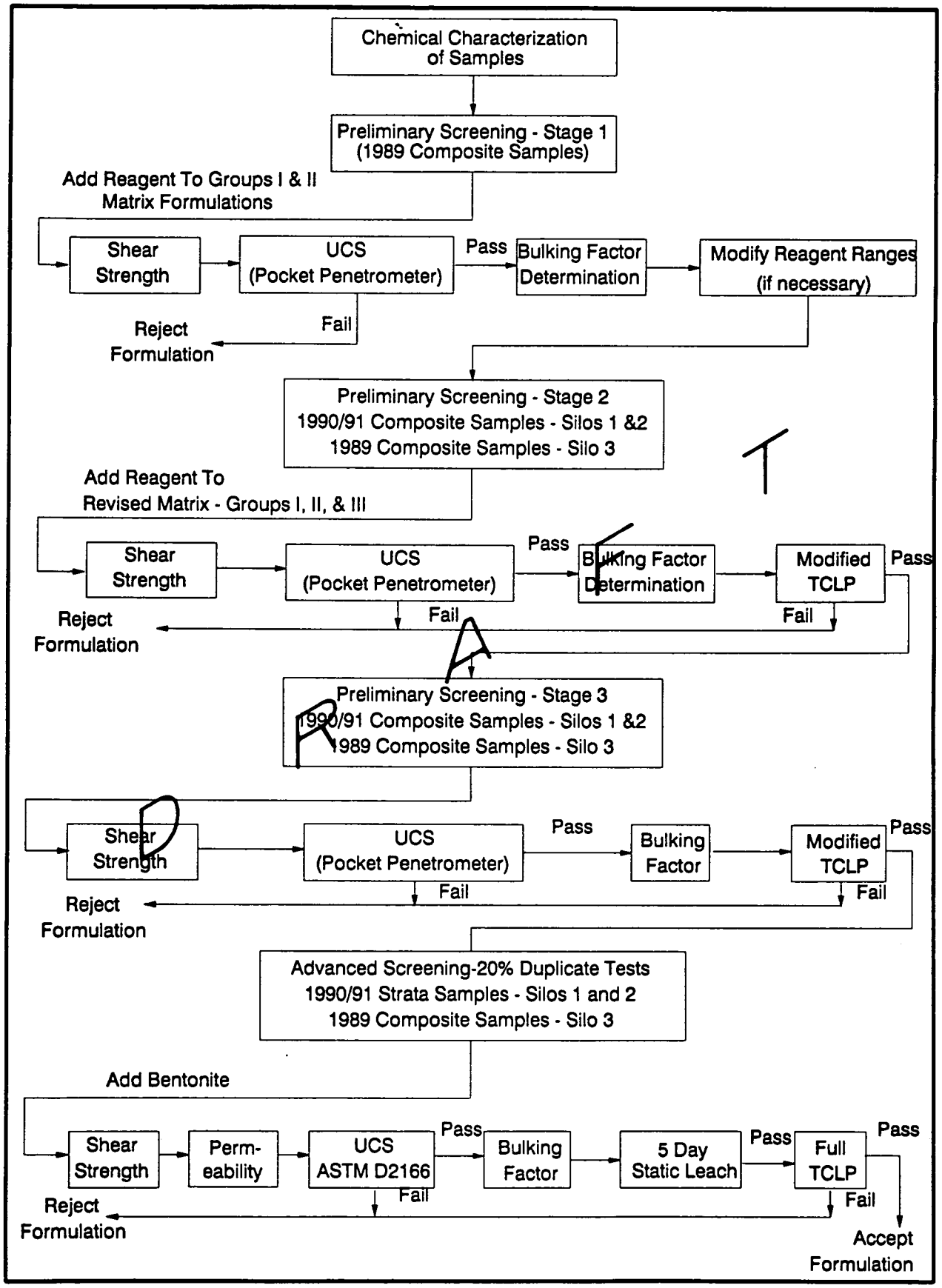

FIGURE 4-1 STABILIZATION FLOWSHEET

The statistical experiments will be used to produce mathematical models to predict results and, if necessary, to design more comprehensive experimental matrices. The single variable matrices will be used to demonstrate the effects of changing the source of fly ash and the amount and type of adsorbents.

In the Group II experiments, site fly ash is substituted for a commercial fly ash. The substitution of site fly ash will allow the stabilization of contaminated material from two operable units at the same time.

Group III experiments are comparisons to Experiment 9 of Group I. The level and type of the adsorbents (attapulgite and clinoptilolite) are changed. This may affect the leachability of the heavy metals and radionuclides in the treated samples.

4.3.1.1 Preliminary Screening - Stage 1

To more efficiently use the material from the 1990-1991 sampling program, a preliminary study using material from the 1989 sampling effort will be tested first. These samples will be treated according to the Group I and II matrices in Table 4-3. The shear strength and waste from temperature rise will be measured within ten minutes after mixing waste and reagents. The penetrometer resistance will be measured using a Soiltest concrete penetrometer CT-421 on Days 0, 7, 14, and 28. The bulking factor will be measured on Day 28.

Approximately 50- to 100-gram samples will be used in these tests. The samples for the pocket penetrometer will be set in plastic containers with lids. Mixing will be by hand using a spatula until the mix has an even consistency without any lumps (two to five minutes).

4.3.1.2 Preliminary Screening - Stage 2

After completion of Stage 1 tests, composite samples from the 1990/1991 sampling effort (1989 samples for Silo 3) will be treated according to the uneliminated combinations in the stabilization matrix (Table 4-3). This set of tests will include Groups I through III of Table 4-3.

The shear strength and waste form temperature rise will be measured within ten minutes after mixing the waste and reagents. The penetrometer resistance will be measured using a Soiltest concrete penetrometer CT-421 on Days 0, 7, 14, and 28. The bulking factor will be measured on Day 28.

Approximately 100-gram samples will be used in these tests. The ranges listed in Table 4-3 may be narrowed depending on the results from the preliminary study with the 1989 samples.

The screening studies on the 3 composite samples will entail up to 39 experiments (3 composite samples x 13 runs). Insight gained from completed studies on one of the composite samples may allow the elimination of specific reagents and conditions from the treatment studies of other composite samples. In this case, the total number of experiments with the composite samples may be reduced. Also, the ranges of the reagents in the matrices may be changed as more is learned about the samples and when experiments are completed. It is expected that 20 to 30 percent of the samples (4 to 8 samples) will meet the 500 psi compressive strength requirement. The bulking factor will be measured on Day 28.

4.3.1.3 Preliminary Screening - Stage 3

If the initial screening tests provide sufficient data to define ideal conditions, then further testing with other reagent mixtures may not be necessary. The results may indicate that a reagent combination(s) is promising, but more data are required to evaluate its performance. If this is the case, additional tests will be designed to gather these data.

The shear strength and waste form temperature rise will be measured within ten minutes after mixing the waste and reagents. The penetrometer resistance will be measured using a Soiltest concrete penetrometer CT-421 on Days 0, 7, 14, and 28. The bulking factor will be measured on Day 28.

The number of experiments may range from 0 to 5 formulations per composite sample.

4.3.2 Advanced Screening - Silos 1 and 2

The most promising one to two formulations (those giving the most penetration resistance, the lowest bulking, and least reagents requirements) from the composite sample study will be tested on the top, middle, and bottom strata of the Silos 1 and 2 (six strata samples) to determine the effect of the variability of the samples' composition on the objective functions. Twenty percent of the samples will be set in duplicate. In these experiments, the UCS sample container will be used. The UCS will be determined with a Soiltest U-610 instrument. TCLP, 5-day static leach test, and permeability will be performed on the samples with a 28-day UCS greater than 500 psi. The bulking factor of the stabilized material with the appropriate UCS will be measured.

Bentonite will be added to Silos 1 and 2 as part of a removal action to act as a sealant to stop or reduce radon emissions from the silos. Therefore, the stabilization tests on the top stratum of both Silos and 1 and will use 20/80 weight percent bentonite/silo material as the feed instead of silo material only. A 10/90 weight percent bentonite/silo material will be used for tests on the middle stratum.

4.3.3 Advanced Screening - Silo 3

Composite samples will be used instead of individual strata samples. The most promising one or two formulations for Silo 3 will be repeated using the UCS sample container. Twenty percent of the samples will be set in duplicate. The UCS will be determined using a Soiltest U-610 instrument. TCLP, 5-day static leach test, and permeability will be performed on the samples with a 28-day UCS greater than 500 psi. The bulking factor of the stabilized material with the appropriate UCS will be measured.

4.3.4 Data Required

The following data will be recorded during cement stabilization preliminary and advanced screening:

- Compressive strength measured by a Soiltest concrete penetrometer CT-421 (for preliminary screening) or UCS (ASTM D2166) with a U-610 instrument
- Permeability (for advanced screening)
- MTCLP (for preliminary screening), or TCLP and 5-day static leach test (for advanced screening) on those mixtures with a compressive strength greater than 500 psi
- Bulking factor
- Waste form temperature rise after waste and reagents are mixed
- General descriptions of the waste before and after reagent addition
- Approximate shear strength measured within 10 minutes after waste and reagents are mixed
- Physical characteristics: percent moisture, bulk density
- The amount of water added to each waste form

4.4 METAL EXTRACTIONS

4.4.1 Leaching

The objective is to determine the effectiveness of various acid/EDTA leaching solutions in removing lead, uranium, thorium, and radium from the material in Silos 1 and 2. (The leaching treatability plan is graphically demonstrated in Figure 4-2.) The preliminary screening consists of up to three sets of tests: Stage 1, Stage 2, and Stage 3. In the Stage 1 and 2 tests, the leachates resulting from the application of the various acid and EDTA solutions to the samples will be analyzed for lead and uranium. Uranium and lead are selected as the target compounds in this study because they are

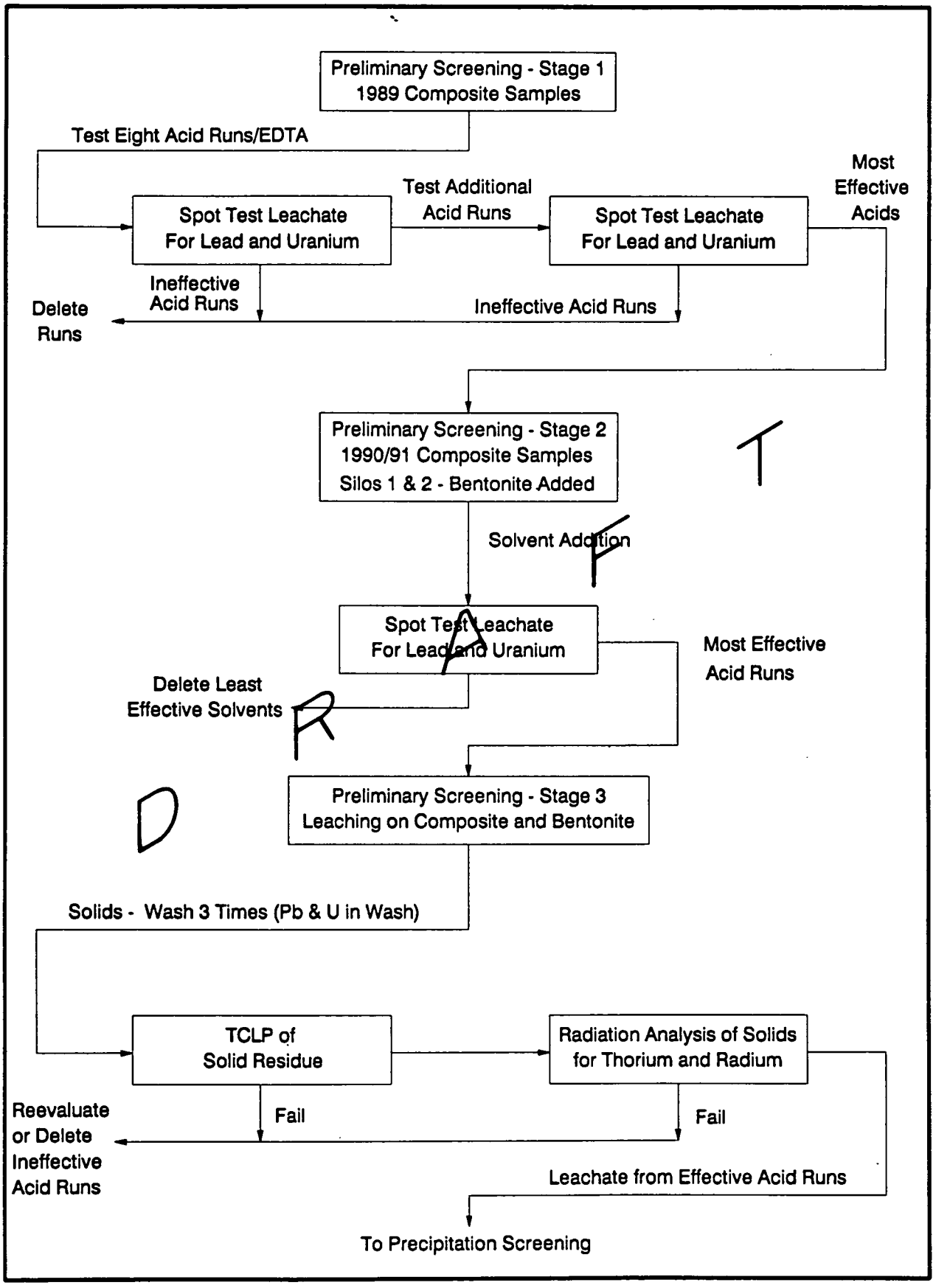

FIGURE 4-2 OVERALL LEACHING FLOWSHEET - SILOS 1 AND 2

present in greater concentrations than thorium or radium. The removal of thorium and radium will be demonstrated in the Stage 3. A typical detailed leaching screening plan is shown in Figure 4-3.

4.4.1.1 Preliminary Screening - Stage 1

As in Section 4.3, a preliminary study using composite samples from the 1989 sampling effort will be performed first. The leaching experiments are listed in Tables 4-4 and 4-5. The complete matrices of experiments will not be performed in these preliminary tests. That is, Run Nos. 1, 2, 6, 7, 12, 13, 14, and 18 in Table 4-4 will be conducted first. Hydrogen peroxide and ferric chloride may be added to improve the effectiveness and kinetics of the process. The appropriate omitted experiments from Table 4-4 may be conducted if the data indicate that they are warranted. Also, if the extraction procedures listed in Table 4-4 are effective, then the EDTA extraction procedures (Table 4-5) will be omitted. The extraction procedures are given in Appendix B.

4.4.1.2 Preliminary Screening - Stage 2

After completion of the Stage 1 tests, composite samples from the 1990-1991 sampling effort will be tested. Bentonite will be added to the samples (20 percent by weight) prior to testing. Run numbers from Tables 4-4 and 4-5 will be selected based on the results of the preliminary tests.

4.4.1.3 Preliminary Screening - Stage 3

The 5 to 10 treatments from the initial screening tests that yield leachates with the greatest concentrations of lead and uranium will be repeated on a larger scale (presumably 100 to 500 grams) in the Stage 3. Composite samples with bentonite added as in the initial screening will be used. The solid material will be washed three times with deionized water to remove the soluble compounds. The wash water will be analyzed for lead and uranium. The solid material from these latter experiments will be analyzed at the IT Analytical Services (ITAS)-Oak Ridge Laboratory. The analyses will include TCLP analysis to establish that the extracted materials are nonhazardous. In addition, thorium, radium, and uranium content will be determined by radiation analyses. The limits for thorium and radium are 5 pCi/g. Limits for uranium will be based on risk.

To evaluate Alternatives 8 and 9, the removal effectiveness of the leaching step is the most important step. The results will provide a rough guide by which the viability of remedial action Alternatives 8 and 9 can be preliminarily evaluated.

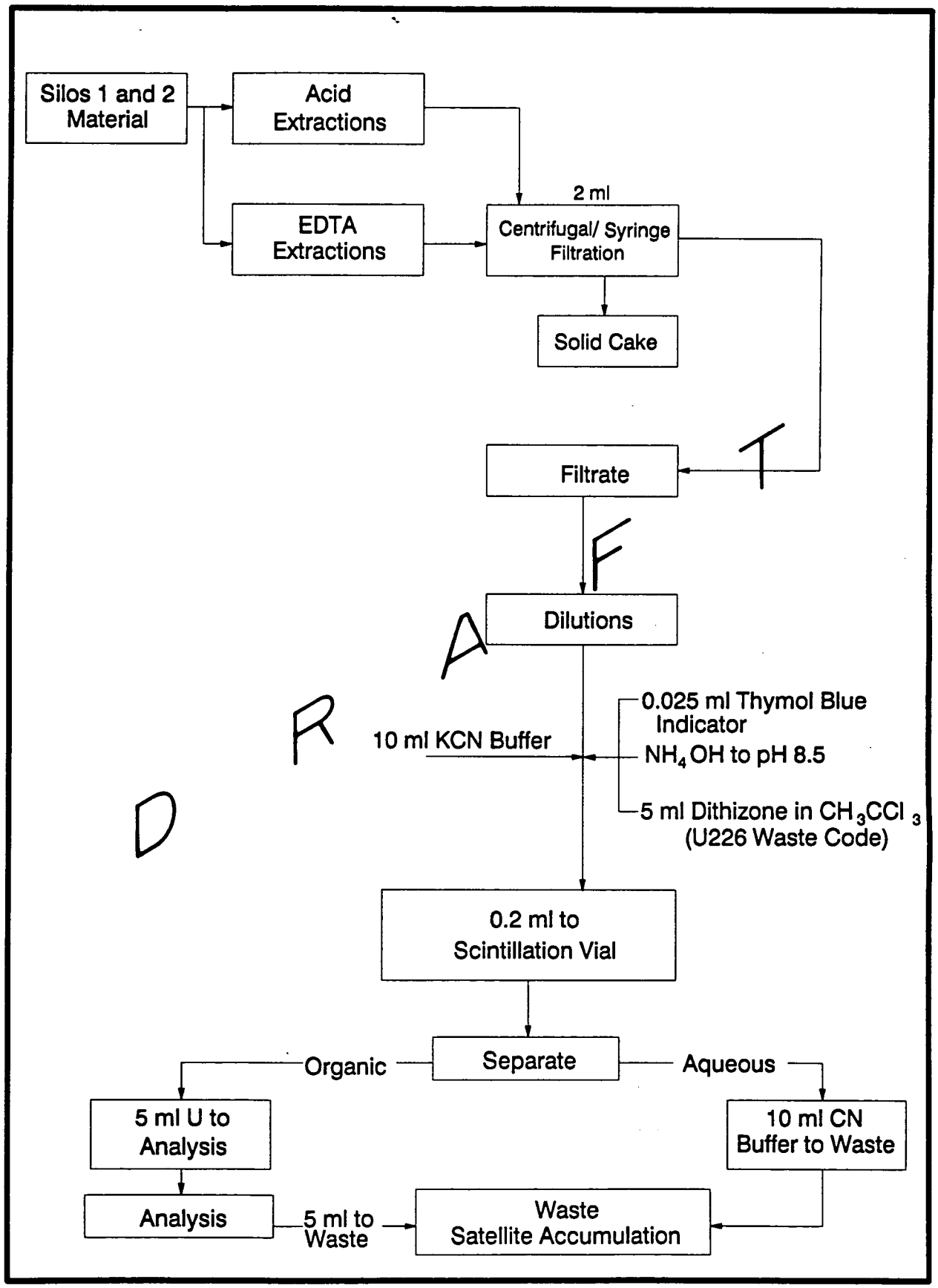

FIGURE 4-3 DETAILED LEACHING PRELIMINARY SCREENING TEST PLAN - TYPICAL FLOWSHEET

TABLE 4-4

MINERAL ACID EXTRACTIONS

Run No.	Acid Nominal Conc.	Dose (weight acid/ weight sample)		Temperature	
		2:1	4:1	Ambient	100°C
1	60% HNO_3[a]	X	X	X	
2	60% HNO_3	X	X		X
3	30% HNO_3	X	X	X	
4	30% HNO_3	X	X		X
5	15% HNO_3	X	X	X	
6	15% HNO_3	X	X		X
7	36% HCl[b]	X	X	X	
8	36% HCl	X	X		X
9	18% HCl	X	X	X	
10	18% HCl	X	X		X
11	9% HCl	X	X	X	
12	9% HCl	X	X		X
13	50% HOAc[c]	X	X	X	
14	50% HOAc	X	X		X
15	25% HOAc	X	X	X	
16	25% HOAc	X	X		X
17	12.5% HOAc	X	X	X	
18	12.5% HOAc	X	X		X

This test program will comprise 108 discrete samples (2 silos X 18 acids X 3 treatments).

[a]Nitric acid.
[b]Hydrochloric acid.
[c]Acetic acid.

TABLE 4-5

EDTA EXTRACTIONS

Run No.	EDTA Conc.	Dose weight acid/ weight sample)		Temperature	
		2:1	4:1	Ambient	100°C
19	0.2M	X	X	X	
20	0.2M	X	X		X

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4.4.2 Vitrification of Leachate

This screening will consist of one stage. Several experiments will be conducted to test the effects of sodium hydroxide, site fly ash, site soil, and locally available soil on the vitrification of the leachate. Table 4-6 is an example of a test matrix. The percentages given in the table are preliminary and will be adjusted, taking into consideration the types and quantities of precipitation reagents that were required. The mixture of reagents and waste mixture to be vitrified will have between 40 to 60 percent silica aluminum oxide combined, and 10 to 20 percent sodium oxide content when dried. Enough sodium hydroxide will be added to cause the mixture to melt in a 1250°C oven. Figure 4-4 presents a vitrification flow sheet.

4.4.3 Precipitation of Metals in the Leachate Solutions

4.4.3.1 Preliminary Screening - Stage 1

Precipitation reagents will be added to aliquots (3 to 5 cc) of the leachate solutions from Section 4.4.1.3. The reagents to be investigated are the sodium or potassium salt solutions of hydroxide, sulfide, sulfate, carbonate, phosphate, alum, ferric sulfate, and aqueous sodium silicate (Na_2O: SiO_2). Alum and ferric sulfate additions will be followed by the appropriate pH adjustments. Slurries of MgO and $Ca(OH)_2$ will also be tested. The solutions will be syringe-filtered through a 0.45-micron filter. The filtrate will be analyzed for uranium and lead as noted in Appendix B.

The reagents will also be added in a sequential order. A list of the tests using sequential addition is in Table 4-7. A flow sheet for precipitation of extracted metals is given Figure 4-5.

4.4.3.2 Preliminary Screening - Stage 2

Larger aliquots (50 to 100 cc) of the leachate solution will be tested with the most promising precipitation reagents from Section 4.4.3.1. Aliquots of these mixtures will be filtered or centrifuged. Solutions from the latter two operations will be tested for uranium and lead content.

Note, if three or more confirmation precipitation tests are necessary, then further composite waste samples (presumably 300 to 500 grams) will need to be extracted to finish the tests.

4.4.4 Stabilization of Precipitated Material

The best stabilization formulations, as determined for the silo material in Section 4.3, will be used in these experiments. Precipitated material generated in the conduct of Section 4.4.2 will be used. UCS will be tested with the pocket penetrometer (CT-421). Volume increase will be measured on Day 28 by water displacement. Samples with a UCS greater than 500 psi will be subjected to MTCLP. If

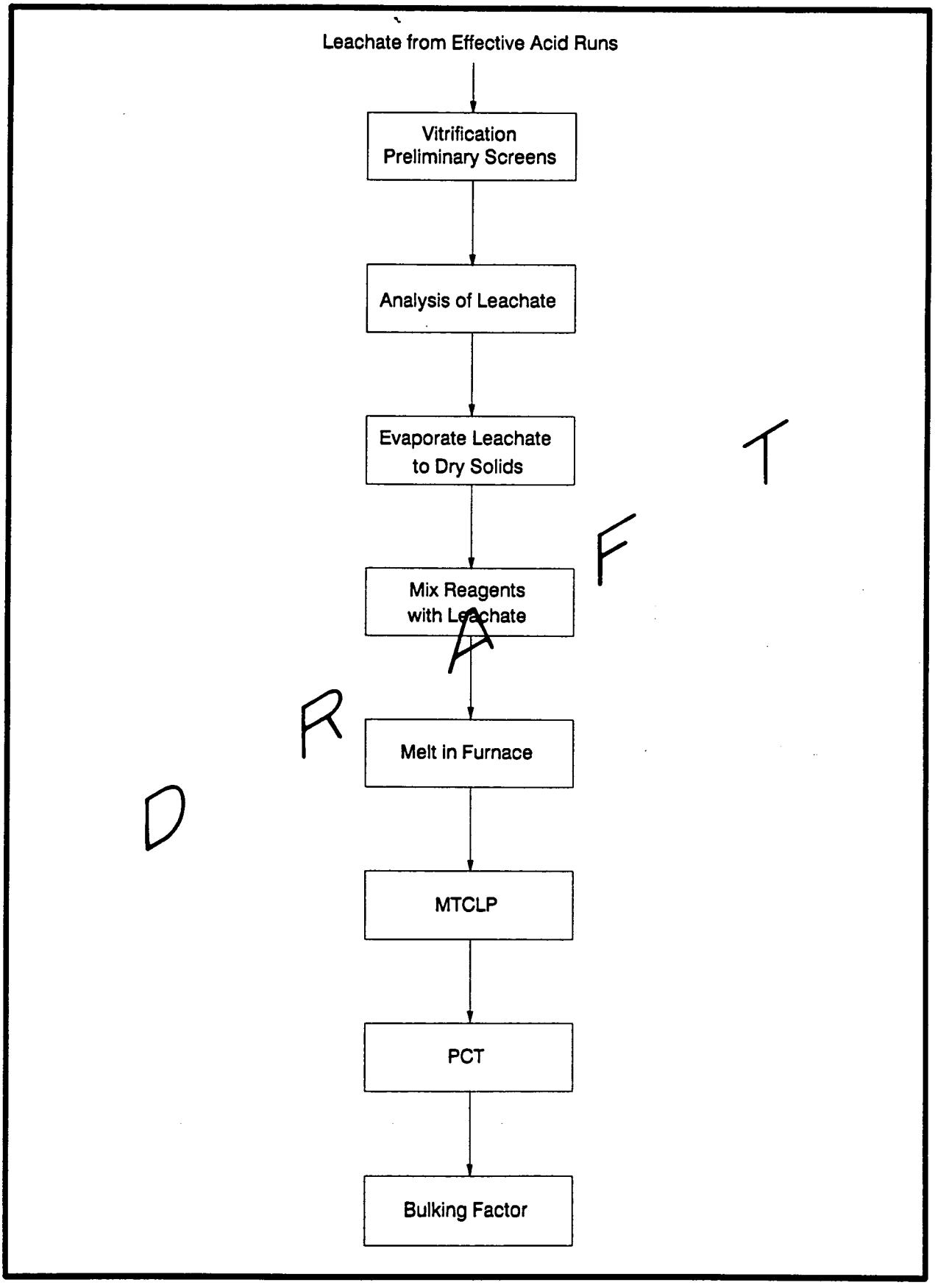

FIGURE 4-4 VITRIFICATION FLOWSHEET

TABLE 4-6. VITRIFICTAION EXPERIMENT MATRIX

Run Number	NaOH[a]	Site Fly Ash[a]	Site Soil[a]	Local Soil[a]
1	0	0	0	0
2	0	10	0	0
3	0	0	10	0
4	0	0	0	10
5	10	10	0	0
6	10	0	10	0
7	10	0	0	10
8	20	10	0	0

[a]Concentration as a percentage of final mixtrue on a dry basis.

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TABLE 4-7

PRECIPITATION OF LEACHATE SOLUTION

First Addition	Second Addition
$Na_2O:SiO_2$	NaOH Na_3PO_4 Na_2CO_3 Na_2S MgO $Ca(OH)_2$
MgO	Na_3PO_4 Na_2CO_3 Na_2S
NaOH	Na_3PO_4 Na_2CO_3 Na_2S
Na_3PO_4	NaOH MgO $Ca(OH)_2$

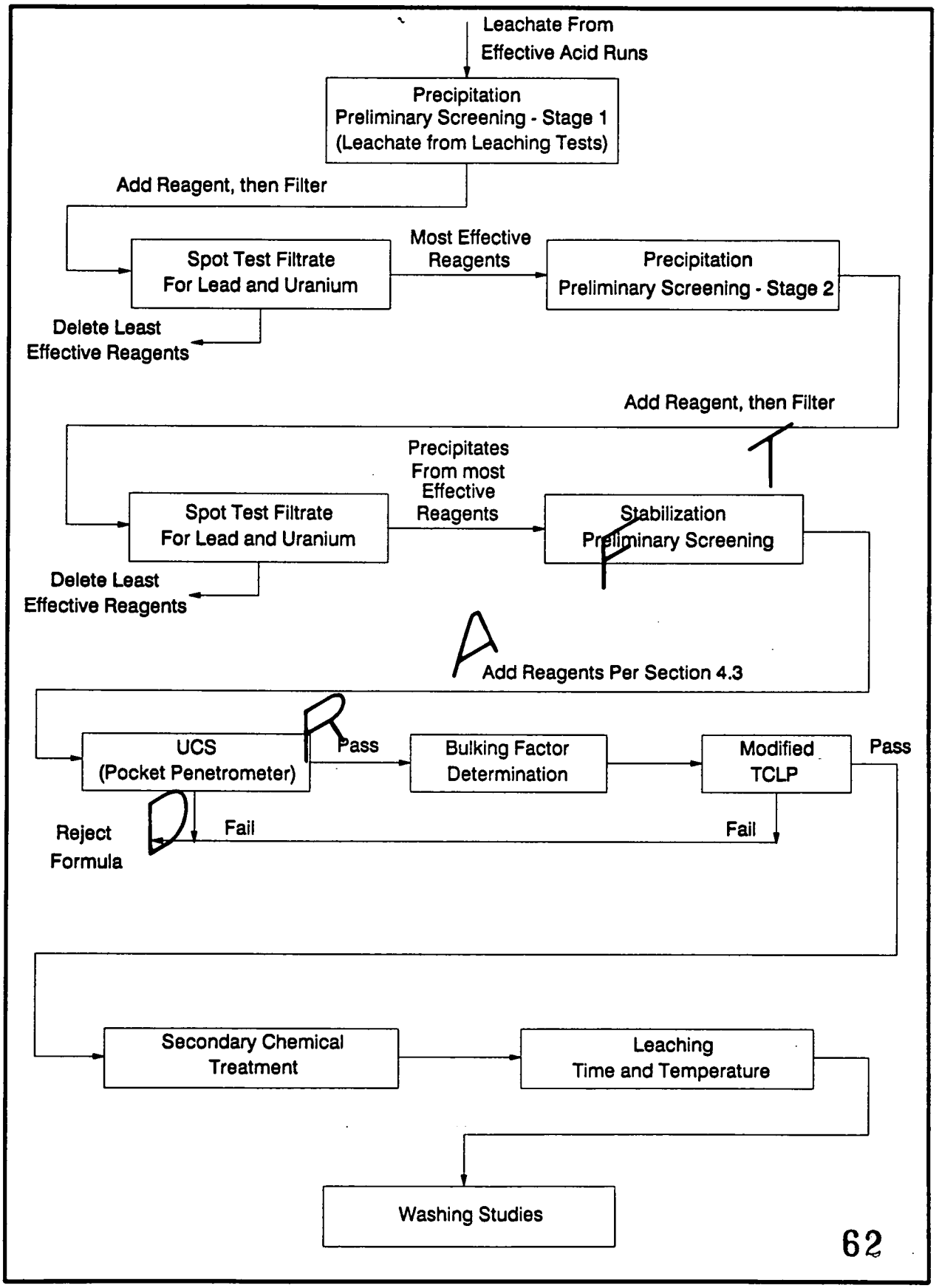

FIGURE 4-5 PRECIPITATION FLOWSHEET

necessary, more waste will be extracted to produce the leachate and metal precipitate for this process. Figure 4-5 shows how stabilization fits into the metals extraction studies.

4.4.5 Secondary Chemical Treatment

4.4.5.1 Settling - Polymer

If necessary, these tests will be conducted. Jar tests will be run using two cationic and two anionic polymers. The polymers will be those recommended by polymer manufacturers active in the field. Four different polymer doses will be tested. The dosages will be 50, 100, 150, and 200 ppm. The coagulant ferric sulfate and Nalmet 8154 will also be tested at two levels, 1000 and 2000 ppm, with the more effective polymer dose and alone, with no polymer at all. Once a polymer and polymer dosage are selected, a settling test will be run. At this point, a supernatant sample will be removed and filtered through a 0.45-micron filter. The filtrate will be tested for uranium and lead as before. The solutions will be analyzed to determine if the process successfully lowered the metal content.

4.4.5.2 Settling - Filter Aid

If necessary, these tests will be conducted. The feed solids concentration will be adjusted to pumpable solids concentration and the body feed concentrations to three different dosages of filter aid. Filter aid concentrations will be those recommended by the manufacturer. The treated samples will be filtered in a buchner funnel. The optimum dose of reagents will be that producing the driest cake and the most filtrate in the shortest time. The filtrate will be analyzed to determine if the process successfully lowered the metal content.

4.4.6 Leaching Time and Temperature

Using the most promising formulations from Section 4.4.1.3, a statistical experiment of leaching time and temperature will be conducted (Table 4-8). Experiment numbers 4 and 6 will be conducted first. From their results, the ranges for the variable "time" in Table 4-8 may be modified. Ten- to twenty-gram composite samples with 20 percent bentonite will be used in these experiments. A mathematical model will be derived from these experiments. An experiment at optimum conditions predicted from the mathematical model will be completed.

4.4.7 Washing Studies

Washing studies of the leached solid will be executed using washing data from Section 4.4.1 as a guide. Fifty grams of sample will be extracted for these tests. The filter cake will be washed 10 times with deionized water in a buchner funnel. The volume of each wash will be half the volume of the leachate solution. The uranium and lead content in each wash liquor will be tested as before.

TABLE 4-8

LEACHING TIME AND TEMPERATURE MATRIX

Experiment No.	Temperature (°C)	Time (hr)
1	25	1
2	25	7
3	100	1
4	100	7
5	62.5	4
6	100	24

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4.4.8 Data Required

The following data will be recorded during the leachant screening:

- Acid (solvent) and concentration
- Quantity of acid
- Quantity of waste
- Description of uranium and lend analyses results
- Percent bentonite in waste
- TCLP of insoluble residue (Stage 3 screening)

The following data will be recorded during the precipitation screening:

- Quantity and type of solvent used to produce leachate
- Precipitation reagents and quantities
- Lead and uranium is filtrate

The following data will be recorded during cement stabilization of precipitated material:

- Compressive strength measured by a Soiltest concrete penetrometer CT-421
- MTCLP
- Bulking factor
- Waste form temperature rise after waste and reagents are mixed
- General descriptions of the waste before and after reagent addition
- Approximate shear strength measured within 10 minutes after waste and reagents are mixed
- Physical characteristics: percent moisture and bulk density
- The amount of water added to each waste form

The following data will be recorded during the vitrification screening:

- MTCLP
- PCT
- Weights of reagents and waste in final waste form
- Temperature of oven
- Time heating sample
- Bulking factor
- General description of the waste before and after melting

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- Physical characteristics: percent moisture, bulk density

The following data will be recorded during the secondary chemical treatment tests:

- Leachate being tested
- Polymers, coagulants, Nalmet 8154, and filter aid added, and their dosages
- Lead and uranium before and after addition of any polymers, coagulants, and filter aid

The following data will be recorded during the leaching time and temperature tests:

- Solvents being tested
- Quantity of waste and solvent being tested
- Lead and uranium in the leachate as a function of time

The following data will be recorded during the washing studies tests:

- Type of solvent used for leaching
- Quantity of leached solid being rinsed
- Quantity of water used for each rinse
- Uranium and lead in each batch of rinse water

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5.0 EQUIPMENT AND MATERIALS

See Table 5-1 for a listing of the major equipment to be used during the laboratory screening.

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TABLE 5-1

EQUIPMENT AND MATERIALS

Item	Description
Multiple	Plastic containers, 5 oz and 8 oz
Multiple	Spatulas
Multiple	Crucibles
1	Cooler box reagent chemicals
1	HACH digital pH meter
1	Glass melter furnace
2	HACH COD digesters Model 45600-00 and associated vial
1	Tek-Mar Laboratory vibrating shaker
1	Thermometer, calibrated and traceable
1	Scale, calibrated
1	Aluminum heating block
Multiple	2 x 4 Jatco Co. plastic molds for UCS
1	Centrifuge
Multiple	50 cc centrifuge tubes
1	Hobart quart ASTM planetary mixer
1	alpha survey meter and beta, gamma scanner
1	Soiltest Torvane
50	TFE bombs

6.0 SAMPLING AND ANALYSIS

The sampling and analysis plan for the samples used for this laboratory screening is contained in "Implementation Plan for the K-65 and Metal Oxide Residue Sampling Project at the Feed Materials Production Center, Fernald, Ohio," Addendum-SAP, October 10, 1990.

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7.0 DATA MANAGEMENT

Two types of laboratory notebooks will be used for this project. All laboratory notebooks are uniquely numbered and permanently bound with sequentially numbered pages.

Project-specific notebooks will be signed out by the facility quality control coordinator (QCC) to the individuals working on the project. All daily laboratory activities associated with the project will be recorded in the project-specific notebooks. Refer to the SOP in Appendix G.

Separate nonproject-specific logbooks will be used to record the injection or introduction of samples into analytical instrumentation. These logbooks are also used to record maintenance or problems with the instrument. Refer to the SOP in Appendix H.

At the completion of the project, the project-specific laboratory notebooks and logbooks will be returned to the facility QCC for retention. Instrument logbooks are returned to the facility QCC when the books are filled.

All records management and reporting will follow standard, QA/QC protocol in the Quality Assurance Project Plan (QAPP) and Volume 4 of the RI/FS Work Plan. Standard QA/QC protocol, as it applies to testing within the laboratory, will adhere to the following guidelines:

- One hundred percent verification on all numerical results - all raw data entries, transcriptions, and calculations are checked and recalculated.
- Data validation through test reasonableness - summaries of all test results for individual reports are reviewed to determine the overall reasonableness of data and to determine the presence of any data that may be considered outliers.
- Routine instrument calibration - will be performed under guidance from the QAPP.
- Use of trained personnel conducting tests - all technicians are trained in the application of standard laboratory procedures for analyses as well as the QA measures implemented for internal QC checks.

8.0 DATA ANALYSIS AND INTERPRETATION

8.1 EFFECTIVENESS OF WASTE FORMS

The results of the leaching tests (MTCLP, TCLP, PCT, and 5-D static) will be used to evaluate the long-term effectiveness of each waste form. The concentrations of radioactive and hazardous constituents in the leachate will be used as input into the geochemical models described in the RI/FS Work Plan Draft Addendum on risk assessment methodology. These models will be used with groundwater fate and transport models will then be used to calculate concentrations of contaminants in the aquifer at the reasonable maximum exposure. These concentrations will in turn be used to calculate the magnitude of that exposure, and the resulting risks to human health and the environment.

8.2 STABILIZATION

The reagent formulation along with the following data will be presented in tabular form for all samples:

- Waste form temperature rise after waste and reagents are mixed
- General descriptions of the waste before and after reagent addition
- Approximate shear strength measured within 10 minutes after waste and reagents are mixed
- Physical characteristics: percent moisture, bulk density
- The amount of water added to each waste form
- Compressive strength measured by a Soil test concrete penetrometer CT-421 (for preliminary screening) or UCS (ASTM D2166) with a U-610 instrument

The following data, also in tabular form, will be presented for samples having a pocket penetrometer on UCS greater than 500 psi:

- Permeability (for advanced screening)
- MTCLP (for preliminary screening), or TCLP and 5-day static leach test (for advanced screening) on those mixtures with a UCS than 500 psi
- Bulking factor

8.3 LEACHING/PRECIPITATION/STABILIZATION/VITRIFICATION

8.3.1 Leaching

The following data will be evaluated and presented in tabular form for all Stage 1 tests:

- Acid (solvent) and concentration
- Quantity of acid
- Quantity of waste
- Description of uranium and lead analyses results

The data recorded for Stage 2 shall be the same parameters as for Stage 1, except that Stage 2 shall also include 20 percent bentonite.

Stage 3 data shall be presented as in Stage 2, with the addition of the following parameters for each test run:

- TCLP of insoluble residue (Stage 3 screening)
- Uranium, thorium, and radium content of insoluble residue

8.3.2 Precipitation

The following data will be presented in tabular form for each experimental run:

- Quantity and type of solvent used to produce leachate
- Precipitation reagents and quantities
- Lead and uranium in filtrate

8.3.3 Stabilization

The following data will be tabulated for each stabilization test of precipitated material:

- Unconfined compressive strength measured by a Soiltest concrete penetrometer CT-421 (for preliminary screening) or UCS (ASTM D2166) with a U-610 instrument
- MTCLP (for preliminary screening), or TCLP and static 5-day leach test (for advanced screening) on those mixtures with a compressive strength greater than 500 psi
- Bulking factor

- Waste form temperature rise after waste and reagents are mixed
- General descriptions of the waste before and after reagent addition
- Approximate shear strength measured within 10 minutes after waste and reagents are mixed
- Physical characteristics: percent moisture, bulk density
- The amount of water added to each waste form

8.3.4 Vitrification

The following data will be tabulated for the vitrification screening:

- MTCLP
- PCT
- Weights of reagents and waste in final waste form
- Temperature of oven
- Heating time of sample
- Bulking factor
- General description of the waste before and after melting
- Physical characteristics: percent moisture, bulk density

8.3.5 Secondary Chemical Treatment

The following data from the secondary chemical treatment tests will be tabulated:

- Leachate being tested
- Polymers, coagulants, Nalmet 8154, and filter aid added, and their dosages
- Lead and uranium before and after addition of any polymers, coagulants, and filter aid

8.3.6 Leaching Time and Temperature

The following data will be presented in tabular form:

- Solvents being tested
- Quantity of waste and solvent being tested
- Lead and uranium in the leachate as a function of time

8.3.7 Number of Washes

The following data will be tabulated for each leached solid being tested:

- Type of solvent used for leaching
- Quantity of leached solid being rinsed
- Quantity of water used for each rinse
- Uranium and lead in each batch of rinse water

8.4 PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

The following are procedures used to assess data precision, accuracy, and completeness:

Calculations of precision, accuracy, and completeness will be used to assess data quality.

Example calculations of precision:

$$RFP = \frac{(C_1 - C_2) \times 100\%}{(C_1 + C_2)/2}$$

where

RPD = relative percent difference
C_1 = larger of the two observed values
C_2 = smaller of the two observed values

Example calculation of accuracy:

$$\%R = \frac{100\% \times (S - U)}{C_{sa}}$$

where

%R = percent recovery
S = measured concentration in spiked aliquot
U = measured concentration in unspiked aliquot
C_{sa} = actual concentration of spike added

Example of calculation of completeness:

$$\%C = 100\% \times \frac{V}{n}$$

where

%C = percent completeness

V = number of measurements judged valid

n = total number of measurements necessary to achieve a specified statistical level of confidence in decision making

An example of the TDL form used for reporting precision of duplicates and accuracy of spikes is given in Figure 8-1.

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Figure 8-1
General QA/QC Report

Analyte:
Matrix:
Sample Number:

	Conc. ( )
Precision of Duplicates Spike Value (b)= Spike Dup. Value (a)=	
Precision (RPD*)	$\frac{\lvert a-b \rvert}{(a+b)/2} \times 100\% =$
Accuracy of Spike Original Value (a)= Observed Spike Value (b)= Spike Level (c)=	
Accuracy=	$\frac{b-a}{c} \times 100\% =$
Accuracy of Spike Dup. Original Value (a)= Observed Spike Dup. Value (b)= Spike Level (c) =	
Accuracy =	$\frac{b-a}{c} \times 100\% =$

9.0 HEALTH AND SAFETY

An alpha-CAM detector will be used to measure radon emissions continuously during testing. The primary purpose of alpha-CAM is for the health and safety of the lab personnel.

The radon emissions will be minimal in the treatability study. This is based on the following assumptions:

- Radon and radium are in secular equilibrium in the contained sample.
- The radium concentration is 192,600 pCi/gm (Operable Unit 4 Remedial Investigation Report).
- Upon opening the sample container, all of the enclosed radon will escape immediately and be captured by the hood.
- After the initial radon cloud is emitted, the contained radium will continue to decay into radon, which will escape immediately and be captured by the hood.
- The initial sample weighs five pounds.

The worst-case calculations indicate that the instantaneous release of radon upon opening the container will be approximately 0.4 mCi, and the radon rate from a single opened sample container will be less than 3.6 µCi/hr. Samples will be handled inside the hood. The hood will use carbon adsorbers and high-efficiency particulate air (HEPA) filtration (in series), which is considered the best available technology to control emissions.

See Appendix A for the site-specific health and safety plan.

10.0 RESIDUALS MANAGEMENT

10.1 STABILIZATION OF SILOS 1 AND 2 AND SILO 3 MATERIALS

The project will generate from 24 to 37 kg of treated solid waste.

10.2 LEACHING/ANALYSIS/DISPOSAL OF SILOS 1 AND AND SILO 3 MATERIALS

The project will generate four basic waste streams. Stream 1 will consist of approximately 2000 to 6600 grams of radioactive waste residue (Silos 1 and 2 material) resulting from the acid/EDTA leaching process. These residues will be sent to IT's Oak Ridge Laboratory for analysis and then will be shipped to WMCO for disposal.

Waste Stream 2 will be the residual leachate, approximately 15 to 30 liters of high lead RCRA waste. This waste will be stored in five-gallon carboy containers in a secondary containment system. Waste Stream 3 will be approximately two to four liters of aqueous cyanide waste generated from the addition of a potassium cyanide (KCN) buffer to the leachate for the lead analysis. The final waste stream, Waste Stream 4, will be approximately one to two liters of uranium/RCRA waste generated from the lead analysis.

10.3 STABILIZATION/VITRIFICATION OF LEACHED WASTE

The total amount of residue will depend on the metal concentration in the waste. Potentially, 10 to 20 kg of solid waste will need to be leached to produce enough leachate for the analysis. This would produce about 3.5 to 7 kg of treated solid waste, 30 to 60 kg of treated leachate, and 30 to 60 kg of treated wash water.

10.4 DISPOSAL

All of the waste materials will be disposed of by WMCO.

11.0 REPORTS

11.1 PRELIMINARY SCREENING

An interim draft report, which will document the results of the stabilization and leaching tests, will be issued following the completion of the preliminary screening. This report will identify the promising stabilization formulation and extraction solutions and will recommend whether those procedures be further tested in the advanced treatability program. To determine the success of the recommended stabilization formulations and extraction solutions, it will be necessary to have the residues and leachates analyzed for radium and thorium at IT's Oak Ridge Laboratory. When the results of these analyses are available, they will be incorporated in the interim draft report, and a final report on the laboratory screening will be issued.

11.2 ADVANCED SCREENING

An interim draft report will be issued following the completion of the experimental portion of the advanced tests that will document the results. This report will identify the stabilization formulations and extraction procedures that are promising and that identify any problems. To determine the success of the recommended stabilization formulations and extraction solutions in removing contaminants, it will be necessary to have the residues analyzed at IT's Oak Ridge Laboratory. When the results of these analyses are available, they will be in the interim draft report and a final report will be issued.

12.0 COMMUNITY RELATIONS

Treatability Studies and community information and involvement activities are required in the CERCLA process. Community Relations activities shall be conducted; 1) to support treatability studies for Operable Unit 4 to explain the role of treatability studies in the RI/FS and 2) to raise the public's confidence in cleanup alternatives and technologies identified in the alternatives screening/ analysis process and in the preferred alternative for this operable unit. The Treatability Study Community Relations activities for Operable Unit 4 will comply with the Community Relations Plan (CRP) -- Remedial Investigation/Feasibility Study and Removal Actions at the U.S. Department of Energy Feed Materials Production Center, Fernald, Ohio, August 1990. At a minimum, the following Community Relations activities will be conducted to explain treatability studies for Operable Unit 4.

- Community Meeting - Held a minimum of three times/year to provide status on cleanup issues, and to ensure that interested area resident have a routine public forum for receiving new information, expressing their views, and getting answers to their questions. The meetings shall focus on operable unit updates, removal actions, major RI/FS documents, and other appropriate topics. During the July 1991 community meeting, an initial discussion of treatability was held to make the community aware of treatability studies underway.

- Publications - RI/FS materials such as progress reports, fact sheets and a community newsletter, Fernald Site Cleanup Report, provide updates of CERCLA-related activities at the FMPC and will include information on treatability study activities for this operable unit.

- Presentations to Community Groups - Information about treatability studies for this operable unit shall be included in briefings to community groups in Ross, Crosby, a Morgan townships, and to Fernald Residents for Environment Safety and Health, as appropriate. Also, this information shall be included in presentations to other organizations, as requested.

Key milestones in treatability studies will be identified and progress reported to the community in these presentations and publications. These milestones include:

- Submittal of work plans to DOE and EPA
- EPA Approval of work plans
- Treatability testing
- Treatability testing report submittal

Other activities identified in Section 4.0 of the CRP may be utilized as appropriate to effectively communicated treatability information to the community. Such activities may include workshops and community roundtables.

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13.0 MANAGEMENT AND STAFFING

The project organization for this laboratory screening is shown in Figure 13-1

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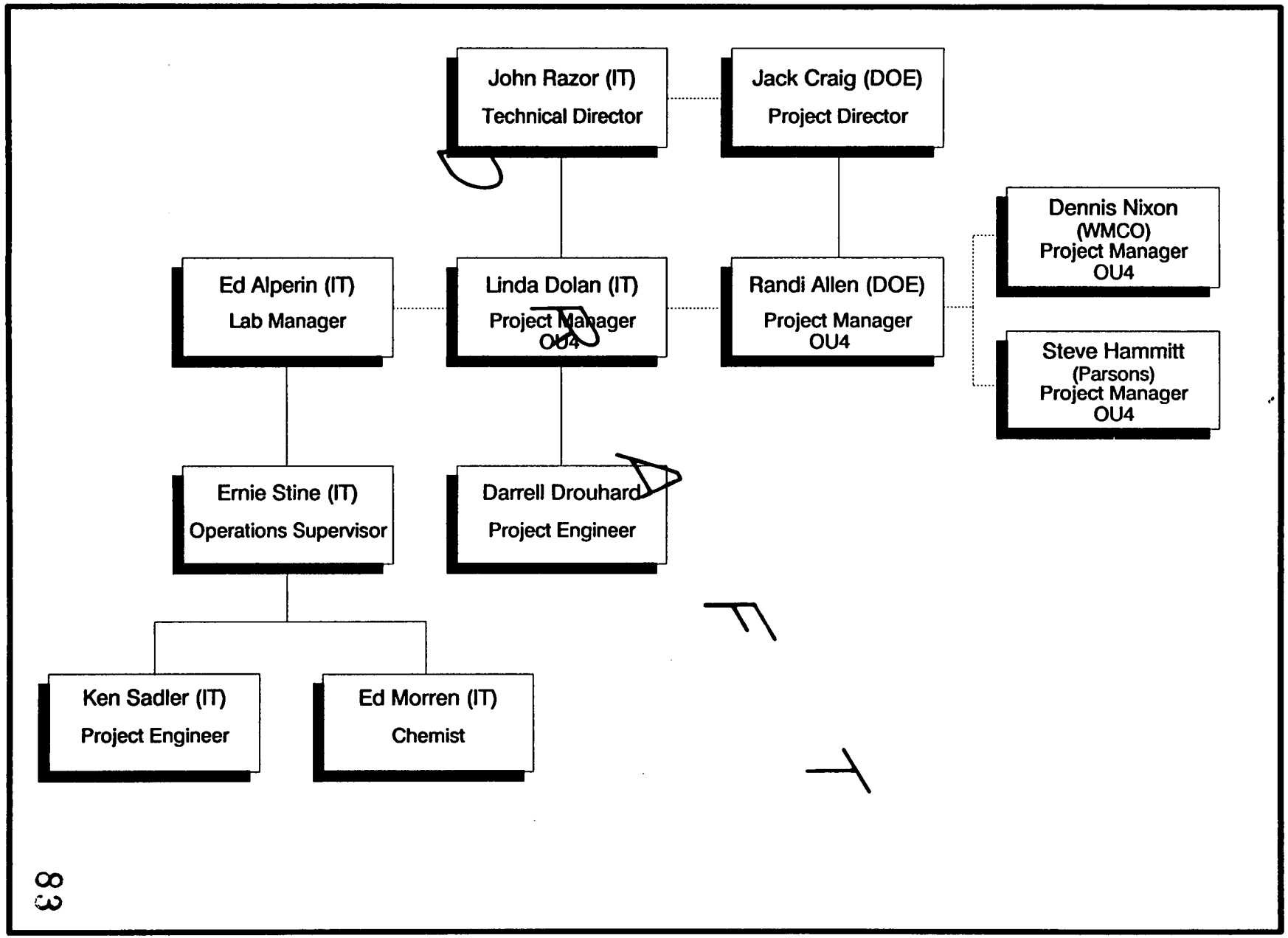

FL/FMPC-0405-2/13-1

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FIGURE 13.1 MANAGEMENT AND STAFFING

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APPENDIX A

SITE-SPECIFIC SAFETY PLAN FOR THE FEED MATERIAL PRODUCTION CENTER SILOS 1 AND 2 TREATABILITY PROGRAM

HEALTH AND SAFETY PLAN

FOR THE

FEED MATERIALS PRODUCTION CENTER

SILOS 1, 2, AND 3 TREATABILITY PROGRAM

PROJECT NUMBER 303317.24.05.20

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1.0 TASKS TO BE PERFORMED

Previously collected samples of the K-65 silo contents will be prepared and analyzed in search of effective treatment methods. All preparations and analyses will be performed in a high-efficiency particulate air (HEPA) filtered hood located in an environmental containment cubicle. The cubicle will be located in the mixed waste testing area in the IT Environmental Technology Development Center.

Job tasks are summarized below. For detailed information, please consult the work plan.

I. Preliminary Characterization

Ia. Samples of both silos will be composited in the hood. This will involve handling the total volume of samples, approximately 100 kg.

Ib. Samples will be weighed, blended, and aliquotted in the hood for subsequent baseline analyses by the IT Oak Ridge laboratory.

II. Metal Extractions

IIa. Acid Extractions - One gram aliquots of each composite will be weighed and placed in HACH digester vials. Room temperature and 100 degree centigrade tests with acid will be run for two hours. Acids used for the extractions will be: nitric (60 to 15 percent), hydrochloric (36 to 9 percent), acetic (50 to 12.5 percent), and hydrochloric/ hydrofluoric acid (HCl/HF) mixture (HCl 36 to 9 percent, HF 5 - 1.25 percent).

Liquids will be diluted 1/1,000 and analyzed for lead content. Reagents involved include potassium cyanide and 1,1,1-trichloroethane. The chemical oxygen demand (COD) vials have been preloaded with potassium cyanide so that the maximum quantity handled at any one time will be five milliliters.

IIb. EDTA Extractions - Extractions will be performed with 0.2 molar ethylenediamine-tetraacetic acid (EDTA).

III. Leaching Kinetics - An optimum treatment will be selected and rerun on five fresh samples.

IV. Solid/Liquid Separations - The optimally treated sample from III will be filtered and analyzed for lead and uranium by the laboratory following the Contract Laboratory Program (CLP) protocol.

V. Leaching Temperature - Three samples from each silo will be treated with the optimum leachate for the optimum time. Three temperatures will be used: optimum temperature, 10 degrees centigrade above optimum, and 20 degrees centigrade above optimum.

VI. Solids Washing

VIa. Filtration - An optimally treated sample will be filtered, and the filter cake will be washed with deionized water. The washes will subsequently be analyzed for lead and uranium.

VIB. Centrifugation - An optimally treated sample will be centrifuged, shaken with deionized water, and recentrifuged.

VII. Precipitation of Leached Materials

VIIa. Carbonate Precipitation - The pH of the optimally treated sample will be adjusted by the addition of sodium carbonate to 9, 10, and 11, and the filtered liquid will be analyzed for lead and uranium. Further analysis will be conducted in the laboratory if separation appears promising.

VIIb. Sulfide Precipitation - If carbonate separation (VIIa) is unacceptable, sodium sulfide will be added to the pH 9 sample and the same separation/analysis will be carried out.

VIII. Solidification/Stabilization of Leached Material - A sample of the optimally leached residue will be subjected to a modified Toxicity Characteristic Leaching Procedure (TCLP) extraction to determine its status with reference to U.S. Environmental Protection Agency (EPA) regulations.

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2.0 K-65 BACKGROUND INFORMATION

The K-65 silos contain waste from the World War II program that produced the first atomic bombs. For this work, a uranium-rich ore called pitchblende was imported from the Belgian Congo. Pitchblende was treated with nitric acid to dissolve the uranium away from the ore. The remaining residues were mixed with water and pumped into the silos, where the solids settled. The liquids at the surface were pumped back out of the silos into a treatment facility. What remains in the silos now is about 9,700 tons of residual solids. The residues in the silos emit radiation. The radioactivity levels of the residues are higher than ordinary tailings from uranium mining and milling. Like other uranium ore tailings, these residues produce radon gas, but in considerably larger quantities.

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3.0 TASK-SPECIFIC HAZARD ASSESSMENT

The following hazard assessment is based on historical information and defined task activities. The treatability team routinely reassesses the hazards before starting work to ensure that conditions have not changed. All newly identified hazards will be addressed with the health and safety engineer to determine the degree of hazard and if any changes to the safety plan are needed.

3.1 PHYSICAL HAZARDS

- Radiological hazards
- Uranium-238 (U-238) and daughters
- Uranium-235 (U-235) and daughters
- Radium-226 (Ra-226) and daughters

Contaminant	Derived Air Concentration	Action Limit .25DAC
Thorium-230	3 X 10^{-12} uCi/mL	7.5 X 10^{-13} uCi/mL
Radium-226	3 X 10^{-11} uCi/mL	7.5 X 10^{-12} uCi/mL
Uranium-238	2 X 10^{-11} uCi/mL	5 X 10^{-12} uCi/mL
Radon Daughters (Polonium-218, Lead-214, Bismuth-214, Polonium-214)	0.3 working level	0.075 working level
Radon-222	60 pCi/L (50 percent equilibrium)	15 pCi/L
Uranium-235 (trace levels of actinium series)	2 X 10^{-11} uCi/mL	5 X 10^{-12} uCi/mL
Uranium-234	2 X 10^{-11} uCi/mL	5 X 10^{-12} uCi/mL

3.2 CHEMICAL HAZARDS

The following chemicals will be present, either in the samples or in the reagents and will pose potential hazards. Other materials, such as fly ash, EDTA, sodium carbonate, sodium sulfide, lime, and cement/sodium silicate will be present but will pose no significant hazard due to their relatively low toxicity and small quantities.

	PEL[a]	
Chemical	TWA[b]	STEL[c]
Sample		
Lead	0.05 mg/m^3	
Reagents		
Acetic acid	10 ppm	
Hydrochloric acid	5 ppm C[d]	
Nitric acid	2 ppm	4 ppm
1,1,1-trichloroethane	350 ppm	450 ppm
Potassium cyanide	5 mg/m^3 (skin)	

[a]PEL - Permissible exposure limit, or maximum airborne exposure allowed by the Occupational Safety and Health Administration (OSHA). Types of PELs include TWAs, STELs, and ceilings.
[b]TWA - Time-weighted average, or average exposure allowed over an 8-hour shift.
[c]STEL - Short-term exposure limit, or maximum average exposure during a 15-minute period.
[d]C - Ceiling, or maximum exposure allowed, even instaneously.

3.3 POTENTIAL ROUTES OF EXPOSURE AND HAZARD ASSESSMENT

The identified site contaminants are either solid or gaseous in nature, and the majority of the reagents to be used are liquids. The routes of entry into the body are inhalation, absorption, and ingestion (in order of importance). Radioisotopes in the sample pose an external and internal exposure hazard. The internal hazard is largely eliminated by the procedures and engineered controls to be utilized. The external hazard will be controlled through monitoring. Direct contact with the corrosives may result in destruction of skin tissue and absorption of other contaminants if in solution. The inorganic lead in the samples poses a potential inhalation hazard, which is minimized by the task procedures. Cyanide-containing reagent poses a potential for the release of hydrogen cyanide (HCN) gas, but the limited quantities per container (less than 5 mL) and the task procedures will prevent any significant hazard unless a spill occurs.

To minimize the potential exposure hazards, nearly all of the operations to be carried out during this project will be performed inside the hood, which is located inside an environmental containment cubicle. This includes acid digestions, sample preparation, pouring reagents, and packaging for disposal. The only operations planned to be performed outside the hood are transport of the silo samples to and from the hood, transport of reagents to the hood, and colorimetric determination of sample results. All container opening will be done inside the hood. Reagents have been prepared and packaged off site to further minimize on-site handling.

There is also a potential that acidic reagents and the potassium cyanide (KCN) reagents might be mixed in a spill. This would liberate HCN gas, which has an OSHA PEL (STEL) of 5 mg/m^3. The treatability team will evacuate if a major spill occurs but will remain to control minor spills. A minor spill is a spill inside the hood of 50 mL or less. This is equivalent to one vial of acid and one vial of KCN. Each KCN vial contains 10 mL of 0.1 percent w/w KCN in water. Therefore, the total CN per vial is:

10,000 mg liquid X 0.001 mg KCN X 26 mg CN/65 mg KCN = 4 mg CN

This quantity of CN mixed with acid would liberate HCN in the following quantity:

4 mg CN X 27 mg HCN/26 mg CN = 4.15 mg HCN

This amount of HCN could be dispersed into one cubic meter of air without exceeding the OSHA PEL.

The use of the hood greatly minimizes any potential for chemical exposure from the silo samples or from the reagents. A potential for some radiation exposure exists and monitoring will be conducted to quantify this exposure and ensure that the procedures in use are appropriate.

4.0 MONITORING

4.1 GOALS

Air monitoring will be performed to ensure that contaminant concentrations in the breathing zone do not exceed the concentrations specified by established exposure levels.

Exposures to chemicals should be kept as low as possible because there are insufficient data to predict the combined effects of most chemical mixtures.

4.2 EXTERNAL RADIATION HAZARD MONITORING

A health physics technician will monitor all locations before start of work and will frequently monitor exposures in all areas that exceed the one millirem (mrem)/hour action limit. Measures such as increasing shielding, increasing distance, or reducing exposure time will be taken to minimize exposures. Radiation monitoring instruments include:

- Ludlum Model 177, or equivalent, with a G-M pancake probe
- Ludlum Model 3, or equivalent, with a ZnS alpha scintillation probe
- Eberline Model Alpha-5A alpha air monitor.

4.3 ACTION LIMITS

The following table provides types, scheduling, and actions for monitoring.

Instrument/chem.	Need	Interval	Limit	Action
Alpha probe	Y	Pre-job and intermittent	20 cpm[a]	HP Review[b]
Beta/gamma probe	Y	Pre-job and intermittent	500 cpm[a]	HP Review
External radiation	Y	Pre-job	>1 mrem/hour	HP review
Continuous air monitor (CAM)	Y	Continuous	>0.075 or >7.5 working level	APR Withdraw
Thermoluminescent dosimetry (TLD) badge	Y	Continuous	NA, no real time results	
TLD ring	Y	Continuous	NA, no real time results	

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[a]Above background.
[b]Full-face air-purifying respirators (APRs) with organic vapor, acid gas, and fume cartridges. Disposable protective clothing, such as Tyvek™ coveralls, and a step-off decontamination pad will also be required at any time APRs are used.

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5.0 TASK-SPECIFIC PERSONAL PROTECTIVE EQUIPMENT

All employees working in the environmental containment cubicles shall wear, as a minimum, safety glasses, lab coat, Tyvek coveralls, and disposable gloves. If certain action limits specified in Section 4.4 are reached, air purifying respirators will be required. The protective equipment needs will be evaluated routinely by the health and safety engineer as the project progresses.

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6.0 LABORATORY ACCESS

6.1 ACCESS

Access to the environmental containment cubicles during treatability studies will be limited to personnel who have completed necessary training and have had required medical exams.

6.2 BIOASSAY SAMPLING

Bioassay Sampling

A baseline 24-hour urine sample will be taken before starting treatability activities. This sample will be analyzed for baseline urine levels.

A post-work, 24-hour urine sample will be submitted upon completion of work and will be analyzed for Th-230 and Ra-226.

Additional urine samples will be required if air samples indicate an acute exposure of 40 DAC-hours (two percent of the annual limit of intake [ALI]). This correlates to a gross alpha activity for the most conservative nuclide, Th-230, of 1 X 10^{-10} uCi/mL averaged over a one-hour exposure. A one-hour exposure leading to 40 DAC-hours for radon daughters is 12.0 WL or 1,200 pCi/L for Rn-222 in 100 percent equilibrium with its daughters. A point worth noting is that no respirator protection factors are built into these action levels.

6.3 MEDICAL MONITORING

In accordance with 29 CFR 1910.120 OSHA requirements, all personnel involved in the treatability study are required to participate in a medial monitoring program that includes:

- A baseline medical examination
- Annual medical examination
- Medical examinations that may be required after potential exposures

6.4 TRAINING REQUIREMENTS

All personnel at the Environmental Technology Development Center (ETDC) involved in the treatability study have the following training:

- IT Chemical Hygiene Plan
- ETDC Emergency Contingency Plan
- General Employee Training - Radiation (Rad) Worker Training

6.5 CONTAMINATION ZONES

The Exclusion Zone is the zone of high potential hazard due to physical, chemical, or radiological dangers. Access to the Exclusion Zone is restricted to employees who are required to enter in order to perform their job functions.

The area inside the environmental containment cubicles is considered to be the Exclusion Zone.

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7.0 EXPOSURE SYMPTOMS

Acute exposure to solvents and corrosives may produce dizziness or irritation. Exposure to low levels of radioactivity do not produce acute exposure symptoms. The potential exposures may cause delayed effects such as cancer. Because biological effects from radiation exposures are cumulative, exposures are to be kept ALARA.

7.1 FIRST AID FOR EXPOSURES

No treatment is anticipated for the predicted contaminants and concentrations. Refer to the Emergency Contingency Plan prepared for the IT ETDC.

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8.0 LABORATORY ENTRY PROCEDURES

- Locate the nearest eyewash/shower before initiating site activities.
- Verify that all instruments are calibrated.
- Visually scan the laboratory for signs of contamination.
- Perform respirator check out and fit test before use (if required).

Note: The Health and Safety Officer and any member of the team have the authority to stop work when imminent or serious safety hazards or conditions exist. Restart of work will be allowed only after the hazard or condition has been abated or reduced to a level deemed acceptable.

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9.0 LABORATORY EXITING PROCEDURE

9.1 CONTAMINATION DETECTION

All personnel are required to decontaminate themselves and then confirm the effectiveness of the decontamination. The effectiveness will be determined by frisking with a hand-held radiation monitor.

The monitor must be held within 1/2 inch of the surface and moved at a rate of approximately one inch per second for effective beta and gamma radiation monitoring. If frisking count exceeds DETECTABLE, additional decontamination is required. This decontamination will be conducted by gently scrubbing with soap and water.

If contamination cannot be removed to below the action levels (100 cpm beta/gamma or detectable alpha radiation, above background), notify the laboratory health and safety officer, Keith Hood.

9.2 DECONTAMINATION

Decontamination reduces contaminant concentrations to acceptable levels, but does not generally totally remove it. Try to avoid contamination where possible by making minimum contact with the contaminant.

Personnel: Dry removal of disposable protective equipment; wash hands, face, and any other exposed area of skin. Detergent and tepid water should be used to gently scrub skin surfaces that have contacted potentially contaminated wastes.

The effectiveness of decontamination must be confirmed by frisking.

Any exposed areas of the equipment surface will be wiped with a damp paper towel/cloth to remove contamination. Wiping with a cloth dampened with detergent solution may be necessary to remove greasy materials.

10.0 OPERATIONALLY DERIVED WASTES

Operationally derived wastes are wastes generated in the performance of various activities. These wastes include, but are not limited to:

- Disposable personal protective equipment such as Tyvek coveralls, gloves, and booties
- Disposable decontamination supplies

Protective clothing will be placed in plastic bags, in a B-25 box, or metal drum for disposal as compactible, potentially contaminated waste by Westinghouse Materials Company of Ohio (WMCO).

Operationally derived wastes are the property of the client and are to be shipped back to Fernald unless otherwise specified in the written contract.

The client will be responsible for proper transport, shipment, or disposal unless otherwise specified in the written contract.

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11.0 CONTINGENCY PLANS

Contingency plans for injuries, spills, releases, fires, and explosions are given in the Emergency Contingency Plan (ECP) for the ETDC. The ECP identifies ETDC emergency coordinators, Tom Geisler and Rick Greene. Agencies that may be requested to provide assistance in an emergency are also listed along with telephone numbers. All employees at the ETDC are provided with a copy of the ETDC ECP.

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APPENDIX B

BULKING FACTOR MEASUREMENT

International Technology Corporation

IT Analytical Services

Technology Development Laboratory Standard Operating Procedure

Title: Bulking Factor Measurement

Prepared by: Charley Morgan Date: 2/7/91

Reviewed by: Ed Oyes Date: 2/8/91
Technical Specialist

Patti B. Carswell Date: 2/8/91
Quality Control Coordinator

Janet M. Jones Date: 3-26-91
Director, Quality and Compliance, ITAS

Approved by: R L Miller Date: 3/27/91
Laboratory Director

Controlled Copy No: Uncontrolled Copy

Key Words:

Revision #	0					
Date	9/16/90				105	

1.0 Purpose and Application

1.1 The purpose of this SOP is to determine the volume increase when additives are mixed with homogenized sludge. This procedure proves to be the best test instead of trying to read the volume increase directly from a plastic or glass container because the sludge tends to stick to the sides, therefore giving an erroneous result.

2.0 References

2.1 ITAS-TDL Chemical Hygiene Plan.

3.0 Associated SOPs and Applicable Methods

3.1 None

4.0 Definitions

4.1 Container Volume (A)

The volume of deionized water that the container will hold.

4.2 Volume of Water Plus Sludge (B)

The amount of deionized water it takes to fill container with a known weight of sludge

4.3 Initial Volume (I)

Initial volume of sludge in cm^3.

4.4 Volume of Water with Treated Sludge (C)

Amount of deionized water needed to fill container that contains treated sludge.

4.5 Treated Sludge

Raw sludge that has been mixed with additives.

4.6 Treated Volume (D)

Treated volume amount of sludge.

4.7 Change in Volume (BF)

Difference of initial volume (I) of sludge and treated volume (D) of sludge.

5.0 Procedure

5.1 Summary

5.1.1 A known volume of deionized water is added to a known weight of a sludge sample. A percent volume change is then calculated.

5.2 Interferences

5.2.1 No known interferences.

5.3 Sample Handling, Preservation, and Holding Time

5.3.1 Application of these procedures on hazardous waste samples must consider the known or suspected hazardous compounds present. Project-specific selection of work area, safe working practices, and personal protective equipment shall be made based upon exposure potential to the hazardous components.

5.3.2 All applicable safety and compliance guidelines set forth by IT Corporation and by federal, state, and local regulations must be followed during performance of this procedure. All work must be stopped in the event of a known or potential compromise to the health or safety of any ITAS Associate, and must be reported immediately to a laboratory supervisor.

5.3.3 There are no holding times applicable to this procedure.

5.3.4 There are no preservation requirements applicable to this procedure.

5.4 Required Equipment

5.4.1 Two 5-oz. S/P Dispo® polypropylene container or equivalent.

5.4.2 Graduated cylinder.

5.5 Reagents/Standards

5.5.1 Deionized water.

5.5.2 Additives.

5.0 Procedure (continued)

5.6 Calibration

5.6.1 Determine the container volume (A). For example, a 5-oz. S/P Dispo® polypropylene container which is graduated from 10 to 140 ml is used. Calibrate the 5-oz container by filling the container with deionized water using a graduate cylinder.

5.7 Analysis/Operation

5.7.1 Add a known weight in grams of raw sludge to a 5-oz container. Tap container with raw sludge to release air bubbles. Add deionized water by a graduate into container until full. Designate the volume of deionized water added as the volume of water plus sludge (B).

5.7.2 In another 5-oz container, add same weight as above of raw sludge plus the percent additives and mix well. Tap container to release air pockets. Fill rest of container using a graduate with deionized water. Designate the volume of deionized water added as volume of water with treated sludge (C).

5.8 Calculations

5.8.1 Initial volume (I) of sludge is equal to (A-B) and units are in cm^3.

$$A - B = I$$

where: A = container volume and
B = volume of water plus sludge.

5.8.2 (A-C) equals treated volume (D).

$$A - C = D$$

where: A = container volume,
C = volume of water with treated sludge, and
D = treated volume.

5.8.3 Calculate the difference of initial volume (I) and treated volume (D). Designate this amount as change in Volume (BF).

$$D - I = BF$$

where: I = initial volume,
D =treated volume, and
BF = change in volume.

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5.0 Procedure (continued)

5.8.4 To get percent change in volume, take (BF) divided by initial volume (I) and multiply by 100.

$$\% \text{ Change in Volume} = BF/I \times 100$$

where: BF = change in volume and
I = initial volume.

5.9 Quality Control

5.9.1 None

6.0 Nonconformance and Corrective Action

6.1 Any failure to follow this procedure will be noted on a nonconformance memo. The corrective action will be verified by the Quality Control Coordinator and approved by the appropriate Operations Manager.

7.0 Records Management

7.1 All data will be recorded in standard laboratory notebooks.

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APPENDIX C

ANALYTICAL PROCEDURE FOR LEAD SPOT TEST ANALYSIS - ERVIN JUNGRERS

1718

QD98.S6J86

Spot Test Analysis

CLINICAL, ENVIRONMENTAL, FORENSIC, AND GEOCHEMICAL APPLICATIONS

ERVIN JUNGREIS

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DRAFT

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The preparation of a plastic rod takes about 5 hours. Castolite is a clear, syrupy liquid that pours easiy and forms a crystal-clear solid extremely resistant to heat and chemicals. The addition of a hardener and cold-setting promoter leads to solidification.

5.3.1. Rapid Screening for Copper, Lead, and Zinc in Soils and Rocks

Many rapid methods have been developed for the estimation of copper, lead, and zinc for geochemical prospecting purposes. In most cases the total amount of these metals is not determined; rather, varying amounts of the metals enter the solution depending on the dissolution procedure. The determination of only the readily soluble copper, lead, and zinc is sometimes of greater diagnostic value for prospecting than are the total values.

Various extraction modes have been used successfully in geochemical prospecting. Samples of hard rock were leached, for example, with dilute sulfuric acid or dilute hydrochloric acid (22) for semiquantitative screening for copper, lead, and zinc. Sediments and soils were screened for the same metals by partially dissolving the samples in dilute nitric acid (23, 24).

In contrast to the above-mentioned leaching procedures, the pyrosulfate fusion screening technique dissolves almost totally the copper, lead, and zinc in almost all naturally occurring soils and rocks.

The chromogen for the colorimetric estimation of zinc and lead is dithizone. Carbon tetrachloride solutions of dithizone form red zinc dithizonate when shaken with a buffered sample. Dithizone is also used as a specific reagent for the determination of lead when the masking agent cyanide is used to sequester the interfering ions. Copper is determined on the basis of the formation of the complex of monovalent copper with 2,2'-biquinoline.

PROCEDURE FOR PREPARATION OF THE SAMPLE SOLUTION. Crush the sample to minus 80 mesh. Scoop a 0.1-g sample into a 16 × 150 mm test tube, add by scooping 0.5 g potassium persulfate powder, mix intimately, and heat. Fuse the mixure for about 2 min after the flux melts. After cooling, add 3 ml 50% HCl to the tube and place the tube in a hot water bath until the melt disintegrates completely. Crushing with a glass rod helps the disintegration process. After removal and cooling, dilute the sample to 10 ml with deionized water. Take aliquots from the sample for the lead, zinc, and copper determinations.

PROCEDURE FOR THE ESTIMATION OF LEAD. Put a 2-ml aliquot of the sample solution into a 125-ml separation funnel containing 10 ml lead buffer solution. Add conc. ammonia dropwise in the presence of thymol

blue indicator until the pH range 8.5–9.0 is reached. This point is indicated by a color change from yellow to blue. Add 5 ml 0.001% dithizone solution in carbon tetrachloride and shake the separating funnel for ~15 sec. Drain the carbon tetrachloride layer into a 25-ml glass-stoppered graduated cylinder containing 10 ml 0.1% potassium cyanide solution. Shake the cylinder briefly. Compare the color of the carbon tetrachloride layer with those of similarly prepared standard solutions.

Lead standards (0, 1, 2, and 3 μg) are prepared by pipetting aliquots from a 10-ppm standard lead solution. Calculation of the metal concentration is according to the general formula:

$$\text{ppm} = \frac{\text{volume of sample solution in ml}}{\text{sample weight taken for analysis in g}} \times \frac{\mu\text{g of trace element found}}{\text{aliquot of sample solution in ml}}$$

PREPARATION OF LEAD BUFFER SOLUTION. Put 50 g ammonium citrate, 10 g potassium cyanide, and 8 g hydroxylamine hydrochloride into a large separation funnel. Add 800 ml deionized water and dissolve the materials by shaking. Add 2 ml 0.2% aqueous thymol blue indicator; then add conc. ammonia until the color turns blue (pH 8.5).

PROCEDURE FOR THE ESTIMATION OF ZINC. Transfer a 2-ml aliquot of the sample solution to a 22 × 175 mm test tube containing 8 ml zinc buffer solution. Add 5 ml 0.001% dithizone solution in carbon tetrachloride, cap the tube, and shake for 30 sec. Compare the color of the carbon tetrachloride layer with those of similarly prepared standard zinc solutions (0, 1, 2, 3, and 4 μg).

PREPARATION OF ZINC BUFFER SOLUTION. Dissolve 125 g sodium thiosulfate in ~400 ml deionized water in a large separation funnel. Remove heavy metals by extracting with 0.01% dithizone solution and discarding the colored extract. Dissolve 300 g sodium acetate in 400 ml deionized water, add 60 ml glacial acetic acid, and remove heavy metals as before. Combine these two solutions and dilute to 2 liters.

In the presence of sodium thiosulfate, elements potentially interfering with the zinc determination are masked. Only palladium and bivalent tin react under similar conditions, but palladium is unlikely to occur in significant concentrations, and tin occurs almost always in the stannic form.

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APPENDIX D

URANIUM ANALYTICAL PROCEDURE SPOT TESTS IN INORGANIC ANALYSIS

1718

SPOT TESTS IN INORGANIC ANALYSIS

by

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Sixth English edition, completely revised and enlarged

ELSEVIER PUBLISHING COMPANY

AMSTERDAM LONDON NEW YORK

1972

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benzidine, is used in the form of its chloride, sulfates are not precipitated, and molybdates are precipitated only from solutions more concentrated than 10 %. Tungsten, however, is precipitated quantitatively as a white amorphous product.

The precipitation of tungstates by chlorides of polyatomic organic bases does not lead to formula-pure tungstates of the particular bases but to adsorption compounds of WO_3-gel and the bases. This is especially true of the precipitation of small amounts of tungsten from acid solution.

Procedure. A drop of the test solution is mixed with a drop of diphenyline hydrochloride in a micro test tube. A precipitate, or cloudiness, indicates the presence of tungstate. For very small amounts, a blank test should be carried out and compared with the test, after both have stood for 15 minutes.

Limit of Identification: 6 γ tungsten
Limit of Dilution: 1 : 8500
Reagent: 1 % solution of diphenyline chloride in 2 N hydrochloric acid

1. F. Feigl, *Rec. trav. chim.*, 58 (1939) 471.
2. G. v. Knorre, *Z. anal. Chem.*, 47 (1908) 37.

URANIUM

(1) Test with 8-hydroxyquinoline (oxine)[1]

In neutral or masked alkaline solutions of uranyl salts, a quantitative precipitation of a red-brown product is obtained by adding 8-hydroxyquinoline (oxine). In contrast to other metal oxinates, which for the most part are inner complex phenolates[2], the uranium compound contains also a molecule of oxine as neutral part according to the formulation $UO_2(C_9H_6NO)_2 \cdot C_9H_6NOH$.[3] Probably this compound should not be viewed as uranyl oxinate but rather as the oxine ester of uranic acid, in other words as oxine uranate.

The precipitation through oxine also occurs from solutions of the complex alkali uranyl double carbonates, which yield $[UO_2(CO_3)_3]^{-4}$ ions. The latter are produced by adding an excess of alkali carbonate to solutions of uranyl salts. Since all metal ions forming oxinates are precipitated by alkali carbonate, it is thus possible to separate the uranium before conducting the test with oxine.

Procedure. The test solution is treated with an excess of ammonium carbonate solution. Any precipitate is filtered off or removed by centrifuging. One drop of the clear liquid is placed on a spot plate or filter paper and treated with a drop of 5 % alcohol solution of oxine. A red-brown precipitate or stain indicates uranium.

Limit of Identification: 10 γ uranium
Limit of Dilution: 1 : 5,000
Reagent: Ammonium carbonate solution: 2 g of the salt is dissolved in 10 ml of concentrated ammonia and diluted with 10 ml of water

1. A. de Sousa, *Mikrochem. ver. Mikrochim. Acta*, 40 (1953) 319.
2. Compare F. J. Welcher, *Organic Analytical Reagents*, Vol. I, New York, 1947, p. 264ff.
3. F. Hecht and W. Reich-Rohrwig, *Monatsh.*, 53, 54 (1926) 596; F. Hecht and H. Krafft-Ebing, *Z. anal. Chem.*, 106 (1936) 321.

Quantitative methods
B. Hök, *Svensk Kem. Tidskr.*, 65 (1953) 106.
A. de Sousa, *Mikrochemie Mikrochim. Acta*, 40 (1953) 319.
A. Claassen and J. Visser, *Rec. trav. chim.*, 65 (1946) 211.
L. Silverman, L. Moudy and D. W. Hawley, *Anal. Chem.*, 25 (1953) 1369.
D. L. Rulfs, A. K. De, J. Lakritz and P. J. Elving, *Anal. Chem.*, 27 (1957) 1802.
A. R. M. Al-Salihy, *Dissertation Abstracts*, 21 (1961) 2091.
K. S. Koppiker and K. B. Gajankush, *At. Energy Comm. India Rept. AEET-225*, 1965.

(2) Test with potassium ferrocyanide

Neutral or acetic acid solutions of uranyl salts give a red-brown precipitate with potassium ferrocyanide. Very dilute solutions give only a coloration. Uranyl potassium ferrocyanide or uranyl ferrocyanide is formed.

This test is specific in the absence of ferric and copper salts which also give colored ferrocyanides. In this case, previous separation with alkali carbonate is necessary (see test (*1*)).

Procedure.[1] A drop of the slightly acid test solution is placed on filter paper impregnated with 3 % potassium ferrocyanide, or a drop of the test solution and then the ferrocyanide are placed on filter paper. According to the concentration of uranium, a more or less intense brown stain is formed.

Limit of Identification: 0.92 γ uranium
Limit of Dilution: 1 : 50,000

Test for uranium in the presence of iron and copper[2]

The test for uranium with potassium ferrocyanide can also be carried out in the presence of ferric and cupric salts, if these metals are converted, before the addition of the ferrocyanide, into the nonreacting cuprous and ferrous forms. Reduction with iodide ions in acid solution serves this purpose:

$$2\ Cu^{+2} + 4\ I^- \rightarrow Cu_2I_2 + I_2$$
$$2\ Fe^{+3} + 2\ I^- \rightarrow 2\ Fe^{+2} + I_2$$

If the liberated iodine is decolorized (reduced) with thiosulfate, the uranium may then be detected with potassium ferrocyanide.

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Procedure. A drop of concentrated potassium iodide solution is placed on filter paper and, after it has soaked in, a drop of the acid test solution is added; iodine is liberated. To complete the reduction, a further drop of potassium iodide is added, and then a drop of sodium thiosulfate to remove the elementary iodine. A drop of potassium ferrocyanide is placed on the decolorized fleck. A more or less deeply colored brown or yellowish circle is formed, according to the amount of uranium present.

Uranyl ions can be separated from interfering ions by extraction with ethyl acetate from HNO_3 solution.[3]

1. F. Feigl and R. Stern, *Z. anal. Chem.*, 60 (1921) 39.
2. N. A. Tananaeff and G. A. Pantschenko, *Z. anorg. allgem. Chem.*, 150 (1926) 164.
3. V. V. Sergovskaya, *Anal. Abstracts*, 10 (1963) 4643.

Quantitative method

F. H. Burgstall and R. P. Linstead, *J. Chem. Soc.*, Suppl. 2 (1949) 311.

(3) Fluorescence test

Uranyl salts fluoresce best in the crystalline form, but only slightly in solution. If a dilute solution of uranium salts is allowed to evaporate slowly on a microscope slide, and the residue examined, single fluorescent crystals can be observed.[1] Traces of impurities or too rapid evaporation of the solution interfere with the test because they prevent the formation of good crystals.

Borax beads containing uranium exhibit an appreciable green fluorescence. Fluoride beads of the alkalis and alkaline earth metals[2] fluoresce especially well. Sodium fluoride beads light up to a deep yellow color and are most striking. They can be used to detect uranium.

The shape of the bead is very important when testing for uranium by means of activated beads. Thin flat beads are better than the round type, because the ultraviolet light penetrates farther. Neither SiO_2, TiO_2 nor sulfates, etc., should be present, nor any other material that liberates hydrofluoric acid or forms complex compounds with fluorides. Iron should be avoided because it makes the bead yellow and so absorbs the ultraviolet light at the surface. Manganese salts, which color the beads blue, do not interfere so much as iron. Thorium salts also greatly reduce the luminosity, but it may still be perceptible provided sodium fluoride is present in excess. Only niobium[3] as well as greater quantities of beryllium (exceeding 1 mg/ml) give a similar fluorescence, but it is relatively so weak as to be of no importance.

Procedure I. Sodium fluoride is fused in a loop of platinum wire (diam. 1 mm). When cold, the bead appears only slightly violet in ultraviolet light (reflected light). By means of a calibrated loop of platinum, 0.001 ml of the neutral test solution is placed on the bead and evaporated. After fusing for a short time, the bead is cooled and examined in ultraviolet light.

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Limit of Identification: 0.001 γ uranium in 0.001 ml
Limit of Dilution: 1 : 1,000,000

Zirconium phosphate ion exchange resin (Ionite 3) (H^+, Na^+ or K^+ form) absorbs uranium^{VI} salts and gives an intense yellow fluorescence under u.v. light. This behavior is used as the basis of a sensitive spot test for uranium.[4]

Procedure II. 4–5 beads of Ionite 3 are added to 1 drop of the sample. The appearance of a yellow fluorescence under u.v. light within 30 minutes indicates the presence of uranium.

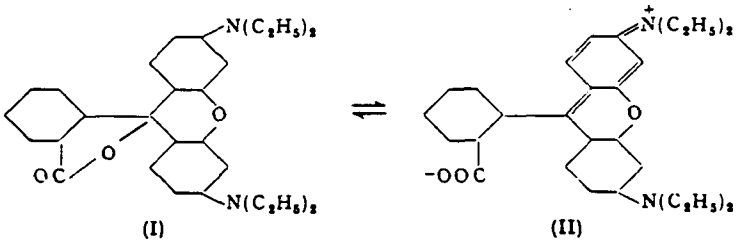
Limit of Identification: 0.06 γ U
Limit of Dilution: 1 : 1,000,000

The sample solution must be less than 0.5 N with respect to acid or less than 0.05 N with respect to alkali. The fluorescence is inhibited by Ag, Fe and Th.

1. F. Hernegger, *Anz. Akad. Wiss. Wien, Math.-naturw. Klasse*, 144 (1935) 217; F. Hernegger and B. Karlik, *Sitzber. Akad. Wiss. Wien, Math.-naturw. Klasse, Abt. IIa*, 144 (1935) 217; *Chem. Abstracts*, 30 (1936) 408.
2. E. L. Nichols and M. K. Slattery, *J. Optical Soc. Am.*, 12 (1926) 449.
3. J. Papisch and L. E. Hoag, *Proc. Natl. Acad. Sci. U.S.*, 13 (1927) 726.
4. H. Kakihana, Y. Mori and Y. Watanabe, *J. Chem. Soc. Japan*, 82 (1961 594; *Anal. Abstracts*, 8 (1962) 4177.

4) Test with Rhodamine B[1]

The red dye Rhodamine B is a sensitive reagent in acid solutions for monobasic complex metal halogeno acids (compare pages 107, 232, 242, 476). Its almost colorless solutions in benzene contain an equilibrium mixture of the lacto form (I) with minimal amounts of the red quinoid form (II):

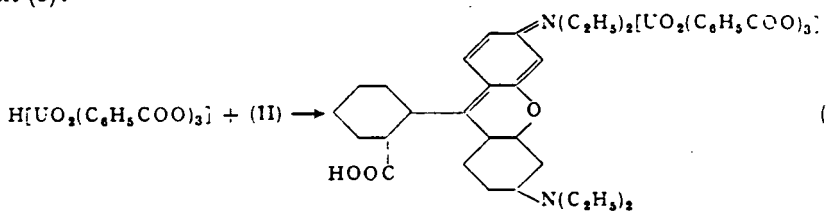


(I) (II) (1)

When neutral solutions of uranyl-, ferric-, or bismuth nitrate (chloride) are shaken with a benzene solution of Rhodamine B, the benzene layer turns red and exhibits an intense orange fluorescence in ultraviolet light. This effect is surprisingly heightened if a little benzoic acid or some other benzene-soluble carboxylic acid is added to the benzene solution. In the case of uranyl salts, the color (fluorescence) reaction is so marked that a sensitive

$$UO_2^{+2} + 3\ C_6H_5COOH \leftrightharpoons H[UO_2(C_6H_5COO)_3] + 2\ H^+ \qquad (2)$$

test can be based on this finding. Although the benzene-soluble uranyl compound has not been isolated, the underlying chemistry is probably that the union of uranyl ions with benzoic acid yields a slight quantity of a complex uranyl benzoic acid (2) which then produces a red benzene-soluble salt (3):



(3)

On this basis, the formation of the benzene-soluble dye salt constantly disturbs equilibria (1) and (2) because the products contained in them are replenished after consumption and so suffice to accomplish the color reaction.

Procedure. The test is conducted in a micro test tube. A drop of the neutral test solution is treated with 5 drops of the reagent solution and shaken. A red or pink benzene layer results if uranium is present, the shade depending on the quantity of the latter.

Limit of Identification: 0.05 γ uranium
Limit of Dilution: 1 : 1,000,000
Reagent: A 0.5 % solution of benzoic acid in benzene is treated with an excess of Rhodamine B, shaken, and then filtered. The solution keeps.

If iron and bismuth are also present, they must be removed because they show analogous behavior. The test solution is warmed with an excess of sodium carbonate and the precipitate removed. The filtrate which contains $[UO_2(CO_3)_2]^{-2}$ is taken to dryness with nitric acid. The residue contains uranyl nitrate, and can be tested by the procedure just described. As little as 0.5 γ uranium can be detected in the presence of 2500 γ iron, starting with one drop and operating within the bounds of spot test technique.

1. F. Feigl, V. Gentil and D. Goldstein, unpublished studies.

Quantitative methods

N. R. Andersen and D. M. Hercules, *Anal. Chem.*, 36 (1964) 2138.
H. H. P. Moeken and W. A. H. van Neste, *Anal. Chim. Acta*, 37 (1967) 480.
L. M. Burtnenko and N. S. Poluektiv, *Zh. Analit. Khim.*, 23 (1968) 700; *Anal. Abstracts*, 18 (1970) 105.

(5) Other tests for uranium

(*a*) A rust-brown fleck is produced by uranium when spotted on paper with 0.2 % solution of quercetin or quercitrin (*Idn. Limit:* 3 γ U).[1] See also page 273.

(*b*) A drop of the neutral test solution on treatment with a drop of 0.5 *N*

Na_3PO_4 gives a precipitate of $(UO_2)_3(PO_4)_2$ which exhibits a strong yellow fluorescence (*Idn. Limit:* 2.5 γ U).[2]

(*c*) The red fluorescence of a drop of an alkaline solution of cochineal disappears on the addition of a drop of a solution of a uranyl salt (*Idn. Limit:* 2.5 γ U).[2]

(*d*) A red color appears if a drop of 0.12 % of fluorescein solution and 3 drops of 5 % ammonium chloride are added to a drop of a uranyl solution on a spot plate (*Idn. Limit:* 0.12 γ UO_2^{+2}).[3]

(*e*) Reduction of the weakly acid test solution yields U^{+4} ions which reduce Fe^{+3} to Fe^{+2}. Accordingly, a red color results if the reduced solution is treated with a $FeCl_3$ solution containing phenanthroline (*Idn. Limit:* 1 γ uranium).[4] If only slight amounts of uranium are suspected, it is advisable to add thorium nitrate after the test solution has been reduced and to precipitate ThF_4 and UF_4 jointly by means of ammonium fluoride. The test is then conducted with the precipitate.

(*f*) Resacetophenone oxime reacts with uranium^{VI} salts in weak mineral acid solution forming a reddish-brown color (*Idn. Limit:* 0.6 γ U at pH 5.6). Al, Zr and Th do not interfere. U^{IV} does not react; trivalent iron gives a deep purple color.[5]

1. E. A. Kocsis, *Mikrochemie*, 25 (1938) 13.
2. H. Gotô, *Sci. Rep. Tohoku Univ.*, 29 (1940) 287.
3. M. Nageswara Rao and Bh. S. V. Raghawa Rao, *Z. anal. Chem.*, 142 (1954) 161.
4. F. Lucena Conde and L. Prat, *Mikrochim. Acta*, (1955) 799.
5. C. Rama Rao, *Talanta*, 9 (1962) 81.

Quantitative method

(*a*) J. Kommenda, *Chem. Listy*, 47 (1953) 531; *Anal. Abstracts*, 1 (1954) 2669.

VANADIUM

A. Metallic Vanadium

Detection by conversion into alkali salts of vanadic acid

For the discussion of the method and the procedure, see metallic molybdenum (page 319).

B. Vanadium^{V} Compounds

(1) Test with hydrogen peroxide[1]

A solution of vanadium containing sulfuric acid turns red-brown to blood-red, or, in very dilute solution, pale brown-pink, on the addition of hydrogen peroxide. Excess hydrogen peroxide causes partial decoloration. The reaction involves the formation of the colored peroxovanadium salt (I) which, in the presence of excess hydrogen peroxide, reacts:

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APPENDIX E

HACH MICRO COD DESIGN

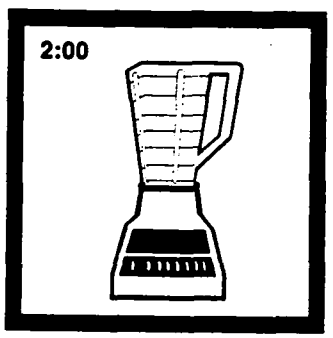
ANALYTICAL PROCEDURES

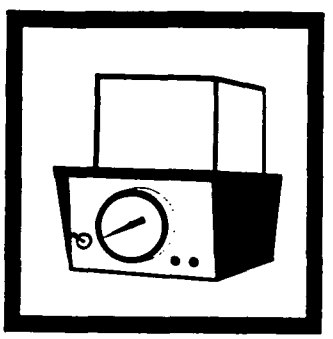
For DR/2000, DR/3000 and DR/3 Instruments

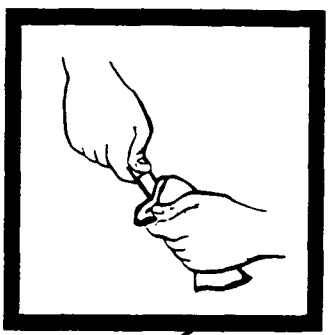
OXYGEN DEMAND, CHEMICAL

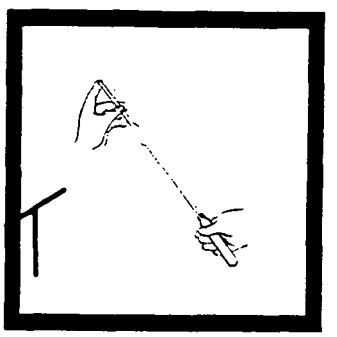
Reactor Digestion Method*, EPA Approved†
0 to 150, 0 to 1,500, 0 to 15,000 mg/L COD
For water, wastewater, seawater

DIGESTION









1. Homogenize 500 mL of sample for 2 minutes in a blender.

0 to 15,000 mg/L Note: *Homogenize 100 mL of sample. Pour the homogenized sample into a 250-mL beaker and stir with a magnetic stirrer.*

Note: *Blending ensures distribution of solids and improves accuracy and reproducibility.*

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these procedures.*

2. Turn on the COD Reactor. Preheat to 150°C. Place the plastic shield on the reactor.

Caution: Ensure safety devices are in place to protect analyst from splattering should reagent leaking occur.

3. Remove the cap of a COD Digestion Reagent Vial of the desired range.

Sample Concentration Range (mg/L)	COD Digestion Reagent Vial Type
0 to 150	Low Range
0 to 1,500	High Range
0 to 15,000	High Range Plus

Note: *The reagent mixture is light sensitive. Keep unused vials in the opaque shipping container, in a refrigerator if possible. The amount of light striking the vials during the test will not affect results.*

4. Hold the vial at a 45-degree angle. Pipet 2.00 mL (0.2 mL for the 0 to 15,000 mg/L range) of sample into the vial.

0 to 15,000 mg/L Note: *Pipet only 0.20 mL of sample, not 2.00 mL, using a TenSette pipet. For greater accuracy a minimum of three replicates should be analyzed and the results averaged.*

Note: *Spilled reagent will affect test accuracy and is hazardous to skin and other materials. Do not run tests with vials which have been spilled. If some spills, wash with running water.*

Note: *For proof of accuracy, use COD standard solutions (preparation given in the Accuracy Check) in place of the sample.*

*Adapted from Jirka, A.M., and Carter, M.J., *Analytical Chemistry*, 47 (8) 1397 (1975)
†*Federal Register*, 45(78) 26811-26812 (April 21, 1980)
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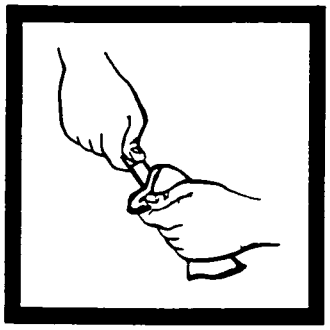
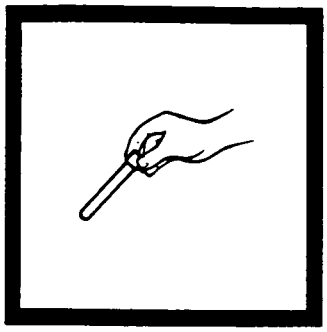
OXYGEN DEMAND, CHEMICAL, continued

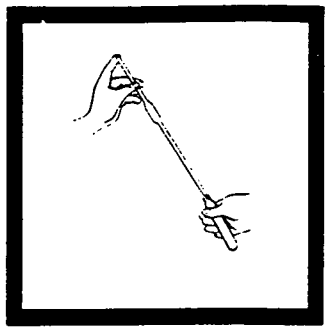


5. Replace the vial cap tightly. Rinse the COD vial with deionized water and wipe the vial clean with a paper towel.



6. Hold the vial by the cap and over a sink. Invert gently several times to mix the contents. Place the vial in the preheated COD Reactor.

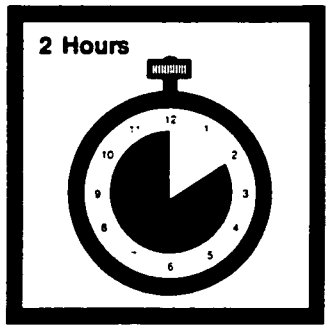
Note: The vial will become very hot during mixing.



7. Prepare a blank by repeating Steps 3 to 6, substituting 2.00 mL (0.2 mL for the 0 to 15,000 mg/L range) deionized water for the sample.

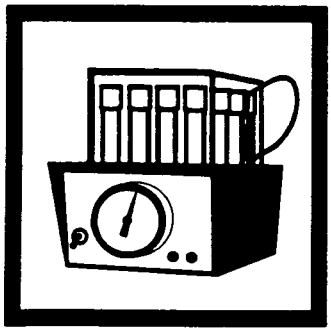
Note: Be sure the pipet is well rinsed, or use a clean pipet.

Note: One blank must be run with each set of samples. All tests (samples and blank) should be run with the same lot of vials. The lot number appears on the container label.

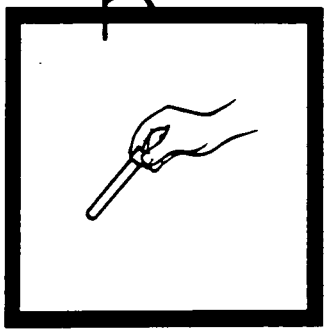


8. Heat the vials for 2 hours.

Note: Many wastewater samples containing easily oxidized materials are digested completely in less than two hours. If desired, measure the concentration (while still hot) at 15 minute intervals until it remains unchanged. At this point, the sample is completely digested. Cool the vials to room temperature for final measurement.



9. Turn the reactor off. Wait about 20 minutes for the vials to cool to 120°C or less.



10. Invert each vial several times while still warm. Place the vials into a rack. Wait until the vials have cooled to room temperature.

Note: If a pure green color appears in the reacted sample, the reagent capacity may have been exceeded. Measure the COD and if necessary, repeat the test with a diluted sample.

Colorimetric
OR
Titrimetric

11. Use one of the following analytical techniques to determine the sample concentration:

Colorimetric determination, 0 to 150 mg/L COD

Colorimetric determination, 0 to 1,500 mg/L COD

Colorimetric determination, 0 to 15,000 mg/L COD

Buret titration

APPENDIX F

UNCONFINED COMPRESSIVE STRENGTH

DRAFT

STANDARD OPERATING PROCEDURE

ITAS-TECHNOLOGY DEVELOPMENT LABORATORY

TITLE:	SOP NO: TDL1109
UNCONFINED COMPRESSIVE STRENGTH	DATE INITIATED: 7/31/89 REVISION NO: 1 DATE REVISED: 3/28/90 PAGE 1 OF 19

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
[illegible]	[illegible]	3/28/90	Janet M Jones	9-4-90

1.0 Purpose and Application

1.1 This test method covers the determination of the unconfined compressive strength of cohesive soil in the undisturbed, remolded, or compacted condition using strain-controlled application of the axial load.

1.2 This test method provides an approximate value of the strength of cohesive soils in terms of total stresses.

1.3 This test method is applicable only to cohesive materials which will not expel bleed water during the loading portion of the test and which will retain intrinsic strength after removal of confining pressures, such as clays or cemented soils.

2.0 References

2.1 Annual Book of ASTM Standards. 1988. "Soil and Rock; Building Stones; Geotextiles. Vol. 4.08.

3.0 Associated SOPs and Applicable Methods

3.1 ASTM D-422.

3.2 ASTM D-854.

3.3 ASTM D-2216.

3.4 ASTM D-2850.

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3.0 Associated SOPs and Applicable Methods (continued)

3.5 ASTM D-4220.

3.6 ASTM D-4318.

4.0 Definitions

4.1 Unconfined compressive strength - the compressive stress at which an unconfined cylindrical specimen of soil will fail in a simple compression test.

4.2 Shear strength - for unconfined compressive strength test specimens, the shear strength is calculated to be one-half of the compressive stress at failure.

4.3 Bleed water - water expelled from the soil due to deformation or compaction.

5.0 Procedure

5.1 ASTM Standard Method D-2166.

6.0 Nonconformance and Corrective Action

6.1 If this procedure cannot be followed for any reason, a nonconformance memo will be filed with the Quality Control Coordinator. Corrective action will be approved by the Operations or Project Manager.

7.0 Records Management

7.1 Data is to be recorded in a standard laboratory notebook with the project it pertains to clearly labeled on the notebook page.

Designation: D 2166 - 85

Standard Test Method for UNCONFINED COMPRESSIVE STRENGTH OF COHESIVE SOIL[1]

This standard is issued under the fixed designation D 2166; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the unconfined compressive strength of cohesive soil in the undisturbed, remolded, or compacted condition, using strain-controlled application of the axial load.

1.2 This test method provides an approximate value of the strength of cohesive soils in terms of total stresses.

1.3 This test method is applicable only to cohesive materials which will not expel bleed water (water expelled from the soil due to deformation or compaction) during the loading portion of the test and which will retain intrinsic strength after removal of confining pressures, such as clays or cemented soils. Dry and crumbly soils, fissured or varved materials, silts, peats, and sands cannot be tested with this method to obtain valid unconfined compression strength values.

NOTE 1.—The determination of the unconsolidated, undrained strength of cohesive soils with lateral confinement is covered by Test Method D 2850.

1.4 This test method is not a substitute for Test Method D 2850.

1.5 The values stated in SI units are to be regarded as the standard. The values stated in inch-pound units are approximate.

1.6 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Applicable Documents

2.1 *ASTM Standards:*

D 422 Method for Particle-Size Analysis of Soils[2]

D 653 Terms and Symbols Relating to Soil and Rock[2]

D 854 Test Method for Specific Gravity of Soils[2]

D 1587 Practice for Thin-Walled Tube Sampling of Soils[2]

D 2216 Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures[2]

D 2487 Test Method for Classification of Soils for Engineering Purposes[2]

D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)[2]

D 2850 Test Method for Unconsolidated, Undrained Compressive Strength of Cohesive Soils in Triaxial Compression[2]

D 4220 Practices for Preserving and Transporting Soil Samples[2]

D 4318 Test Method for Liquid Limit, Plastic Limit, and Plasticity Index of Soils[2]

3. Terminology

3.1 Refer to Terms and Symbols D 653 for standard definitions of terms.

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[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.05 on Structural Properties of Soils.

Current edition approved July 26, 1985. Published September 1985. Originally published as D 2166 - 63T. Last previous edition D 2166 - 66 (1979)[ε1].

[2] *Annual Book of ASTM Standards*, Vol 04.08.

3.2 *Descriptions of Terms Specific to this Standard:*

3.2.1 *unconfined compressive strength (q_u)*—the compressive stress at which an unconfined cylindrical specimen of soil will fail in a simple compression test. In this test method, unconfined compressive strength is taken as the maximum load attained per unit area or the load per unit area at 15 % axial strain, whichever is secured first during the performance of a test.

3.2.2 *shear strength (s_u)*—for unconfined compressive strength test specimens, the shear strength is calculated to be ½ of the compressive stress at failure, as defined in 3.2.1.

4. Significance and Use

4.1 The primary purpose of the unconfined compression test is to quickly obtain the approximate compressive strength of soils that possess sufficient cohesion to permit testing in the unconfined state.

4.2 Samples of soils having slickensided or fissured structure, samples of some types of loess, very soft clays, dry and crumbly soils and varved materials, or samples containing significant portions of silt or sand, or both (all of which usually exhibit cohesive properties), frequently display higher shear strengths when tested in accordance with Test Method D 2850. Also, unsaturated soils will usually exhibit different shear strengths when tested in accordance with Test Method D 2850.

4.3 If both an undisturbed and a remolded test are performed on the same sample, the sensitivity of the material can be determined. This method of determining sensitivity is suitable only for soils that can retain a stable specimen shape in the remolded state.

NOTE 2—For soils that will not retain a stable shape, a vane shear test or Test Method D 2850 can be used to determine sensitivity.

5. Apparatus

5.1 *Compression Device*—The compression device may be a platform weighing scale equipped with a screw-jack-activated load yoke, a hydraulic loading device, or any other compression device with sufficient capacity and control to provide the rate of loading prescribed in 7.1. For soil with an unconfined compressive strength of less than 100 kPa (1.0 ton/ft^2) the compression device shall be capable of measuring the compressive stress to within 1 kPa (0.01 ton/ft^2). For soil with an unconfined compressive strength of 100 kPa (1.0 ton/ft^2) or greater, the compression device shall be capable of measuring the compressive stress to the nearest 5 kPa (0.05 ton/ft^2).

5.2 *Sample Extruder*, capable of extruding the soil core from the sampling tube in the same direction of travel in which the sample entered the tube, at a uniform rate, and with negligible disturbance of the sample. Conditions at the time of sample removal may dictate the direction of removal, but the principal concern is to keep the degree of disturbance negligible.

5.3 *Deformation Indicator*—The deformation indicator shall be a dial indicator graduated to 0.03 mm (0.001 in.) or better and having a travel range of at least 20 % of the length of the test specimen, or some other measuring device, such as an electronic deformation measuring device, meeting these requirements.

5.4 *Dial Comparator*, or other suitable device, for measuring the physical dimensions of the specimen to within 0.1 % of the measured dimension.

NOTE 3—Vernier calipers are not recommended for soft specimens, which will deform as the calipers are set on the specimen.

5.5 *Timer*—A timing device indicating the elapsed testing time to the nearest second shall be used for establishing the rate of strain application prescribed in 7.1.

5.6 *Balance*—The balance used to weigh specimens shall determine the mass of the specimen to within 0.1 % of its total mass.

5.7 *Equipment*, as specified in Method D 2216.

5.8 *Miscellaneous Apparatus*, including specimen trimming and carving tools, remolding apparatus, water content cans, and data sheets, as required.

6. Preparation of Test Specimens

6.1 *Specimen Size*—Specimens shall have a minimum diameter of 30 mm (1.3 in.) and the largest particle contained within the test specimen shall be smaller than one tenth of the specimen diameter. For specimens having a diameter of 72 mm (2.8 in.) or larger, the largest particle size shall be smaller than one sixth of the specimen diameter. If, after completion of a test on

an undisturbed specimen, it is found, based on visual observation, that larger particles than permitted are present, indicate this information in the remarks section of the report of test data (Note 4). The height-to-diameter ratio shall be between 2 and 2.5. Determine the average height and diameter of the test specimen using the apparatus specified in 5.4. Take a minimum of three height measurements (120° apart), and at least three diameter measurements at the quarter points of the height.

NOTE 4—If large soil particles are found in the sample after testing, a particle-size analysis performed in accordance with Method D 422 may be performed to confirm the visual observation and the results provided with the test report.

6.2 *Undisturbed Specimens*—Prepare undisturbed specimens from large undisturbed samples or from samples secured in accordance with Practice D 1587 and preserved and transported in accordance with the practices for Group C samples in Practices D 4220. Tube specimens may be tested without trimming except for the squaring of ends, if conditions of the sample justify this procedure. Handle specimens carefully to prevent disturbance, changes in cross section, or loss of water content. If compression or any type of noticeable disturbance would be caused by the extrusion device, split the sample tube lengthwise or cut it off in small sections to facilitate removal of the specimen without disturbance. Prepare carved specimens without disturbance, and whenever possible, in a humidity-controlled room. Make every effort to prevent any change in water content of the soil. Specimens shall be of uniform circular cross section with ends perpendicular to the longitudinal axis of the specimen. When carving or trimming, remove any small pebbles or shells encountered. Carefully fill voids on the surface of the specimen with remolded soil obtained from the trimmings. When pebbles or crumbling result in excessive irregularity at the ends, cap the specimen with a minimum thickness of plaster of paris, hydrostone, or similar material. When sample condition permits, a vertical lathe that will accommodate the total sample may be used as an aid in carving the specimen to the required diameter. Where prevention of the development of appreciable capillary forces is deemed important, seal the specimen with a rubber membrane, thin plastic coatings, or with a coating of grease or sprayed plastic immediately after preparation and during the entire testing cycle. Determine the mass and dimensions of the test specimen. If the specimen is to be capped, its mass and dimensions should be determined before capping. If the entire test specimen is not to be used for determination of water content, secure a representative sample of cuttings for this purpose, placing them immediately in a covered container. The water content determination shall be performed in accordance with Method D 2216.

6.3 *Remolded Specimens*—Specimens may be prepared either from a failed undisturbed specimen or from a disturbed sample, providing it is representative of the failed undisturbed specimen. In the case of failed undisturbed specimens, wrap the material in a thin rubber membrane and work the material thoroughly with the fingers to assure complete remolding. Avoid entrapping air in the specimen. Exercise care to obtain a uniform density, to remold to the same void ratio as the undisturbed specimen, and to preserve the natural water content of the soil. Form the disturbed material into a mold of circular cross section having dimensions meeting the requirements of 6.1. After removal from the mold, determine the mass and dimensions of the test specimens.

6.4 *Compacted Specimens*—Specimens shall be prepared to the predetermined water content and density prescribed by the individual assigning the test (Note 5). After a specimen is formed, trim the ends perpendicular to the longitudinal axis, remove from the mold, and determine the mass and dimensions of the test specimen.

NOTE 5—Experience indicates that it is difficult to compact, handle, and obtain valid results with specimens that have a degree of saturation that is greater than 90 %.

7. Procedure

7.1 Place the specimen in the loading device so that it is centered on the bottom platen. Adjust the loading device carefully so that the upper platen just makes contact with the specimen. Zero the deformation indicator. Apply the load so as to produce an axial strain at a rate of ½ to 2 %/min. Record load, deformation, and time values at sufficient intervals to define the shape of the stress-strain curve (usually 10 to 15 points are sufficient). The rate of strain should be chosen so that the time to failure does not exceed about

15 min (Note 6). Continue loading until the load values decrease with increasing strain, or until 15 % strain is reached. The rate of strain used for testing sealed specimens may be decreased if deemed desirable for better test results. Indicate the rate of strain in the report of the test data, as required in 9.1.7. Determine the water content of the test specimen using the entire specimen, unless representative cuttings are obtained for this purpose, as in the case of undisturbed specimens. Indicate on the test report whether the water content sample was obtained before or after the shear test, as required in 9.1.2.

NOTE 6—Softer materials that will exhibit larger deformation at failure should be tested at a higher rate of strain. Conversely, stiff or brittle materials that will exhibit small deformations at failure should be tested at a lower rate of strain.

7.2 Make a sketch, or take a photo, of the test specimen at failure showing the slope angle of the failure surface if the angle is measurable.

7.3 A copy of a sample data sheet is included in Appendix X1. Any data sheet can be used, provided the form contains all the required data.

8. Calculations

8.1 Calculate the axial strain, ϵ_1, to the nearest 0.1 %, for a given applied load, as follows:

$$\epsilon_1 = \Delta L / L_0$$

where:

ΔL = length change of specimen as read from deformation indicator, mm (in.), and

L_0 = initial length of test specimen, mm (in.).

8.2 Calculate the average cross-sectional area, A, for a given applied load, as follows:

$$A = A_0/(1 - \epsilon_1)$$

where:

A_0 = initial average cross-sectional area of the specimen, mm² (in.²), and

ϵ_1 = axial strain for the given load, %.

8.3 Calculate the compressive stress, σ_c, to three significant figures, or nearest 1 kPa (0.01 ton/ft²), for a given applied load, as follows:

$$\sigma_c = (P/A)$$

where:

P = given applied load, kPa (ton/ft²),

A = corresponding average cross-sectional area mm² (in.²).

8.4 *Graph*—If desired, a graph showing the relationship between compressive stress (ordinate) and axial strain (abscissa) may be plotted. Select the maximum value of compressive stress, or the compressive stress at 15 % axial strain, whichever is secured first, and report as the unconfined compressive strength, q_u. Whenever it is considered necessary for proper interpretation, include the graph of the stress-strain data as part of the data reported.

8.5 If the unconfined compressive strength is determined, the sensitivity, S_T, is calculated as follows:

$$S_T = \frac{q_u \text{ (undisturbed specimen)}}{q_u \text{ (remolded specimen)}}$$

9. Report

9.1 The report should include the following:

9.1.1 Identification and visual description of the specimen, including soil classification, symbol, and whether the specimen is undisturbed, remolded, compacted, etc. Also include specimen identifying information, such as project, location, boring number, sample number, depth, etc. Visual descriptions shall be made in accordance with Practice D 2488.

9.1.2 Initial dry density and water content (specify if the water content specimen was obtained before or after shear, and whether from cuttings or the entire specimen).

9.1.3 Degree of saturation (Note 7), if computed.

NOTE 7—The specific gravity determined in accordance with Test Method D 854 is required for calculation of the degree of saturation.

9.1.4 Unconfined compressive strength and shear strength.

9.1.5 Average height and diameter of specimen.

9.1.6 Height-to-diameter ratio.

9.1.7 Average rate of strain to failure, %.

9.1.8 Strain at failure, %.

9.1.9 Liquid and plastic limits, if determined, in accordance with Test Method D 4318.

9.1.10 Failure sketch or photo.

9.1.11 Stress-strain graph, if prepared.

9.1.12 Sensitivity, if determined.

9.1.13 Particle size analysis, if determined, in accordance with Method D 422, and

9.1.14 *Remarks*—Note any unusual conditions or other data that would be considered necessary to properly interpret the results obtained, for example, slickensides, stratification,

shells, pebbles, roots, or brittleness, the type of failure (that is, bulge, diagonal shear, etc.).

10. Precision and Bias

10.1 No method presently exists to evaluate the precision of a group of unconfined compression tests on undisturbed specimens due to specimen variability. Undisturbed soil specimens from apparently homogeneous soil deposits at the same location often exhibit significantly different strength and stress-strain properties.

10.2 A suitable test material and method of specimen preparation have not been developed for the determination of laboratory variances due to the difficulty in producing identical cohesive soil specimens. No estimates of precision for this test method are available.

APPENDIX

(Nonmandatory Information)

X1. Example Data Sheet

UNCONFINED COMPRESSION TEST—UI

Name _____ Date _____ Job No. _____

Location _____

Boring No. _____ Sample No. _____ Depth/Elev. _____

Description of Sample _____

_____

Proving Ring No. _____ Apparatus No. _____

Water Content Determination

Tare No. _____

Wt. Specimen Wet + Tare _____

Wt. Specimen Dry + Tare _____

Wt. Water _____

Wt. Tare _____

Wt. Specimen Wet _____

Wt. Specimen Dry _____

Water Content in % Dry Wt.

at 105°C _____

Wet Density _____

Dry Density _____

Unconfined Compressive Strength

Initial Diameter D_o _____

Initial Area A_o _____

Initial Height L_o _____

Initial Volume V_o _____

Specific Gravity _____

$$\text{Stress} = \frac{\text{Load}}{\text{Corr. Area}}$$

Data sheet continued

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D 2166

Test Data

Unit Strain $\frac{\Delta L}{L_o}$ $\frac{L}{D}$ = _____ Corr. Area = $\frac{A_o}{1 - \text{Unit Strain}}$

Elapsed Time-min	Load Dial	Axial Load	Strain Dial	Total Strain	Unit Strain	Corrected Area	Stress	

Type of Sample _____

Strain Rate _____ In./Min

Remarks _____

Attach a photo or sketch of the specimen after failure to this form

APPENDIX XI:

UNCONFINED COMPRESSION TEST

1. INTRODUCTION. The unconfined compression test is used to measure the unconfined compressive strength of a cohesive soil. The unconfined compression test is applicable only to coherent materials such as saturated clays or cemented soils that retain intrinsic strength after removal of confining pressure; it is not a substitute for the Q test. Dry or crumbly soils, fissured or varved materials, silts, and sands cannot be tested meaningfully in unconfined compression. In this test, a laterally unsupported cylindrical specimen is subjected to a gradually increased axial compression load until failure occurs. The unconfined compression test is a form of triaxial test in which the major principal stress is equal to the applied axial stress, and the intermediate and minor principal stresses are equal to zero. The unconfined compressive strength, q_u, is defined as the maximum unit axial compressive stress at failure or at 15 percent strain, whichever occurs first. The undrained shear strength, s_u, is assumed to be equal to one-half the unconfined compressive strength. The axial load may be applied to the specimen either by the controlled strain procedure, in which the stress is applied to produce a predetermined rate of strain, or by the controlled stress procedure, in which the stress is applied in predetermined increments of load.

2. APPARATUS. The apparatus consists of the following:

a. Equipment for Preparing Specimen. A trimming frame as described in paragraph 3e of Appendix X, TRIAXIAL COMPRESSION TESTS, or a trimming cylinder with beveled cutting edges may be used for trimming specimens. The equipment should include wire saws and knives of various sizes and types for use with the trimming frame. A motorized soil lathe may be used advantageously under certain circumstances. A miter box or cradle is required to trim the specimen to a fixed length and to ensure that the ends of the specimen are parallel with each other and perpendicular to the vertical axis of the specimen.

b. Loading Device. A number of commercially available controlled-strain or controlled-stress types of loading devices are suitable for applying the axial loads in the unconfined compression test. In

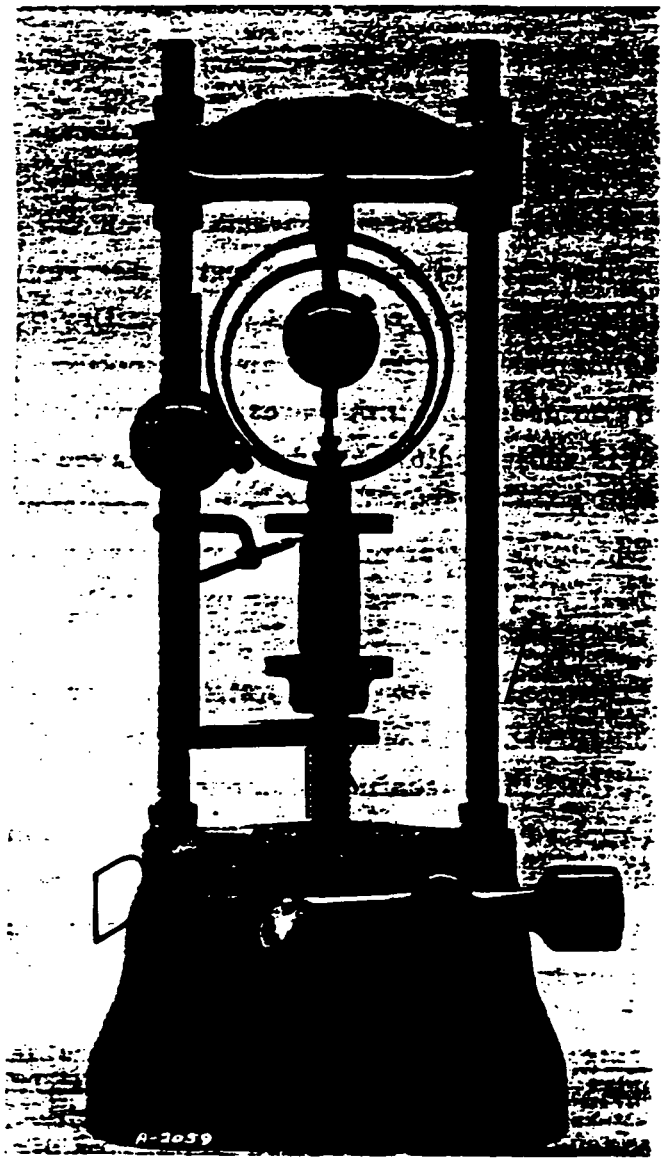

Figure 1. Typical unconfined compression test apparatus

general, controlled-strain type loading devices are preferable, and the procedures described herein are based on the use of this type of equipment. If available, an automatic stress-strain recorder may be used to measure and record applied axial loads and displacements. A typical loading device is shown in Figure 1. Any equipment used should be calibrated so that the loads actually applied to the soil specimen can be determined. The required sensitivity of stress-measuring equipment for both controlled-stress and controlled-strain testing will vary with the strength characteristics of the soil. For relatively weak soils (compressive strengths less than 1.0 ton per sq ft), the unit load should be measurable to within 0.01 ton per sq ft. For soils with compressive strengths of 1.0 ton per sq ft or greater, the loads should be measurable to the nearest 0.05 ton per sq ft.

c. Measuring equipment, such as dial indicators and calipers, suitable for measuring the dimensions and axial deformation of a specimen

to the nearest 0.001 in.

d. Timing device, either a watch or clock with second hand.

e. Balances, sensitive to 0.1 g.

f. Other. Apparatus necessary to determine water content and specific gravity (see Appendixes I, WATER CONTENT - GENERAL, and IV, SPECIFIC GRAVITY).

3. PREPARATION OF SPECIMENS. a. Specimen Size. Unconfined compression specimens shall have a minimum diameter of 1.0 in. (preferably 1.4 in.), and the largest particle in any test specimen will be no greater than one-sixth the specimen diameter. The height-to-diameter ratio shall be not less than 2.1. Commonly used diameters of unconfined compression specimens are 1.4 and 2.8 in. Specimens of 1.4-in. diameter are generally used for testing cohesive soils which contain a negligible amount of gravel.

b. Undisturbed Specimens. Generally, undisturbed specimens are prepared from undisturbed tube or chunk samples of a larger size than the test specimen. Core or thin-wall tube samples of relatively small diameter may be tested without further trimming except for squaring the ends, if the condition of the soil requires this procedure. Specimens must be handled carefully to prevent remolding, changes in cross section, or loss of moisture. To minimize disturbance caused by skin friction between samples and metal sampling tubes, the tubes should be cut into short lengths before ejecting the samples. Sample ejection should be accomplished with a smooth continuous, and fairly rapid motion in the same direction that the sample entered the tube. All specimens shall be prepared in a humid room to prevent evaporation of moisture. The specimen shall be prepared as follows:

(1) From the undisturbed sample cut a section somewhat larger in length and diameter than the desired specimen size.

It is generally desirable to prepare duplicate specimens for unconfined compression testing, and selection of material for testing should be made with this in mind.

(2) Carefully trim the specimen to the required diameter using a trimming frame and various trimming tools (see Fig. 7, Appendix X, TRIAXIAL COMPRESSION TESTS). Remove any small shells or pebbles encountered during the trimming operations. Carefully fill voids on the surface of the specimen with remolded soil obtained from the trimmings. Cut the specimen to the required length, using a miter box (see Fig. 8, Appendix X, TRIAXIAL COMPRESSION TESTS). Where the presence of pebbles or crumbling results in excessive irregularity at the ends, cap the specimens with a minimum thickness of plaster of Paris, hydrostone, or other support material. Care must be taken to insure that the ends of the specimen are parallel with each other and perpendicular to the vertical axis of the specimen.

(3) From the soil trimmings obtain 200 g of material for specific gravity and water content determinations (see Appendixes I, WATER CONTENT - GENERAL, and IV, SPECIFIC GRAVITY).

(4) Weigh the specimen to an accuracy of ±0.01 g for 1.4-in.-diameter specimens and ±0.1 g for 2.8-in.-diameter specimens. If specimens are to be capped, they should be weighed before capping.

(5) Measure the height of the specimen with calipers or a scale and the diameter with calipers or circumference measuring devices. If the specimen is cut to a fixed length in a miter box, the length of the miter box can be taken as the height of specimen for routine tests, and additional height measurements are not usually necessary. It is always advisable to measure the diameter of the specimen after trimming, even though specimens are cut to a nominal diameter in a trimming frame. Make all measurements to the nearest ±0.01 in. Determine the average initial diameter, D_o, of the specimen using the diameters measured at the top, D_t, center, D_c, and bottom, D_b, of the specimen, as follows:

$$D_o = \frac{D_t + 2D_c + D_b}{4}$$

(6) If the specimen is not tested immediately after preparation, precautions must be taken to prevent drying and consequent development of capillary stresses. When drying before or during the test is anticipated, the specimen may be covered with a thin coating of grease such as petrolatum. This coating cannot be used if the specimen is to be used in a subsequent remolded test.

c. Remolded Specimens. Remolded specimens usually are prepared in conjunction with tests made on undisturbed specimens after the latter has been tested to failure. The remolded specimens are tested to determine the effects of remolding on the shear strength of the soil. The remolded specimen should have the same water content as the undisturbed specimen in order to permit a comparison of the results of the tests on the two specimens. The remolded specimen shall be prepared as follows:

(1) Place the failed undisturbed specimen in a rubber membrane and knead it thoroughly with the fingers to assure complete remolding of the specimen. Take reasonable care to avoid entrapping air in the specimen and to obtain a uniform density.

(2) Remove the soil from the membrane and compact it in a cylindrical mold with inside dimensions identical with those of the undisturbed specimen. The compaction effort is not critical since the water contents of soils subjected to remolded tests are always considerably wetter than optimum. Care must be taken, however, to insure uniform density throughout the specimen. A thin coat of petrolatum on the inside of the molding cylinder will assist in the removal of the specimen after compaction.

(3) Carefully remove the specimen from the mold, preferably by means of a close fitting piston, and plane off the top of the specimen. The specimen is then ready for testing.

(4) Follow the steps outlined in paragraphs 3b(4) and 3b(5).

4. PROCEDURE. The procedure small consist of the following steps:

a. Record all identifying information for the sample such as project, boring number, visual classification, and other pertinent data on the data sheet (see Plate XI-1 which is a suggested form). The data sheet is also used for recording test observations described below.

b. Place the specimen in the loading device so that it is centered on the bottom platen; then adjust the loading device carefully so that the loading ram or upper platen barely is in contact with the specimen. If a proving ring is used for determining the axial load, contact of the platen and specimen is indicated by a slight deflection of the proving ring dial. Attach a dial indicator, sensitive to 0.001 in., to the loading ram to measure vertical deformation of the specimen. Record the initial reading of the dial indicator on the data sheet (Plate XI-1). Test the specimen at an axial strain rate of about 1 percent per minute. For very stiff or brittle materials which exhibit small deformations at failure, it may be desirable to test the specimen at a slower rate of strain. Observe and record the resulting load corresponding to increments of 0.3 percent strain for the first 3 percent of strain and in increments of 1 or 2 percent of strain thereafter. Stop the test when the axial load remains constant or when 20 percent axial strain has been produced.

c. Record the duration of the test, in minutes, to peak strength (time to failure), type of failure (shear or bulge), and a sketch of specimen after failure on the data sheet (Plate XI-2).

d. After the test, place the entire specimen or a representative portion thereof in a container of known weight and determine the water content of the specimen in accordance with Appendix I, WATER CONTENT - GENERAL.

5. COMPUTATIONS. The computations consist of the following steps:

a. From the observed data, compute and record on the data sheet (Plate XI-1) the water content, volume of solids, void ratio, degree of

saturation, and dry density, using the formulas presented in Appendix II, UNIT WEIGHTS, VOID RATIO, POROSITY, AND DEGREE OF SATURATION.

b. Compute and record on the data sheet the axial strain, the corrected area, and the compressive stress, at each increment of strain by using the following formulas:

$$\text{Axial strain, } \varepsilon = \frac{\Delta H}{H_o}$$

$$\text{Corrected area of specimen, } A_{corr}\text{, sq cm} = \frac{A_o}{1 - \varepsilon}$$

$$\text{Compressive stress, tons per sq ft} = \frac{P}{A_{corr}} \times 0.465$$

where

ΔH = change in height of specimen during test, cm

H_o = initial height of specimen, cm

A_o = initial area of specimen, sq cm

P = applied axial load, lb

6. PRESENTATION OF RESULTS. The results of the unconfined compression test shall be recorded on the report form shown as Plate XI-2. Pertinent information regarding the condition of the specimen, method of preparing the specimen, or any unusual features of each specimen (such as slickensides, stratification, shells, pebbles, roots, or brittleness) should be shown under "Remarks." The applied compressive stress shall be plotted versus the axial strain in Plate XI-2. The unconfined compressive strength, q_u, of the specimen shall be taken as the maximum or peak compressive stress. For tests continued to 20 percent strain without reduction of axial load occurring, the unconfined compressive strength as a rule shall be taken as the compressive stress at 15 percent strain.

Where the unconfined compressive strength of a specimen is also obtained after remolding, the sensitivity ratio, S_t, shall also be calculated and reported. The sensitivity ratio is defined as follows:

$$S_t = \frac{q_u \text{ (undisturbed)}}{q_u \text{ (remolded)}}.$$

7. POSSIBLE ERRORS. Following are possible errors that would cause inaccurate determinations of unconfined compressive strength:

a. Test not appropriate to type of soil.

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b. Specimen disturbed while trimming.

c. Loss of initial water content. A small change in water content can cause a larger change in the strength of a clay, so it is essential that every care be taken to protect the specimen against evaporation while trimming and measuring, during the test, and when remolding a specimen to determine the sensitivity.

d. Rate of strain or rate of loading too fast.

8. USE OF OTHER TYPES OF EQUIPMENT FOR UNDRAINED SHEAR STRENGTH DETERMINATIONS. Various other types of laboratory equipment, such as cone penetrometers and vane shear apparatus, may be used advantageously in the laboratory as a supplement to the basic unconfined compression test equipment for determining the undrained shear strength of cohesive soils. The use of these testing devices generally results in savings in cost and time. However, the devices should be used with caution until sufficient data and procedural details are established to assure their successful application. Use of such testing apparatus, as a rule,

should be preceded by careful correlations with the results of tests with the basic unconfined compression test equipment on the same type of soil, and correlations developed for a given type of soil should not be used indiscriminately for all soils.

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UNCONFINED COMPRESSION TEST

Date _____

Project _____

Boring No. _____ Sample No. _____

Specimen No.			Classification				
Tare No.			Height	H_o	in.	H_o	cm
Weight, w: Tare plus wet specimen			Average diameter	D_o	in.	D_o	cm
Tare plus dry specimen			Initial area	A_o	sq in.	A_o	sq cm
Water	W_w		Volume in cc = A_oH_o			V_o	
Tare			Volume of solids in cc = $W_s \div G_s$			V_s	
Wet specimen	W		Void ratio = $(V_o - V_s) \div V_s$			e_o	
Dry specimen	W_s		Saturation in % = $G_s w_o \div e_o$			S_o	%
Water content	w_o	%	Dry dens., lb/cu ft = $62.4\ (W_s \div V_o)$			γ_d	
Specific gravity of solids	G_s		Note: If only a portion of the specimen is used for the water content determination, $W_s = W \div \left(1 + \frac{w_o}{100}\right)$				

Elapsed Time min	Dial Reading 10^{-} in.	Cumulative Change, ΔH 10^{-} in.	Proving Ring Dial Reading 10^{-} in.	Axial Load, P lb	Axial Strain $\epsilon = \Delta H/H_o$	$1 - \epsilon$	$A_{corr} = A_o/(1-\epsilon)$ sq cm	Compr Stress tons/sq ft $0.465(P/A_{corr})$

Remarks _____

_____ Type Failure _____

Technician _____ Computed by _____ Checked by _____

PLATE XI-1 ENG FORM 1 JUN 65 3857

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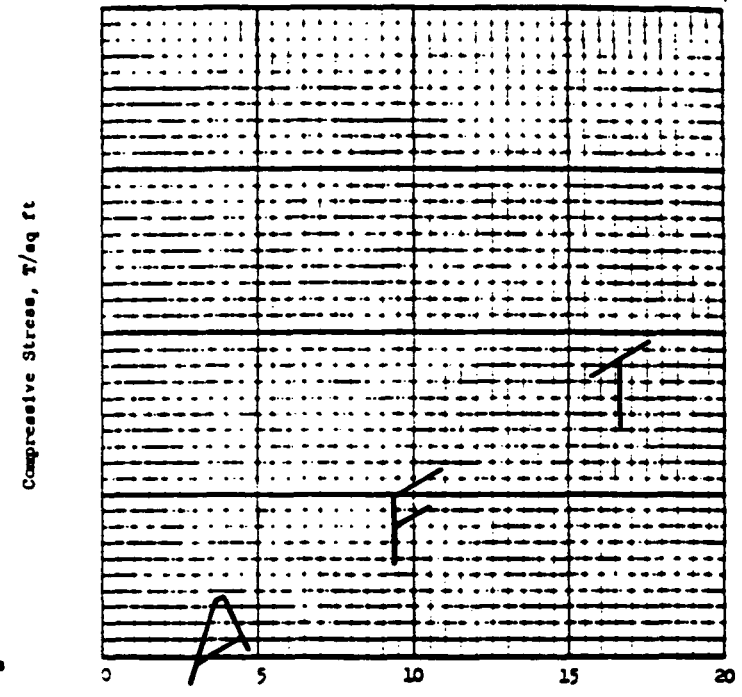
Failure Sketches



☐ Controlled stress
☐ Controlled strain

Test No.					
Type of specimen					
Water content	w_o	%	%	%	%
Void ratio	e_o				
Saturation	S_o	%	%	%	%
Dry density, lb/cu ft	γ_d				
Time to failure, min	t_f				
Unconfined compressive strength, T/sq ft	q_u				
Undrained shear strength, T/sq ft	s_u				
Sensitivity ratio	S_t				
Initial specimen diameter, in.	D_o				
Initial specimen height, in.	H_o				

Classification

LL	PL	PI	G_s

Remarks

Project	
Area	
Boring No.	Sample No.
Depth ft in	Date

UNCONFINED COMPRESSION TEST REPORT

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ENG FORM 1 JUN 68 3659

PLATE XI-2

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APPENDIX G

LABORATORY NOTEBOOK RECORDING PROCEDURES

International Technology Corporation

IT Analytical Services

Technology Development Laboratory Standard Operating Procedure

Title: Laboratory Notebook Recording Procedures

Prepared by: Patti B. Carswell Date: 2/7/91

Reviewed by: Bruce F. Wagner Date: 2/7/91
Technical Specialist

Patti B. Carswell Date: 2/7/91
Quality Control Coordinator

Janet M. Jones Date: 3-26-91
Director, Quality and Compliance, ITAS

Approved by: R. Miller Date: 3/27/91
Laboratory Director

Controlled Copy No: Uncontrolled Copy

Key Words: NOTEBOOK

Revision #	0					
Date	1-21-91				146	

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1.0 Purpose and Application

1.1 The purpose of this method is to describe the required methods of data entry in Technology Development Laboratory notebooks.

1.2 This procedure applies to laboratory notebooks used for project-specific and non-project-specific documentation.

1.3 The purpose of each entry in your notebook is to provide a complete record of your work, one that would enable a co-worker to repeat, if necessary, exactly what you did and produce the same results, without having to ask any questions.

2.0 References

2.1 Writing the Laboratory Notebook, Howard M. Kanare, 1985.

3.0 Associated SOPs and Applicable Methods

3.1 ITAS SOP No. TDL1503, "Analytical Logbook Recording Procedures."

4.0 Definitions

4.1 None

5.0 Procedure

5.1 Safety

5.1.1 All applicable safety and compliance guidelines set forth by IT Corporation and by federal, state, and local regulations must be followed during performance of this procedure. All work must be stopped in the event of a known or potential compromise to the health or safety of any ITAS Associate, and must be reported immediately to a laboratory supervisor.

5.1.2 All laboratory notebooks must be kept free of chemical contamination while being used on benchtops, in field settings, etc.

5.2 Summary

5.2.1 All laboratory notebooks are the property of the International Technology Corporation (IT) Technology Development Laboratory (TDL). It is assigned to you so that you may keep a complete, careful, chronological record of your work. The work which you do and the data which you enter in the notebook are confidential; they must not be disclosed to unauthorized persons. The notebook's security and maintenance are your responsibility. In case of damage, loss, or disappearance, report the

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5.0 Procedure (continued)

facts to your supervisor at once. When the notebook is filled or upon termination of your employment, it must be returned to the laboratory quality/operation files.

5.3 Procedure

5.3.1 All data is to be recorded directly into the notebook. Recording of original data on loose pieces of paper for later transcription into the logbook is to be avoided. Should loose paper be necessary for proper conduct of an experiment:

5.3.1.1 Write on the logbook page itself identification of what is affixed to that page.

5.3.1.2 Firmly affix the loose paper with clear tape

5.3.1.3 Initial and date over the edge of the tape.

5.3.2 All entries must be made in black ink. Red ink is reserved for Quality Control (QC) checking purposes only. Erasures, blacking out, or use of correction fluid is not permitted. If a mistake is made, draw a single line through the erroneous material and make a corrected entry, initial, and date the correction.

5.3.3 It is necessary to fill each page and keep the sequence of entries in chronological order. Several pages may be reserved for a particular experiment. However, if the continuity of pages for a particular experiment is broken for lack of reserved space, notations will be made on both sides of the break. The unused balance of a page will be cancelled by a diagonal line. Spaces intentionally left blank in tables or logs will contain horizontal lines.

5.3.4 Stock or standard solutions must reference:

5.3.4.1 Source
5.3.4.2 Lot number
5.3.4.3 Date received
5.3.4.4 Notebook and page numbers whenever available.

5.3.5 When reference is made to samples, the TDL sample number must be used. Additional sample identification may be offered, but not to the exclusion of the TDL sample number.

5.3.6 A co-worker performs a QC check on your calculations by recalculating 20 percent and verifying the formula used. Have him make a check in red ink beside each answer which was recalculated and sign and date

5.0 Procedure (continued)

calculations that lead to the generation of a result which is reported to the client either verbally or in writing. Any values which have not had a 20 percent QC check (one of every five calculations has been checked) are considered "preliminary" and will be marked as such on any material leaving the TDL lab. If an error is found during the 20 percent check, then a 100 percent QC check will be performed.

5.3.7 If one of your co-workers has witnessed an experiment you have conducted, to an extent that enables him to state of his own knowledge what you did and what results you secured, have him sign and date the notebook page(s) as "Witnessed and understood by." If the experiment seems to you to be of sufficient importance (i.e., is potentially patentable), arrange to have it witnessed for content and date of entry.

5.4 Project Documentation Requirements

5.4.1 Every page of the notebook will contain project name, project number, date, and initials of persons entering data. Each project will then be described by the following entries:

5.4.1.1 Objective - briefly describe the planned experiment and the expected or desired result.

5.4.1.2 Plan - give an overview of what you intend to do.

5.4.1.3 Calibrations and Standards - list frequency of calibration, acceptance limits, and concentrations.

5.4.1.4 Analytical Methods - state SOP, standard reference or give a brief description.

5.4.1.5 Experimental Set-ups - sketch and describe the set-up.

5.4.1.6 Data and Observations - provide tables including units and space for observations within or below.

5.4.1.7 Results - include formula and calculations which are necessary to produce results from raw data.

5.4.1.8 Conclusion - how objective was met and any interpretation of results.

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6.0 Nonconformance and Corrective Action

6.1 A nonconformance is a deficiency in procedure sufficient to render the quality of an item unacceptable or indeterminate or any event which is beyond the limits documented and established for laboratory operation. A nonconformance may include data recording errors, transcription errors, and failure to document. A nonconformance memo associated with this procedure will be filed with the QC Coordinator.

7.0 Records Management

7.1 TDL Notebooks are the property of IT Corporation.

7.2 Document control of TDL Notebooks is handled by the QC Coordinator (QCC). The QCC will issue all notebooks. All completed notebooks will be returned to the QCC.

7.3 All returned Laboratory Notebooks are filed in TDL Central Files.

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APPENDIX H

ANALYTICAL LOGBOOK RECORDING PROCEDURES

International Technology Corporation

IT Analytical Services

Technology Development Laboratory Standard Operating Procedure

Title: Analytical Logbook Recording Procedures

Prepared by: Patti B. Carswell Date: 2/7/91

Reviewed by: Bruce F. Wagner Date: 2/7/91
Technical Specialist

Patti B. Carswell Date: 2/7/91
Quality Control Coordinator

Janet M. Jones Date: 3-26-91
Director, Quality and Compliance, ITAS

Approved by: R. S. Miller Date: 6/7/91
Laboratory Director

Controlled Copy No: Uncontrolled Copy

Key Words: Logbook, Notebook

Revision #	0					
Date	1-21-91				152	

Technology Development Laboratory
304 Directors Drive ▪ Knoxville, Tennessee 37923 ▪ (615) 690-3211 ▪ FAX (615) 694-9573
IT Corporation is a wholly owned Subsidiary of International Technology Corporation.

1.0 Purpose and Application

1.1 The purpose of this method is to describe the required methods of data entry in Technology Development Analytical Logbooks.

1.2 This procedure applies to analytical logbooks such as instrument injection logbooks, maintenance logbooks, and balance logs.

2.0 References

2.1 Writing the Laboratory Notebook, Howard M. Kanare, 1985.

3.0 Associated SOPs and Applicable Methods

3.1 ITAS SOP No. TDL1504, "Laboratory Notebook Recording Procedures."

4.0 Definitions

4.1 None

5.0 Procedure

5.1 Safety

5.1.1 All applicable safety and compliance guidelines set forth by IT Corporation and by federal, state, and local regulations must be followed during performance of this procedure. All work must be stopped in the event of a known or potential compromise to the health or safety of any ITAS Associate, and must be reported immediately to a laboratory supervisor.

5.1.2 All analytical logbooks must be kept free of chemical contamination while being used on benchtops, in field settings, etc.

5.2 Summary

5.2.1 All logbooks are the property of the International Technology Corporation (IT) Technology Development Laboratory (TDL). It is assigned to you so that you may keep a complete, careful, chronological record of your work. The work which you do and the data which you enter in this book are confidential; they must not be disclosed to unauthorized persons. The logbook's security and maintenance are your responsibility. In case of damage, loss, or

5.0 Procedure (continued)

disappearance, report the facts to your supervisor at once. When the logbook is filled, or upon termination of your employment, it must be returned to the laboratory quality/operation files.

5.3 Procedure

5.3.1 Briefly define in the front pages of the book what type of log is contained within. Definitions of column headings, references, and acceptance limits will be addressed on the first pages as well.

5.3.2 All entries are to be recorded directly into the logbook. Recording of original data on loose pieces of paper for later transcription into the logbook is to be avoided. Should loose paper be necessary for proper conduct of an experiment:

5.3.2.1 Write on the logbook page itself identification of what is affixed to that page

5.3.2.2 Firmly affix the loose paper with clear tape

5.3.2.3 Initial and date over the edge of the tape.

5.3.3 All entries must be made in black ink. Red ink is reserved for Quality Control (QC) checking purposes only. Erasures, blacking out, or use of correction fluid is not permitted. If a mistake is made, draw a single line through the erroneous material and make a corrected entry, initial, and date the correction.

5.3.4 It is necessary to fill each page and keep the sequence of entries in chronological order. Any unused section of a page will be cancelled with a diagonal line. Spaces intentionally left blank in tables or logs will contain horizontal lines.

5.3.5 When reference is made to samples, the TDL sample number will be used. Additional sample identification may be offered, but not to the exclusion of the TDL sample number.

5.3.6 Use a ruler to draw lines defining columns. Label columns including units when appropriate. Injection logs, balance logs, and other similar logs will include columns for the operators' initials and date.

5.3.7 Each entry in an analytical logbook is to be initialed and dated. The "Completed by" is signed by the last person to make entry on a given page and indicates that the page has been checked for completeness of entries.

6.0 Nonconformance and Corrective Action

6.1 A nonconformance is a deficiency in procedure sufficient to render the quality of an item unacceptable or indeterminate or any event which is beyond the limits documented and established for laboratory operation. A nonconformance may include data recording errors, transcription errors, and failure to document. A nonconformance memo associated with this procedure will be filed with the QC Coordinator.

7.0 Records Management

7.1 TDL Analytical Logbooks are the property of IT Corporation.

7.2 Document control of TDL Logbooks is handled by the QC Coordinator (QCC). The QCC will issue all notebooks. All completed logbooks will be returned to the QCC.

7.3 All returned Laboratory Logbooks are filed in TDL Central Files.

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APPENDIX I

APPLICABLE MSDSs

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1718

ATTN: SAFETY DIRECTOR
IT CORPORATION
REGIONAL ACCOUNTING
312 DIRECTORS DRIVE
KNOXVILLE TN 37923
DWAYNE ROOT

DATE: 04/02/87
CUST # 427195 P.O. # 307376

MATERIAL SAFETY DATA SHEET

IDENTIFICATION

PRODUCT # 29899-9 NAME: 1,1,1-TRICHLOROETHANE, ANHYDROUS, UNINHIBITED, 99+%

CAS # 71-55-6

TOXICITY HAZARDS

RTECS # KJ2975000
ETHANE, 1,1,1-TRICHLORO-

IRRITATION DATA

EYE-MAN 450 PPM/8H	BJIMAG 28,286,71
SKN-RBT 5 GM/12D-I MLD	AIHAAP 19,353,58
SKN-RBT 500 MG/24H MOD	28ZPAK -,28,72
EYE-RBT 100 MG MLD	AIHAAP 19,353,58
EYE-RBT 2 MG/24H SEV	28ZPAK -,28,72

TOXICITY DATA

IHL-MAN LCLO:27 GM/M3/10M	JOCMA7 8,358,66
ORL-RAT LD50:10300 MG/KG	NTIS** PB257-185
IHL-RAT LC50:18000 PPM/4H	28ZPAK -,28,72
IPR-RAT LD50:5100 MG/KG	NTIS** PB257-185
ORL-MUS LD50:11240 MG/KG	NTIS** PB257-185
IHL-MUS LC50:3911 PPM/2H	SAIGBL 13,226,71
IPR-MUS LD50:4700 MG/KG	TXAPA9 13,287,68
ORL-DOG LD50:750 MG/KG	FMCHA2 -,C242,83
IPR-DOG LD50:3100 MG/KG	TXAPA9 10,119,67
ORL-RBT LD50:5660 MG/KG	AIHAAP 19,353,58
ORL-GPG LD50:9470 MG/KG	AIHAAP 19,353,58

REVIEWS, STANDARDS, AND REGULATIONS

CARCINOGENIC REVIEW:ANIMAL INDEFINITE IMEMDT 20,515,79
MSHA STANDARD-AIR:TWA 350 PPM (1900 MG/M3) DTLVS* 3,161,71
OSHA STANDARD-AIR:TWA 350 PPM FEREAC 39,23540,74
NIOSH REL TO 1,1,1-TRICHLOROETHANE-AIR:CL 350 PPM/15M MMWR** 34(1S), 29S,85
EPA GENETIC TOXICOLOGY PROGRAM, JANUARY 1984
REPORTED IN EPA TSCA INVENTORY, 1983
EPA TSCA 8(A) PRELIMINARY ASSESSMENT INFORMATION, FINAL RULE FEREAC 47,26992,82
EPA TSCA SECTION 8(E) STATUS REPORT 8EHQ-0283-0471 S;8EHQ-0980-0365; 8EHQ-0982-0457
EPA TSCA SECTION 8(E) STATUS REPORT 8EHQ-0979-0310
NIOSH CURRENT INTELLIGENCE BULLETIN 27, 1978
"NIOSH MANUAL OF ANALYTICAL METHODS, 3RD ED." SEE: METHOD 1003
NCI CARCINOGENESIS BIOASSAY (GAVAGE);RESULTS INDEFINITE:MOUSE,RAT (NCITR* NCI-CG-TR-3,77)
NCI CARCINOGENESIS BIOASSAY COMPLETED;RESULTS NEGATIVE:MOUSE, RAT (NCITR* NCI-CG-TR-3,77)
NTP CARCINOGENESIS STUDIES; TEST COMPLETED, JUNE 1986
MEETS CRITERIA FOR PROPOSED OSHA MEDICAL RECORDS RULE FEREAC 47,30420, 82

ONLY SELECTED REGISTRY OF TOXIC EFFECTS OF CHEMICAL SUBSTANCES (RTECS) DATA IS PRESENTED HERE. SEE ACTUAL ENTRY IN RTECS FOR COMPLETE INFORMATION.

HEALTH HAZARD DATA

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ACUTE EFFECTS

MAY BE HARMFUL BY INHALATION, INGESTION, OR SKIN ABSORPTION.
VAPOR OR MIST IS IRRITATING TO THE EYES, MUCOUS MEMBRANES AND UPPER RESPIRATORY TRACT.

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aldrich chemical co.
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CATALOG # 29899-9 NAME: 1,1,1-TRICHLOROETHANE, ANHYDROUS, UNINHIBITED, 99+%

CAUSES SKIN IRRITATION.
EXPOSURE TO AND/OR CONSUMPTION OF ALCOHOL
MAY INCREASE TOXIC EFFECTS.
PROLONGED EXPOSURE CAN CAUSE:
NARCOTIC EFFECT.
DERMATITIS

CHRONIC EFFECTS
DAMAGE TO THE LIVER
DAMAGE TO THE KIDNEYS

FIRST AID
IN CASE OF CONTACT, IMMEDIATELY FLUSH EYES WITH COPIOUS AMOUNTS OF WATER FOR AT LEAST 15 MINUTES.
IN CASE OF CONTACT, IMMEDIATELY WASH SKIN WITH SOAP AND COPIOUS AMOUNTS OF WATER.
IF INHALED, REMOVE TO FRESH AIR. IF NOT BREATHING GIVE ARTIFICIAL RESPIRATION, PREFERABLY MOUTH-TO-MOUTH. IF BREATHING IS DIFFICULT, GIVE OXYGEN.
CALL A PHYSICIAN.
WASH CONTAMINATED CLOTHING BEFORE REUSE.

PHYSICAL DATA

MELTING POINT: -35 C
BOILING POINT: 74 C TO 76 C
SPECIFIC GRAVITY: 1.338

FIRE AND EXPLOSION HAZARD DATA

FLASH POINT: NONE

EXTINGUISHING MEDIA
WATER SPRAY.
CARBON DIOXIDE, DRY CHEMICAL POWDER, ALCOHOL OR POLYMER FOAM.

SPECIAL FIRE FIGHTING PROCEDURES
WEAR SELF-CONTAINED BREATHING APPARATUS AND PROTECTIVE CLOTHING TO PREVENT CONTACT WITH SKIN AND EYES.

UNUSUAL FIRE AND EXPLOSION HAZARDS
EMITS TOXIC FUMES UNDER FIRE CONDITIONS.

REACTIVITY DATA

INCOMPATIBILITIES
STRONG OXIDIZING AGENTS
ALUMINUM
AND ITS ALLOYS.
MAGNESIUM
ZINC
STRONG BASES

REACTS VIOLENTLY WITH:
POTASSIUM
SODIUM

HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS
TOXIC FUMES OF:
CARBON MONOXIDE, CARBON DIOXIDE
HYDROGEN CHLORIDE GAS
PHOSGENE GAS

SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED
WEAR SELF-CONTAINED BREATHING APPARATUS, RUBBER BOOTS AND HEAVY RUBBER GLOVES.
COVER WITH DRY LIME OR SODA ASH, PICK UP, KEEP IN A CLOSED CONTAINER AND HOLD FOR WASTE DISPOSAL.
VENTILATE AREA AND WASH SPILL SITE AFTER MATERIAL PICKUP IS COMPLETE.

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MATERIAL SAFETY DATA SHEET PAGE: 3

CATALOG # 29899-9 NAME: 1,1,1-TRICHLOROETHANE, ANHYDROUS, UNINHIBITED, 99+%

WASTE DISPOSAL METHOD
DISSOLVE OR MIX THE MATERIAL WITH A COMBUSTIBLE SOLVENT AND BURN IN A CHEMICAL INCINERATOR EQUIPPED WITH AN AFTERBURNER AND SCRUBBER.

OBSERVE ALL FEDERAL, STATE & LOCAL LAWS.

--- PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE ----

CHEMICAL SAFETY GOGGLES.
RUBBER GLOVES
OSHA/MSHA-APPROVED RESPIRATOR.
SAFETY SHOWER AND EYE BATH.
MECHANICAL EXHAUST REQUIRED.
AVOID CONTACT AND INHALATION.
DO NOT GET IN EYES, ON SKIN, ON CLOTHING.
WASH THOROUGHLY AFTER HANDLING.
IRRITANT.
KEEP TIGHTLY CLOSED.
STORE IN A COOL DRY PLACE.

---------- ADDITIONAL PRECAUTIONS AND COMMENTS --------

ADDITIONAL INFORMATION
1,1,1-TRICHLOROETHANE HAS BEEN REPORTED TO REACT VIOLENTLY WITH ACETONE, NITRITES, AND OXYGEN.

THE ABOVE INFORMATION IS BELIEVED TO BE CORRECT BUT DOES NOT PURPORT TO BE ALL INCLUSIVE AND SHALL BE USED ONLY AS A GUIDE. ALDRICH SHALL NOT BE HELD LIABLE FOR ANY DAMAGE RESULTING FROM HANDLING OR FROM CONTACT WITH THE ABOVE PRODUCT. SEE REVERSE SIDE OF INVOICE OR PACKING SLIP FOR ADDITIONAL TERMS AND CONDITIONS OF SALE.

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J.T.BAKER INC. 222 RED SCHOOL LANE, PHILLIPSBURG, NJ 08865
M A T E R I A L S A F E T Y D A T A S H E E T
24-HOUR EMERGENCY TELEPHONE -- (201) 859-2151
CHEMTREC # (800) 424-9300 -- NATIONAL RESPONSE CENTER # (800) 424-8802

3660 D04 NITRIC ACID PAGE: 1
EFFECTIVE: 05/01/89 ISSUED: 05/16/89

J.T.BAKER INC., 222 RED SCHOOL LANE, PHILLIPSBURG, NJ 08865

SECTION I - PRODUCT IDENTIFICATION

PRODUCT NAME: NITRIC ACID
COMMON SYNONYMS: HYDROGEN NITRATE; AZOTIC ACID
CHEMICAL FAMILY: INORGANIC ACIDS
FORMULA: HNO3
FORMULA WT.: 63.01
CAS NO.: 7697-37-2
NIOSH/RTECS NO.: QU5775000
PRODUCT USE: LABORATORY REAGENT
PRODUCT CODES: 4801,9597,5113,9602,5371,9598,9605,9600,9615,6901,9606,9601

PRECAUTIONARY LABELING

BAKER SAF-T-DATA* SYSTEM

HEALTH	-	3	SEVERE (POISON)
FLAMMABILITY	-	0	NONE
REACTIVITY	-	3	SEVERE (OXIDIZER)
CONTACT	-	4	EXTREME (CORROSIVE)

LABORATORY PROTECTIVE EQUIPMENT

GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES

U.S. PRECAUTIONARY LABELING

POISON DANGER

SPILLAGE MAY CAUSE FIRE OR LIBERATE DANGEROUS GAS. HARMFUL IF INHALED AND MAY CAUSE DELAYED LUNG INJURY. STRONG OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE. LIQUID AND VAPOR CAUSE SEVERE BURNS. MAY BE FATAL IF SWALLOWED OR INHALED.
KEEP FROM CONTACT WITH CLOTHING AND OTHER COMBUSTIBLE MATERIALS. DO NOT STORE NEAR COMBUSTIBLE MATERIALS. DO NOT GET IN EYES, ON SKIN, ON CLOTHING. DO NOT BREATHE VAPOR. KEEP IN TIGHTLY CLOSED CONTAINER. USE WITH ADEQUATE VENTILATION. WASH THOROUGHLY AFTER HANDLING. IN CASE OF FIRE, USE WATER SPRAY. IN CASE OF SPILL, NEUTRALIZE WITH SODA ASH OR LIME.

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PRECAUTIONARY LABELING (CONTINUED)

INTERNATIONAL LABELING

VOID CONTACT WITH EYES. AFTER CONTACT WITH SKIN, WASH IMMEDIATELY WITH LENTY OF WATER. KEEP CONTAINER TIGHTLY CLOSED.

AF-T-DATA* STORAGE COLOR CODE: YELLOW (REACTIVE)

SECTION II - COMPONENTS

OMPONENT	CAS NO.	WEIGHT %	OSHA/PEL	ACGIH/TLV
ITRIC ACID	7697-37-2	65-71	2 PPM	2 PPM
ATER	7732-18-5	29-35	N/E	N/E

SECTION III - PHYSICAL DATA

OILING POINT: 121 C (249 F)
(AT 760 MM HG)

VAPOR PRESSURE (MMHG): 9
(20 C)

ELTING POINT: -42 C (-43 F)
(AT 760 MM HG)

VAPOR DENSITY (AIR=1): N/A

PECIFIC GRAVITY: 1.41
(H2O=1)

EVAPORATION RATE: N/A

OLUBILITY(H2O): COMPLETE (100%)

% VOLATILES BY VOLUME: 100
(21 C)

H: N/A

OOR THRESHOLD (P.P.M.): N/A

PHYSICAL STATE: LIQUID

OEFFICIENT WATER/OIL DISTRIBUTION: N/A

PPEARANCE & ODOR: CLEAR, COLORLESS LIQUID. SUFFOCATING ODOR.

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SECTION IV - FIRE AND EXPLOSION HAZARD DATA

FLASH POINT (CLOSED CUP): N/A NFPA 704M RATING: 3-0-0 OXY

AUTOIGNITION TEMPERATURE: N/A

FLAMMABLE LIMITS: UPPER - N/A LOWER - N/A

FIRE EXTINGUISHING MEDIA
USE WATER SPRAY.

SPECIAL FIRE-FIGHTING PROCEDURES
FIREFIGHTERS SHOULD WEAR PROPER PROTECTIVE EQUIPMENT AND SELF-CONTAINED BREATHING APPARATUS WITH FULL FACEPIECE OPERATED IN POSITIVE PRESSURE MODE. MOVE EXPOSED CONTAINERS FROM FIRE AREA IF IT CAN BE DONE WITHOUT RISK. USE WATER TO KEEP FIRE-EXPOSED CONTAINERS COOL; DO NOT GET WATER INSIDE CONTAINERS.

UNUSUAL FIRE & EXPLOSION HAZARDS
STRONG OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE. REACTS WITH MOST METALS TO PRODUCE HYDROGEN GAS, WHICH CAN FORM AN EXPLOSIVE MIXTURE WITH AIR. A VIOLENT EXOTHERMIC REACTION OCCURS WITH WATER. SUFFICIENT HEAT MAY BE PRODUCED TO IGNITE COMBUSTIBLE MATERIALS.

TOXIC GASES PRODUCED
OXIDES OF NITROGEN, HYDROGEN

EXPLOSION DATA-SENSITIVITY TO MECHANICAL IMPACT
NONE IDENTIFIED.

EXPLOSION DATA-SENSITIVITY TO STATIC DISCHARGE
NONE IDENTIFIED.

SECTION V - HEALTH HAZARD DATA

THRESHOLD LIMIT VALUE (TLV/TWA): 5 MG/M3 (2 PPM)

SHORT-TERM EXPOSURE LIMIT (STEL): 10 MG/M3 (4 PPM)

PERMISSIBLE EXPOSURE LIMIT (PEL): 5 MG/M3 (2 PPM)

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SECTION V - HEALTH HAZARD DATA (CONTINUED)

OXICITY OF COMPONENTS

NTRAPERITONEAL MOUSE LD50 FOR WATER 190 G/KG
NTRAVENOUS MOUSE LD50 FOR WATER 25 G/KG
ARCINOGENICITY: NTP: NO IARC: NO Z LIST: NO OSHA REG: NO

ARCINOGENICITY
NONE IDENTIFIED.

EPRODUCTIVE EFFECTS
NONE IDENTIFIED.

FFECTS OF OVEREXPOSURE

INHALATION: SEVERE IRRITATION OR BURNS OF RESPIRATORY SYSTEM, COUGHING, DIFFICULT BREATHING, CHEST PAINS, PULMONARY EDEMA, LUNG INFLAMMATION, UNCONSCIOUSNESS, AND MAY BE FATAL

SKIN CONTACT: SEVERE IRRITATION OR BURNS

EYE CONTACT: SEVERE IRRITATION OR BURNS

SKIN ABSORPTION: NONE IDENTIFIED

INGESTION: NAUSEA, VOMITING, SEVERE BURNS, ULCERATION - MOUTH, THROAT, STOMACH, AND MAY BE FATAL

CHRONIC EFFECTS: DAMAGE TO LUNGS, TEETH

ARGET ORGANS
EYES, SKIN, MUCOUS MEMBRANES, RESPIRATORY SYSTEM, LUNGS, TEETH, GI TRACT

EDICAL CONDITIONS GENERALLY AGGRAVATED BY EXPOSURE
DAMAGED SKIN, EYE DISORDERS, CARDIOPULMONARY DISEASE, LUNG DISEASE

RIMARY ROUTES OF ENTRY
INHALATION, INGESTION, EYE CONTACT, SKIN CONTACT

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J.T.BAKER INC. 222 RED SCHOOL LANE, PHILLIPSBURG, NJ 08865

MATERIAL SAFETY DATA SHEET

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SECTION V - HEALTH HAZARD DATA (CONTINUED)

EMERGENCY AND FIRST AID PROCEDURES

INGESTION: CALL A PHYSICIAN. IF SWALLOWED, DO NOT INDUCE VOMITING. IF CONSCIOUS, GIVE WATER, MILK, OR MILK OF MAGNESIA.

INHALATION: IF INHALED, REMOVE TO FRESH AIR. IF NOT BREATHING, GIVE ARTIFICIAL RESPIRATION. IF BREATHING IS DIFFICULT, GIVE OXYGEN.

SKIN CONTACT: IN CASE OF CONTACT, IMMEDIATELY FLUSH SKIN WITH PLENTY OF WATER FOR AT LEAST 15 MINUTES WHILE REMOVING CONTAMINATED CLOTHING AND SHOES. WASH CLOTHING BEFORE RE-USE.

EYE CONTACT: IN CASE OF EYE CONTACT, IMMEDIATELY FLUSH WITH PLENTY OF WATER FOR AT LEAST 15 MINUTES.

SARA/TITLE III HAZARD CATEGORIES AND LISTS

ACUTE: YES CHRONIC: YES FLAMMABILITY: YES PRESSURE: NO REACTIVITY: NO

EXTREMELY HAZARDOUS SUBSTANCE:	YES	CONTAINS NITRIC ACID (RQ = 1,000 LBS, TPQ = 1,000 LBS)
CERCLA HAZARDOUS SUBSTANCE:	YES	CONTAINS NITRIC ACID (RQ = 1000 LBS)
TOXIC CHEMICALS:	YES	CONTAINS NITRIC ACID
GENERIC CLASS:		C16
TSCA INVENTORY:	YES	

SECTION VI - REACTIVITY DATA

STABILITY: STABLE HAZARDOUS POLYMERIZATION: WILL NOT OCCUR

CONDITIONS TO AVOID: HEAT, LIGHT, MOISTURE

INCOMPATIBLES: STRONG BASES, CARBONATES, SULFIDES, CYANIDES, COMBUSTIBLE MATERIALS, ORGANIC MATERIALS, STRONG REDUCING AGENTS, MOST COMMON METALS, POWDERED METALS, CARBIDES, AMMONIUM HYDROXIDE, WATER, ALCOHOLS

DECOMPOSITION PRODUCTS: OXIDES OF NITROGEN, HYDROGEN

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SECTION VII - SPILL & DISPOSAL PROCEDURES

STEPS TO BE TAKEN IN THE EVENT OF A SPILL OR DISCHARGE
WEAR SELF-CONTAINED BREATHING APPARATUS AND FULL PROTECTIVE CLOTHING. STOP LEAK IF YOU CAN DO SO WITHOUT RISK. VENTILATE AREA. NEUTRALIZE SPILL WITH SODA ASH OR LIME. WITH CLEAN SHOVEL, CAREFULLY PLACE MATERIAL INTO CLEAN, DRY CONTAINER AND COVER; REMOVE FROM AREA. FLUSH SPILL AREA WITH WATER.
KEEP COMBUSTIBLES (WOOD, PAPER, OIL, ETC.) AWAY FROM SPILLED MATERIAL.

J. T. BAKER NEUTRASORB(R) OR TEAM* 'LOW NA+' ACID NEUTRALIZERS ARE RECOMMENDED FOR SPILLS OF THIS PRODUCT.

DISPOSAL PROCEDURE
DISPOSE IN ACCORDANCE WITH ALL APPLICABLE FEDERAL, STATE, AND LOCAL ENVIRONMENTAL REGULATIONS.

EPA HAZARDOUS WASTE NUMBER: D001, D002 (IGNITABLE, CORROSIVE WASTE)

SECTION VIII - INDUSTRIAL PROTECTIVE EQUIPMENT

VENTILATION: USE GENERAL OR LOCAL EXHAUST VENTILATION TO MEET TLV REQUIREMENTS.

RESPIRATORY PROTECTION: RESPIRATORY PROTECTION REQUIRED IF AIRBORNE CONCENTRATION EXCEEDS TLV. AT CONCENTRATIONS UP TO 100 PPM, A CHEMICAL CARTRIDGE RESPIRATOR WITH ACID CARTRIDGE IS RECOMMENDED. ABOVE THIS LEVEL, A SELF-CONTAINED BREATHING APPARATUS IS ADVISED.

EYE/SKIN PROTECTION: SAFETY GOGGLES AND FACE SHIELD, UNIFORM, PROTECTIVE SUIT, NEOPRENE GLOVES ARE RECOMMENDED.

SECTION IX - STORAGE AND HANDLING PRECAUTIONS

SAF-T-DATA* STORAGE COLOR CODE: YELLOW (REACTIVE)

STORAGE REQUIREMENTS
KEEP CONTAINER TIGHTLY CLOSED. STORE SEPARATELY AND AWAY FROM FLAMMABLE AND COMBUSTIBLE MATERIALS. ISOLATE FROM INCOMPATIBLE MATERIALS. KEEP PRODUCT OUT OF LIGHT.

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SECTION X - TRANSPORTATION DATA AND ADDITIONAL INFORMATION

OMESTIC (D.O.T.)

PROPER SHIPPING NAME: NITRIC ACID (OVER 40%)
HAZARD CLASS: OXIDIZER
N/NA: UN2031 REPORTABLE QUANTITY: 1000 LBS.
ABELS: OXIDIZER, CORROSIVE
EGULATORY REFERENCES: 49CFR 172.101; 173.268

NTERNATIONAL (I.M.O.)

PROPER SHIPPING NAME: NITRIC ACID
HAZARD CLASS: 8 I.M.O. PAGE: 8107
N: UN2031 MARINE POLLUTANTS: NO PACKAGING GROUP: II
ABELS: CORROSIVE
EGULATORY REFERENCES: 49CFR 172.102; PART 176; IMO

IR (I.C.A.O.)

ROPER SHIPPING NAME: NITRIC ACID
AZARD CLASS: 8
N: UN2031 PACKAGING GROUP: II
ABELS: CORROSIVE
EGULATORY REFERENCES: 49CFR 172.101; 173.6; PART 175; ICAO/IATA

.S. CUSTOMS HARMONIZATION NUMBER: 2808000000

/A = NOT APPLICABLE OR NOT AVAILABLE
/E = NOT ESTABLISHED
--
HE INFORMATION IN THIS MATERIAL SAFETY DATA SHEET MEETS THE
EQUIREMENTS OF THE UNITED STATES OCCUPATIONAL SAFETY AND HEALTH ACT AND
EGULATIONS PROMULGATED THEREUNDER (29 CFR 1910.1200 ET. SEQ.) AND THE
ANADIAN WORKPLACE HAZARDOUS MATERIALS INFORMATION SYSTEM. THIS DOCUMENT
S INTENDED ONLY AS A GUIDE TO THE APPROPRIATE PRECAUTIONARY HANDLING OF
HE MATERIAL BY A PERSON TRAINED IN, OR SUPERVISED BY A PERSON TRAINED
N, CHEMICAL HANDLING. THE USER IS RESPONSIBLE FOR DETERMINING THE
RECAUTIONS AND DANGERS OF THIS CHEMICAL FOR HIS OR HER PARTICULAR
PPLICATION. DEPENDING ON USAGE, PROTECTIVE CLOTHING INCLUDING EYE AND
ACE GUARDS AND RESPIRATORS MUST BE USED TO AVOID CONTACT WITH MATERIAL
R BREATHING CHEMICAL VAPORS/FUMES.
XPOSURE TO THIS PRODUCT MAY HAVE SERIOUS ADVERSE HEALTH EFFECTS. THIS
HEMICAL MAY INTERACT WITH OTHER SUBSTANCES. SINCE THE POTENTIAL USES

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RE SO VARIED, BAKER CANNOT WARN OF ALL OF THE POTENTIAL DANGERS OF USE
R INTERACTION WITH OTHER CHEMICALS OR MATERIALS. BAKER WARRANTS THAT
HE CHEMICAL MEETS THE SPECIFICATIONS SET FORTH ON THE LABEL.
AKER DISCLAIMS ANY OTHER WARRANTIES, EXPRESSED OR IMPLIED WITH REGARD
O THE PRODUCT SUPPLIED HEREUNDER, ITS MERCHANTABILITY OR ITS FITNESS
OR A PARTICULAR PURPOSE.
HE USER SHOULD RECOGNIZE THAT THIS PRODUCT CAN CAUSE SEVERE INJURY AND
VEN DEATH, ESPECIALLY IF IMPROPERLY HANDLED OR THE KNOWN DANGERS OF USE
RE NOT HEEDED. READ ALL PRECAUTIONARY INFORMATION. AS NEW DOCUMENTED
ENERAL SAFETY INFORMATION BECOMES AVAILABLE, BAKER WILL PERIODICALLY
EVISE THIS MATERIAL SAFETY DATA SHEET. IF YOU HAVE ANY QUESTIONS,
LEASE CALL CUSTOMER SERVICE (1-800-JTBAKER) FOR ASSISTANCE.
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