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# TREATABILITY STUDY WORK PLAN FOR OPERABLE UNIT 4 OCTOBER 1991

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REPORT

# TREATABILITY STUDY WORK PLAN FOR OPERABLE UNIT 4

FERNALD ENVIRONMENTAL MANAGEMENT PROJECT
FERNALD, OHIO

REMEDIAL INVESTIGATION and FEASIBILITY STUDY

OCTOBER 1991

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 |
| ASTM | American Society of Testing and Materials |
| CERCLA | Comprehensive Environmental Response, Compensation, and Liability Act |
| CIS | Characterization Investigation Study |
| CLP | Contract Laboratory Program |
| CLRDL | contract laboratory required detection limit |
| COD | chemical oxygen demand |
| DLRL | derived leachate reference level |
| DOE | U.S. Department of Energy |
| DQO | data quality objective |
| EDTA | ethylenediaminetetraacetic acid |
| EPA | U.S. Environmental Protection Agency |
| FEMP | Fernald Environmental Management Project |
| FFCA | Federal Facilities Compliance Agreement |
| FMPC | Feed Materials Production Center |
| FS | feasibility study |
| GC | gas chromatography |
| HEPA | high efficiency particulate air |
| HSL | Hazardous Substances List |
| IC | ion chromatography |
| ICP | inductively coupled plasma |

## LIST OF ACRONYMS
(Continued)

| | |
|---|---|
| ITAS | IT Analytical Services |
| MCL | maximum contaminant level |
| MS | mass spectrometry |
| MTCLP | modified toxicity characteristic leaching procedures |
| QA | quality assurance |
| QAPP | Quality Assurance Project Plan |
| QC | quality control |
| QCC | Quality Control Coordinator |
| PCT | (nuclear waste) product consistency test |
| PRG | preliminary remediation goals |
| RA | remedial action |
| RAO | remedial action objective |
| RCRA | Resource Conservation and Recovery Act |
| RfD | toxicity based reference doses |
| RG | remediation goals |
| RI | remedial investigation |
| SAP | Sampling and Analysis Plan |
| SOP | Standard Operating Procedure |
| TBC | to be considered |
| TCLP | toxicity characteristic leaching procedure |
| TDL | Technology Development Laboratory |
| UCS | unconfined compression strength |
| WL | working level |
| WEMCO | Westinghouse Environmental Management Company of Ohio |

## LIST OF ACRONYMS
## (Continued)

| | |
|---|---|
| WMCO | Westinghouse Materials Company of Ohio |
| XRF | X-ray fluorescence |

## LIST OF 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 |

## LIST OF CHEMICAL ABBREVIATIONS
## (Continued)

| | |
|---|---|
| $MgO$ | magnesium oxide |
| $Ca(OH)_2$ | 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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# 1.0 PROJECT DESCRIPTION

The Fernald Environmental Management Project (FEMP), formerly 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 FEMP 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 human health environmental impacts associated with past and present activities at the FEMP 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, which 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.

## 1.1 BACKGROUND INFORMATION

### 1.1.1 Site Description

A variety of chemical and metallurgical processes were utilized at the FEMP for manufacturing uranium products. Uranium compounds were introduced into the FEMP processes at several points During the manufacturing process. Impure starting materials were dissolved in nitric acid, and the uranium was purified through solvent extraction to yield a solution of uranyl nitrate. Evaporation and heating converted the nitrate solution to uranium trioxide ($UO_3$) powder. This compound was reduced with hydrogen to uranium dioxide ($UO_2$) and then converted to uranium tetrafluoride ($UF_4$) by reaction with anhydrous hydrogen fluoride. Uranium metal was produced by reacting $UF_4$ and magnesium metal in a refractory-lined vessel. This primary uranium metal was then remelted with scrap uranium metal to yield a purified uranium ingot.

From 1953 through 1955, the FEMP refinery processed pitchblende ore from the Belgian Congo. Pitchblende ore contains all daughter products of the uranium decay chains and is particularly high in radium. No chemical separation or purification was performed on the ore before its arrival at the FEMP. Beginning in 1956, the refinery feedstock consisted of uranium concentrates (yellowcake) from Canada and the United States. Canadian concentrates were not processed after 1960. In the production of these concentrates, most of the uranium daughters had been removed. Radium-226 (Ra-226) and thorium-230 (Th-230), however remained in the yellowcake in amounts that varied with the process.

Small amounts of thorium were produced at the FEMP on several occasions from 1954 through 1975. Thorium operations were performed in the metals fabrication plant, the recovery plant, the special project plant, and the pilot plant. The FEMP currently serves as the thorium repository for DOE and maintains long-term storage facilities for a variety of thorium materials.

Large quantities of liquid and solid waste were generated by the various operations at the FEMP. Before 1984, disposal of solid and slurried waste from FEMP processes was in the on-property waste storage area. This area, which is located west of the production facilities, includes seven low-level radioactive waste storage pits and a clearwell; two earthen-bermed concrete silos containing K-65 waste that are high-specific activity and low-level radium-bearing residues resulting from the pitch-blende refining process; one concrete silo containing metal oxides (raffinate solids disposed of in the pits are similar to those initially dried and pneumatically transferred to that silo) and one unused concrete silo; two lime sludge ponds; and a sanitary landfill. The waste storage area is addressed under Operable Units 1, 2, and 4.

An inactive fly ash disposal area and an active fly ash pile, addressed under Operable Unit 2, are located approximately 3000 feet south-southeast of the waste storage area. One pile remains active for the disposal of fly ash from the FEMP coal-fired boiler plant. Fly ash from this area will be tested in the Operable Unit 1 treatability studies. An area between and adjacent to the fly ash areas, known as the Southfield, is believed to be the disposal site for construction debris and possibly other types of solid waste from FEMP operations. The Southfield is also being addressed as a solid waste unit under Operable Unit 2.

### 1.1.2 Operable Unit 4 Description

Operable Unit 4 is located south of the waste pit area and consists of four concrete silos. 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 4 feet below the top of the vertical wall.

Historic analysis of the Silos 1 and 2 residues indicates that approximately 11,200 kilograms (kg) of uranium (0.71 percent uranium-235 [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 Environmental Management Company of Ohio (WEMCO), formerly Westinghouse Materials Company of Ohio (WMCO), for Silos 1 and 2 indicate that the concentration of 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. 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.

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 overall program goals, i.e, 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. The primary purposes of RAOs are to ensure site-wide compliance with:

- Chemical-specific applicable or relevant and appropriate requirements (ARARs) and to be considered (TBC) guidelines
- EPA guidance for risk to public health from hazardous chemicals
- Regulatory standards for control of radiation and radioactivity in the environment

The RAOs for Operable Unit 4 must cover all constituents (radiological and chemical) that contribute to a reasonable maximum exposure (RME) scenario. RAOs for Operable Unit 4 are given in Figure 1-1. Alternatives for remediation must meet airborne RAOs and direct radiation RAOs at a point immediately adjacent to the silos, as well as drinking water RAOs in perched water that might be encountered directly below the silos. The treatability study goals are given in Section 1.4.

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(s). Further details of the alternatives are given in Section 2.0.

## 1.3 JUSTIFICATION

The justification to conduct these tests is provided by EPA in "Guide for Conducting Treatability Studies Under CERCLA" (EPA 1989). 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 by reducing its volume, toxicity, or mobility. The RAOs and treatability goals for Operable Unit 4 are discussed in Sections 1.2 and 1.4.

Westinghouse is conducting vitrification tests on the Silos 1 and 2 materials. The stabilization tests in this work plan are required so that comparisons of vitrification and stabilization that will be made in the FS and in subsequent engineering designs can be based on fact rather than on conjuncture.

Because the Silo 3 wastes were produced at the FEMP 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 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 nonhazardous as defined by the Resource Conservation and Recovery Act (RCRA). 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, formerly National Lead Company of Ohio (Bettis et al.). These analyses are not sufficient in detail to support a metals extraction decision as feasible or not feasible.

| MEDIA | | REMEDIAL ACTION OBJECTIVES |
|---|---|---|
| 1. SILO CONTENTS | 1-1 | For Human Health: Prevent exposures to non-carcinogens which would result in a Hazard Index less than or equal to unity (1), and/or combined risks from exposure to carcinogens less than or equal to 1.0E-04, using 1.0E-06 as the point of departure. |
| | 1-2 | Prevent migration of contaminants which would result in groundwater concentrations greater than the MCLs or that would result in a Hazard Index less than or equal to unity (1), and/or combined risks from exposure to carcinogens less than or equal to 1.0E-04, using 1.0E-06 as the point of departure. |
| | 1-3 | Prevent current and future direct radiation doses from exceeding 100 mrem/yr. |
| | 1-4 | For Environmental Protection: Prevent migration of contaminants that would result in surface water levels greater than ambient water quality criteria. |
| | 1-5 | Prevent current and future direct radiation doses from causing detectable chronic effects. |
| 2. AIR | 2-1 | For Human Health: Prevent inhalation of contaminants which would result in a Hazard Index less than or equal to unity (1), and/or combined risks from exposure to carcinogens less than or equal to 1.0E-04, using 1.0E-06 as the point of departure. |
| | 2-2 | Prevent doses from radionuclide emissions at the FEMP from exceeding 10 mrem/yr, and radon flux from exceeding 20pCi/square meter-second. |
| | 2-3 | For Environmental Protection: Prevent current and future radiation emissions from causing detectable chronic effects. |

**FIGURE 1-1. REMEDIAL ACTION OBJECTIVES**

| MEDIA | | REMEDIAL ACTION OBJECTIVES |
|---|---|---|
| 3. SOILS | 3-1 | For Human Health:<br>Prevent inhalation of/ingestion of/direct contact with soils surrounding the silos which would result in a Hazard Index less than or equal to unity (1), and/or combined risks from exposure to carcinogens less than or equal to 1.0E-04, using 1.0E-06 as the point of departure. |
| | 3-2 | Prevent migration of contaminants which would result in groundwater concentrations greater than the MCLs or that would result in a Hazard Index less than or equal to to unity (1), and/or combined risks from exposure to carcinogens less than or equal to 1.0E-04, using 1.0E-06 as the point of departure. |
| | 3-3 | Prevent contact with radium and thorium above 5 pCi/g in the first 15 cm of soil, and 15 pCi/g at lower depths. Prevent contact with other nuclides at concentrations resulting in doses greater than 100 mrem/yr. |
| | 3-4 | For Environmental Protection:<br>Prevent migration of contaminants that would result in surface water contamination levels greater than ambient water quality criteria. |
| 4. SEDIMENTS | 4-1 | For Human Health:<br>Prevent ingestion of/direct contact with sediment contaminants which would result in a Hazard Index less than or equal to to unity (1), and/or combined risks from exposure to carcinogens less than or equal to 1.0E-04, using 1.0E-06 as the point of departure. |
| | 4-2 | For Environmental Protection:<br>Prevent releases of contaminants from sediments that would result in surface water contamination levels greater than ambient water quality criteria. |

**FIGURE 1-1.**
**(CONTINUED)**

| MEDIA | | REMEDIAL ACTION OBJECTIVES |
|---|---|---|
| 5. SURFACE WATER | 5-1 | For Human Health:<br>Prevent exposures to non-carcinogens which would result in a Hazard Index less than or equal to unity (1), and/or combined risks from exposure to carcinogens less than or equal to 1.0E-04, using 1.0E-06 as the point of departure. |
| | 5-2 | For Environmental Protection:<br>Restore surface water to below ambient water quality criteria. |
| 6. GROUNDWATER | 6-1 | For Human Health:<br>Prevent ingestion of water having contaminant levels greater than the MCLs or TBCs, or which would result in a Hazard Index less than or equal to to unity (1), and/or combined risks from exposure to carcinogens less than or equal to 1.0E-04, using 1.0E-06 as the point of departure. |
| | 6-2 | For Environmental Protection:<br>Restore groundwater aquifer to contaminant concentrations below the MCLs. |

**FIGURE 1-1.**
**(CONTINUED)**

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 treatability 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 must 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.

## 1.4 GOALS OF TREATABILITY STUDY

The primary goal of the treatability study is to support remedy selection during the FS. It supports the FS by providing data about the waste treatment under consideration by the FS. This information is used to select the most promising treatment technologies for further consideration, in conjunction with other aspects of the proposed alternative designs.

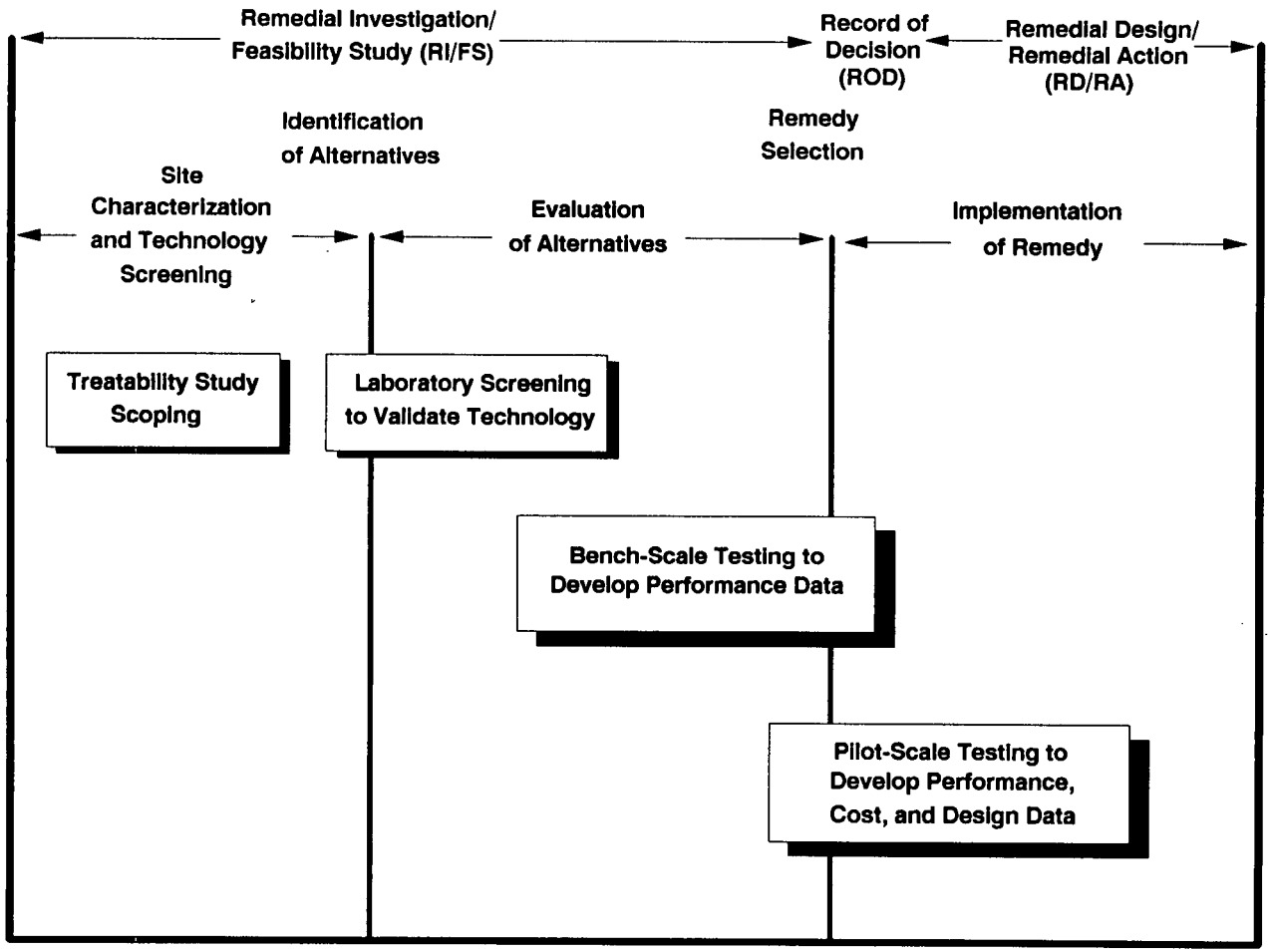
This treatability study is designed to provide data for technologies that lower the leachability of contaminants by chemically fixing them in an altered material matrix. These data will be compared to preliminary remediation goals (PRGs), toxic constituent regulatory limits (toxicity characteristic leaching procedure [TCLP] limits), and site background concentrations to determine if attainment of any or all of these goals is feasible using the technologies listed in Section 1.5. These quantitative goals are developed in Section 3.0, which outlines the treatability study's specific performance objectives.

## 1.5 TREATABILITY STUDY

### 1.5.1 EPA Treatability Guidance

EPA's "Guide for Conducting Treatability Studies Under CERCLA" (EPA 1988) outlined a three-tiered approach to conducting treatability studies for a Superfund site. This original interpretation of the approach can be seen in Figure 1-2. The remedy evaluation phase of the RI/FS, in accordance with the EPA guidance, may require a minimum of three tiers of treatability testing:

- Remedy screening
- Remedy selection
- Remedy design




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

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

Figure 1-3 reflects the approach recommended by dePercin, Bates, and Smith of EPA in their article "Designing Treatability Studies for CERCLA Sites: Three Critical Issues," (1990). 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.

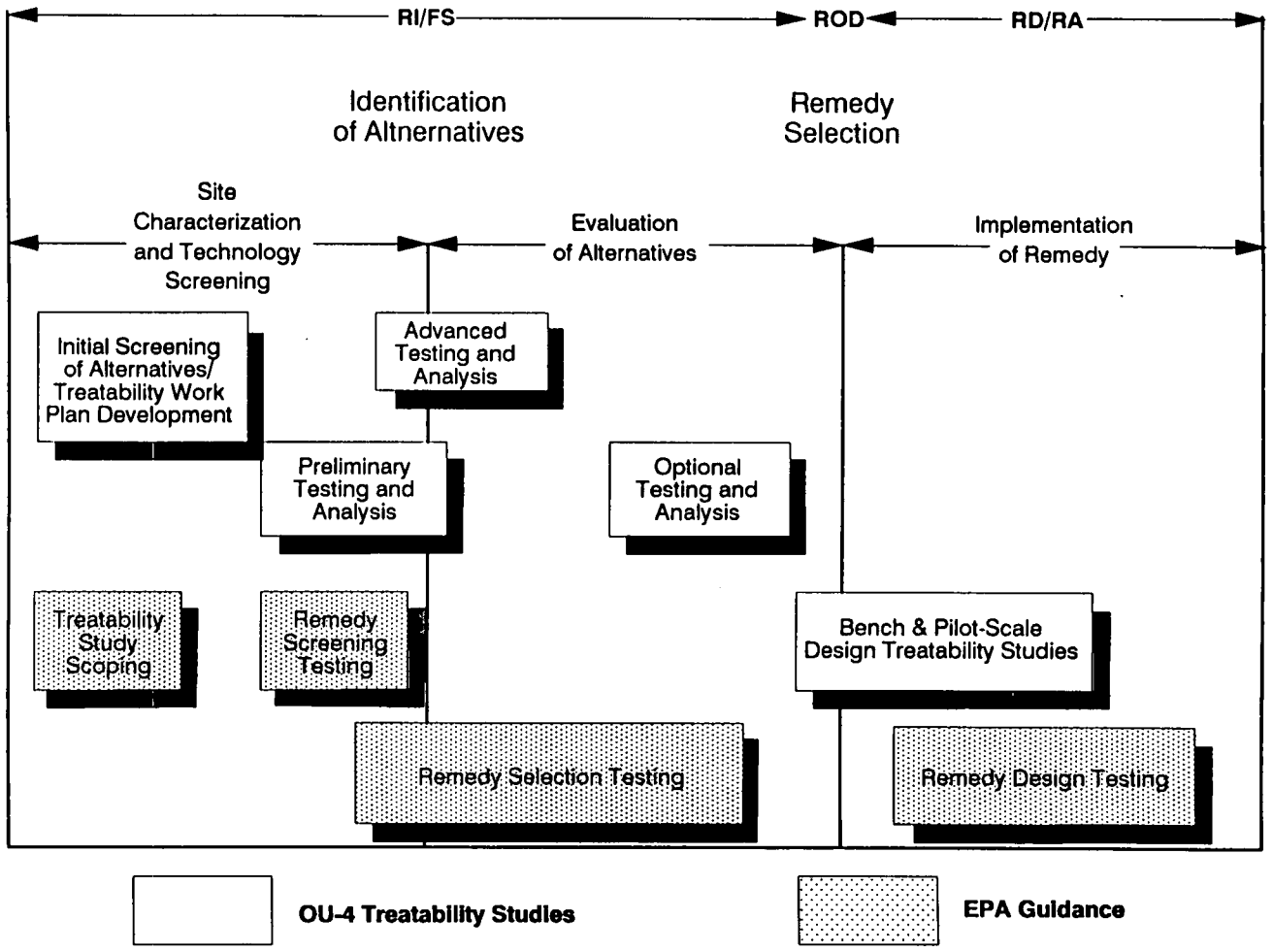
The detailed analysis of alternatives phase of the RI/FS follows the development and screening of alternatives and precedes the actual selection of a remedy in the ROD. During the detailed analysis, all remedial alternatives are evaluated based on nine RI/FS evaluation criteria. These criteria are as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

- Overall protection of human health and the environment
- Compliance with ARARs
- Long-term effectiveness and permanence
- Reduction of toxicity, mobility, and volume through treatment
- Short-term effectiveness
- Implementability
- Cost
- State acceptance
- Community acceptance

These criteria are described in detail in "Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA" (EPA 1988).

The relationship between the evaluation criteria and the data that will be generated during treatability studies as shown in Table 1-1. For example, the ability of a particular waste formulation or technology (cement stabilization versus vitrification) to provide protection of human health and the environment would be determined by evaluating factors such as concentration of contaminants in the leachate, the durability of the waste form, its compressive strength as it relates to disposal and handling, permeability, and intrinsic properties of the waste form (glass versus cement).

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 of 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




**Source for EPA Guidance: dePercin, P.E.; Bates, E.; Smith, D., 1991,"Designing Treatability Studies for CERCLA Sites: Three Critical Issues," Journal of the Air and Waste Management Assn., Vol.41,No.5**

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

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**TABLE 1-1**

**RELATIONSHIP OF TREATABILITY DATA TO FS EVALUATION CRITERIA**

| | Treatability Data | | | | | |
|---|---|---|---|---|---|---|
| FS Evaluation Criteria | Unconfined Compressive Strength - UCS | Leachate Analysis - TCLP - Static Leach | Bulking Factor (% volume change) | Permeability | Durability - PCT - Shear Strength - Waste Form Temperature Rise | Treatment Method - Cement Stabilization - Metal Extraction - Vitrification |
| • Overall protection of human health and the environment | X | X | | X | X | X |
| • Compliance with ARARs | X | X | | | | X |
| • Long-term effectiveness and permanence | X | X | | X | X | X |
| • Reduction of toxicity, mobility, or volume through treatment | | X | X | X | | X |
| • Short-term effectiveness | | | X | | | X |
| • Implementability | | | X | | X | X |
| • Cost | | | X | | | X |
| • State acceptance | X | X | X | X | X | X |
| • Community acceptance | X | X | X | X | X | X |

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 provide information, which will be used to determine whether a treatment alternative can meet the operable units' cleanup criteria and at what cost. This tier generates 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.

1.5.2 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 cement stabilization of the Silos 1 and 2 and Silo 3 material and the leaching, leachate stabilization, and leachate purification of the Silos 1 and 2 wastes. Because of the differences in the hazardous and radioactive substances found between Silos 1 and 2 and Silo 3, these materials will be handled as separate treatability study samples. See Figure 1-4 for overall flowsheet for this treatability study. TCLP, unconfined compressive strength (UCS), radiological analysis, modified TCLP (MTCLP), and product consistency test (PCT) will be used to compare the effectiveness of the various stabilization formulations.

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 phase studies and the remediation selection is performed after the advanced phase treatability studies. The preliminary phase 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 phase experiments to minimize total experiments, cost, and waste generation. The effect of silo material

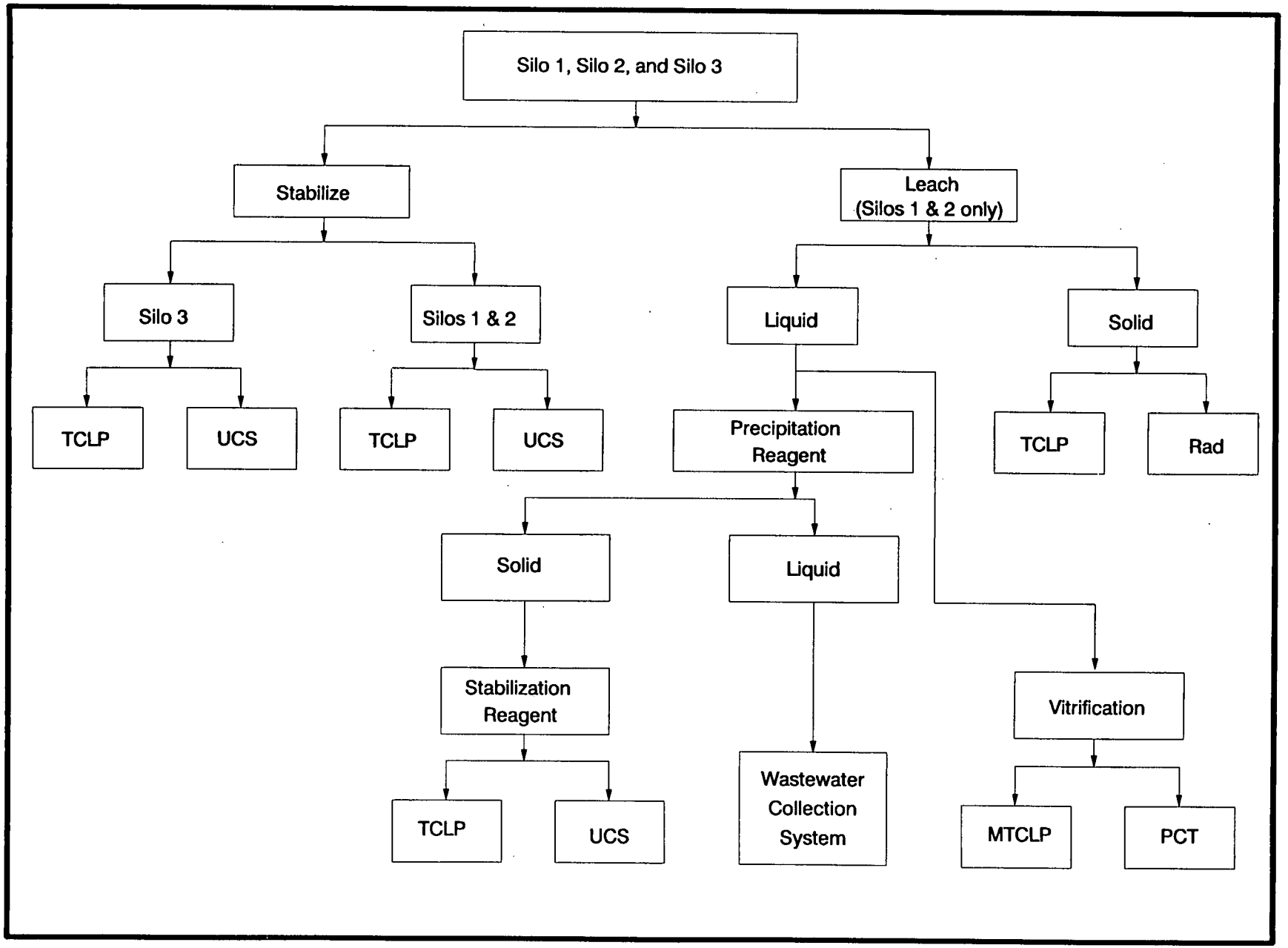


**FIGURE 1-4. Treatability Flowsheet**

variability will be evaluated in the advanced phase studies by testing the formulations and/or leaching on the top, middle, and bottom layers from each silo.

1.5.3 Stabilization of Untreated Silo Material

In the preliminary phase, 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 1990 archive 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 phase studies. Samples from the 1990-1991 sampling effort will be used in the advanced phase studies. Figure 1-5 illustrates the phases and stages of testing to be performed. The analytical tests to be performed in each stage are listed in Table 1-2.

From the available analytical data and the process history of the waste, the organic compound concentrations should be low. The work plan was written to reflect the known constituents in the waste. It is expected that the inorganic inhibitors (e.g., $MgF_2$ and inorganic or organic phosphate compounds) will cause more problems than the organic contaminants. Due to the anticipated problems resulting from the inorganic inhibitors and the potential organic constituents, a wide range of cement and fly ash concentrations will be investigated in the preliminary phase. In Stage 1, the proposed range of reagents (see Tables 4-2 and 4-3) land will be investigated on archive samples. The experiments were designed such that trends could be identified and utilized in the subsequent experiments in this treatability study. When possible, contour maps of UCS and MTCLP results versus reagent loadings will be created to aid in visualization of the trends. Based on the results of the tests, the ranges for each reagent may be adjusted before Stage 2. In Stage 2, contour maps will also be used. The contour maps will separately plot UCS, bulking factor, and MTCLP results versus reagent loadings.

The general procedure of this work plan is an iterative process where the results from matrices of experiments are used to determine the course of the next set of experiments.

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.

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

The work plan was customized to the limited availability of samples from each silo. This limitation restrains the depth of experimentation with the sample. The treatability study will determine the proof of principle of the leaching process. In the remedy design phase, the details of the process may be investigated. If the matrix of experiments indicates that multiple extractions are needed, this will be

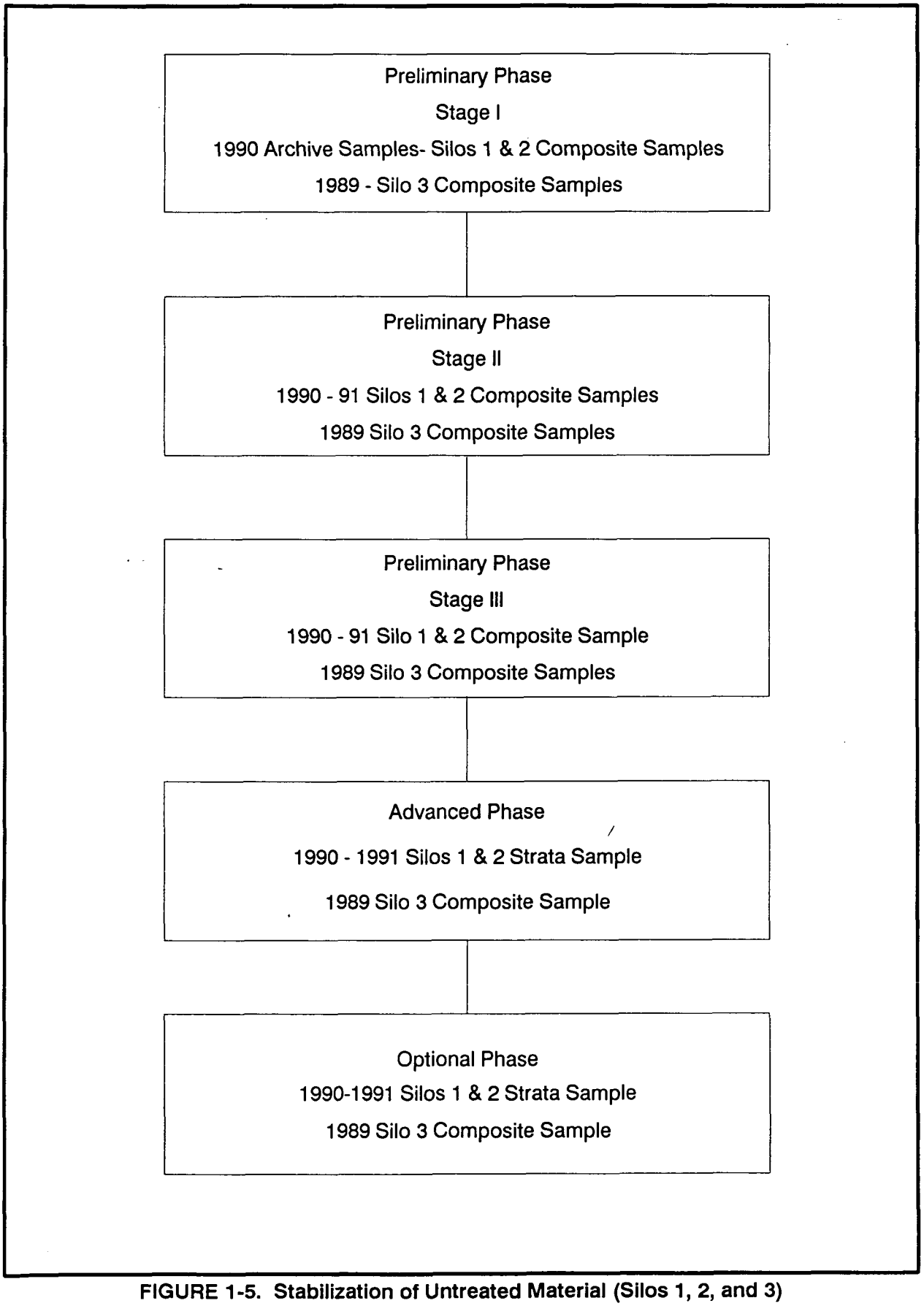


**FIGURE 1-5. Stabilization of Untreated Material (Silos 1, 2, and 3)**

**TABLE 1-2**

**ANALYTICAL TESTS - STABILIZATION OF UNTREATED SILO MATERIAL**

| | Preliminary Phase | | | Advanced Phase |
|---|---|---|---|---|
| | Stage 1 | Stage 2 | Stage 3 | |
| Bulking factor | X | X | X | X |
| UCS | X | X | X | X |
| Temperature rise | X | X | X | X |
| Shear strength | X | X | X | X |
| MTCLP - metals | X | X | X | |
| MTCLP - Gross alpha - beta | X | X | X | |
| MTCLP - U by IC | X | X | X | |
| TCLP - organic | | | | X |
| TCLP - metals | | | | X |
| TCLP - radionuclide | | | | X |
| TCLP - general chemistry | | | | X |
| 5-Day Static - metals* | | | | X |
| 5-Day Static - radionuclide | | | | X |
| 5-Day Static - general chemistry | | | | X |
| Permeability | | | | X |

*Optionally, after extraction for 5 days, the samples will be soaked for an additional 85 days. The sample may be inspected for physical degradation.

noted in the report. Also, if there is sample available and the investigator discretion, a few experiments with multiple extractions may be investigated. 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 (hydrochloric, nitric, and acetic acids) and ethylenediaminetetraacetic acid (EDTA) leaching schemes. Hydrochloric and nitric acids were selected as a result of their use in the uranium mining industry and because most metal chloride and nitrate salts are soluble. Nitric acid has the additional benefit of being able to oxidize $UO_2$ to a more soluble hexavalent uranium complex. Acetic acid was selected due to its mild complexing ability that may accentuate the metal solubilities.

A flow diagram showing phases and stages of experiments to be performed is presented in Figure 1-6. The analytical tests to be performed in each stage are listed in Table 1-3. The general procedure of this work plan is an iterative process where the results from matrices of experiments are used to determine the course of the next set of experiments.

The most promising leaching methods, as determined in the preliminary phase, will be applied in the advanced phase analysis. The treatability study will also study vitrification of the leachate, leaching kinetics, solids washing, solid/liquid separation, precipitation of remaining metals in the leachate solution, and stabilization of the material precipitated from the leachate. The leachate will be vitrified by first removing the liquid by evaporation followed by heating the dried waste combined with glass former/modifiers at 1250°C. The glass former/modifiers tested in this study are alumina-silicates (soil and fly ash) and sodium hydroxide. The most effective stabilization reagents determined from the screening that is described in Section 1.3.3 will be used as a guide in determining the formulations to investigate. Up to 10 formulations will be examined with the precipitated material.

The precipitation of the leachate experiments are preliminary phase tests to determine which type(s) of precipitation reagents will be needed to remove the majority of the hazardous and radioactive metals from the leachate before the liquid is sent to the site-wide water purification system. The subsequent stabilization or vitrification of the leachate are also preliminary phase tests. They will be used to determine if the treatment of the precipitated material has a reasonable chance of success and to provide preliminary cost data for analysis of the total leaching alternative. MTCLP will be conducted to determine the RCRA metal leachability of the treated material. A PCT to measure durability will also be performed. If the leaching alternative is carried forward, a full TCLP should be conducted during the remedy design phase when the actual precipitating reagents and larger volumes are used.

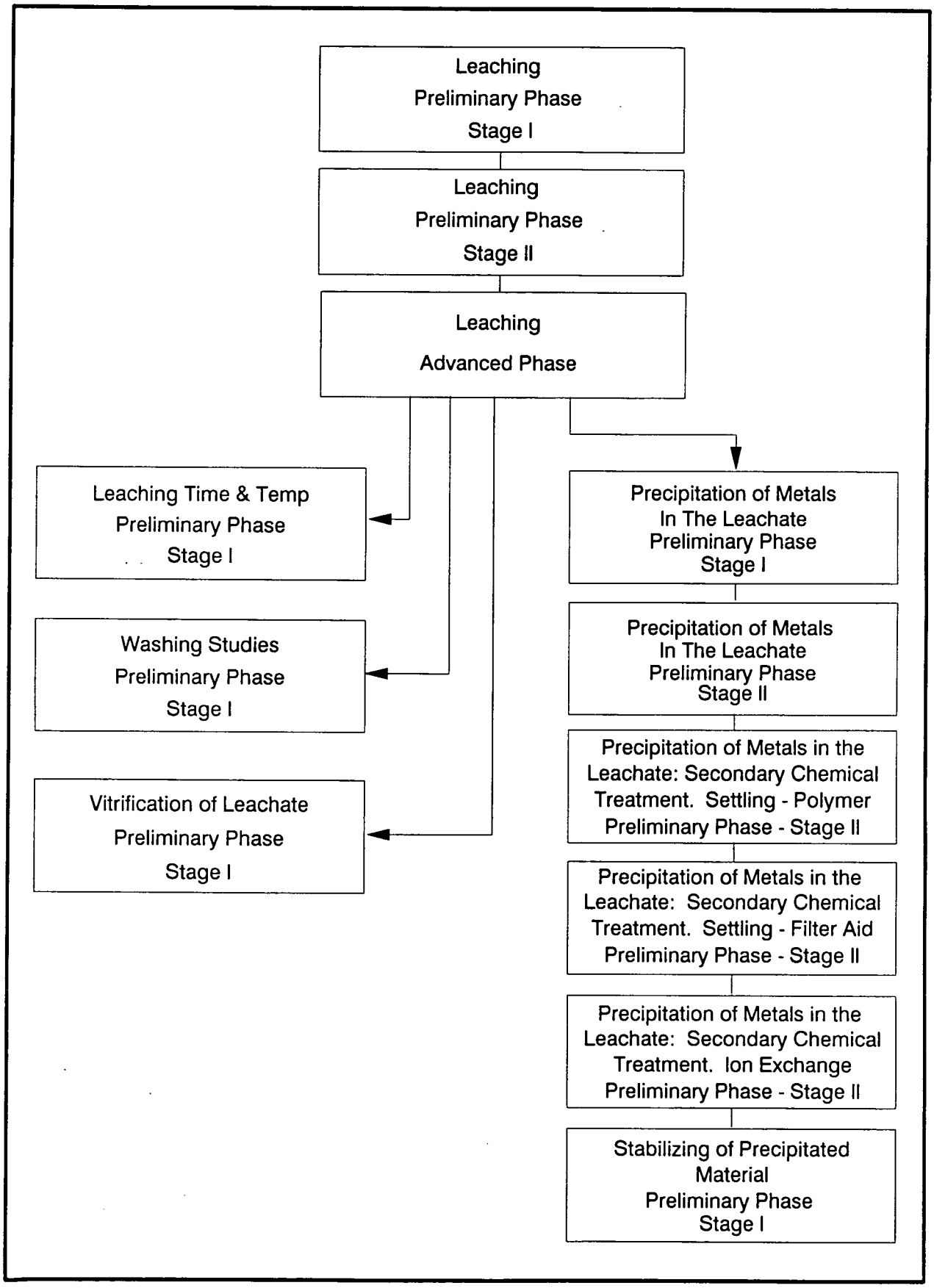


**FIGURE 1-6. Metal Extraction of Composite Samples From Silos 1 and 2**

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## TABLE 1-3

## ANALYTICAL TESTS - METAL EXTRACTION OF SILOS 1 AND 2

| Analytical Tests | Leaching | | | | | |
|---|---|---|---|---|---|---|
| | Preliminary | | | Preliminary Stage 1 | | |
| | Stage 1 | Stage 2 | Advanced | Leaching Time and Temp. | Washing Studies | Vitrification of Leachate |
| RAD - U, Th, Ra, Pb, Po | | | X | | | |
| Pb by Spot | X | X | X | X | X | |
| U by Spot | X | X | X | X | X | |
| U by IC | | X | X | X | X | |
| Pb by ICP | | X | X | X | X | |
| TCLP metals | | | X | | | |
| Leachate Characterization for Vitrification | | | | | | X |
| MTCLP - metals | | | | | | X |
| MTCLP - Gross alpha - beta | | | | | | X |
| MTCLP - U by IC | | | | | | X |
| PCT - metals | | | | | | X |
| PCT - general chemistry | | | | | | X |
| PCT - Gross alpha - beta | | | | | | X |
| PCT - U by IC | | | | | | X |
| Visual | | | | | | X |
| Bulking factor | | | | | | X |

TABLE 1-3

(Continued)

| Precipitation of Metals | | | | | |
|---|---|---|---|---|---|
| Analytical Tests | Stage 1 | Stage 2 | Stage 3 | | Stabilization of Precipitation |
| | | | Polymer | Filter Aid | |
| Pb by spot | X | | | | |
| U by spot | X | | | | |
| Settling tests | | X | X | | |
| Pb by ICP | | X | X | X | |
| U by IC | | X | X | X | |
| Bulking factor | | | | | X |
| UCS | | | | | X |
| Temperature rise | | | | | X |
| Shear strength | | | | | X |
| MTCLP - Gross alpha - beta | | | | | X |
| MTCLP - U by IC | | | | | X |
| MTCLP - metals | | | | | X |

1.5.5 General Selection Criteria

During these pre-ROD treatability studies, the most promising cement-based formulations will meet at a minimum the following standards: a UCS of approximately 500 pounds per square inch (psi), pass all of the TCLP leaching standard, and have a minimum volume increase after treatment.

The third criteria will be a secondary requirement. For vitrification, the formulations should pass all of the TCLP leaching requirements, form a durable glass, and have minimum volume increase. In addition, the leaching data will also be inspected from a risk assessment perspective as a key consideration in the selection of the most promising formulations.

The best technology will be determined by comparison of multiple criteria during the detailed analysis. The detailed analysis of the 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 ARARs
- Long-term effectiveness and permanence
- Reduction of toxicity, mobility, and volume through treatment
- Short-term effectiveness
- Implementability
- Cost
- State acceptance
- Community acceptance

The relationship between the evaluation criteria and the data that will be generated during treatability studies was shown in Table 1-1. For example, the ability of a particular waste formulation or technology (cement stabilization versus vitrification) to provide protection of human health and the environment would be determined by evaluating factors such as concentration of contaminants in the leachate, the durability of the waste form, its compressive strength as it relates to disposal and handling, permeability, and intrinsic properties of the waste form (glass versus cement).

Compliance with ARARs would be determined by whether the treated material meets compressive strength requirements for disposal, whether this leachate exceeds established discharge standards, and on factors relating to waste form. A full evaluation of the technology for compliance with ARARs will be performed in the FS.

Treatability testing that relates to a technology's long-term effectiveness and permanence includes its shear strength and durability for handling and disposal purposes, its solubility as measured by leachability, and based on permeability, the extent to which it transmits water. The waste form itself (glass or cement) also influences long-term stability. A glass for instance, would tend to be a more stable waste form provided the glass is of good quality.

The ability of a technology or formulation to reduce the toxicity, mobility, or volume will be measured by indicators such as bulking factor for volume reduction, leachate analysis for toxicity and mobility, permeability, and waste form for mobility reduction.

Short-term effectiveness is impacted primarily by bulking factor, which is an indicator of the volume of treated waste that must be handled and disposed of and by the specific technology chosen. The short-term impacts associated with implementing cement stabilization would be different from vitrification because these technologies have significantly different requirements to construct, operate, and maintain during remediation.

The implementability of a particular technology is influenced by the volume of waste to be handled as measured by bulking factor and by the waste form itself (glass versus cement). As with implementability, cost is impacted by the technology selected and the volume of waste to be generated. Because cement stabilization and vitrification are radically different processes, each will require different equipment and facilities.

The final two evaluation criteria, state and community acceptance, are influenced by the results of all the data and by the other seven criteria.

Additional information on the use of the evaluation criteria and treatability data in the feasibility study process can be found in "Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA" (EPA 1988).

# 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 (DOE 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 be classified as a solid waste under Ohio law and could then be disposed of in a sanitary landfill.

## 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. It does however 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. In situ vitrification was however 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 (1976), Mound Laboratories, Rawlings (1951), and NLO, Inc. 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, nonradioactive solid 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.

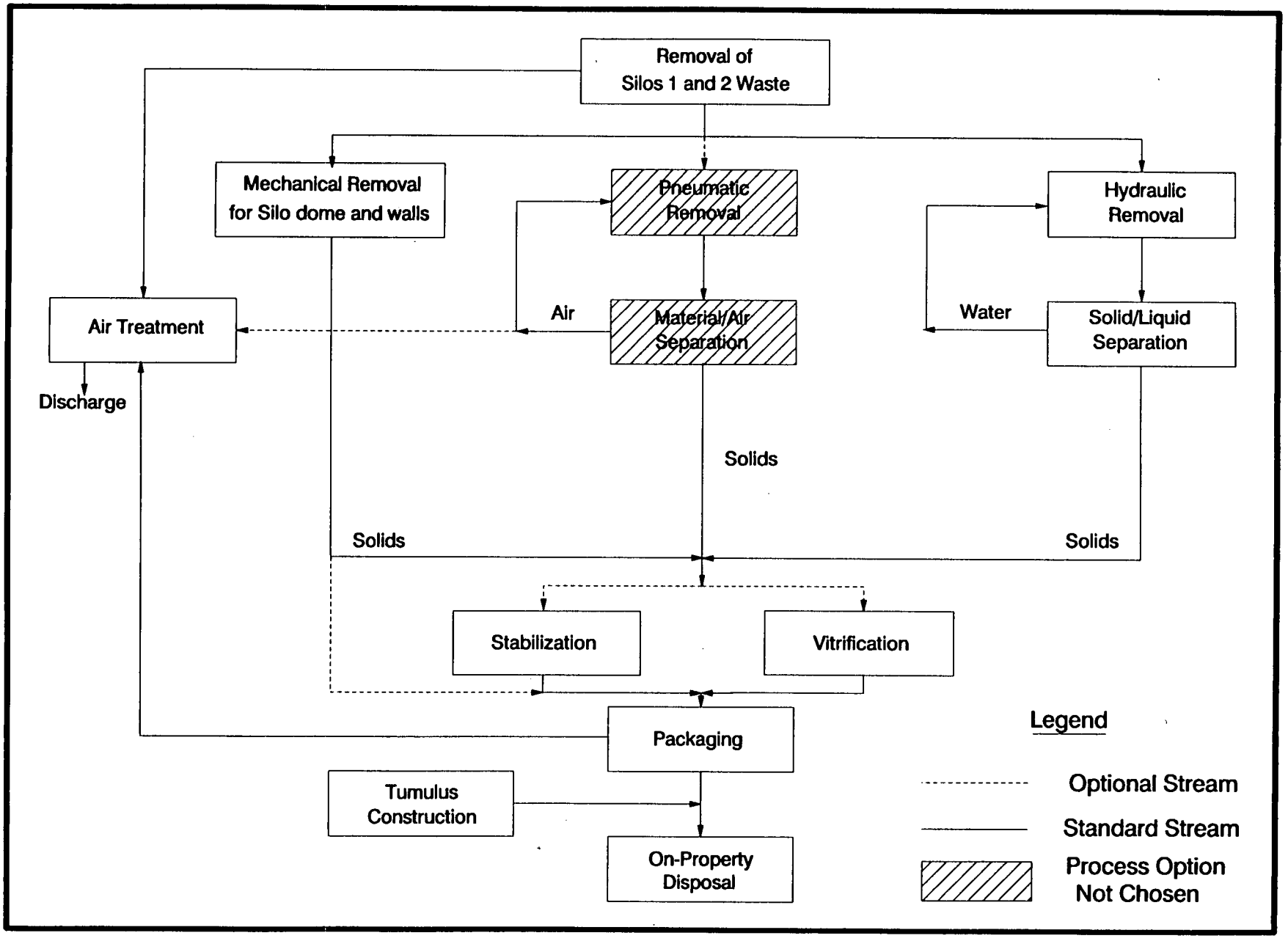


FIGURE 2-1 ALTERNATIVE 6 - REMOVAL, TREATMENT, ON-PROPERTY DISPOSAL - SILOS 1 AND 2

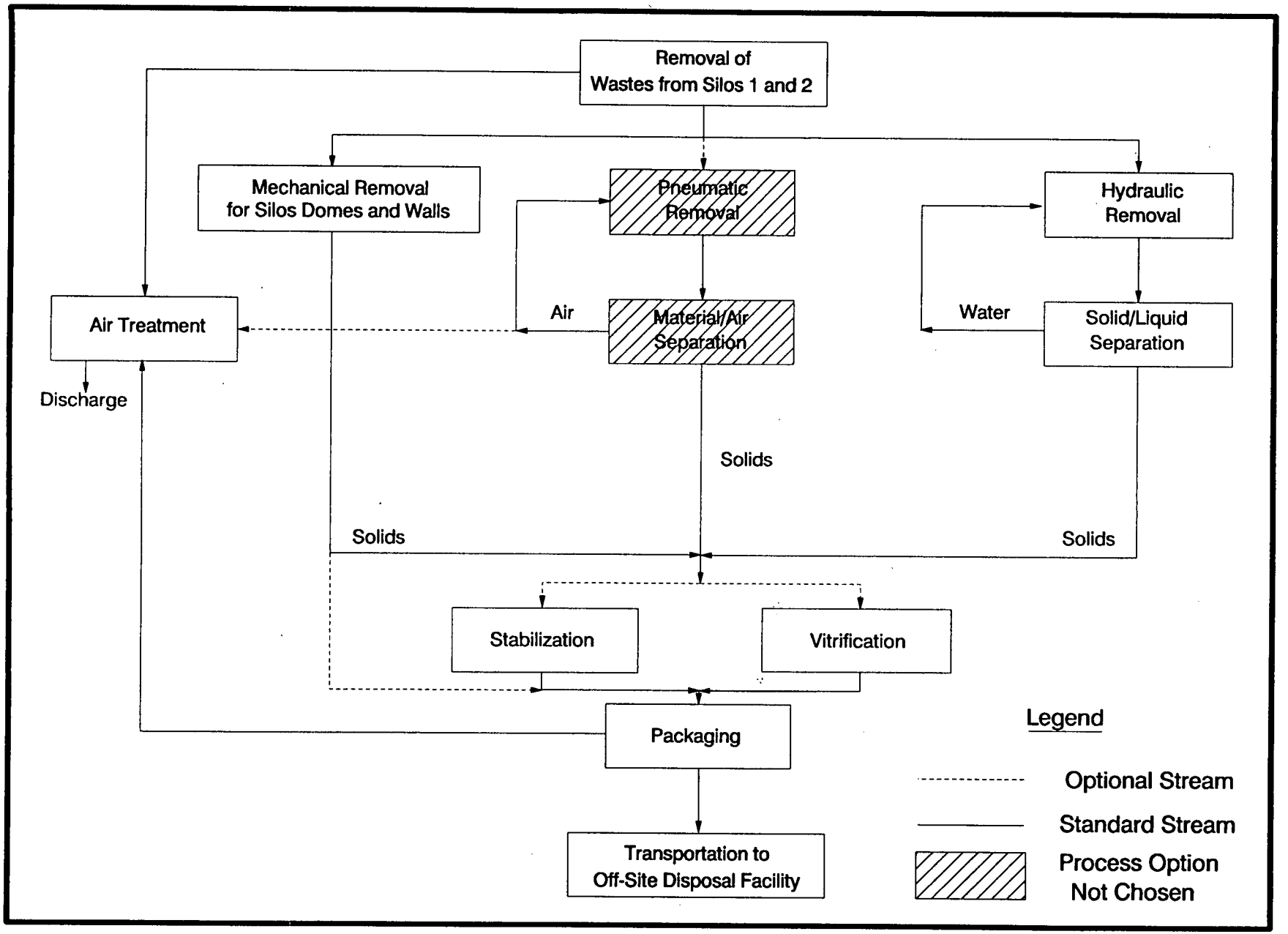


FIGURE 2-2 ALTERNATIVE 7 - REMOVAL,TREATMENT,OFF-SITE DISPOSAL - SILOS 1 AND 2

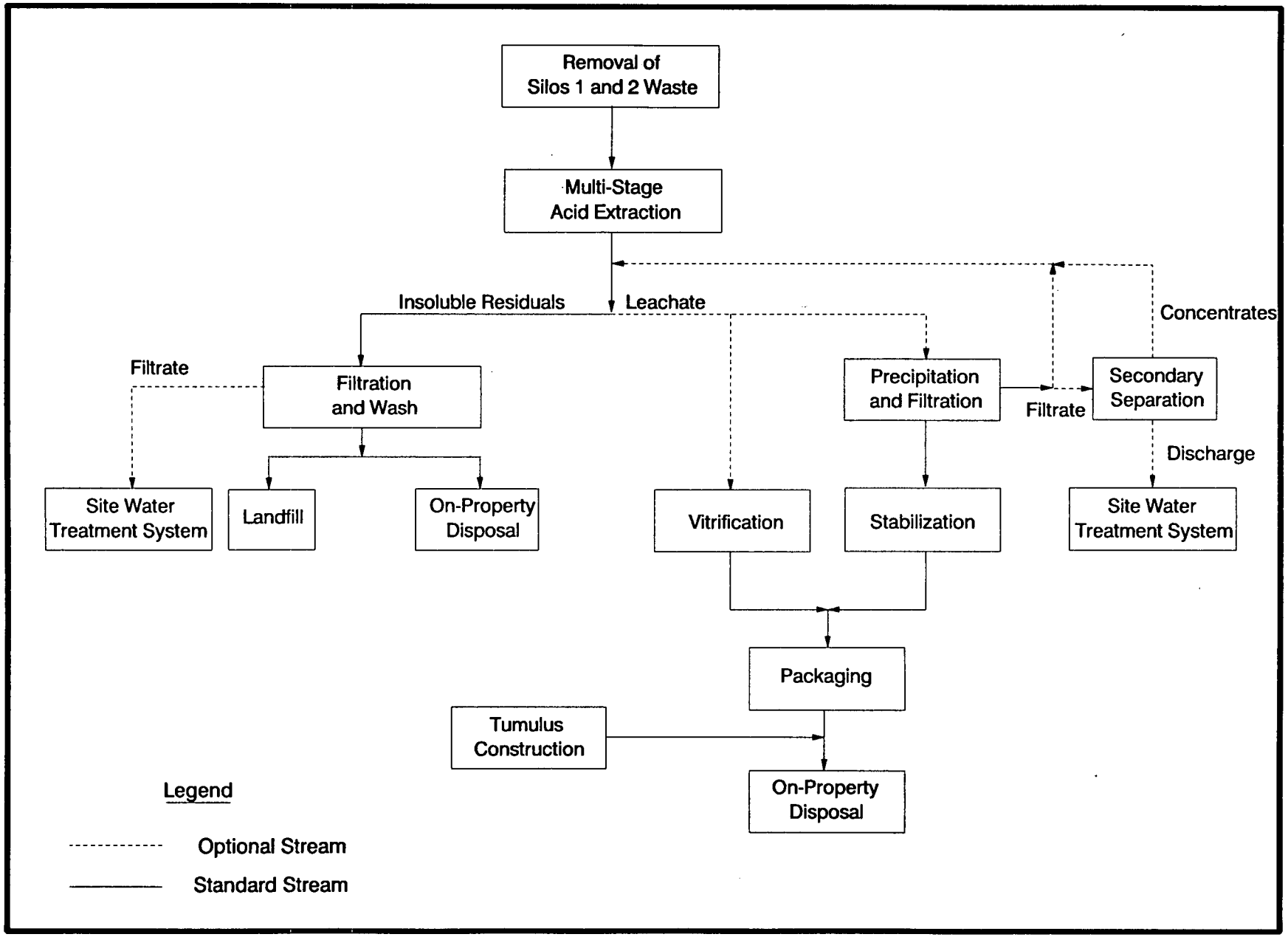


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**FIGURE 2-3 ALTERNATIVE 8 - REMOVAL, CONTAMINANT SEPARATION, ON-PROPERTY DISPOSAL - SILOS 1 AND 2**

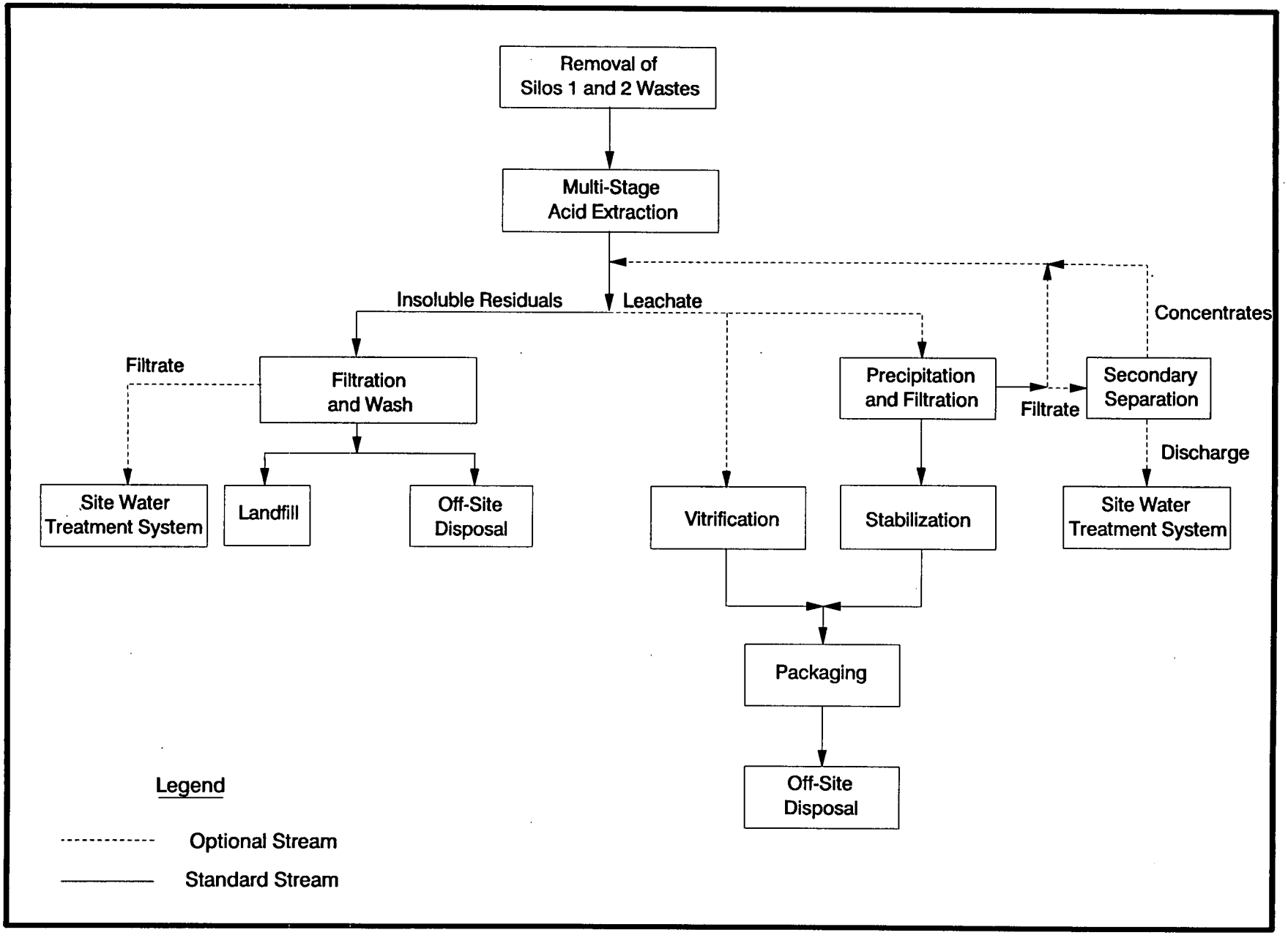


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**FIGURE 2-4 ALTERNATIVE 9 - REMOVAL, CONTAMINANT SEPARATION, OFF-SITE DISPOSAL - SILOS 1 AND 2**



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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 1-3).

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 study 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).

Concentration-based performance objectives and the resulting DQOs for the advanced phase of the treatability testing are driven by the remediation goals (RGs) established for the site. RGs are chemical-specific, medium-specific numerical concentration limits that should address all contaminants and all pathways found to be of concern during the baseline risk assessment process. The baseline risk assessment for Operable Unit 4 has not been completed, but PRGs based on chemical-media-specific concentrations have been developed using results of the RI/FS investigation presently available. These PRGs are based on a $10^{-6}$ risk level (as a point of departure) and are presented in Tables 3-1, 3-2, and 3-3 for radiological and chemical constituents, respectively.

Although these PRGs are used to provide preliminary goals for evaluating the effectiveness of the treatment technology, they are not intended to provide final action levels for contaminants in leachate, soils, or waste residues. Therefore, if the technology does not achieve individually specified levels, it should not be judged ineffective solely for that reason. The technology may later be determined to be the best available technology for treating the silo contents.

Additional information has been provided in Tables 3-1, 3-2, and 3-3 to give some perspective on how the listed PRGs compare with detection limits, background concentrations, and existing ARARs. These tables also contains a column titled "DLRL," which stands for Derived Leachate Reference Level. The DLRL numbers were calculated using the same methodology as that used by EPA to determine the regulatory levels of toxic constituents published in Table IV-3 of the Federal Register (FR Vol. 55, No. 61, pp. 11796-11877). The DLRL concentrations will be used as minimum performance criteria during initial screening, keeping in mind that the PRGs are the current proposed action levels for the FEMP. Background concentrations and detection limits are provided for comparative purposes only.

TABLE 3-1

COMPARISON OF ARARs, TBCs, PRELIMINARY REMEDIATION GOALS, DERIVED LEACHATE REFERENCE LEVELS, BACKGROUND CONCENTRATIONS, AND CONTRACT LABORATORY REQUIRED DETECTION LIMITS FOR WATER AND SURFACE SOIL

| Radionuclides | Water Concentrations | | | | | Surface Soil Concentrations | | | |
|---|---|---|---|---|---|---|---|---|---|
| | TBC/ARAR Based[a] (pCi/L) | PRGs[b] (pCi/L) | DLRL[c] (pCi/L) | FEMP Background[d] (pCi/L) | QAPP Detection Limits (pCi/L) | ARAR Based[e] (pCi/g) | PRGs[f] (pCi/g) | Background[g] (pCi/g) | QAPP Detection Limits (pCi/g) |
| Ac-227 | 0.4 | 0.056 | 56 | ~0.02 | NA | 1.0 | 0.12 | ~0.06 | NA |
| Pa-231 | 0.5 | 0.1 | 100 | ~0.02 | NA | 3 | 0.24 | ~0.06 | NA |
| Pb-210 | 1 | 0.03 | 30 | ~1 | NA | 5 | 0.6 | ~1 | NA |
| Po-210 | 3 | 0.075 | 75 | ~1 | NA | 14 | 1.5 | ~1 | NA |
| Ra-224 | 15 | 0.41 | 410 | 3 | NA | NA | 8.2 | 1 | NA |
| Ra-226 | 5[h] | 0.16 | 160 | 1 | 1 | [i] | 0.33 | 1.5 | 0.3 |
| Ra-228 | 5[h] | 0.2 | 200 | 3 | 3 | [i] | 3.9 | 1 | 0.5 |
| Rn-220 | 300 | NA | NA | NA | NA | [k] | NA | 0 | NA |
| Rn-222 | 300[j] | 1.5 | 1500 | ~1 | NA | [k] | NA | 0 | NA |
| Th-228 | 14 | 1.3 | 1300 | 1 | 1 | 20 | 0.13 | 1 | 0.6 |
| Th-230 | 10 | 0.82 | 820 | 0.1 | 1 | 21 | 0.32 | 1.4 | 0.6 |

(Continued)

| Radionuclides | Water Concentrations | | | | | Surface Soil Concentrations | | | |
|---|---|---|---|---|---|---|---|---|---|
| | TBC/ARAR Based[a] (pCi/L) | PRGs[b] (pCi/L) | DLRL[c] (pCi/L) | FEMP Background[d] (pCi/L) | QAPP Detection Limits (pCi/L) | ARAR Based[e] (pCi/g) | PRGs[f] (pCi/g) | Background[g] (pCi/g) | QAPP Detection Limits (pCi/g) |
| Th-232 | 2 | 0.89 | 890 | 1 | 1 | 4 | 0.32 | 1 | 0.6 |
| U-234 | 1 | 0.14 | 140 | 0.3 | 1 | 52 | 0.36 | 1.4 | 0.6 |
| U-235 | 1 | 0.15 | 150 | 0.02 | 1 | 56 | 0.39 | 0.06 | 0.6 |
| U-238 | 1 | 0.15 | 150 | 0.3 | 1 | 58 | 0.41 | 1.4 | 0.6 |

NA - not available.

[a]Based on doses from drinking water pathway. Calculated using 4 mrem/yr dose limit from DOE Order 5400.5 and assuming 730 L/year for 70 years

[b]Risks of 1 x $10^{-6}$ from the drinking water pathway using HEAST methodology and assuming 730 L/year for 70 years

[c]Derived leachate reference level. Calculated using the same methodology as that used by EPA to determine regulatory levels found in 40CFR261 et al. (Federal Register Vol. 55, No. 61, 11796 - 11877

[d]Site-specific RI/FS data from the FEMP groundwater report

[e]Based on doses from inhalation of resuspended dust. Calculated using an inhalation rate of 7300 $m^3$/year, a dust loading rate of 0.0002 mg/$m^3$, and the 40CFR61 dose limit of 10 mrem/year

[f]Risks of 1 x $10^{-6}$ from the inhalation and soil ingestion pathways using HEAST methodology and assuming 51100 $m^3$ of air inhaled or 2660 g of soil ingested per lifetime

[g]All fission products and transuranics are assumed to be zero. Ra-226, Th-232, and U-238 concentrations are from Myrick, T.E., et al., (1983). All daughter nuclides are assumed to be in equilibrium with their long-lived progenitors. Natural isotopic ratios are assumed for uranium.

[h]Combined radium limit in community water systems 40CFR141.15 and 141.16

[i]40CFR192 combined limit for Ra-226 and Ra-228 in surface soil is 5 pCi/g

[j]Proposed MCL for radon in drinking water

[k]40CFR61 fluence limit for radon is 20 pCi/$m^2$-sec

[l]20 mg/L total uranium is the published preliminary maximum concentration

## TABLE 3-2

## COMPARISON OF ARARs, TBCs, PRELIMINARY REMEDIATION GOALS, DERIVED LEACHATE REFERENCE LEVELS, FEMP BACKGROUND CONCENTRATIONS, AND CONTRACT LABORATORY REQUIRED DETECTION LIMITS FOR SOIL

| Chemical | PRGs[a] (mg/kg) | DRLR[b] (mg/kg) | FEMP Background | CLRDL[c] (mg/kg) |
|---|---|---|---|---|
| Aluminum | d | | 57000 | 20 |
| Arsenic | $8.00 \times 10^{1}$ | 8000 | 7.4 | 1 |
| Barium | $4.00 \times 10^{3}$ | 400000 | 420 | 20 |
| Beryllium | $1.63 \times 10^{-1}$ | 16 | 0.85 | 0.5 |
| Cadmium (soil) | $8.00 \times 10^{1}$ | 8000 | 1.7 | 0.5 |
| Chromium | $4.00 \times 10^{2}$ | 40000 | 52 | 1 |
| Cobalt | e | | 9.2 | 5 |
| Copper | e | | 22 | 2.5 |
| Lead | $5.60 \times 10^{1}$ | 5600 | 17 | 0.5 |
| Magnesium | e | | 4600 | 500 |
| Manganese | $8.00 \times 10^{3}$ | 800000 | 640 | 1.5 |
| Mercury | $2.40 \times 10^{3}$ | 2400 | 0.12 | 0.02 |
| Nickel | $1.60 \times 10^{3}$ | 160000 | 18 | 4 |
| Selenium | e | | 0.45 | 0.5 |
| Silver | $2.40 \times 10^{2}$ | 24000 | 2.8 | 1 |
| Thallium | 5.60 | 560 | NA | 1 |
| Uranium | $2.40 \times 10^{2}$ | 24000 | 4.2 | NA |
| Vanadium | $5.60 \times 10^{2}$ | 56000 | 66 | 5 |
| Zinc | $1.60 \times 10^{4}$ | 1600000 | 52 | 2 |

NA - not available

[a]PRG for a noncarcinogen in soil calculated from: Cleanup Level = (RFD*Body Weight)/(Intake*Absorption Factor); for an intake of 0.2 gram/day for a 16 kg child and an absorption factor of 1. Federal Register, 7/27/90, Vol. 55, No. 145, p. 30870

PRG for a carcinogen in soil calculated from: Cleanup Level = (Risk Level *Body Weight*Assumed Lifetime)/(CSF*Intake*Absorption Factor*Exposure Duration); for a soil intake of 0.1 gram/day for a 70

**TABLE 3-2**

**(Continued)**

kg adult/70-year lifetime exposure. The risk level used was $10^{-6}$, the absorption factor was 1, and the exposure duration was 70 years. See document cited in footnote "a"?

Lowest resulting soil concentration is reported as PRG

[b]Derived leachate reference level. Calculated using the same methodology used by EPA to determine regulatory levels found in 40CFR261. The dilution attenuation factor used was 100. (Federal Register Vol. 55, No. 61, 11796 - 11877)

[c]Contract laboratory required detection limit (CLRDL)

[d]Toxicity data were inadequate for risk-based calculation (EPA 1990)

## TABLE 3-3

## COMPARISON OF ARARs, TBCs, PRELIMINARY REMEDIATION GOALS, DERIVED LEACHATE REFERENCE LEVELS, FEMP BACKGROUND CONCENTRATIONS, AND CONTRACT LABORATORY REQUIRED DETECTION LIMITS FOR WATER

| Chemical | TBC/ARAR[a] (mg/L) | PRGs[b] (mg/L) | DRLR[c] (mg/L) | FEMP Background | CLRDL[d] (mg/L) |
|---|---|---|---|---|---|
| Arsenic | 0.050 | $3.50 \times 10^{-2}$ | 3.5 | NA | 0.01 |
| Barium | 2.000 | 1.75 | 175 | 0.3 | 0.2 |
| Beryllium | 0.001[e] | $8.14 \times 10^{-6}$ | 0.0008 | NA | 0.005 |
| Cadmium | 0.005 | $1.75 \times 10^{-2}$ | 0.5 | NA | 0.005 |
| Chromium | 0.100 | $1.75 \times 10^{-1}$ | 10 | 0.02 | 0.01 |
| Copper | 1.300[f] | g | 130 | 0.02 | 0.025 |
| Lead | 0.005 | $2.45 \times 10^{-2}$ | 0.5 | NA | 0.005 |
| Manganese | NA | 3.50 | 350 | 0.02 | 0.015 |
| Mercury | 0.002 | $1.05 \times 10^{-2}$ | 0.2 | 0.003 | 0.0002 |
| Nickel | 0.100[f] | $7.00 \times 10^{-1}$ | 10 | NA | 0.004 |
| Selenium | 0.050 | g | 5 | NA | 0.005 |
| Thallium | 0.001[f] | $2.45 \times 10^{-3}$ | 0.1 | NA | 0.01 |
| Uranium | 0.020[f] | $1.05 \times 10^{-1}$ | 2 | 1.0 | NA |
| Vanadium | NA | $2.45 \times 10^{-1}$ | 2.45 | NA | 0.05 |
| Zinc | NA | 7.00 | 700 | NA | 0.02 |

NA - not available

[a]ARARs are from 7/18/91 memo, "Drinking water MCLs and HAs.", from J. Dee. ARARs listed for Aroclor-1242, -1248, -1254, and -1260 are for total PCBs

[b]RAO for a noncarcinogen in water calculated from: Cleanup Level = (RFD*Body Weight)/Intake; for an intake of 2 liters/day for 70 kg adult. (HEAST)

RAO for a carcinogen in water calculated from: Cleanup Level = (Risk Level*Body Weight)/(CSF*Intake); for a water intake of 2 liters/day for a 70 kg adult and a risk level of $10^{-6}$. (HEAST)

Lowest resulting water concentration was reported as the PRG

**TABLE 3-3**

**(Continued)**

[c]Derived leachate reference level. Calculated using the same methodology used by EPA to determine regulatory levels found in 40CFR261. The dilution attenuation factor used was 100. (Federal Register Vol. 55, No. 61, 11796 - 11877.)

[d]Contract laboratory required detection limit (CLRDL).

[e]Proposed maximum contaminant level

[f]Current drinking water standard

[g]Toxicity data were inadequate for risk-based calculation (EPA 1990)

## 3.1 PERFORMANCE OBJECTIVES AND DESIRED DATA - STABILIZATION OF UNTREATED MATERIAL

Specific test objectives have been established so that the performance of the various stabilization mixtures can be evaluated in the areas of leachability, 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 this treatability program are as follows:

- To develop a database of stabilization reagents and corresponding hazardous and radioactive materials leachability for stabilized waste forms
- To determine the cement stabilization reagents and relative quantities required to minimize leachate concentrations of radionuclides and Hazardous Substance List (HSL) constituents from the final waste form
- To determine the cement stabilization reagents and relative quantities required so that the final waste form achieves a UCS of approximately 500 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 general stabilization: shear strength, waste form temperature rise with reagent addition, general description of waste before and after reagent addition, permeability of stabilized percent water in the waste, pH of the leachate solutions, and evolution of gas during mixing or during curing process
- To provide chemical and radiological data as shown in Table 3-4
- Establish the proof of process and applicability of the selected stabilization technology
- Screen a large number of parameters and identify those that will be critical for later bench-scale studies
- To provide data for evaluation of Silos 1 and 2 alternatives:
  - 2A - Nonremoval, In Situ Stabilization, and Cap
  - 6 - Removal, Treatment, and On-Property Disposal
  - 7 - Removal, Treatment, and Off-Site Disposal

TABLE 3-4

CHEMICAL AND RADIOLOGICAL INFORMATION TO BE ACQUIRED

PRELIMINARY PHASE

| Modified TCLP List (MTCLP) for Vitrification | | PCT List for Vitrification | | Modified TCLP List for Cement Stabilization | |
|---|---|---|---|---|---|
| Metals | Radionuclides | Metals | Radionuclides | Metals | Radionuclides |
| Arsenic (As)<br>Barium (Ba)<br>Cadmium (Cd)<br>Chromium (Cr)<br>Lead (Pb)<br>Selenium (Se)<br>Silver (Ag) | Uranium by IC<br>Gross alpha<br>Gross beta | Aluminum (Al)<br>Boron (B)<br>Iron (Fe)<br>Potassium (K)<br>Sodium (Na) | Uranium by IC<br>Gross alpha<br>Gross beta | Arsenic (As)<br>Barium (Ba)<br>Cadmium (Cd)<br>Chromium (Cr)<br>Lead (Pb)<br>Selenium (Se)<br>Silver (Ag) | Uranium by IC<br>Gross alpha<br>Gross beta |
| | | General chemistry<br>Chloride<br>Nitrate<br>Sulfate | | | |
| Physical parameters<br>Bulking factor<br>Temperature of oven<br>Time of sample heating<br>pH, Eh | | | | Physical parameters<br>Bulking factor<br>Temperature rise<br>UCS<br>pH, Eh, shear strength | |

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TABLE 3-4

(Continued)

ADVANCED PHASE

| TCLP Organic List[a] | Five-Day Static Leach Test, PCT, and TCLP Inorganic List | | | |
|---|---|---|---|---|
| | Metals | | Radionuclides | General Chemistry |
| TCL[b] Volatiles<br>TCL Semivolatiles<br>TCL Pesticides/PCBs | Aluminum (Al)<br>Antimony (Sb)<br>Arsenic (As)<br>Barium (Ba)<br>Beryllium (Be)<br>Boron (B)<br>Calcium (Ca)<br>Cadmium (Cd)<br>Chromium (Cr)<br>Cobalt (Co)<br>Copper (Cu)<br>Cyanide (CN)<br>Lead (Pb)<br>Lithium | Magnesium (Mg)<br>Manganese (Mn)<br>Mercury (Hg)<br>Molybdenum (Mo)<br>Potassium (K)<br>Nickel (Ni)<br>Selenium (Se)<br>Silicon (Si)<br>Silver (Ag)<br>Sodium (Na)<br>Thallium (Tl)<br>Vanadium (V)<br>Zinc (Zn) | Ra-total<br>Th-total<br>U-total<br>Pb-210<br>Po-210<br>Ac-227<br>Pa-231 | alkalinity<br>chloride<br>reactivity<br>fluoride<br>ammonia<br>nitrate<br>pH<br>phosphorus<br>sulfate |
| Physical Parameters :<br>pH and Eh<br>Bulking factor<br>Temperature rise (cement only)<br>Shear Strength (cement only)<br>UCS (cement only)<br>Permeability (cement only)<br>Temperature of oven (vitrification only)<br>Time of sample heating (vitrification only) | | | | |

[a]TCLP organics will not be analyzed if the compounds are not found in the characterization study portion of the work plan (Section 6.0).
[b]Target Compound List (TCL).

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 OF UNTREATED MATERIAL

The data quality needs are used to establish DQOs. 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.

DQO analytical levels are defined in EPA's "Guide for Conducting Treatability Studies Under CERCLA" (EPA 1989). 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 3-5. A list of tests and associated DQOs for stabilization are listed in Table 3-6. In addition, the appendices that contain the descriptions of the procedures are listed. Standard Operating Procedures (SOPs) and nonstandard test methods are described in Appendices B and C, respectively. In Table 3-6, two different appendices are listed for bulking factor. If the untreated waste is a slurry, the bulking factor will be determined according to the SOP in Appendix B. If the untreated waste is a solid, the bulking factor will be calculated using densities in accordance with Appendix C. (See Table 1-2 for a list of procedures for each phase and stage of the project.)

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. See Section 4.0 for a detailed discussion of the experimental design and lists of desired data.

## 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 meet TCLP standards, i.e., produce a nonhazardous residue as defined by RCRA (Table 3-1)

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**TABLE 3-5**
**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. CLP analysis. |
| 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 3-6

## STABILIZATION TEST DQOS

| PRELIMINARY (REMEDY SCREENING) | | | |
|---|---|---|---|
| TEST | APPENDIX | DQO/COMMENT | DQO LEVEL |
| Bulking Factor | B or C | Minimize waste volume increase.<br>To estimate the volume of waste that will be generated. | V |
| | | | |
| Modified Toxicity Characteristic Leaching Procedure (MTCLP) | C | During the screening phases, to determine the relative leachability of hazardous and radiological constituents associated with the various stabilization reagent formulations. | V |
| | | | |
| Waste Form Temperature | C | Preliminary Process Parameters | I |
| | | | |
| Shear Strength | C | Preliminary Process Parameters | I |
| | | | |
| Unconfined Compressive Strength (UCS) | B | To determine the unconfined compressive strength associated with each of the reagent formulations | II |
| | | | |
| pH, Eh | C | Preliminary process parameter | I |
| | | | |

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TABLE 3-6

(Continued)

| ADVANCED (REMEDY SELECTION) | | | |
|---|---|---|---|
| TEST | APPENDIX | DQO/COMMENT | DQO LEVEL |
| Bulking Factor | B or C | Minimize waste volume increase. To estimate the volume of waste that will be generated. | V |
| | | | |
| Unconfined Compressive Strength | B | To determine the unconfined compressive strength associated with each of the stabilization reagent formulations. | III |
| Full TCLP | See QAPP | To determine leachability of each of the stabilization reagent formulations. To provide data for the FS risk assessment calculations. | III |
| | | | |
| 5-Day Static Leach Test | C | To provide data for the FS risk assessment calculations | V |
| | | | |
| Permeability | C | To provide data for the FS risk assessment calculations | III |
| | | | |
| Waste Form Temperature Rise | C | To provide preliminary process parameters | I |
| | | | |
| Shear Strength | C | To provide preliminary process parameters | I |
| | | | |
| Radon Emanation | C | To determine effect of stabilization on radon emanation | V |
| | | | |
| pH, Eh | C | Preliminary process parameter | I |

- To reduce the level of radioactive components in the insoluble residue and achieve PRGs where possible (Table 3-4)
- To determine the leaching time required
- To determine effect of different waste-to-leach solution ratios on the extractions
- To determine the reagents and conditions necessary to precipitate the metals in the leachate solution
- To determine the cement stabilization reagents and relative quantities required so that the final waste form achieves an UCS of approximately 500 psi
- To determine the leachability of all radionuclides and HSL constituents from the final waste form
- To determine the cement stabilization reagents and relative quantities required to minimize leachate concentrations of radionuclides and HSL constituents from the final waste form
- 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, percent water in the waste, pH of leachate solutions, and indications of gas evolution during mixing and curing

  - 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, locations of procedure descriptions, and associated DQOs for metal extraction/precipitation/stabilization/vitrification are in Table 3-7. See Table 1-3 for a list of procedures for each phase and stage of the project. All screening will be done using composite samples. Qualitative

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TABLE 3-7

METALS EXTRACTIONS TEST DQOs

| PRELIMINARY (REMEDY SCREENING) | | | |
|---|---|---|---|
| TEST | APPENDIX | DQO/COMMENT | DQO LEVEL |
| Bulking Factor | B or C | Minimize waste volume increase during stabilization and vitrification. Estimate the volume of waste that will be generated. | V |
| Modified Toxicity Characteristic Leaching Procedure (MTCLP) | C | During the screening phases, to determine the relative leachability of hazardous and radiological constituents associated with the various stabilization and vitrification reagent formulations. | V |
| Waste Form Temperature Rise | C | Preliminary Process Parameters (Cement Stabilization) | I |
| Shear Strength | C | Preliminary Process Parameters (Cement Stabilization) | I |
| PCT | C | To determine the durability of the glass formulations. To provide data on the relative leachability of radionuclides and glass components with the various reagent formulations. | V |
| Unconfined Compressive Strength (UCS) | B | To determine the unconfined compressive strength associated with each of the reagent formulations. | II |
| | | | |
| Uranium and Lead Spot Tests | D | To qualitatively compare the effectiveness of various solvents and reagents during leaching and precipitation experiments. | I |
| Uranium By IC and Lead By ICP | C and SW-846 | To quantitatively compare the effectiveness of various solvents and reagents during leaching and precipitation experiments. | III |

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(Continued)

| PRELIMINARY (REMEDY SCREENING) | | | |
|---|---|---|---|
| TEST | APPENDIX | DQO/COMMENT | DQO LEVEL |
| | | | |
| pH, Eh | C | Preliminary process parameter | I |
| | | | |
| Temperature | C | To determine the effect temperature has on the rate of dissolution of the metals | III |
| ADVANCED (REMEDY SELECTION) | | | |
| Radon Emanation | C | To determine effect stabilization and vitrification have on radon emanation. | V |
| | | | |
| Radiological | | To quantify the residual radionuclide concentrations in the insoluble residue resulting from tests with the most effective solvents. This will be used to grade solvents pass/fail. | IV |
| 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. | IV |
| | | | |
| Uranium and Lead Spot Tests | D | To qualitatively compare the effectiveness of various solvents and reagents during leaching and precipitation experiments. | I |
| | | | |
| Uranium by IC and Lead by ICP | C and SW-846 | To quantitatively compare the effectiveness of various solvents and reagents during leaching and precipitation experiments. | II |
| | | | |
| Temperature | C | To determine the effect temperature has on the rate of dissolution of the metals | III |

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.

The 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, polonium and lead. These tests will identify the most effective solvents.

If the leaching process is successful (i.e., the insoluble residue from the leaching has favorable TCLP and risk-based radiological test results), 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. The precipitated material from the most effective precipitation reagents will be subjected to stabilization tests and vitrification experiments. See Section 4.0 for a detailed discussion of the experimental design and lists of desired data.

# 4.0 EXPERIMENTAL DESIGN AND PROCEDURES

## 4.1 STABILIZATION OF UNTREATED MATERIAL

### 4.1.1 Preliminary Phase

In the preliminary phase, 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 1990 archive and 1990-1991 silo sampling programs will be tested. The data produced will be used to define the scope of the advanced phase better. A stabilization flow sheet is given in Figure 4-1. The preliminary phase 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 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 4-1. 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 4-1, 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 4-1, 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.

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.

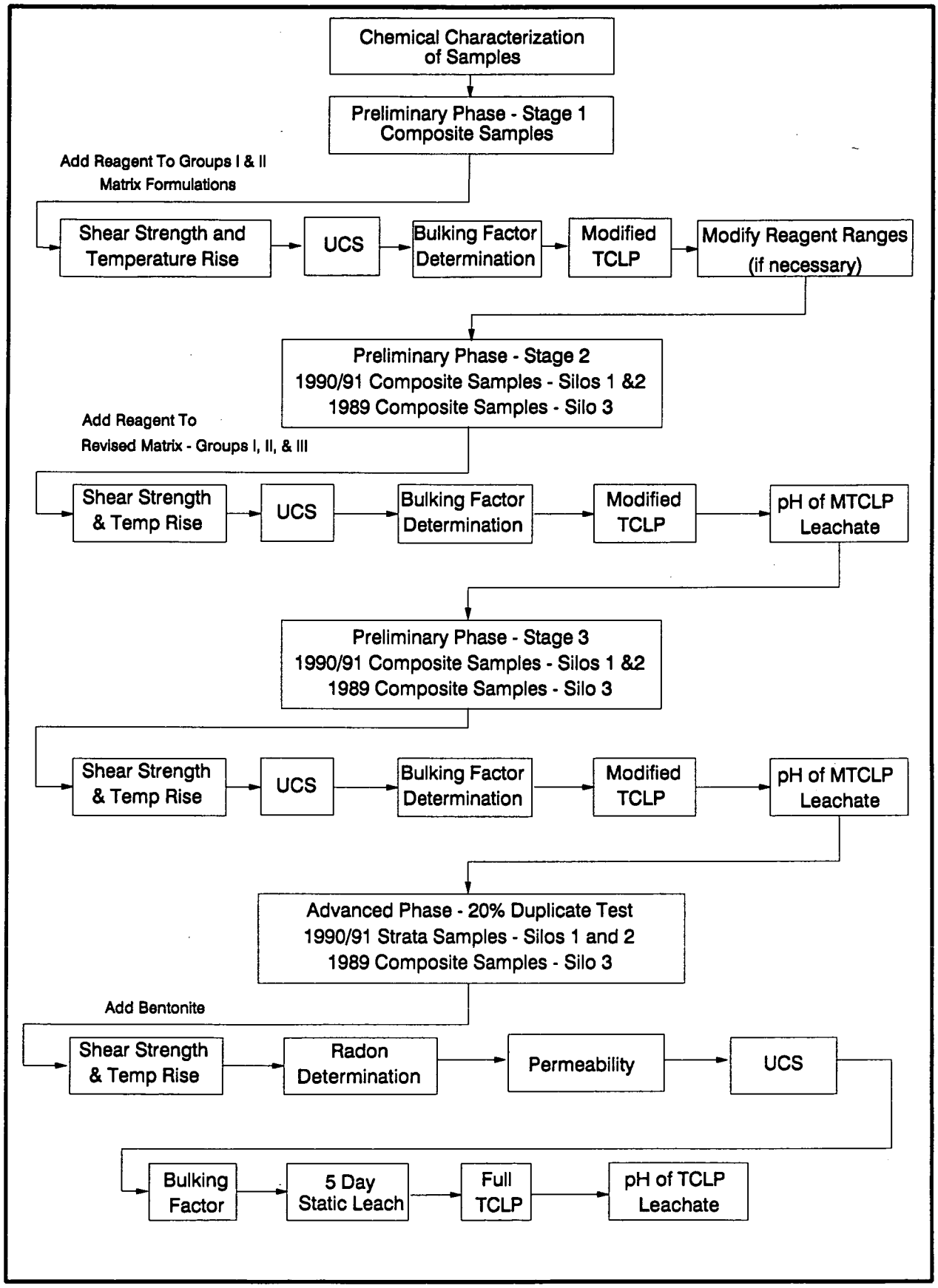


**FIGURE 4-1. Stabilization Flowsheet**

## TABLE 4-1

## 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 - 65 |
| 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 |

(Continued)

| 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[a] (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 |

[a]12A and 12C: Add 12 grams of attapulgite and clinoptilolite, respectively.

4.1.1.1 Preliminary Phase - Stage 1

Samples from the 1990 archive for Silos 1 and 2 will be treated according to the Group I and II matrices in Table 4-1. The shear strength and waste form temperature rise will be measured within 10 minutes of mixing waste and reagents. The UCS MTCLP and bulking factor will be measured on Day 28.

In addition, the following observations, measurements, or tests will be performed: general description of waste before and after reagent addition, permeability of treated sample, percent water in waste, pH of stabilized waste analytical leachate solutions, and indication if there is gas evolution during mixing or during the curing process.

A metals TCLP analysis of a blank consisting of reagent with sand or quartz will be conducted.

4.1.1.2 Preliminary Phase - Stage 2

After completion of the Stage 1 tests, separate composited samples from Silos 1 and 2 from the 1990/1991 sampling period and from Silo 3 from the 1989 sampling period will be treated according to the stabilization matrix (Table 4-1). This series of tests will include Groups 1 through III of Table 4-1.

The shear strength and waste form temperature rise will be measured within 10 minutes of mixing the waste and reagents. The UCS and the bulking factor will be measured on Day 28. MTCLP for metals will also be run on the samples. In addition, the following observations, measurements, or tests will be performed: general description of waste before and after reagent addition, permeability of treated sample, percent water in waste, pH of stabilized waste analytical leachate solutions, and indication if there is gas evolution during mixing or during the curing process.

Approximately 100-gram samples will be used in these tests. The ranges listed in Table 4-1 may be narrowed depending on the results Stage 1.

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 goal. The bulking factor will be measured on Day 28.

4.1.1.3 Preliminary Phase - Stage 3

The most promising formulations from Stages 1 and 2 are those with a high UCS (approximately 500 psi), low leachability for hazardous and radioactive constituents, minimum volume increase of the resultant waste, and low cost of reagents.

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 mathematical models developed in Stages 1 and 2 will be used to aid in the development of these experiments.

The shear strength and waste form temperature rise will be measured within 10 minutes of mixing the waste and reagents. The UCS and the bulking factor will be measured on Day 28. MTCLP for metals will also be run on the samples. In addition, the following observations, measurements, or tests will be performed: general description of waste before and after reagent addition, permeability of treated sample, percent water in waste, pH of stabilized waste analytical leachate solutions, and indication if there is gas evolution during mixing or during the curing process.

The number of experiments may range from zero to five formulations per composite sample.

4.1.2 Advanced Phase - Silos 1 and 2

The most promising two formulations (those giving the best combination of leachability, UCS, the lowest bulking, and lowest reagent loading) from the composite sample study will be tested on the top, middle, and bottom strata (Zones A, B, C) 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 and tested in duplicate. The UCS will be determined with a Soiltest U-590 or U-610 instrument. TCLP, 5-day static leach test, and permeability will be performed on the samples. The bulking factor of the stabilized material will be measured. Radon emission tests will be conducted. In addition, the following observations, measurements, or tests will be performed: general description of waste before and after reagent addition, permeability of treated sample, percent water in waste, pH of stabilized waste analytical leachate solutions, and indication if there is gas evolution during mixing or during the curing process.

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 1 and 2 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.

4.1.3 Advanced Phase - Silo 3

Composite samples will be used instead of individual strata samples. The most promising two formulations for Silo 3 will be repeated. Twenty percent of the samples will be set in duplicate. The UCS will be determined using a Soiltest U-590 or U-610 instrument. TCLP, 5-day static leach test, and permeability will be performed on the samples. The bulking factor of the stabilized material with the appropriate UCS will be measured. In addition, the following observations, measurements, or tests will be performed: general description of waste before and after reagent addition, permeability of treated sample, percent water in waste, pH of stabilized waste analytical leachate solutions, and indication if there is gas evolution during mixing or during the curing process. Radon emission tests will be conducted.

4.1.4 Advanced Experiments - Optional

It is possible that some waste forms that appear to be promising will fail TCLP, or exhibit other traits casting doubt on the formulations. If this occurs, optional experiments might be designed. Waste forms from optional tests would, as a minimum, be subjected to the same tests used in Stages 1 and 2 of the advanced experiments. The treated sample from the 5-day static test may be inspected for physical degradation after 90 days of leaching. The leachate may be analyzed as during the advanced phase.

4.1.5 Data Required

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

- UCS measured by a Soiltest U-590 or U-610 instruments (SOP No. TCL 1109, Appendix B)
- Permeability (for advanced phase)
- MTCLP (for preliminary phase), or TCLP and 5-day static leach test (for advanced phase)
- Bulking factor
- Waste form temperature rise after waste and reagents are mixed, and the time between mixing and temperature measurements
- Approximate shear strength measured within 10 minutes of when waste and reagents are mixed
- Physical characteristics: percent moisture, bulk density
- Amount of water added to each waste form

- The maximum particle size treated; weight and percentage of material sieved from the raw waste before treatment
- General description of the waste form before and after reagents are mixed. This includes a description of any grinding of the sample to meet particle size requirements for UCS
- Description of vapor or gas released during mixing and during curing of mixture
- Physical appearance of mold after 90-day soak in deionized water in optional phase
- pH and Eh of the reagent waste mixture before adding mixture to molds
- pH of MTCLP and TCLP extraction fluids, pH of TCLP extraction fluid determination test
- pH of 5-day static leach solution
- pH of 90-day leach solution in optional phase
- pH and Eh of slightly wet water waste mixture
- Radon emission test results (advanced phase)
- TCLP metals results for reagents combined with clean sand or quartz

## 4.2 METAL EXTRACTIONS

### 4.2.1 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 phase 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 present in greater concentrations than thorium or radium. The removal of thorium, uranium, lead, polonium, and radium will be demonstrated in the advanced phase. A typical detailed leaching screening plan is shown in Figure 4-3.

#### 4.2.1.1 Leaching Preliminary Phase - Stage 1

1990 archive will be investigated during this stage. The acid and EDTA leaching experiments are listed in Tables 4-2 and 4-3, respectively. Selected experiments from Table 4-2 will be conducted first to determine which acids have promise and the effects of temperature and acid concentration on the

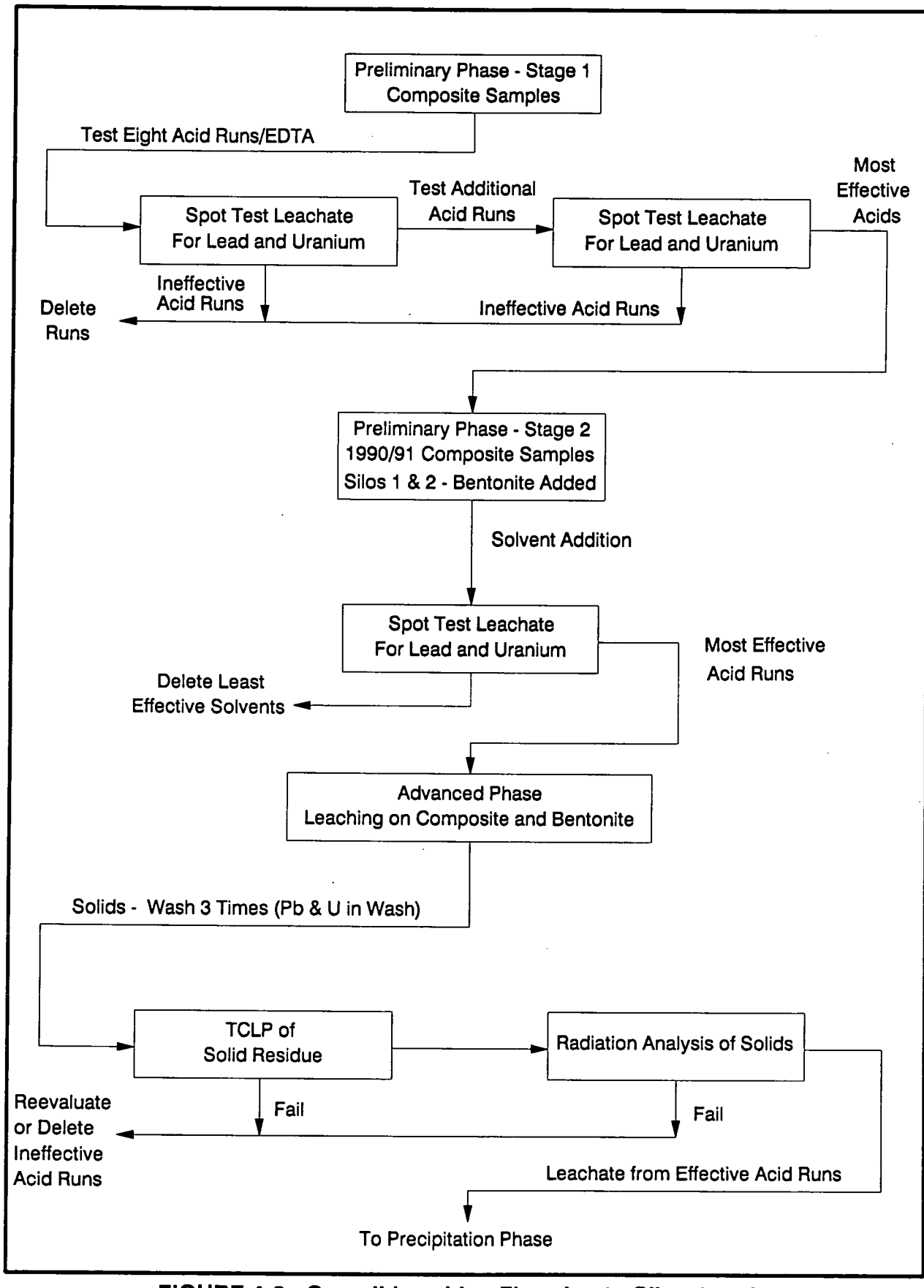


**FIGURE 4-2. Overall Leaching Flowsheet - Silos 1 and 2**

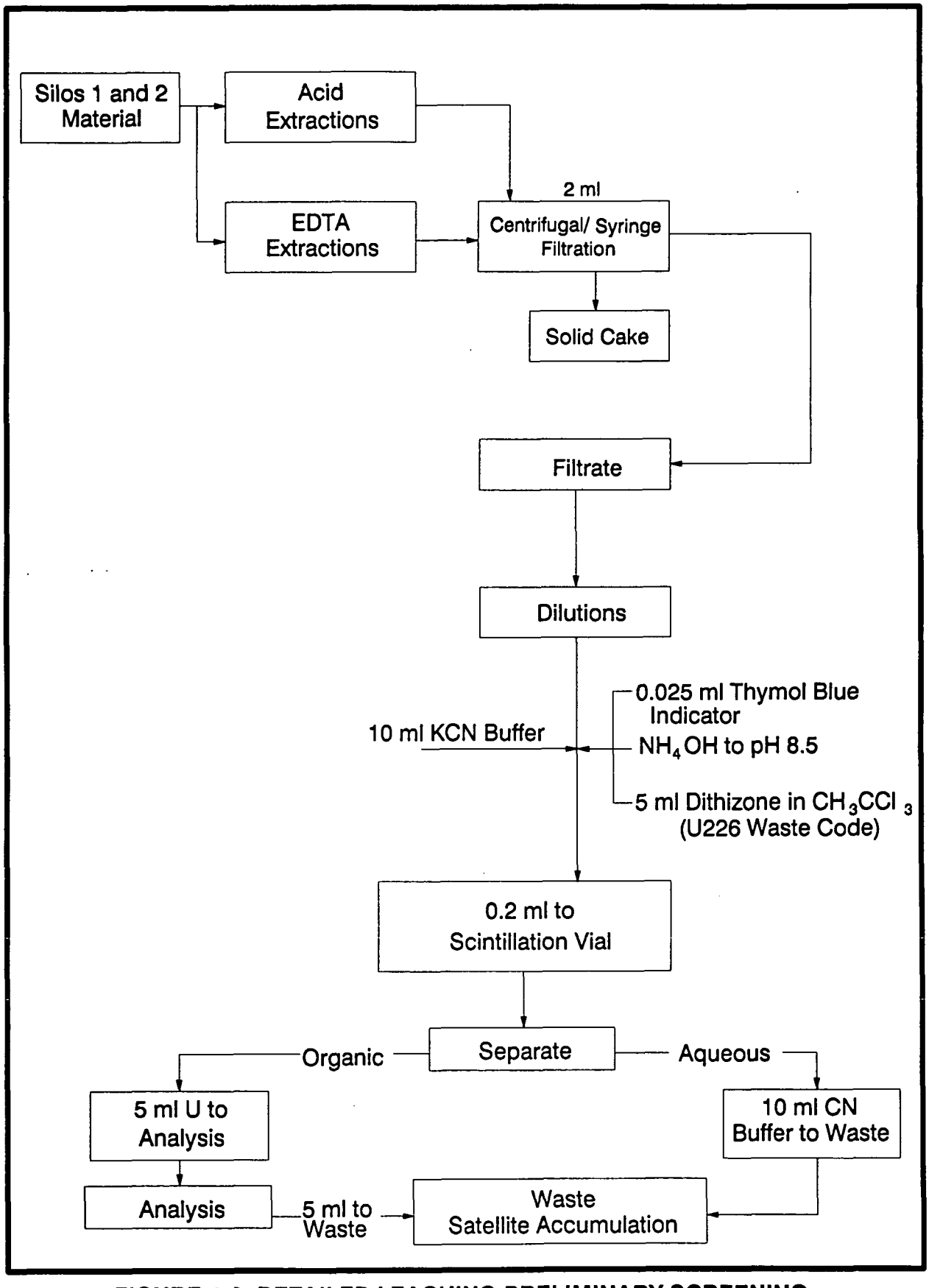


**FIGURE 4-3 DETAILED LEACHING PRELIMINARY SCREENING**
TEST PLAN - TYPICAL FLOWSHEET

## TABLE 4-2

## ACID EXTRACTIONS

| Run No. | Acid Nominal Conc. | Dose (weight acid/ weight sample) | | Temperature | |
|---|---|---|---|---|---|
| | | 2:1 | 4:1 | Ambient | 80°C |
| 1 | 60% $HNO_3$[a](13N) | X | X | X | |
| 2 | 60% $HNO_3$ (13N) | X | X | | X |
| 3 | 30% $HNO_3$ (5.6N) | X | X | X | |
| 4 | 30% $HNO_3$ (5.6N) | X | X | | X |
| 5 | 15% $HNO_3$ (2.6N) | X | X | X | |
| 6 | 15% $HNO_3$ (2.6N) | X | X | | X |
| 7 | 36% HCl[b] (11.6N) | X | X | X | |
| 8 | 36% HCl (11.6N) | X | X | | X |
| 9 | 18% HCl (5.4N) | X | X | X | |
| 10 | 18% HCl (5.4N) | X | X | | X |
| 11 | 9% HCl (2.6N) | X | X | X | |
| 12 | 9% HCl (2.6N) | X | X | | X |
| 13 | 50% HOAc[c] (8.8N) | X | X | X | |
| 14 | 50% HOAc (8.8N) | X | X | | X |
| 15 | 25% HOAc (4.3N) | X | X | X | |
| 16 | 25% HOAc (4.3N) | X | X | | X |
| 17 | 12.5% HOAc (2N) | X | X | X | |
| 18 | 12.5% HOAc (2N) | 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 4-3**

**EDTA EXTRACTIONS**

| Run No. | EDTA Conc. | Dose (weight ext. liquid/ weight sample) | | Temperature | |
|---|---|---|---|---|---|
| | | 2:1 | 4:1 | Ambient | 80°C |
| 19 | 0.2M | X | X | X | |
| 20 | 0.2M | X | X | | X |

metal solubilities. In these initial tests, the effect of temperature is measured with the concentrated acids by testing them at ambient and 80°C. The effect of acid concentrations is being measured by testing concentrated acidand dilute acid at elevated temperatures. For each acid this entails three test points; that is, Run Nos. 1, 2, 6, 7, 8, 12, 13, 14, and 18 in Table 4-2 will be conducted first.

Hydrogen peroxide, chlorine bleach (NaOCl), and ferric chloride will be added if it is apparent that uranium is not extracting from the solid. Hydrogen peroxide and bleach are added to oxidize lower valence uranium species to more soluble uranium (VI) species. Ferric chloride is a catalyst for this oxidation reaction.

During this stage, a matrix of experiments is being conducted to determine trends of solubilities. If it is apparent from the analytical results that a particular acid is not successfully leaching the metals, the acid will be eliminated from further testing. If the analytical results indicate that a particular leachant(s) extracts more uranium and lead than another leachant, then it is considered promising. The promising leachant may be investigated further to define better the effect of acid concentrations and temperature on the solubilities.

The appropriate omitted experiments from Table 4-2 may be conducted if the results indicate that they are warranted. Also, if the extraction procedures listed in Table 4-2 are effective, then the EDTA extraction procedures (Table 4-3) will be omitted.

4.2.1.2 Leaching - Preliminary Phase - 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 4-2 and 4-3 will be selected based on the Stage 1 results.

4.2.1.3 Leaching - Advanced Phase

The objective of the advanced phase is to demonstrate on larger samples that the leached material is a nonhazardous material as defined by RCRA and that uranium, lead, thorium, polonium, and radium were successfully leached from the solid. The 5 to 10 treatments from the preliminary phase tests that yield leachates with the greatest concentrations of lead and uranium will be repeated on a larger scale (presumably 100 to 500 grams). Composite samples with bentonite added will be used. The solid material will be filtered and washed three times with deionized water to remove the soluble compounds. The leachate and 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 as defined by RCRA. In addition, lead, thorium, radium, polonium, and uranium content will be determined by radiation analyses.

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.

4.2.2 Vitrification of Leachate - Preliminary Phase - Stage 1

This laboratory screening will consist of one phase - preliminary phase - Stage 1. The effects of the adding sodium hydroxide, site fly ash, and site soil will be demonstrated. Except for tests on the dried leachate, no experiments will be conducted until the chemical characterization of the leachate, soil, and fly ash are completed. As a target, the reagent waste mixture will have between 40 to 60 percent combined $SiO_2$ and $Al_2O_3$ content and 10 to 20 percent sodium oxide content when dried. It is expected that this range of $SiO_2$ and $Al_2O_3$ content will produce a durable glass. The melting point of the glass mixture can be lowered by increasing the sodium oxide content of the glass. Sodium hydroxide may be added to the mixture before heating to increase the sodium oxide content of the vitrified waste. Sodium hydroxide is converted to sodium oxide during the vitrification process. Enough sodium hydroxide will be added to cause the mixture to melt at 1250°C in a muffle furnace. This temperature was chosen to give a reasonable compromise between the cost of adding sodium oxide content to lower the melting point, the expected increase in leachability as the melting point of mixture is lowered, and the energy cost to melt and form the vitrified material. It is generally recognized in the glass manufacturing industry by companies such as Corning that to form homogenous and durable glass mixture with hazardous waste melt temperatures between 1250° and 1350°C are needed. If this process is carried forward to the remedy design phase, the effect of melt temperature may be investigated.

Figure 4-4 presents a flow sheet for the vitrification process. The leachate will be analyzed on a dry basis for the content of total aluminum as alumina, silicon as silica, and sodium as sodium oxide. The leachate will be slowly dried in a beaker on a hot plate. Using the chemical analyses of the leachate, fly ash, and soil as guide, a series of range-finding experiments will be performed. Various amounts of sodium hydroxide will be added to mixtures of waste, fly ash, and soil to determine the sodium hydroxide concentration needed to lower the melting point temperature to about 1250°C. These range-finding experiments will be followed by an experimental matrix similar to Table 4-4. The ranges given in Table 4-4 may be changed after completion of the range-finding experiments and consideration of the chemical analysis of the leachate, soil, and fly ash.

According to Table 4-4, sodium hydroxide will be added at three levels: 0 percent, 10 percent, and 20 percent of the dry weight of the waste. The site fly ash and soil will be added at 50 percent of the dry weight of the waste.

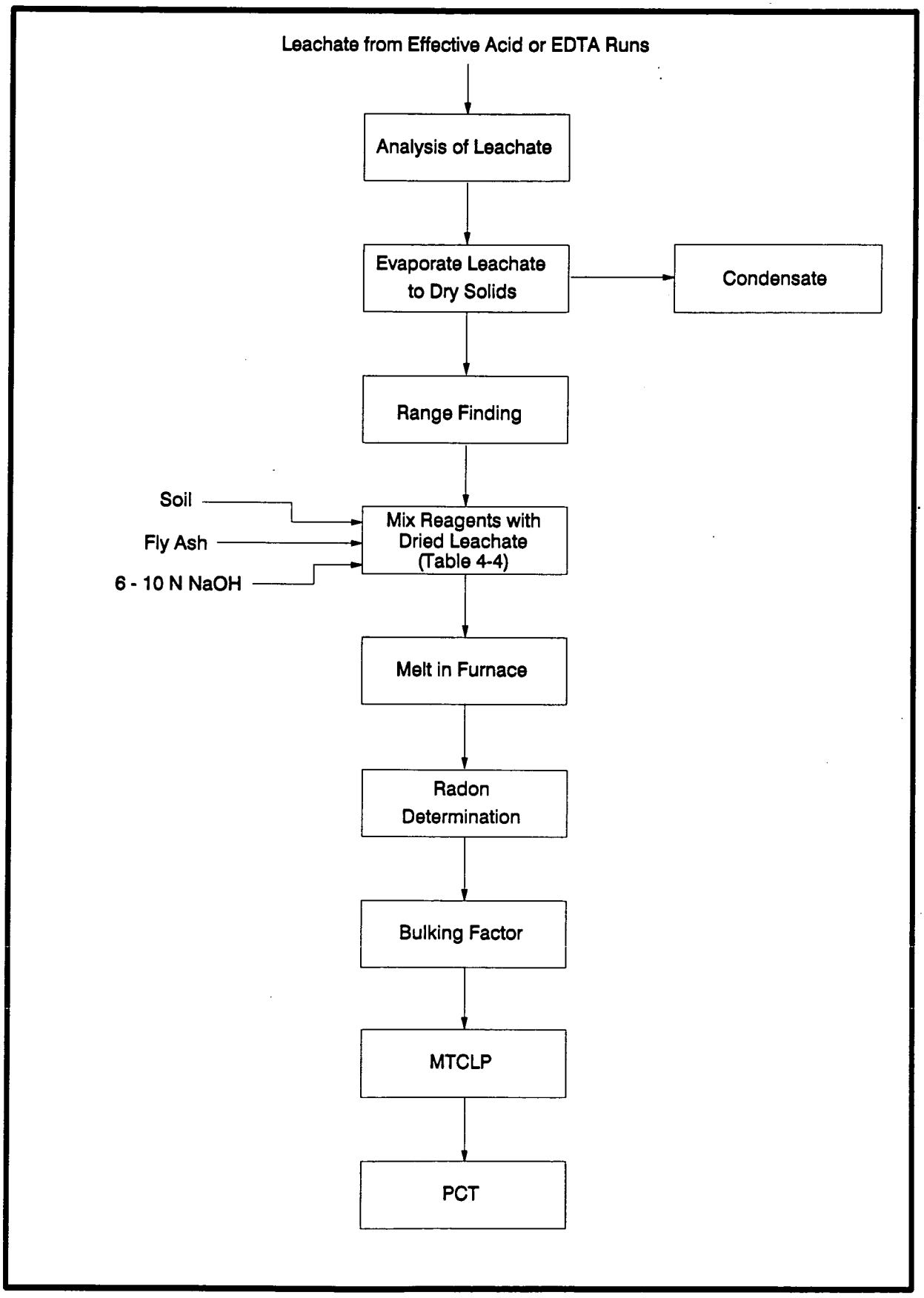


**FIGURE 4-4. Vitrification Flowsheet**

## TABLE 4-4

## VITRIFICATION EXPERIMENT MATRIX

| Run Number | NaOH[a] | Site Fly Ash[a] | Site Soil[a] |
|---|---|---|---|
| 1 | 0 | 0 | 0 |
| 2 | 0 | 50 | 0 |
| 3 | 0 | 0 | 50 |
| 4 | 10 | 50 | 0 |
| 5 | 10 | 0 | 50 |
| 6 | 20 | 50 | 0 |
| 7 | 20 | 0 | 50 |

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

For each of the experiments that are not range-finding experiments, the bulking factor will be recorded. MTCLP and PCT leaching tests will be performed. Radon emission tests will be conducted.

4.2.3 Leaching Time and Temperature - Preliminary Phase - Stage 1

This set of experiments will use the most promising formulation from Section 4.2.1.3. Initial range-finding experiments will be conducted to determine the maximum time the samples will be extracted in the later statistical experiments. The samples will be extracted at 80°C for 7 and 24 hours. Uranium will be analyzed by ion chromatography (IC). Lead will be checked with the inductively coupled plasma (ICP). If the concentrations of uranium and lead in the leachate are similar for the two experiments, the seven-hour extraction times will be used as the maximum extraction time in the statistical study. Otherwise, the maximum time will be 24 hours. The range-finding experimental matrix is in Table 4-5A.

The proposed statistical matrix is in Table 4-5B. Experiment Numbers 1 through 5, in Table 4-5B, are constructed in a two by two factorial experimental design matrix with a center point. The minimum temperature and time of extraction are 25°C and one hour. The maximum temperature and time of extraction are 80°C and seven hours. The proposed maximum time of extraction may be increased as a result of the range-finding experiments.

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 the optimum conditions predicted from the mathematical model will be completed.

4.2.4 Washing Studies - Preliminary Phase - Stage 1

Washing studies of the leached solid will be executed using washing data from Section 4.2.1 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 by IC and ICP, respectively.

4.2.5 Precipitation of Metals in the Leachate Solutions

4.2.5.1 Precipitation of Metals in the Leachate Solutions - Preliminary Phase - Stage 1

Acid Extractions Solution

Precipitation reagents will be added to aliquots (3 to 5 cc) of the leachate solutions from Section 4.2.1.3. The reagents to be investigated are the sodium or potassium salt solutions of hydroxide,

## TABLE 4-5A

## RANGE-FINDING LEACHING TIME MATRIX

| Experiment No. | Temperature (°C) | Time (hr) |
|---|---|---|
| 1 | 100 | 7 |
| 2 | 100 | 24 |

## TABLE 4-5B

## 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 |

sulfide, sulfate, carbonate, and phosphate. Alum, ferric sulfate, and aqueous sodium silicate ($Na_2O$: $SiO_2$) will also be investigated. Alum and ferric sulfate additions will be followed by the appropriate pH adjustments. Slurries of magnesium oxide and calcium hydroxide and dolomitic lime will also be tested. The solutions will be either syringe-filtered or filtered through a centrifugal microfilter using a 0.45-micron filter. The filtrate will be analyzed for uranium and lead as noted in Appendix B.

A 0.45- micron filter is used to determine if a removable precipitate is formed. If larger particulates are needed to improve filtrations or settling, polymer addition and a filter aid may be used.

A series of reagents will also be added in a sequential order where the "first addition" reagent is added and allowed to react before the "second addition" reagent is added. A list of the tests using sequential addition is in Table 4-6. A flow sheet for precipitation of extracted metals is given Figure 4-5.

The most promising reagent formulations will be determined by use of professional judgment. The experiments will note the appearance of turbidity and precipitation in the solution. Correlations between change in pH and onset of turbidity and precipitation, and correlations of pH with volume or weight of titrant added will be noted. The experiments will also note the rate of setting and which reagents lower the uranium and the lead the most. The general procedure of this work plan is an iterative process where the results from matrices of experiments are used to determine the course of the next set of experiments.

<u>EDTA Chelant Extraction Liquid Decontamination</u>

The metal-laden chelant solution from the most promising extraction treatment will be treated for metals removal from the liquid by the following methods. The methods are listed in order of testing sequence. If one of the bulleted methods work, the methods listed in subsequent bullets may not be tested.

- Alkaline precipitation - Tests will be performed by addition of sodium hydroxide, $Na_2CO_3$, or $Na_3PO_4$ to the liquid. Filtration and subsequent analysis of the treated liquid will determine the effectiveness of the treatment. IF none of the above are successful, a preliminary treatment with $Fe^{3+}$ (to displace other metals) will be used, followed by alkaline precipitation.

- Insoluble chelant treatment - Tests will include treatment with and without $Fe^{3+}$ preliminary addition at a pH 3 of 6 (to displace other metals), followed by addition of another organic chelant that forms a stronger insoluble complex. The correct pH (using sodium hydroxide addition) will be determined empirically based on previous experience.

- Electrochemical treatment - An electrochemical cell can be used to remove metals while regenerating the chelant extraction liquid. This process consists of an electrochemical

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**TABLE 4-6**

**PRECIPITATION OF LEACHATE SOLUTION**

| First Addition | Second Addition |
|---|---|
| $Na_2O:SiO_2$<br>$Na_2O:SiO_2$<br>$NA_2O:SiO_2$<br>$Na_2O:SiO_2$<br>$Na_2O:SiO_2$<br>$Na_2O:SiO_2$ | NaOH<br>$Na_3PO_4$<br>$Na_2CO_3$<br>$Na_2S$<br>MgO<br>$Ca(OH)_2$ |
| MgO<br>MgO<br>MgO | $Na_3PO_4$<br>$Na_2CO_3$<br>$Na_2S$ |
| NaOH<br>NaOH<br>NaOH | $Na_3PO_4$<br>$Na_2CO_3$<br>$Na_2S$ |
| $Na_3PO_4$<br>$Na_3PO_4$<br>$Na_3PO_4$ | NaOH<br>MgO<br>$Ca(OH)_2$ |

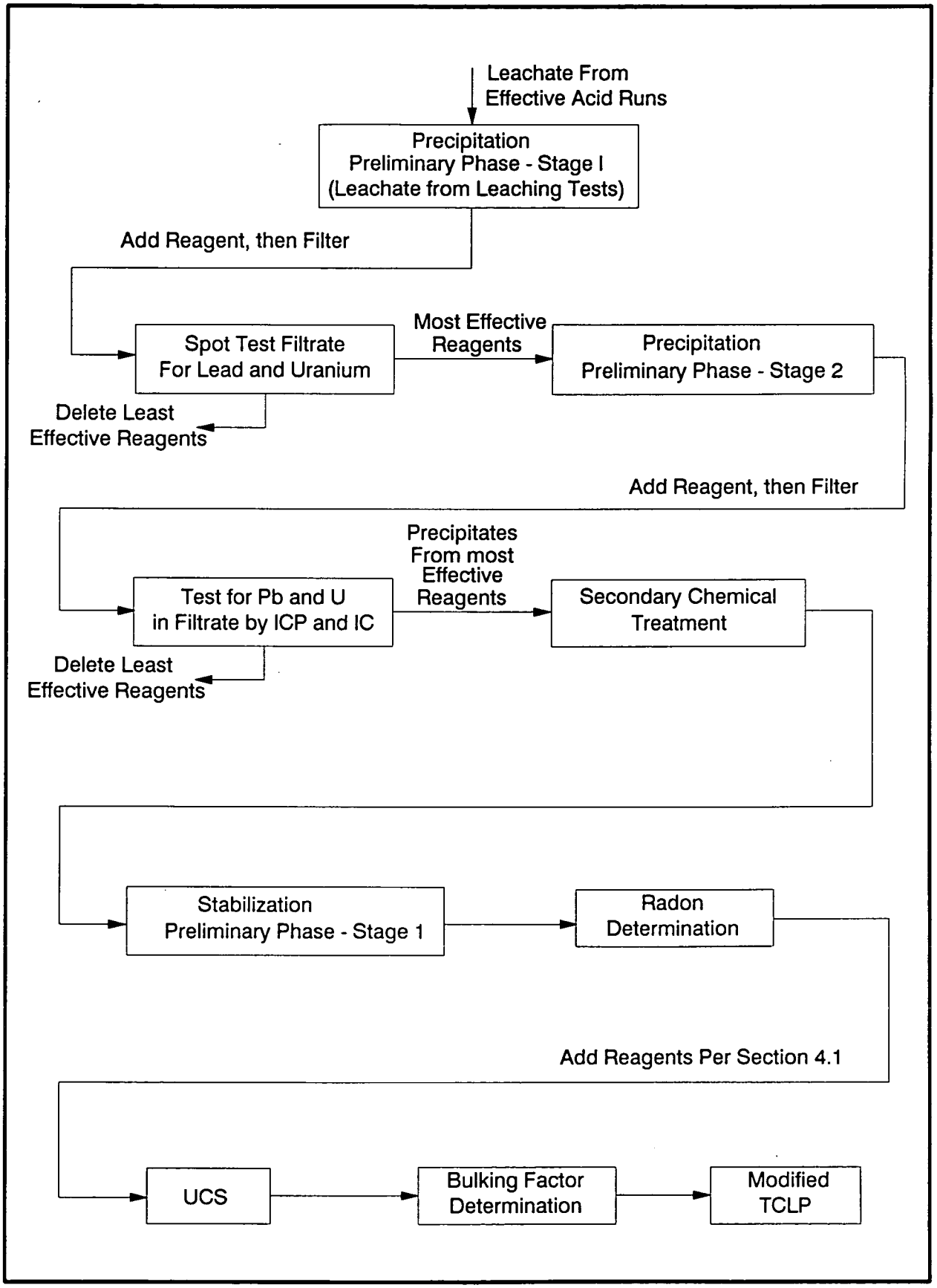



**FIGURE 4-5. Precipitation Flowsheet**

cell divided into two chambers by a cationic ion exchange membrane. One chamber contains the cathode and metal chelate solution, while the second contains $Na_2CO_3$ and the anode. During the process, metals are plated at the cathode while, $Na^+$ ions migrate across the cationic exchange membrane to place the working chelant in the $Na^+$ form.

- Sodium sulfide treatment - If none of the above treatments are successful, sodium sulfide will be added to the metal chelate liquid to produce the insoluble metal sulfides. After filtration of the precipitate, samples will be analyzed for metals.

4.2.5.2 Precipitation of Metals in the Leachate Solutions - Preliminary Phase - Stage 2

Larger aliquots (50 to 100 cc) of the leachate solution will be tested with the most promising precipitation reagents from Section 4.2.5.1. Settling rates will be determined. 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 precipitation tests are necessary, then further composite waste samples (presumably 300 to 500 grams) will need to be extracted to finish the tests.

4.2.5.3 Precipitation of Metals in the Leachate Solution - Settling - Polymer - Preliminary Phase - Stage 2

If settling or filtration rates are very slow, then jar tests using inorganic coagulants (such as ferric sulfate) and/or organic polymers (such as Nalco #7768 anionic polymer). Preliminary range finder tests will be performed with up to 10 different reagent combinations, incrementally adding the reagents until the appearance of floc. The most promising treatment, based on dosage versus sludge volume and effluent quality, will be tested at four different dosages to determine the most effective reagent dosage. A settling test will be run on the best treatment and dosage. The clear supernatant liquid will be sampled and analyzed for total and dissolved lead and uranium.

4.2.5.4 Precipitation of Metals in the Leachate Solutions - Settling - Filter Aid - Preliminary Phase - Stage 2

If the filtration rates are slow, 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.

4.2.5.5 Precipitation of Metals in the Leachate Solutions - Ion Exchange - Preliminary Phase - Stage 2

Ion exchange will be tested as a final polishing step for precipitation/filtration-treated extraction liquid. This testing will consist of 10 isotherms using several different ion exchange resins.

4.2.6 Stabilization of Precipitated Material - Preliminary Phase - Stage 1

The most effective stabilization reagents determined from the screening described in Section 4.1 will be used as a guide in determining the formulations to investigate. Up to 10 formulations will be examined with the precipitated material. Precipitated material generated in the conduct of Section 4.2.2 will be used. Shear strength and temperature rise will be recorded within 10 minutes of mixing. Volume increase will be measured on Day 28 by water displacement. Samples with a UCS of approximately 500 psi will be subjected to MTCLP. Radon emission tests will be conducted. If 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.

4.2.7 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 lead 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 in filtrate

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

- UCS as measured by a soiltest U-590 or U-610 instrument (SOP No. TCL 1109, Appendix B)
- MTCLP
- Bulking factor
- Waste form temperature rise after waste and reagents are mixed and the time between mixing and temperature measurement

- Approximate shear strength measured within 10 minutes of when waste and reagents are mixed
- Physical characteristics: percent moisture and bulk density
- Amount of water added to each waste form
- The maximum particle size treated; weight and percentage of material sieved from the raw waste before treatment
- General description of the waste form before and after reagents are mixed. This includes a description of any grinding of the sample to meet particle size requirements for UCS
- Description of vapor or gas released during mixing and during curing of mixture
- pH and Eh of mixture before adding mixture to molds
- pH of MTCLP extraction fluids
- Radon emission test results for the solidified material

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
- Physical characteristics: percent moisture, bulk density
- Radon emission tests results

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

## 5.0 EQUIPMENT AND MATERIALS

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

## TABLE 5-1

## EQUIPMENT AND MATERIALS

| Item | Description |
|---|---|
| Multiple | Plastic containers, 5 oz and 8 oz |
| Multiple | Spatulas |
| Multiple | Crucibles |
| 1 | HACH digital pH meter |
| 1 | Glass melter furnace |
| 2 | HACH COD digesters Model 45600-00 and associated vial |
| 1 | Soiltest laboratory vibrating table |
| 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 or equivalent planetary mixer |
| 1 | alpha survey meter and beta, gamma scanner |
| 1 | Soiltest Torvane |
| 50 | TFE bombs |

Note: This equipment list does not include analytical instrumentation for leachate analyses; equipment for TCLP, PCT, or 5-day static leach tests; equipment for radion determination; or general laboratory equipment.

# 6.0 SAMPLING AND ANALYSIS

A review of the Characterization Investigation Study (CIS) (Weston 1986) data revealed additional requirements for Silos 1 and 2. These data are needed for the final design of the remedial actions and also for the evaluation of the risks associated with remediation. Consequently, a Sampling and Analysis Plan (SAP) for resampling Silos 1 and 2 has been prepared and approved. Actual field sampling ended in August 1991. The samples taken in this sampling program will be used for this laboratory screening.

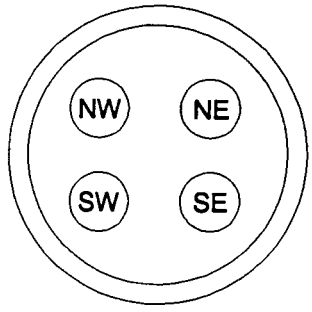
A total of 24 samples were taken from Silos 1 and 2 under the sampling program (Figures 6-1, 6-2, and 6-3). The spatial variability of the silo contents considered both horizontal and vertical variability. The known disposal history indicated that the K-65 residuals are homogeneous in the horizontal direction and nonhomogeneous in the vertical direction. The 1990 resampling program established, through a visual observation of archive samples recovered from the southwest manways of Silos 1 and 2, that there is not a continuous strata variability in the vertical direction.

The sampling technique involved sampling each silo manway in one-third attempts. With the homogeneous nature and minimal horizontal variability of the silo content material, a composite sample for analytical and physical analysis from each one-third sample attempt would yield sufficient data to characterize the K-65 silo material in Silos 1 and 2.

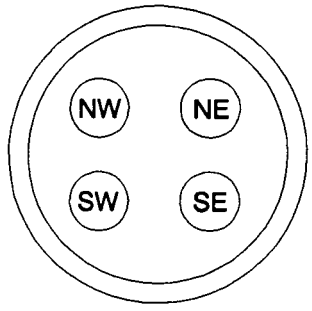
Each one-third sampling effort yielded one sample composite per sampling attempt. With a required high radiological sample per manway core, a total of four samples was retrieved from each silo manway. Three manways per silo were sampled with the fourth manway (southwest) completed during the 1990 resampling program for archival purposes only. Each silo shall yield 12 composite samples for analytical and engineering analysis. A total of 18 composite samples with 6 radiological samples were collected for Silos 1 and 2 analytical analyses.

The physical analysis will be performed on zone composites. A zone is considered to be the material retrieved in a single one-third sampling attempt. An additional sample is a silo composite, which is a composite of each zone for all three manways for each silo. Three zone composites and a silo composite will be submitted for physical analysis for each silo. Undisturbed samples from each manway sampled has been retrieved for geotechnical analysis.

According to the SAP, a full range of radionuclide, organic, and inorganic analyses will be conducted on the retrieved samples. These analyses are listed in Table 6-1. For the material to be treated, this study requires that the presence and concentrations of a number of analytes be known as well as a number of physical parameters. The analytes and physical parameters are of interest because their




SILO #1 (S1)




SILO #2 (S2)

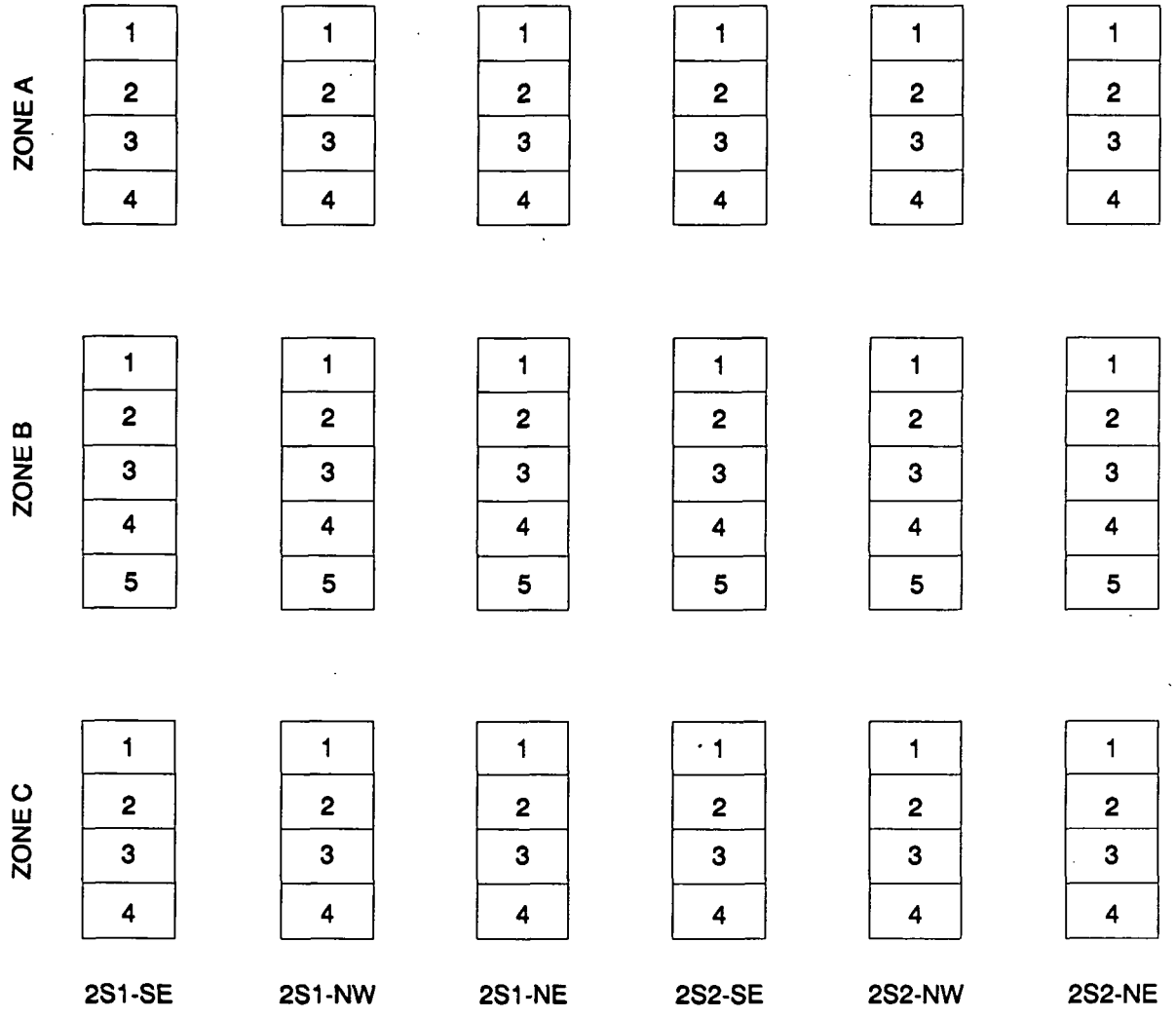
General Sample nomenclature is as follows:

Silo Number - Manway I.D. - Zone I.D. - Section I.D.

Example: 2S1-SW-A-1 indicates second sampling period,

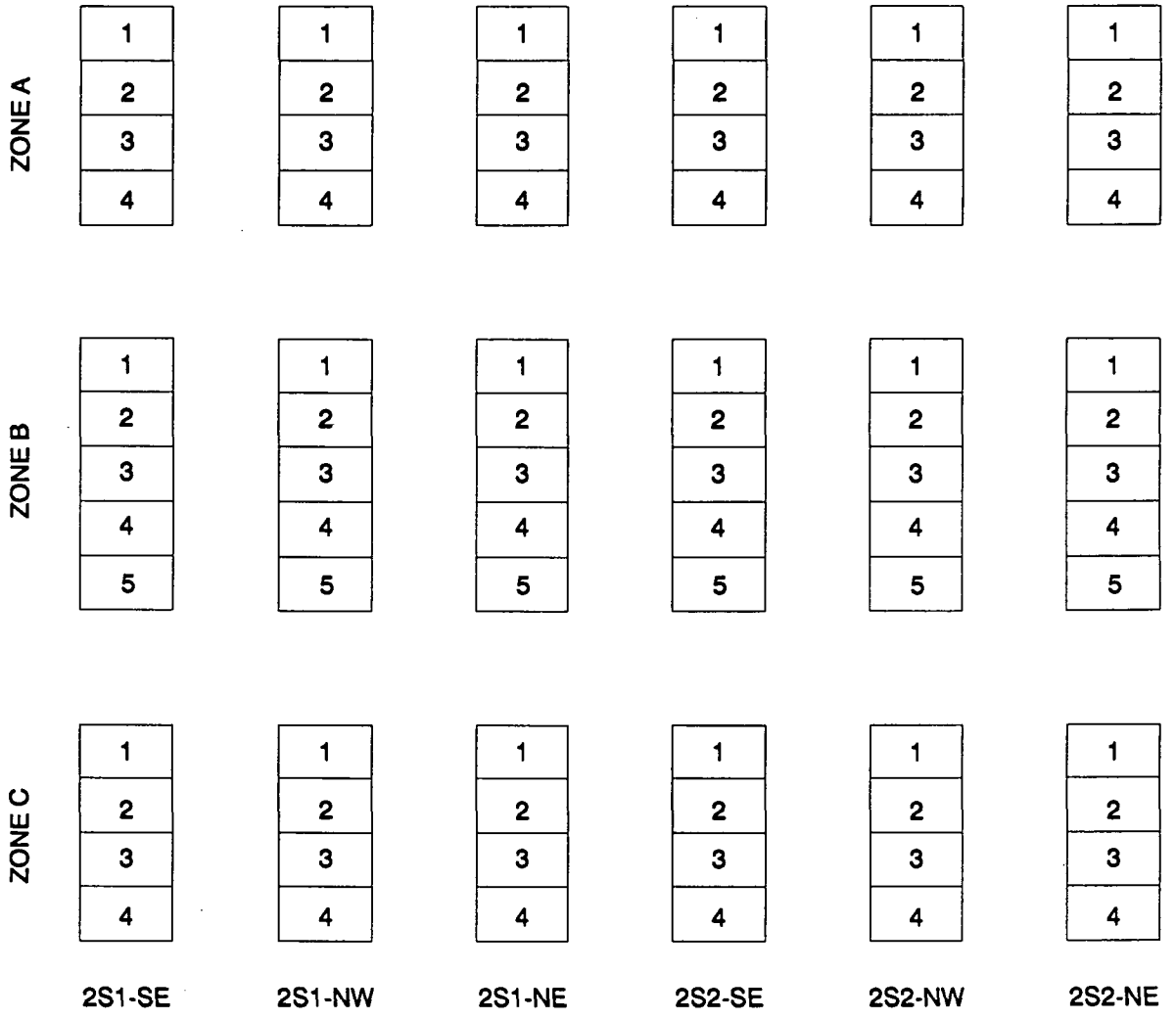
Silo 1 - Southwest manway - Zone A - Section 1

**FIGURE 6-1. Identification of Core Samples**




SILO CONTENT MATERIAL SAMPLE CORE SUBSAMPLES SHALL BE TAKEN FROM ALL OF THE SPECIFIED ZONES ABOVE. THESE ZONES SHALL BE DETERMINED BY THE ONE-THIRD INCREMENT IN WHICH EACH MANWAY IS SAMPLED. ALL SECTIONS SHALL BE BETWEEN 12 AND 18 INCHES IN LENGTH. A COMPOSITE SAMPLE SHALL BE COLLECTED FROM EACH ZONE FOR ANALYTICAL TESTS SUCH AS HSL INORGANICS, HSL ORGANICS, TCLP ORGANICS, TCLP METALS, AND RADIONUCLIDES. A HIGH RADIOLOGICAL SAMPLE SHALL BE TAKEN FROM ONE SECTION PER MANWAY CORE.

**FIGURE 6-2. Sectioning of SE, NW, and NE Sample Cores**




THE SE, NW, AND NE SAMPLE CORES WILL BE SUBSAMPLED FOR ENGINEERING TESTS. THREE COMPOSITED SAMPLES FROM EACH SILO WILL BE MADE UP OF SUBSAMPLES FROM THE SAME HORIZONTAL LAYERS (ZONES). CRITERIA TO SELECT SPECIFIC ZONES FROM EACH CORE FOR SAMPLING WILL BE BASED ON SAME CRITERIA USED IN SECTIONING NE, SE, AND NW CORES LESS THE RADIOLOGICALLY MOST ACTIVE ZONE CRITERIA.

**FIGURE 6-3. Subsampling of Sample Cores for Engineering Tests**

## TABLE 6-1

## ANALYTICAL CHARACTERIZATION PARAMETERS FOR SILOS 1 AND 2 IN OPERABLE UNIT 4

| Required Analyses | No. of Samples |
|---|---|
| Radiological:<br>• Isotopic uranium<br>• Isotopic thorium<br>• Isotopic radium<br>• Lead-210<br>• Gamma spectroscopy<br>• Total uranium<br>• Polonium-210<br>• Protactinium-231<br>• Actinium-227 | 24 |
| Chemical:<br>• TAL inorganics*<br>• HSL volatiles<br>• HSL semivolatiles and tributylphosphate<br>• HSL pesticides and PCBS (if positive hits, confirm by GC/MS)<br>• TCLP metals<br>• TCLP organics | 24 |
| General Chemistry:<br>• Total phosphorous<br>• Total organic carbon<br>• Ammonia<br>• Total Kjeldahl nitrogen<br>• Total organic nitrogen<br>• Oil and grease<br>• Soil pH<br>• Bromide (By Ion Chromatography)<br>• Chloride (By Ion Chromatography)<br>• Nitrate (By Ion Chromatography)<br>• Sulfate (By Ion Chromatography) | 18 |

*Plus boron, cobalt, and thallium

presence and/or high concentrations may have adverse effects on the proposed cement stabilization, chemical separation, and vitrification testing.

The tests to determine physical parameters are listed in Table 6-2.

Silo 3 was sampled under the 1989 program carried out by WEMCO. Results of the analyses for radionuclides, inorganics, and organics are given in Tables 6-3, 6-4, and 6-5.

## TABLE 6-2

## GEOTECHNICAL/PHYSICAL TESTS

| ASTM[a] Designation | Method Title | Minimum No. of Tests |
|---|---|---|
| D2216-80 | Water Content Determination | 8 |
| D4318-84 | Atterberg Limits | 8 |
| D854-83 | Specific Gravity Determination | 8 |
| D422-63 | Grain Size Distribution with Hydrometer Analysis | 8 |
| D2435-80 | One-Dimensional Consolidation | 8 |
| D4253-83 | Maximum Index Drained Triaxial Density | 6 |
| D4254-83 | Minimum Index Granular Soils | 6 |
| No ASTM Designation | In Situ Soils Density Determination | 6 |
| D698-78 | Standard Proctor | 6 |
| D1557-78 | Modified Proctor | 8 |
| No ASTM Designation Department of Army EM 1110-2-1906 | Consolidated Undrained Triaxial with Pore Pressure | 6 |

[a]American Society of Testing and Materials

TABLE 6-3

RADIONUCLIDE CONCENTRATIONS IN SILO 3

| Radionuclide | pCi/g |
|---|---|
| Ac-227 | 412 - 1363 |
| Pa-231 | 266 - 931 |
| Th-228 | ND - 998 |
| Th-230 | 21,010 - 71,650 |
| Th-232 | ND - 911 |
| Ra-224 | 64 - 453 |
| Ra-226 | 467 - 6435 |
| Ra-228 | ND - 559 |
| Pb-210 | 454 - 6427 |
| U-234 | 348 - 1935 |
| U-235/236 | ND - 127 |
| U-238 | 320 - 2043 |
| U-Total (ppm) | 738 - 4554 |

ND - not detected

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TABLE 6-4

INORGANICS CONCENTRATIONS IN SILO 3
(1989 Sampling Program)

| Contaminant (ppm) | Silo 3 |
|---|---|
| Aluminum | 10800 - 23700 |
| Antimony | ND |
| Arsenic | 532 - 6380 |
| Barium | 118 - 332 |
| Beryllium | 10.0 - 39.9 |
| Cadmium | 21.5 - 204 |
| Calcium | 21300 - 39900 |
| Chromium | 139 - 560 |
| Cobalt | ND - 3520 |
| Copper | 1610 - 7060 |
| Iron | 13900 - 67600 |
| Lead | 646 - 4430 |
| Magnesium | 38200 - 80900 |
| Manganese | 2420 - 6500 |
| Mercury | ND - 0.69 |
| Nickel | 1200 - 6170 |
| Potassium | 1300 - 22800 |
| Selenium | 101 - 349 |
| Silver | 9.2 - 23.8 |
| Sodium | 22900 - 51700 |
| Thallium | 3.1 - 73.9 |
| Vanadium | 418 - 4550 |
| Zinc | 301 - 672 |
| Cyanide | ND |

ND = not detected
Note: Data validation is currently in progress

## TABLE 6-5

## ORGANICS CONCENTRATIONS IN SILO 3

| Contaminant | Silo 3 |
|---|---|
| VOLATILE ORGANICS ANALYSIS DATA (ppb) | |
| Methylene Chloride | 1000 - 2800 |
| Acetone | 3400 - 12000 |
| Chloroform | 560 - 810 |
| 2-Butanone | 9700 - 16000 |
| 4-Methyl-2-Pentanone | ND |
| Toluene | 180 - 6800 |
| Trichloroethane | ND |
| Chloromethane | ND - 140 |
| Styrene | ND |
| Total Xylenes | ND |
| SEMIVOLATILE ORGANICS ANALYSIS DATA (ppb) | |
| Bis(2-Ethylhexyl)Phthalate | ND - 40 |
| Di-n-Octyl Phthalate | ND |
| PESTICIDE ORGANICS ANALYSIS DATA (ppb) | |
| Aroclor-1248 | ND |
| Aroclor-1254 | ND |

ND - not detected

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# 7.0 DATA MANAGEMENT

## 7.1 GENERAL

This section pertains to work performed at the Technology Development Laboratory (TDL) only. 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 B.

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 B.

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 - 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.

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## 7.2 STABILIZATION

### Spikes

- TCLP - During the site characterization, the TCLP leachate from the sample will be spiked. Spike recovery will be calculated separately for each silo (1, 2, 3) and for each zone (A, B, C). These spike recovery values will be used with all subsequent TCLP results.

### Blanks

- Reagent blank - Solidify sand or quartz, run TCLP on solidified mass
- Radionuclide test will use a water blank
- TCLP will use the ORL laboratory blank

### Duplicate Analysis

- There will be a 20 percent experimental duplicate of all tests during the advanced phase.

## 7.3 LEACHING/PRECIPITATION/STABILIZATION/VITRIFICATION

### Spikes

- TCLP - During the site characterization, the TCLP leachate from the sample will be spiked. Spike recovery will be calculated separately for each silo (1, 2, 3) and for each zone (A, B, C). These spike recovery values will be used with all subsequent TCLP results.

### Blanks

- Radionuclide test will use a water blank
- TCLP will use the ORL laboratory blank

### Duplicate Analysis

- There will be 20 percent experimental duplicate of all tests during the advanced phase

# 8.0 DATA ANALYSIS AND INTERPRETATION

## 8.1 EFFECTIVENESS OF WASTE FORMS

The results of the leaching tests (MTCLP, TCLP, PCT, and 5-day static) will be used to evaluate the long-term effectiveness of each waste form. The concentrations of radioactive and hazardous constituents in the TCLP leachate (and possibly PCT and 5-day static) will be used as input into the geochemical models described in the draft RI/FS Risk Assessment Work Plan Addendum on Risk Assessment methodology. These models will be used with groundwater fate and transport models, which 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. Fate and transport models are discussed in the draft "Risk Assessment Work Plan Addendum" (DOE 1991).

## 8.2 STABILIZATION

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

- Waste form temperature rise after waste and reagents are mixed, and time between mixing and temperature measurements
- General descriptions of the waste before and after reagent addition. This includes a description of any grinding of the sample to meet particle size requirements for UCS.
- Approximate shear strength measured within 10 minutes of when waste and reagents are mixed
- Physical characteristics: percent moisture, bulk density
- Amount of water, raw waste, and reagents added to each waste form
- UCS (SOP TDL 1109)
- Permeability (for advanced screening)
- Bulking factor
- The maximum particle size treated; weight and percentage of material sieved from the raw waste before treatment
- Description of gases or vapors released during mixing and during curing of mixture
- Physical appearance of mold after 90-day soak in deionized water in optional phase
- pH and Eh of the reagent waste mixture before adding mixture to molds

- pH of MTCLP and TCLP extraction fluids, pH of TCLP extraction fluid determination test
- pH of 5-day static leach solution
- pH and Eh of slightly wet water mixture
- pH of 90-day leach solution in optional phase
- Radon emission test results in advanced phase
- MTCLP (for preliminary phase)
- 5-day static (for advanced phase)
- TCLP (for advance phase). TCLP results will be reported three ways: (1) actual analysis of extract, (2) results corrected for spike recovery, and (3) results corrected for spike recovery and dilution by stabilization reagents.

## 8.3 LEACHING/PRECIPITATION/STABILIZATION/VITRIFICATION

### 8.3.1 Leaching

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

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

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

Advanced phase data will be presented as in Stage 2, with the addition of the following parameters for each test run:

- TCLP of insoluble residue
- Uranium, thorium, radium, and lead 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

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

8.3.3 Stabilization

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

- UCS measured according to SOP TDL 1109
- MTCLP
- Bulking factor
- Waste form temperature rise after waste and reagents are mixed and the time between mixing and temperature measurement
- General descriptions of the waste before and after reagent addition
- Approximate shear strength measured within 10 minutes of when waste and reagents are mixed
- Physical characteristics: percent moisture, bulk density
- Amount of water, treated waste, and reagents added to each waste form
- Radon emissions test results for the solidified material
- Maximum particle size treated; weight and percent of material sieved from the raw waste before treatment
- Description of gases or vapors released during mixing and during curing of mixture
- Physical appearance of mold after 90-day soak in deionized water
- pH and Eh of the reagent waste mixture before adding mixture to molds
- pH of MTCLP extraction fluids

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
- Radon emissions test results

8.3.5 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

8.3.6 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. These formulas can be found in "Preparing Perfect Project Plans" (EPA 1989b).

Example calculations of precision:

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

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:

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

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:

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

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.

Figure 8-1
General QA/QC Report

Analyte:
Matrix:
Sample Number:

| | | Conc. ( ) |
|---|---|---|
| Precision of Duplicates<br>Spike Value (b)=<br>Spike Dup. Value (a)= | | |
| Precision (RPD[a]) | $\frac{\lvert a-b \rvert}{(a+b)/2} \times 100\% =$ | |
| Accuracy of Spike<br>Original Value (a)=<br>Observed Spike Value (b)=<br>Spike Level (c)= | | |
| Accuracy= | $\frac{b-a}{c} \times 100\% =$ | |
| Accuracy of Spike Dup.<br>Original Value (a)=<br>Observed Spike Dup. Value (b)=<br>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 2 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 or other QAPP laboratory for analysis and then will be shipped to DOE's FEMP integrator or environmental remediation management contractor 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 shipped to DOE's FEMP integrator or environmental remediation management contractor for disposal.

# 11.0 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, (2) to explain the role of treatability studies in the RI/FS, and (3) 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 "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 residents have a routine public forum for receiving new information, expressing their views, and getting answers to their questions, meetings will focus on operable unit updates, removal actions, major RI/FS documents, and other appropriate topics.

- Publications - RI/FS materials such as progress reports, fact sheets, a community newsletter *(Fernald Site Cleanup Report)*, and updates of CERCLA-related activities at the FEMP and will include information on treatability study activities for Operable Unit 4.

- Presentations to community groups - Information about treatability studies for this operable unit will be included in briefings to community groups in Ross, Crosby, and Morgan townships, and to Fernald Residents for Environmental Safety and Health, as appropriate. Also, this information will 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 the work plan to DOE and EPA
- EPA approval of the work plan
- Treatability testing
- Submittal of the treatability study report

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

# 12.0 REPORTS

An interim draft report, which will document the results of the stabilization and leaching tests, will be issued following the completion of the preliminary phase. This report will identify the promising stabilization formulation and extraction solutions and will recommend whether those procedures be 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. In addition, all raw data will be presented in a tabular format.

The advanced phase report will be issued following the completion of the experimental portion of the advanced tests. 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. The following outline can be used as a guide when preparing the reports.

SUGGESTED ORGANIZATION OF TREATABILITY STUDY REPORT

1.0 Introduction
- 1.1 Site description
  - 1.1.1 Site name and location
  - 1.1.2 History of operations
  - 1.1.3 Prior removal and remediation activities
- 1.2 Waste stream description
  - 1.2.1 Waste matrices
  - 1.2.2 Pollutants/chemicals
- 1.3 Remedial technology description
  - 1.3.1 Treatment process and scale
  - 1.3.2 Operating features
- 1.4 Previous treatability studies at the site

2.0 Conclusions and recommendations
- 2.1 Conclusions
- 2.2 Recommendations

3.0 Treatability Study Approach
- 3.1 Test objectives and rationale

3.2 Experimental design and procedures 1
3.3 Equipment and materials 2
3.4 Sampling and analysis 3
3.4.1 Waste stream 4
3.4.2 Treatment process 5
3.5 Data management 6
3.6 Deviations 7
4.0 Results and discussion 8
4.1 Data analysis and interpretation 9
4.1.1 Analysis of waste stream characteristics 10
4.1.2 Analysis of treatability study data 11
4.1.3 Comparison to test objectives 12
4.2 Quality assurance/quality control 13
4.3 Costs/schedule for performing the treatability study 14
4.4 Key contacts 15
References 16
Appendices 17
A. Data summaries 18
B. Standard operating procedures 19

# 13.0 SCHEDULE

The schedule to complete all treatability related activities is shown in Figure 13-1. The activities and dates are based on the Operable Unit 4 Consent Agreement Schedule.

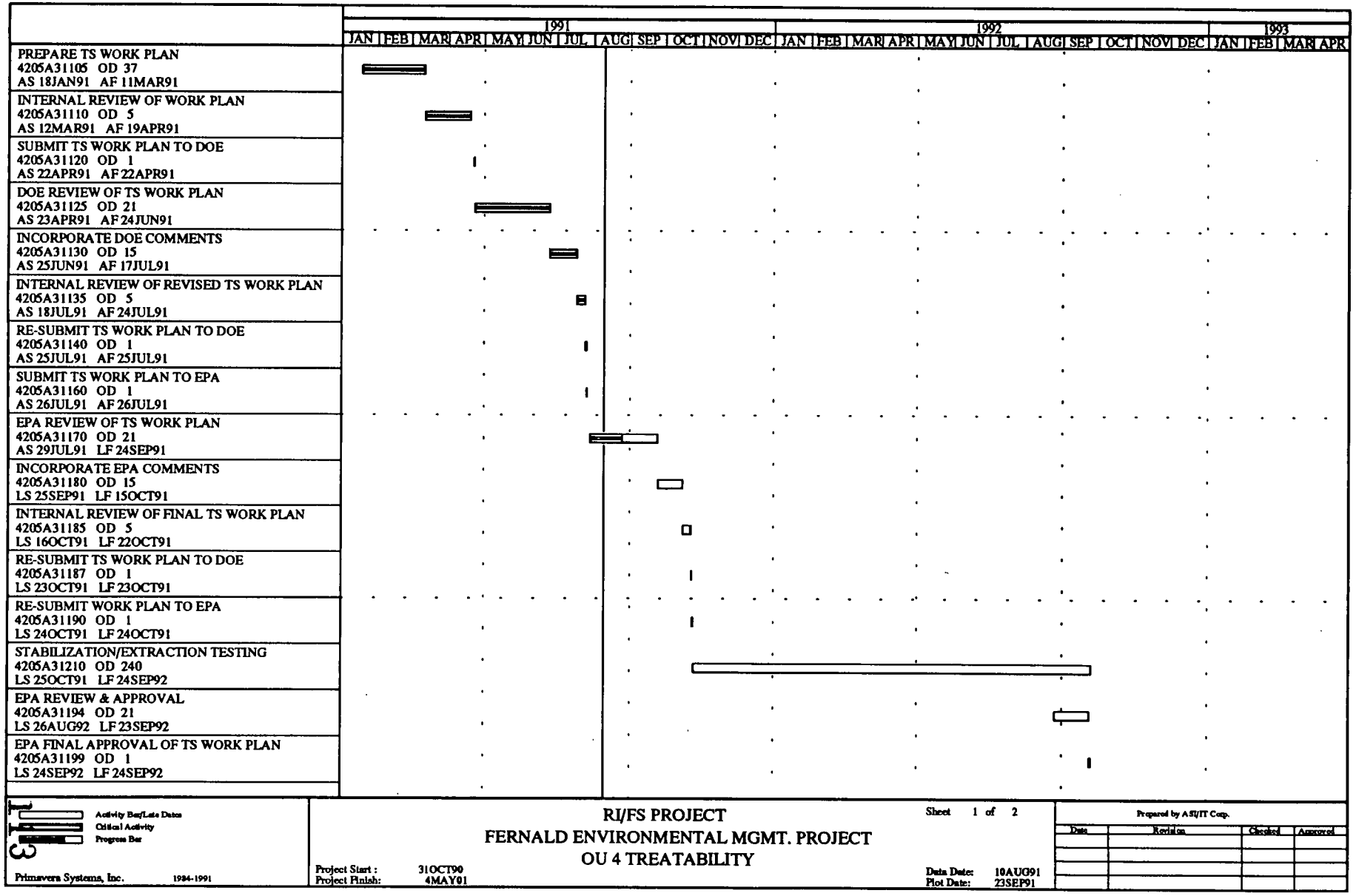


**FIGURE 13-1. Operable Unit 4 Treatability Schedule**

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| | 1991 | | | | | | | | | | | | 1992 | | | | | | | | | | | | 1993 | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | JAN | FEB | MAR | APR | MAY | JUN | JUL | AUG | SEP | OCT | NOV | DEC | JAN | FEB | MAR | APR | MAY | JUN | JUL | AUG | SEP | OCT | NOV | DEC | JAN | FEB | MAR | APR |
| COMPILE TEST RESULTS FROM TS<br>4205A31220 OD 10<br>LS 4DEC92 LF 17DEC92 | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| PREPARE TS REPORT<br>4205A31230 OD 5<br>LS 18DEC92 LF 24DEC92 | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| INTERNAL REVIEW OF TS REPORT<br>4205A31240 OD 5<br>LS 25DEC92 LF 31DEC92 | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| DOE REVIEW OF TREAT. STUDIES<br>4205A31250 OD 21<br>LS 26MAR93 LF 23APR93 | | | | | | | | | | | | | | | | | | | | | | | | | | | | |

Activity Bar/Late Dates
Critical Activity
Progress Bar

Primavera Systems, Inc. 1984-1991

RI/FS PROJECT
FERNALD ENVIRONMENTAL MGMT. PROJECT
OU 4 TREATABILITY

Sheet 2 of 2

Project Start : 31OCT90
Project Finish: 4MAY01

Data Date: 10AUG91
Plot Date: 23SEP91

Prepared by ASI/IT Corp.

| Date | Revision | Checked | Approved |
|---|---|---|---|
| | | | |

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**FIGURE 13-1. Continued**

## 14.0 MANAGEMENT AND STAFFING

An organizational chart for the management of the Operable Unit 4 treatability study is provided in Figure 14-1. The principle parties include: DOE Fernald, WEMCO, ASI/IT, and IT Technology Development Laboratory.

Personnel involved in the management of the entire RI/FS include: Jack Craig, DOE RI/FS Project Director; John Wood, ASI/IT's Project Director for the RI/FS consultant; and ASI/IT's John Razor, who serves as Deputy Project Director and is responsible for the technical content within all of the RI/FS consultant's documents.

Additional personnel involved in the management of RI/FS treatability programs for all operable units include Briand Wu, ASI/IT's Technical Integration Manager, who is responsible for the RI, NEPA, and Treatability. Also, Sam Wolinsky serves as treatability coordinator for all operable unit treatability studies performed by the RI/FS consultant.

Those personnel specifically involved in Operable Unit 4 include: Randi Allen, the DOE Operable Unit manager; Dennis Nixon, WEMCO's (the integration contractor) Operable Unit manager; and Steve Hammitt, operable unit manager for Parsons, the remedy design contractor. Susan Rhyne of ASI/IT serves as the RI/FS consultant (acting) Operable Unit manager and is the focal point for supervision of the laboratory performing the treatability study.

The IT Technology Development Laboratory personnel will perform the actual treatability testing. Those personnel include Ed Alperin, laboratory manager, who is responsible for all of the treatability testing programs within the treatability laboratory. Darrell Drouhard, project manager/engineer, coordinates all treatability laboratory work between labs and site. Ernie Stine, operations supervisor, is responsible for the technical aspects of the treatability programs at the laboratory; Ken Sadler and Ed Morren perform most of the experiments; Patti Carswell is responsible for all QA activities and reports directly to Jack Hall, the Lab Director.

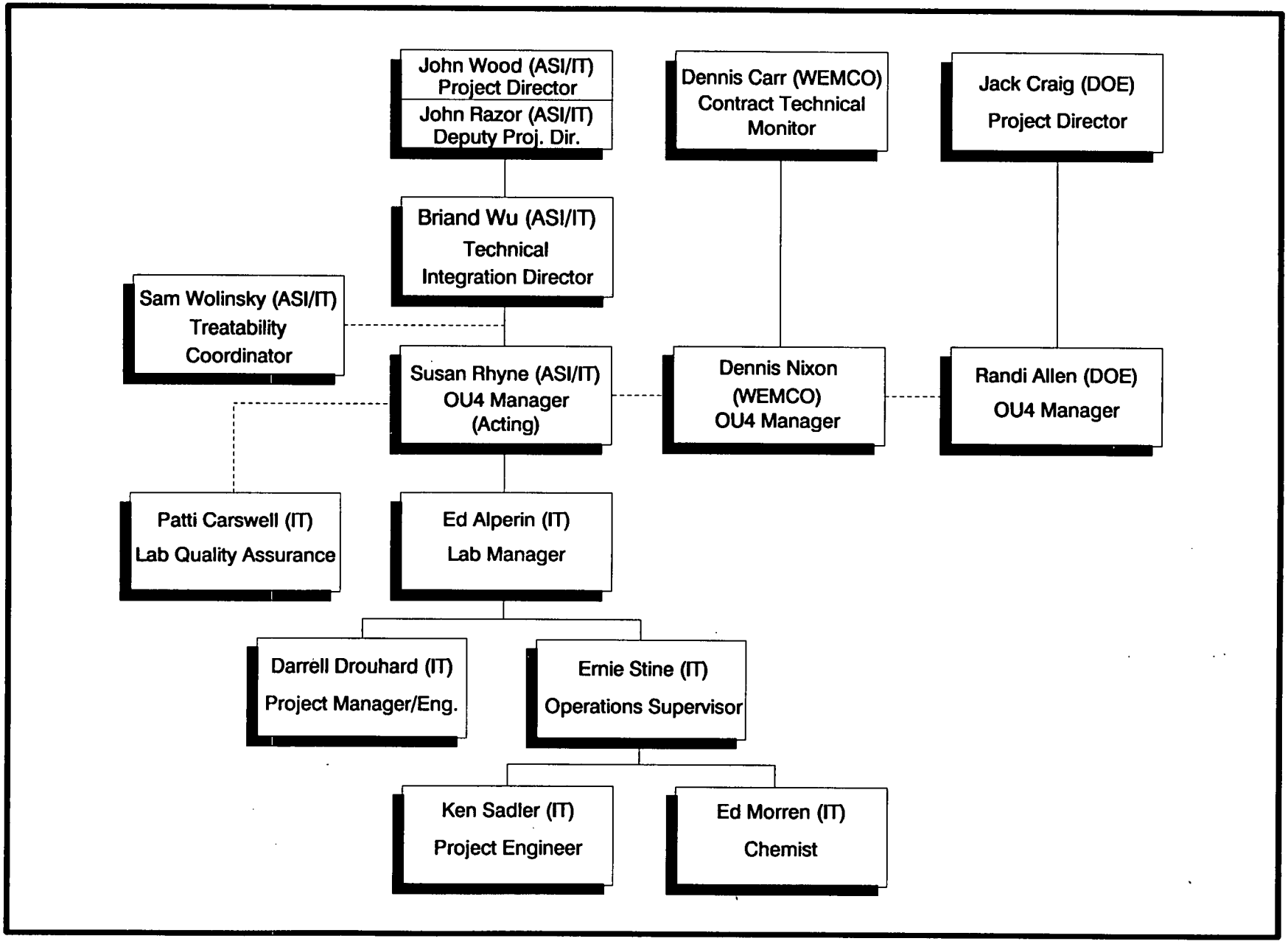


**FIGURE 14-1. Treatability Study Management and Staffing**

# REFERENCES

# REFERENCES

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# APPENDIX A

## SITE-SPECIFIC SAFETY PLAN FOR THE FEED MATERIAL PRODUCTION CENTER SILOS 1, 2, AND 3 TREATABILITY PROGRAM

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# APPENDIX A

## A.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. Stabilization

The following procedures will be conducted in a hood. Samples from each of the silos will be sieved through a 3/8-inch screen. The sieved material will be mixed with stabilizing reagents in a planetary mixer and then placed in molds.

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).

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 Time and Temperature - Leaching test with varying time and temperature using the most promising leaching solution will be conducted. The leachate will be analyzed to determine time and temperature effects.

IV. Washing Studies - The insoluble residue from the leaching experiments will be rinsed several times with deionized water. Each rinse will be analyzed.

V. Precipitation of Leached Materials - Reagents such as sodium or potassium salt solutions of hydroxide, sulfide, sulfate, carbonate, and phosphate will be tested to determine effectiveness of precipitating the metals. Also, alum, ferric sulfate, aqueous sodium silicate, magnesium oxide, and calcium hydroxide may be tested. The supernatant will be filtered and analyzed.

VIII. Solidification/Stabilization of Leached Material - Some of the leachate will be dried to solids, mixed with sodium hydroxide, soil, and other reagents, as appropriate, and melted in an oven. Some of the precipitated solids from V above will be mixed with cement, fly ash, and other suitable stabilizing reagents. These vitrified and stabilized samples 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.

## A.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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## A.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.

### A.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 | $3 \times 10^{-12}$ uCi/mL | $7.5 \times 10^{-13}$ uCi/mL |
| Radium-226 | $3 \times 10^{-11}$ uCi/mL | $7.5 \times 10^{-12}$ uCi/mL |
| Uranium-238 | $2 \times 10^{-11}$ uCi/mL | $5 \times 10^{-12}$ 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) | $2 \times 10^{-11}$ uCi/mL | $5 \times 10^{-12}$ uCi/mL |
| Uranium-234 | $2 \times 10^{-11}$ uCi/mL | $5 \times 10^{-12}$ uCi/mL |

### A.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.

### A.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:

$$10{,}000 \text{ mg liquid} \times 0.001 \text{ mg KCN} \times 26 \text{ mg CN}/65 \text{ mg KCN} = 4 \text{ mg CN}$$

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

$$4 \text{ mg CN} \times 27 \text{ mg HCN}/26 \text{ mg CN} = 4.15 \text{ mg HCN}$$

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.

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## A.4.0 MONITORING

### A.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.

### A.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 or equivalent

### A.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 | 4MPC-hours of Th-230 | Withdraw |
| Thermoluminescent dosimetry (TLD) badge | Y | Continuous | NA, no real time results | |

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| Instrument/chem. | Need | Interval | Limit | Action |
|---|---|---|---|---|
| TLD ring | Y | Continuous | NA, no real time results | |

[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.

## A.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.

## A.6.0 LABORATORY ACCESS

### A.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.

### A.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 uranium and Ra-226. If significant uptake of radioactivity is suspected, fecal samples will be analyzed for Th-230.

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]). 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.

### A.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

### A.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

A.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.

## A.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.

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.

## A.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.

## A.9.0 LABORATORY EXITING PROCEDURE

### A.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.

### A.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.

## A.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 DOE's FEMP integrator or environmental remediation management contractor.

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.

## A.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.

# APPENDIX B

# TECHNOLOGY DEVELOPMENT LABORATORY

# STANDARD OPERATING PROCEDURES

# APPENDIX B
## TABLE OF CONTENTS

Laboratory Notebook Recording Procedures
Analytical Logbook Recording Procedure
Standard Laboratory Sieves: Specification, Calibration, and Maintenance
Bulking Factor Measurement for Pourable Sludge
Calibration of Thermometers
Unconfined Compressive Strength

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 | | | | | |

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

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

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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.

# IT Analytical Services

# Technology Development Laboratory Standard Operating Procedure

itle: Analytical Logbook Recording Procedures

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

eviewed 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*

pproved by: R S Miller Date: 6/7/91
*Laboratory Director*

ontrolled Copy No: Uncontrolled Copy

ey Words: Logbook, Notebook

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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 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

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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.

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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 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.

INTERNATIONAL TECHNOLOGY CORPORATION

# IT Analytical Services

# Technology Development Laboratory Standard Operating Procedure

**Title:** STANDARD LABORATORY SIEVES : SPECIFICATION, CALIBRATION, AND MAINTENANCE

**Prepared by:** Coy A. Lauer Date: March 5, 1991

**Reviewed by:** Tom Leist Date: 3/6/91
*Technical Specialist*

Patti B. Carswell Date: 3/13/91
*Quality Control Coordinator*

Janet M Jones Date: 6-24-91
*Director, Quality and Compliance, ITAS*

**Approved by:** John M Hall Date: 7/5/91
*Laboratory Director*

**Controlled Copy No:** Uncontrolled Copy

**Key Words:** Sieve, ASTM E 11-87, Geotechnical

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| Date | 03/05/91 | | | | | |

# LABORATORY SIEVES
# SPECIFICATION, CALIBRATION, AND MAINTENANCE

## 1.0 Purpose and Application

1.1 This SOP defines the standards for standard laboratory sieves used in the Geotechnical Analysis Laboratory. It also describes calibration requirements and maintenance of the sieves.

## 2.0 References

2.1 ASTM E 11-87, Standard Specification For Wire Cloth Sieves For Testing Purposes.

## 3.0 Associated SOPs

3.1 None.

## 4.0 Definitions

4.1 None.

## 5.0 Procedure

5.1 All standard sieves will meet the specifications in ASTM E 11-87, Standard Specifications for Wire Cloth Sieves For Testing Purposes. Upon receipt, each sieve will be checked for a label which has the ASTM specification, sieve size, and a identification number or serial number. If the ASTM specification is not on the sieve, that sieve will be returned to the vendor and not used. If the sieve size or a serial number is not on the label, prepare a permanent label with the appropriate information and affix it to the side of the sieve. Due to the corrosive nature of some samples, brass sieves with stainless steel mesh are preferred.

5.2 Sieves put into use prior to this SOP do not require a serial number.

5.3 Calibration certificates should be provided by the manufacturer. If a calibration certificate did not come with the sieve, either return it, or get a certificate from the vendor. Calibration certificates will be kept in the Quality/Operations files maintained

by the lab QC Coordinator.

5.4 If a sieve calibration is suspect, it shall be either checked or replaced. Due to the amount of time involved in checking sieve calibration, replacement is usually the preferred alternative. AASHTO proficiency samples may also be used as an indication of sieve calibration. If the results from a proficiency sample are too far out of line (as determined by the lab supervisor), the suspect sieve shall be pulled for calibration or replacement.

5.5 Sieves with a mesh size of #200 or smaller will be replaced one year after initially being placed into service. Each sieve will be labeled with the replacement date at the time it is placed into service.

5.6 Prior to use, each sieve will be visually inspected for holes, broken mesh, or any other condition which may make the sieve unsuitable for use. Sieves which are clogged will be cleaned with a suitable brush. Caution shall be used when cleaning fine sieves with a wire bristle brush as this may damage the sieve. Any sieve deemed unsuitable for use will be immediately discarded.

5.7 Sieves used in washing samples or sieves used with corrosive samples will be cleaned with water and a brush after use. It may be useful to place the sieve in a drying oven (<120 °C) to dry. This will help to keep corrosion to a minimum.

5.8 Sieves will be stored in a clean, dry environment.

6.0 <u>Nonconformance and Corrective Action</u>

6.1 Sieves which do not meet the required specifications, are damaged, or otherwise unsuitable for use will be discarded or returned to the vendor if newly purchased. If a sieve is discovered nonuseable during use, the sample(s) will be retested and a nonconformance memo generated to describe the problem with the sieve and the fact that the sample(s) are being retested.

7.0 Records Management/Documentation

7.1 Sieve calibration records will be kept in the Quality/Operations files by the QA coordinator.

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 Olyer Date: 2/8/91
*Technical Specialist*

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

James 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:**

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| Date | 9/16/90 | | | | | 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 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.

# IT Analytical Services

# Technology Development Laboratory Standard Operating Procedure

**Title:** CALIBRATION OF THERMOMETERS

**Prepared by:** David McNeil **Date:** 5/17/91

**Reviewed by:** Walter W. [illegible]
*Technical Specialist* **Date:** 5/29/91

Patti B. Carswell
*Quality Control Coordinator* **Date:** 5/29/91

Janet M. Jones
*Director, Quality and Compliance, ITAS* **Date:** 6-24-91

**Approved by:** James A. [illegible]
*Laboratory Director* **Date:** 6-22-91

**Controlled Copy No:** Uncontrolled Copy

**Key Words:** CALIBRATION,THERMOMETER,NBS

| Revision # | 1 | | | | | |
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| Date | 02/27/91 | | | | | 157 |

## 1.0 Purpose and Application

1.1 The purpose of this SOP is to detail proper procedures for the calibration of all laboratory thermometers, such that temperature measurements are accurate and traceable.

1.2 This procedure applies to any thermometer used in the laboratory directly or indirectly in the preparation, storage or analysis of samples.

1.3 Working thermometers in the laboratory shall be calibrated annually against reference thermometers that have initial NBS traceability and that are recertified every three years with equipment directly traceable to the NBS.

## 2.0 References

2.1 ITAS-SW SOP No. MW104R0, "Calibration of Thermometers."

## 3.0 Associated SOPs and Applicable Methods

3.1 ITAS System Procedure No. 9014-HSC-01, "General Health and Safety Practices for Tasks Performed in the Laboratory."

## 4.0 Definitions

4.1 None.

## 5.0 Procedure

5.1 Copies of the NBS traceable certification of reference thermometers will be kept in the Quality/Operations files.

5.2 Every three years reference thermometers will be recertified with equipment directly traceable to the NBS. A record of the date of this certification will be kept in the Equipment Maintenance and Calibration files by the QCC.

5.3 Each working thermometer in use in the laboratory will be assigned a unique number and will be calibrated annually against a reference thermometer using the calibration methods listed below as appropriate for the specific use of the thermometer:

5.0 Procedure (continued)

5.3.1 Calibration Method 1:

5.3.1.1 Working thermometer and reference thermometers are allowed to remain together in the same room for at least 24 hours. The bulbs are then put together on desk top for at least 30 minutes and read.

5.3.2 Calibration Method 2:

5.3.2.1 A one-liter beaker is filled with regular refrigerator ice cubes prepared with deionized water. The remainder of space in beaker is filled with deionized water. The working thermometer and reference thermometer are immersed with bottom of bulbs at same level. Wait at least 30 minutes and read.

5.3.3 Calibration Method 3:

5.3.3.1 Fill a one liter glass beaker with deionized water and bring to a boil on a hot plate. The working and reference thermometer are immersed with bottom of bulbs at same level. At least the whole bulb on each thermometer must be completely immersed. Wait 5 minutes and read.

5.3.4 Calibration Method 4:

5.3.4.1 Working thermometers and a reference thermometer are allowed to remain together in a freezer for at least one hour. After one hour, read the thermometers.

5.4 A Thermometer Calibration form (Figure TDL102-1) shall be completed for each working thermometer calibrated and placed in the Quality/Operation files.

5.5 Any thermometer that does not meet the acceptance criteria (± 1°C) shall be tagged to prevent inadvertent use. New thermometers that do not meet the acceptance criteria will be sent back to the vendor. Old thermometers that do not meet the acceptance criteria will be removed from the lab.

5.6 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.

## 6.0 Nonconformance and Corrective Action

6.1 Any thermometer that does not meet the acceptance criteria (± 1°C) shall be tagged to prevent inadvertent use. New thermometers that do not meet the acceptance criteria will be sent back to the vendor. Old thermometers that do not meet the acceptance criteria will be removed from the lab.

## 7.0 Records Management

7.1 A Thermometer Calibration form (Figure TDL102-1) shall be completed for each working thermometer calibrated and placed in the Quality/Operation files.

# FIGURE TDL102-1

## ITAS TECHNOLOGY DEVELOPMENT LABORATORY

## THERMOMETER CALIBRATION

Date: _____

Number of thermometer being calibrated: _____

Description of thermometer being calibrated: _____

_____

Date last calibrated: _____

Time since last calibration _____

Description of reference thermometer: _____

_____

_____

| Calibration Method Number | Temperature Reading | |
|---|---|---|
| | Reference Thermometer | Thermometer Being Calibrated |
| | | |
| | | |

Working range: _____

Acceptance criteria: _____ ± _____ °C

Signed: _____

STANDARD OPERATING PROCEDURE

ITAS-TECHNOLOGY DEVELOPMENT LABORATORY

| TITLE: | SOP NO: TDL1109 |
|---|---|
| **UNCONFINED COMPRESSIVE STRENGTH** | DATE INITIATED: 7/31/89<br>REVISION NO: 1<br>DATE REVISED: 3/28/90<br>PAGE 1 OF 18 |

| 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.

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. Referenced Documents

2.1 *ASTM Standards:*

D 422 Method for Particle-Size Analysis of Soils[2]
D 653 Terminology Relating to Soil, Rock, and Contained Fluids[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 Terminology D 653 for standard definitions of terms.

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

[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.

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, $\epsilon_1$, to the nearest 0.1 %, for a given applied load, as follows:

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

where:

$\Delta L$ = 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, mm$^2$ (in.$^2$), and

$\epsilon_1$ = axial strain for the given load, %.

8.3 Calculate the compressive stress, $\sigma_c$, to three significant figures, or nearest 1 kPa (0.01 ton/ft$^2$), for a given applied load, as follows:

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

where:

$P$ = given applied load, kPa (ton/ft$^2$),

$A$ = corresponding average cross-sectional area mm$^2$ (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.

D 2166

# 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 _____

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

Test Data

Unit Strain $\frac{\Delta L}{L_o}$

$\frac{L}{D}$ = _____

$$\text{Corr. Area} = \frac{A_o}{1 - \text{Unit Strain}}$$

| Elapsed Time-min | Load Dial | Axial Load | Strain Dial | Total Strain | Unit Strain | Corrected Area | Stress | |
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Type of Sample _____

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

Strain Rate _____ %/Min

Remarks _____

*The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.*

*This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.*

# 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

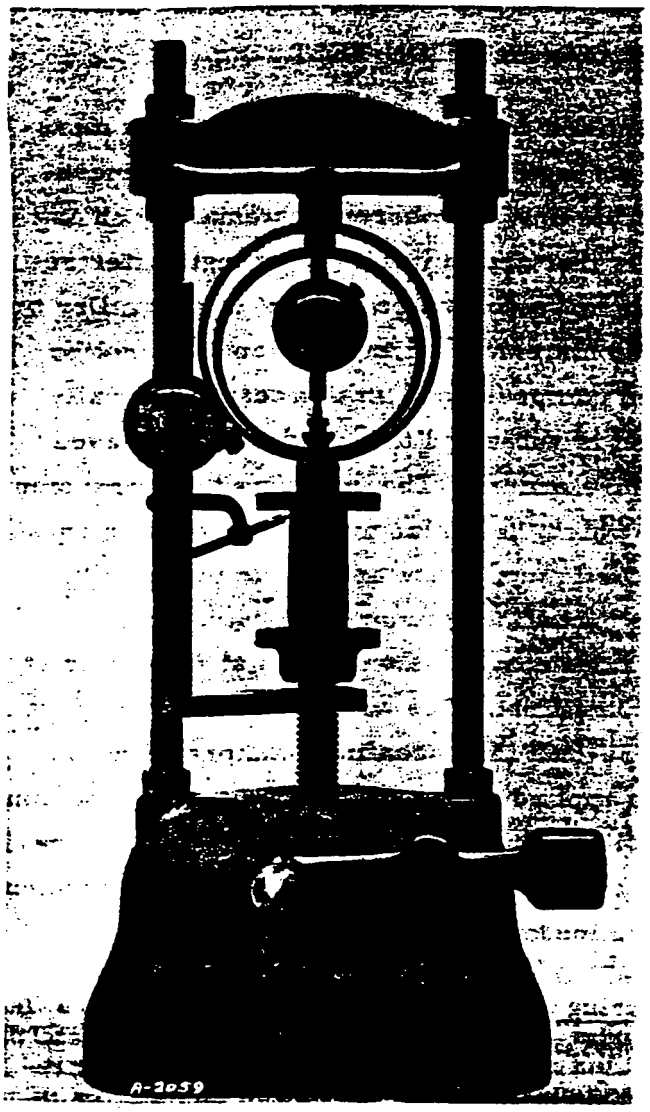


Figure 1. Typical unconfined compression test apparatus

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 shall 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

$\Delta H$ = 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.

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, g: 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 = $62.4\ (W_s \div V_o)$ | | | $\gamma_d$ | |
| 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, $\Delta H$ $10^{-}$ 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 $0.465(P/A_{corr})$ |
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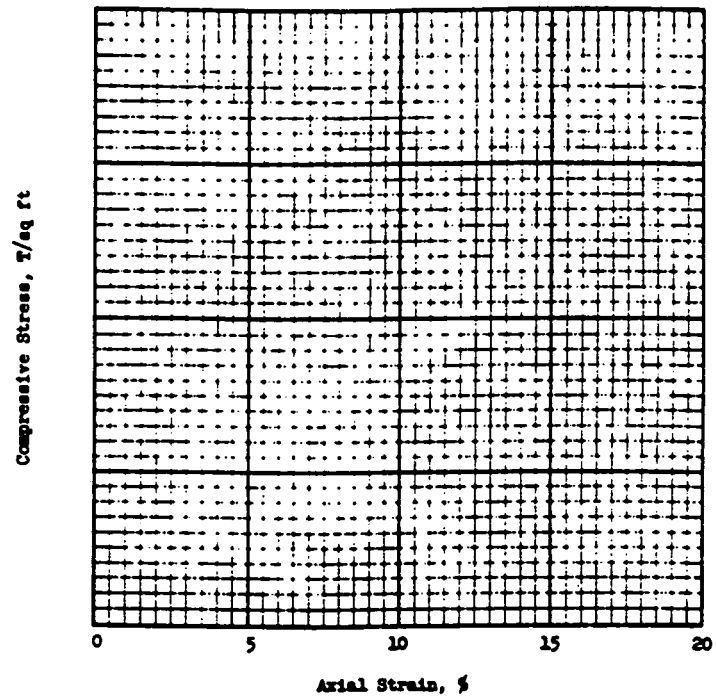
Remarks _____

_____ Type Failure _____

Technician _____ Computed by _____ Checked by _____

PLATE XI-1 ENG FORM 1 JUN 65 3857

Failure Sketches




☐ Controlled stress
☐ Controlled strain

| | | | | | |
|---|---|---|---|---|---|
| Test No. | | | | | |
| Type of specimen | | | | | |
| Initial | Water content | $w_o$ | % | % | % | % |
| | Void ratio | $e_o$ | | | | |
| | Saturation | $S_o$ | % | % | % | % |
| | Dry density, lb/cu ft | $\gamma_d$ | | | | |
| 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 El | Date |
| UNCONFINED COMPRESSION TEST REPORT | |

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PLATE XI-2

# APPENDIX C

# PROCEDURES

# APPENDIX C
# OTHER OPERATING PROCEDURES
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# NUCLEAR WASTE GLASS PRODUCT CONSISTENCY TEST - VERSION 3.0 (U)

A durability test, designated for Product Consistency Test (PCT), has been developed for glasses produced in the Defense Waste Processing Facility (DWPF).[1] The test is designed to meet the requirements of the Waste Acceptance Preliminary Specifications (WAPS) 1.3 and 1.4.[2] Specification 1.3 requires the DWPF to demonstrate control of the radionuclide release properties of the final waste form. Changes in phase composition due to devitrification do not greatly alter the rate of release of material from the glass[3] of the type that will be produced in DWPF. The WAPS Specification 1.4 however requires that the release properties of devitrified glass be similar to those determined in Specification 1.3. The DWPF is responsible for relating the results of the PCT to a repository site-specific release test, or alternatively, for performing the repository site-specific release tests.

The PCT has been developed, in part, to satisfy the WAPS requirements by providing a test which is (1) sensitive to glass composition and homogeneity, and (2) has the potential to be related to repository site-specific release tests. The test was designed to provide confirmation of the consistency of DWPF glass under the following considerations:

- Sensitivity of the test to glass composition and homogeneity
- Time necessary to demonstrate product quality
- Ease of sample preparation for radioactive glass
- Ease of test procedure for remote operation
- Precision of the test results
- Acceptance of waste form developers and repository projects

During PCT development, sample size was limited to 100-200 mesh (149-74 m) crushed glass because leaching of finer mesh sizes can cause overestimation of saturation concentrations, e.g. if finer powders are used, mass balance calculations need to be used to determine the maximum saturation concentration expected from a given particle size.[4] Fine particles also contribute larger errors to the estimation of the sample surface area than coarser sized samples. Moreover, use of a coarser mesh crushed glass simplifies sample preparation for radioactive service.

One test temperature, 90°C, was chosen for the PCT. This temperature is representative of the anticipated temperature in a repository because of the heat of decay of the radionuclides in DWPF waste glass. A single leachant, American Society for Testing and Materials (ASTM) Type I water, was specified so that the test would be dominated by elemental species leached from the glass.

The $v_{soln}/m_{solid}$ ratio for the PCT was chosen as 10 mL/g and test durations of 1, 3, 7, 14, and 28 days were evaluated. Seven days was chosen as the minimum test duration that optimized test precision but did not sacrifice discrimination.[1]

Leachate filtration to <0.45μm was determined to improve the precision of the PCT. Filtering is advantageous because it removes colloidal species that would otherwise dissolve during the leachate acidification step and erroneously be measured as soluble elemental species. Filtering the leachate also removes the potential for fine glass particulates to become entrained in the leachate acidification.[5] Such a dissolved particulate of glass would give an erroneously high soluble leachate concentration or contribute excessive radioactivity to the leachate.

PCT sample preparation specifies that the sieved glass should be washed in ASTM Type I water and absolute ethyl alcohol to remove electrostatically adhering fine particles. Comparisons of B.E.T. specific surface area measurements of alcohol washed and unwashed crushed basalt demonstrated that there was less than a 5 percent difference in the total surface area.[5] Other studies[6-9] have demonstrated that the <1μm fine particles only affect the initial non-linear kinetics of dissolution, e.g. the first 24-hour period. Thereafter, the fines are consumed with no further effect on the bulk dissolution. The amount of fines adhering to a glass sample however, is an uncontrollable quantity and, hence, sample washing was included in the PCT. Later experimental studies verified that sample washing improved the precision and the accuracy of the PCT.

An Savannah River Laboratory (SRL) internal round robin[1] and a seven-laboratory external round robin were completed[10] to determine the precision and accuracy of the PCT. Confirmatory testing on radioactive samples was also performed.[11] These studies indicated that the PCT was very reproducible, yielded reliable results rapidly, and could be easily performed in shielded cell facilities with radioactive samples.

This draft was submitted to ASTM subcommittee C26.13 on Repository Waste Package Materials Testing in January 1990.

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## BULKING FACTOR PROCEDURE FOR NONSLUDGE TYPE WASTE

The bulking factor is the measured percent volume increase/decrease of the treated waste, relative to the original waste volume. The bulking factor measurement for a pourable waste sludge will follow the Standard Operating Procedure (SOP) in Appendix B. For a nonsludge material, the bulking factor will be determined by using bulk density values. The bulking factor will be calculated by using the following equation:

$$BF = 100 * \frac{[(100 + A)/P_t - 100/P_r]}{100/P_r} \qquad (1)$$

where

BF = percent change in volume relative to untreated waste
A = percent additives relative to untreated waste (weight to weight)
$P_t$ = density of treated waste
$P_r$ = density of raw waste

The bulk density of the raw waste will be determined in the site characterization. The bulk density of the treated waste will be calculated by dividing the weight of the unconfined compressive strength (UCS) solid cylinder (e.g., 1.5 by 3 or 2 by 4 inches) by its volume. (See "Stabilization/Solidification of CERCLA and RCRA Wastes," (EPA/625/6-89/022), Section 4.2.4 for a description of bulk density measurement of stabilized waste.)

Bulk density of the raw waste values used in the treatability study will be averaged values from several locations in each pit. These average values will be used in the bulking factor calculation.

The BF equation was derived as follows:

BF is defined as the percent change in volume resulting from treatment to the initial volume. This change can be presented mathematically as follows:

$$BF = 100 \frac{V_t - V_r}{V_r} \qquad (2)$$

where

$V_t$ = volume of waste after treatment

$V_r$ = volume of waste before treatment

Volume can be expressed as a function of density.

$$V = \frac{m}{P} \tag{3}$$

where

m = mass of waste
P = density of waste

Equation (2) can be used to express $V_t$ and $V_r$.

$$V_r = \frac{m}{P_r} \text{ and} \tag{4}$$

$$V_t = \frac{m + t}{P_t} \tag{4}$$

where

t = mass of reagents added

Substituting equations (3) and (4) into (1) gives:

$$BF = 100 \frac{[(m + t)/P_t - m/P_r]}{m/P_r} \tag{5}$$

This can be reduced as follows:

$$BF = 100 \frac{[(1 + \frac{t}{m})/P_t - 1/P_r]}{1/P_r} \tag{6}$$

$\frac{t}{m}$ is the fraction of reagents relative to the untreated waste. This can also be expressed as a percentage and redefined as follows:

$$\frac{100\ t}{m} = A \tag{7}$$

Using equation (7) in (6) gives

$$BF = 100\ \frac{[(100 + A)/P_t - 100/P_r]}{100/P_r} \tag{8}$$

## 5-DAY STATIC LEACH TEST PROCEDURE

The 5-day static leach test uses a monolith and demineralized water. These conditions are more representative of what would be expected for waste placed in a disposal facility. The 5-day static leach test is a modification of the American National Standard Measurement of the Leachability of Solidified Low-Level Radioactive Waste by a Short-Term Procedure. The 5-day static leach test differs from the ANSI/ANS-16.1-1986 as follows: the treated sample is leached for a 5 days continuously instead of 12 wash-leach periods over 90 days, the sample is supported in the leaching solution by a permeable polymeric material or a Teflon® cage, the effective diffusion coefficient will not be calculated, and the concentration of the metals in the treated sample before leaching will not be analyzed. Optionally, the sample may be soaked in another batch of deionized water leachant for an additional 85 days. The physical appearance of the sample would be noted after the cumulative 90-day leaching. The leaching solution may be analyzed as with the 5-day leaching solution.

## MODIFIED TCLP LEACH TEST PROCEDURE

The modified toxicity characteristic leaching procedure (MTCLP) leach test is a modification of the TCLP test. The TCLP procedure is in Federal Register, Vol. 55, No. 126, pages 26,986 through 26,998. The MTCLP screening data will be acquired in the initial stage(s) to minimize costs and waste generation.

The same leachant to solid ratio and leachants (TCLP Type 1 and 2) are used in both procedures. The MTCLP differs from the standard TCLP as follows: the MTCLP uses 2.5 grams of material instead of 100 grams; the MTCLP generates 50 milliliters of leachate instead of 2 liters; and the leachate from the MTCLP is analyzed for metals only rather than metals and organics.

## WASTE AND REAGENT MIXING PROCEDURE

The waste will be sieved through a 3/8-inch-mesh screen before testing. Obvious debris such as chunks of wood and metal will be removed. The percent weight and visual observation of removed debris will be noted. In the preliminary phase, 100 to 110 grams of waste and correct amounts of reagents will be mixed in a plastic container or a metal mixing bowl. The amount of water added will be determined empirically. Enough water will be added to make the mixture into a paste. Mixing will be done by hand with a spatula until the mixture has an even consistency without any lumps or mixed in a Planetary mixer. The mixture will be compacted using a vibrating table. The plastic container will be filled approximately half full and vibrated at least 1 minute. The remainder of the container will be filled and vibrated for another 1 minute. The vibrating table will be set at approximately 38 percent maximum power. The container will be sealed with a lid and taped. The treated samples will be cured at room temperature for 28 days in the sealed containers.

In the advanced phase, approximately 300 grams of waste per mold will be mixed with the correct amount of reagents in Planetary mixer. The mixture will be placed into a 2- by 3-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.

## STABILIZATION WASTE FORM TEMPERATURE RISE GENERIC PROCEDURE

1. Measure room temperature (A).

2. Mix waste and reagents thoroughly to homogenize the mixture.

3. Place 50 to 100 grams of homogenized mixture in a separate container. If the sample is cohesive, press the mixture into a mass along the side of the container. Place the thermometer near the center of the mass.

4. Monitor the mixture temperature. Record the temperature when the temperature reaches a peak and starts to decline (B).

5. Calculate the temperature rise (dT): $dT = B - A$.

The measured temperature rise is a qualitative test. It is conducted as a screening test to alert of potential problems and hazards during scale-up. Further investigations of the actual temperature rise may be made during the remedy design phase when larger equipment, which has a design similar to the full-scale equipment, will be used.

## PERMEABILITY

The permeability of the treated samples will be determined by using procedures in EPA SW-846 and EM-1110-2-1906 as guidelines. There are several methods to choose from, depending on the sample matrix, and sample constraints (e.g., radioactivity and hazardous contaminants, sample condition on receipt, and clients' end use).

The method of choice for determining permeability of treated samples is described in SW-846, Method 9100, Section 2.8. This is the constant-head method using a triaxial-cell with back pressure. This method is applicable to cohesive samples, which are supplied in a molded form.

The constant head triaxial cell method may take a couple of days longer to run, but there is more control over sample conditions during the test, and a wide range of field conditions can be simulated.

There will be one slight modification to the method. A permeability cell will be substituted for the triaxial cell. The permeability cell is similar to the triaxial cell but does not have the plunger for applying a load to the sample. This plunger is not used in permeability testing, and its absence has no effect on the test.

It is anticipated that all of the samples for permeability testing will be of the cohesive, molded type. If a sample is in a form that precludes the above test, there are several options available in the referenced method. Items that would preclude the above test may include: small sample size due to radioactivity level, noncohesive sample, loose sample requiring remolding, and chemicals in the sample that are incompatible with the latex membrane.

A small sample size may require permeability testing in a consolidation cell. This method is not addressed in SW-846, but is found in the Army Corps of Engineers Manual EM 1110-2-1906, Appendix VII, paragraph 8.

Noncohesive samples will require the use of a solid wall permeameter, such as a compaction or standard permeameter. These methods are found in SW-846, Method 9100, Sections 2.5, 2.6, and 2.7, and include both constant-head and falling-head methods. The selection of constant- or falling-head methods is not critical as both methods provide similar results. These methods are also applicable to samples containing chemicals incompatible with the latex membrane.

If a sample requires remolding, a remolding density should be supplied. A moisture/density relationship curve can be generated to aid in the determination of remolding density. The permeability of remolded samples may be determined by any of the aforementioned methods. If the sample is

cohesive, the constant-head method, using a triaxial cell with back pressure, is again the method of choice.

# GENERIC PH AND EH PROCEDURE

I. Single Component Sample

1. Calibrate electrode as specified by the vendor. Record calibration data.

2. Place a few grams of material in a container (e.g., a 5-ounce plastic container).

3. Add water to mixture and stir with a spatula until a wet slurry is produced. There should be free water present. Enough water must be added to allow insertion of electrode in liquid phase with minimal contact with the solid phase. This procedure will minimize damage to the electrode.

4. Insert pH or Eh probe in liquid phase.

5. Take reading when measurement stabilizes.

II. Multicomponent Sample

The procedure is the same with the single component sample except that the sample is mixed before it is added to the container.

## PROPOSED RADON EMISSIONS FROM STABILIZED SOLIDS

1.0 Purpose and Application

A radon emission measurement technique is proposed for determining radon emissions from treated Operable Unit 4 materials. The test will determine the activity of radon emitted from the material's final form by measuring the radon activity in the air flowing through a chamber containing the waste form.

2.0 Definitions

2.1 See Figure C-1

3.0 Procedure

3.1 Summary

3.1.1 A cylinder of solidified material, having a known volume and surface area, is placed in a sealed container having one inlet and one outlet. Air is pumped through the chamber until equilibrium is reached. The radon in the exhaust stream is then measured. The radon emitted from the solidified material during a known time will be equal to the radon removed in the chamber's exhaust stream.

3.2 Interference

No known inteferences.

3.3 Sample Handling, Preservation, and Holding Time

3.3.1 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.

3.3.2 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 if a known or potential compromise to the health or safety of any IT Analytical Services (ITAS) Associate, and must be reported immediately to a laboratory supervisor.

3.3.3 There are no holding times applicable to this procedure.

3.3.4 There are no preservation requirements applicable to this procedure.

3.4 Required equipment

3.4.1 Air-tight test chamber of known volume.

3.4.2 One (1) small fan.

3.4.3 One (1) diaphragm pump (Brailsford TD-3LL or equivalent).

3.4.4 One (1) rotameter.

3.4.5 Two (2) activated carbon radon canisters.

3.4.6 One (1) desiccant canister.

3.4.7 One (1) metering valve (Swagelok B-SS4 or equivalent).

3.4.8 Tubing, fitting, and connectors.

3.4.9 One (1) continuous flow radon detector (Pylon AB-5 or equivalent).

3.5 Operation

3.5.1 Assemble test equipment as shown in Figure C-1.

3.5.2 Place treated solid in test chamber with fan.

3.5.3 Start fan.

3.5.4 Open value "A," and close valve "B."

3.5.5 Start pump.

3.5.6 Start radon detector in continuous counting mode.

3.5.7 Monitor detector until counts stabilize.

3.5.8 Switch detector to integrated count and count for 10 minutes. Record count.

3.5.9 Repeat step 3.5.7 two (2) times and record counts each time, for a total of three recorded measurements.

3.5.10 Open valve "B" and close valve "A."

3.5.11 Repeat steps 3.5.6 through 3.5.8.

3.5.12 Remove solid and store in air-tight container.

3.5.13 Switch radon detector to continuous mode.

3.5.14 Continue operating system until count rate returns to background levels.

3.5.6 Quality Control

3.5.6.1 None.

4.0 Nonconformance and Corrective Action

4.1 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.

5.0 Records Management

5.1 All data will be recorded in standard laboratory notebooks.

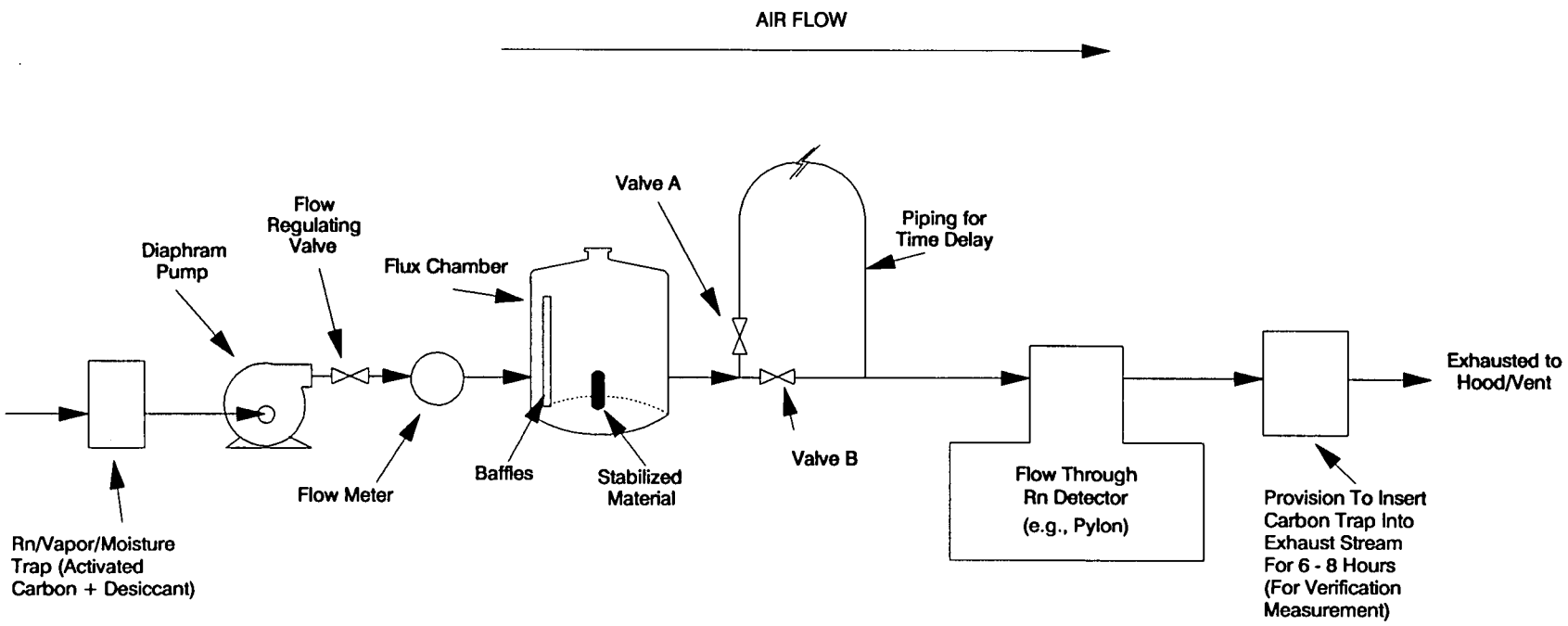


**FIGURE C-1. Proposed Rn-222 Collection/Measurement System**

Calculations:

The radon emitted from the solidified form will be calculated using the following equation:

$$A\ (pCi) = C\ (pCi/L) * Q\ (L/min) * T\ (min)/M\ (g) \qquad (1)$$

where

| | |
|---|---|
| A = | Radon activity emitted per gram of sample over time, t (pCi/g) |
| C = | Measured concentration of radon in exhaust air at equilibrium (pCi/L) |
| M = | Initial mass of sample in solidified material (g) |
| Q = | Flow rate (L/min) |
| T = | Time of count (10 min) |

Example calculation:
Assuming the measured concentration of radon from a 200 gram sample (M = 200) is 100 pCi/L (C = 100) during a 10-minute count (T = 10) at a flow rate of 1 L/min (Q = 1), A becomes:

$$A = 100\ pCi/L * 1\ L/min * 10\ min/200\ g$$

and

$$A = 5\ pCi/g$$

## SHEAR STRENGTH

The following is a procedure to determine shear strength.

MODEL CL-600A 2247

TORVANE SHEAR DEVICE

# Technical Data

SOILTEST®

Soiltest, Inc. • 86 Albrecht Drive • P.O. Box 8004
Lake Bluff, Illinois 60044-8004 U.S.A.
Telephone (708) 295-9400
Telex: 687-1537 SOILT UW • FAX (708) 295-9414

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## 1. GENERAL

The CL-600A Torvane is a scientifically designed soil testing instrument for the rapid determination of shear strength of cohesive soils, either in the field or in the laboratory.

The Torvane permits the rapid determination of a large number of strength values with different orientation of failure planes. It is simple to use and sample trimming is eliminated. All that is required is a reasonably flat two-inch minimum diameter surface.

The Torvane, ideally suited to field usage, is an invaluable addition to the inspector's kit or to the consulting engineer. Here are some suggested applications for evaluation of shear strength.

1. Ends of Shelby tube samples.
2. Standard penetration samples.
3. Split spoon samples.
4. Chunk samples from test pits and backhoe excavations.
5. Sides of test pits.

The instrument has a stress range of zero to 2.5 kg./sq. cm (tons/sq. ft.). This is also the approximate range of torque that can be easily applied by the fingers. It should be used only for fully saturated cohesive soils whose undrained strength is independent of normal pressure. The stress range permits it to be used for clays varying in consistency from very soft to stiff. The dial head is equipped with a mechanism to hold the maximum reading after release. The instrument is supplied with three vanes. The standard vane (1 inch diameter) is for a range of 0 to 1.0 kg./sq. cm. The sensitive vane (1 7/8 inch diameter) is for a range of 0 to 0.2 kg./sq. cm. When this vane is used, multiply the scale reading by 0.2 to get the shear strength of the material. The high capacity vane (3/4 inch diameter) is for the range of 0 to 2.5 kg./sq. cm. When this vane is used, multiply the reading by 2.5.

2.4 Press the Torvane carefully into the soil with the stem at right angles to the surface, to the depth of the blades.

2.5 Maintaining a constant vertical load by finger pressure, slowly turn the knob at a constant rate to provide a torque on the vane. Note: A rate of rotation such that failure develops in 5 to 10 seconds is recommended.

2.6 After sample fails, read Torvane shear strength on the circular scale just against the index.

2.7 Multiply the reading by the proper scale factor to get the shear strength. (For the high capacity vane, the smallest, the scale factor is 2.5; for the sensitive vane, the largest, the scale factor is 0.2; for the standard vane, medimum size, the scale factor is 1.)

2.8 Before making another test, re-zero the scale by rotating it with finger tip in the counter clockwise direction until it stops at the index.

2.9 Take readings at different spots (if possible) on the surface and calculate the average value.

## 3. LAB USES

3.1 Before conducting unconfined compression tests or triaxial tests on undisturbed samples, cut the sample into segments 1/2 inch longer than the desired length, and perform Torvane test on each end. Then trim the material disturbed by the test. It is easier to do the test while the specimen is in the sampling tube, after trimming at one end.

3.2 Use the Torvane test as a control test to determine the shear strength prior to other testing.

3.3 In consolidation testing, after the specimen has been consolidated under a desired normal stress, remove the upper porous stone and determine the consolidated shear strength of the specimen using the Torvane.

# METAL EXTRACTIONS

## 1.0 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 2 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 2 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 or not to carry them into the next phase. 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 1-pint container for disposal. Liquids will be syringe-filtered (0.45 micron) into 8-mL scintillation vials. The filtered samples will be diluted (ca 1:1000 to 1:10000) 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).

## 2.0 Ethylenediaminetetraacetic Acid Extractions

Literature results using ethylenediaminetetraacetic acid (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.

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 2 hours. Room temperature will be the actual temperature inside the hood, and this value will be recorded in a standard laboratory notebook. The extractions will be carried out in a HACH Micro COD Digester (Appendix E). The sample will be extracted for 2 hours in the HACH Digester within the hood. After extraction, 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 or not to carry them into the next phase. This will be based on the lead and uranium content of the extract. If not processed further, solids will be transferred to a 1-pint container for disposal.

Samples will be analyzed for lead as before (EDTA samples may require pretreatment nitric acid digestion) and for uranium using a modified Feigl test (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.

## 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 initial precipitation reagents to be investigated are sodium or potassium solutions of hydroxide, sulfide, sulfate, carbonate, and phosphate. Also calcium hydroxide, magnesium hydroxide, alum, ferric sulfate, and aqueous sodium silicate will be investigated. The mixture may be centrifuged to settle the solids so that the liquid can be decanted.

## VITRIFICATION OF LEACHATE

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.

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# GENERIC URANIUM BY ION CHROMATOGRAPHY WITH POST-COLUMN REACTION AND PHOSPHORESCENCE OR FLUORESCENCE DETECTION

This method uses ion chromatography in the cation-exchange mode to separate the uranium as $UO_2^{+2}$ (uranyl ion) from interferences. As the uranyl ion leaves the analytical column it is mixed with 39 percent $H_3PO_4$ to give a final concentration of approximately 19 percent $H_3PO_4$. The addition of $H_3PO_4$ enhances the fluorescence of the uranyl ion. Finally, the post-column reaction mixtures pass through a flow-through cell mounted in a fluorescence detector. Response has been found to be linear over the range studies (10 to 500 parts per billion [ppb]). The equipment and conditions for this method are listed below:

- High performance liquid chromatography (HPLC) pump - LDC/Milton Roy Constametric III
- Post-column reagent pump - LDC/Milton Roy Constametric III
- Injection valve - Altex 210
- Sample loop size - 147 uL
- Analytical columns - Dionex HPIC-CG2 Cation Guard
- Analytical columns - Dionex HPIC-CG2 Cation Analytical
- Post column reactor (PCR) - 1/16-inch SS low dead volume "TEE" and 12-inch coil, heated 60°C with a water bath
- Detector - Perkin Elmer 204 - S Fluorescence Detector
- Detector excitation wavelength - 275 nm
- Detector emission wavelength - 515 nm
- Eluant - 0.1 M $H_3PO_4$
- Eluant Flow - 1.5 mL/min
- PCR reagent - 39 percent weight $H_3PO_4$ (1 volume 85 percent $H_3PO_4$ to two volumes $H_2O$)
- PCR reagent flow rate - 1.1 mL/min

The concentrations of $H_3PO_4$ and brands of equipment are for examples only. They may be modified during the study.

# APPENDIX D

# SPOT TESTS

## APPENDIX D
## TABLE OF CONTENTS

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Analytical Procedure for Lead Spot Test Analysis - Ervin Jungreis
Uranium Analytical Procedure Spot Tests in Inorganic Analysis - F. Feigl

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QD98.S6J86

# Spot Test Analysis

## CLINICAL, ENVIRONMENTAL, FORENSIC, AND GEOCHEMICAL APPLICATIONS

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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 $\mu$g) 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 $\mu$g).

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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# SPOT TESTS IN INORGANIC ANALYSIS

*by*

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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 $\gamma$ 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 $\gamma$ 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 $\gamma$ 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.

**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.

*Limit of Identification:* 0.001 $\gamma$ 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.

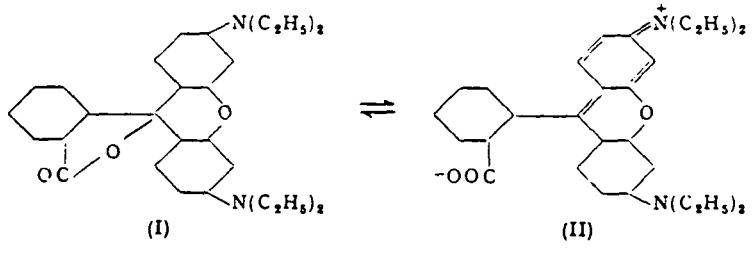
*Limit of Identification:* 0.06 $\gamma$ U
*Limit of Dilution:* 1 : 1,000,000

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):

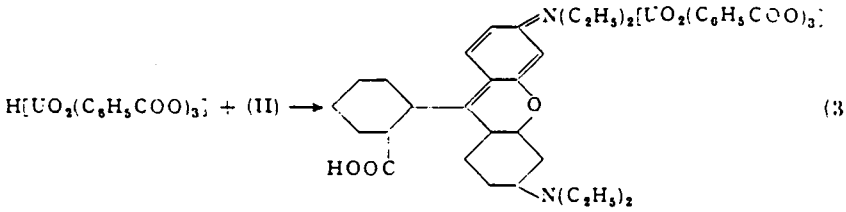



(1)

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 $\gamma$ 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 $\gamma$ uranium can be detected in the presence of 2500 $\gamma$ 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 $\gamma$ 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 $\gamma$ 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 $\gamma$ 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 $\gamma$ $UO_2^{+2}$).[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 $\gamma$ 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 $\gamma$ 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 Ph. 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:

# APPENDIX E

# HACH MICRO COD DESIGN

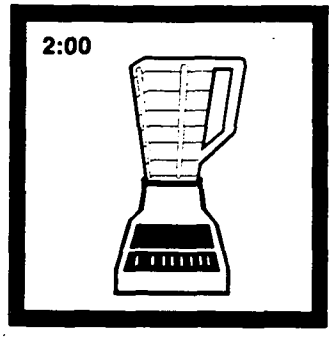
# ANALYTICAL PROCEDURES

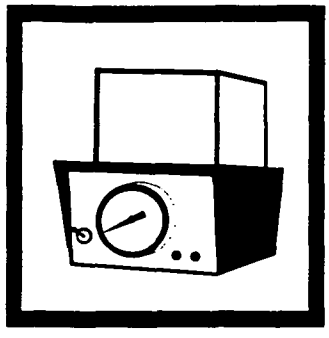
## For DR/2000, DR/3000 and DR/3 Instruments

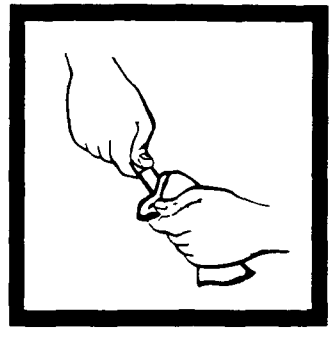
# OXYGEN DEMAND, CHEMICAL

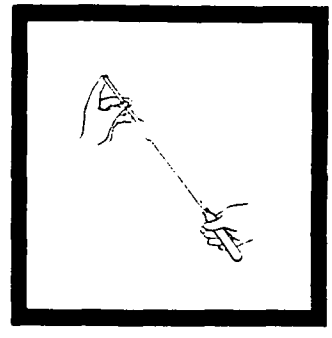
**Reactor Digestion Method*, EPA Approved†**
**0 to 150, 0 to 1,500, 0 to 15,000 mg/L COD**
**For water, wastewater, seawater**

## 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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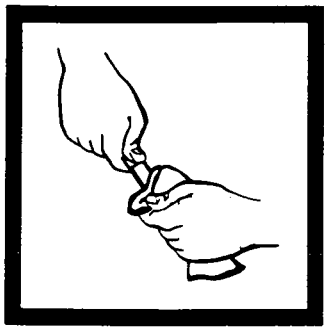
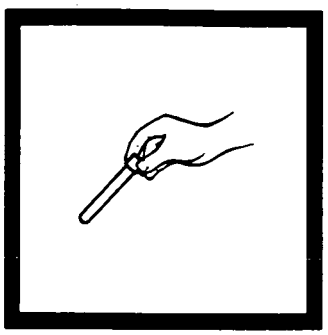
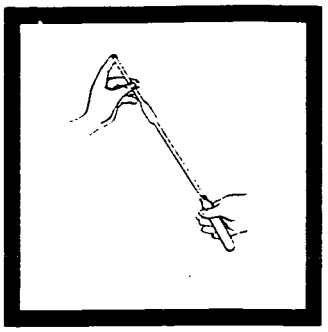


**5.** Replace the vial cap tightly. Rinse the COD vial with deionized water and wipe the vial clean with a paper towel.



**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.

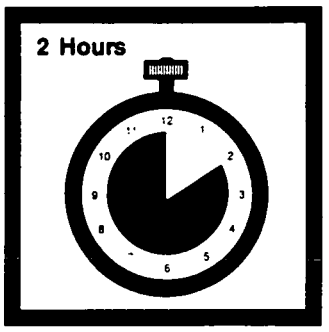
*Note: The vial will become very hot during mixing.*



**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.

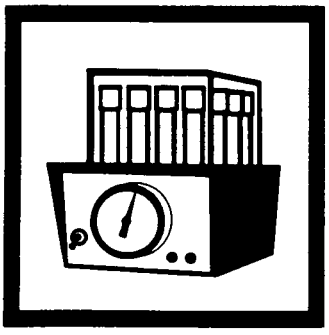
*Note: Be sure the pipet is well rinsed, or use a clean pipet.*

*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.*

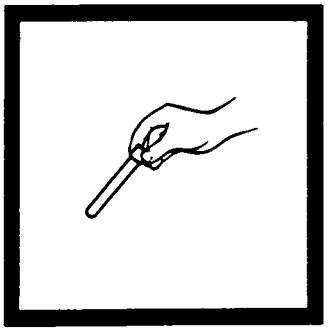



**8.** Heat the vials for 2 hours.

*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.*

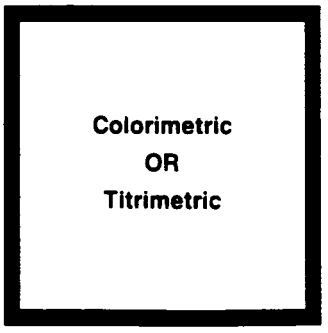


**9.** Turn the reactor off. Wait about 20 minutes for the vials to cool to 120°C or less.



**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.*




**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

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# APPENDIX F

## JUSTIFICATION FOR USING A MINIMUM UCS VALUE OF 500 psi AND A PORTLAND CEMENT/FLY ASH MIXTURE

## F.1.0 JUSTIFICATION FOR USING A MINIMUM UCS VALUE OF 500 psi

Portland cement mortars, which comprise mixtures of cement, lime, silica, sand, and water, are readily capable of achieving compressive strengths of 5000 to 6000 pounds per square inch (psi); that is approximately two orders of magnitude greater than the minimum compressive strength required to resist deformation under load in current low-level waste burial trenches. Therefore, to provide greater assurance that there will be sufficient cementitious material present in the waste form to not only withstand the burial loads, but also to maintain general "dimensions and form" (i.e., to not disintegrate) over time, it is recommended that cement-stabilized waste forms possess compressive strengths that are representative of the values that are reasonably achievable with current cement solidification processes. Taking into consideration the fact that low-level radioactive waste material constituents are not in most cases capable of providing the physical and chemical functions of silica sand in a cement mortar, a mean compressive strength equal to or greater than 500 psi is recommended for waste form specimens cured for a minimum of 28 days. This value of compressive strength is recommended as a practical strength value that is representative of the quality of cementitious material that should be used in the waste form to provide assurance that it will maintain integrity and thus possess the long-term structural capability required by Part 61.

# F.2.0 JUSTIFICATION FOR USING A PORTLAND CEMENT/FLY ASH MIXTURE

## F.2.1 INTRODUCTION

This appendix provides additional justification for choosing stabilization/solidification using a portland cement/fly ash mixture as the treatment process option to treat the pits. The wastes would be solidified using the fly ash from the Active Fly Ash Pile, although solidification using fly ash from the Inactive Fly Ash Disposal Area will be examined on a limited basis.

The additional justification will be provided by discussing results from a literature search of solidification technology. The literature search provides information that indicates solidification of the wastes will provide a waste form that could pass Toxicity Characteristics Leaching Procedure (TCLP) tests and allows mixed wastes to be disposed of as nonhazardous or low-level wastes. Also discussed in this appendix will be the reasoning for using the cement to fly ash ratios and water to cement ratios indicated in this study.

## F.2.2 TYPES OF SOLIDIFICATION

Various solidification processes exist that could be used to solidify waste. Systems that could be used for solidification are the portland cement-based process, the portland cement/soluble silicate process, the lime/fly ash-based systems, the kiln dust and fly ash-based process, and the portland cement/fly ash process.

### F.2.2.1 Portland Cement-Based Process

With the portland cement process, water from the waste reacts chemically with the cement to form a hardened concrete-like material. Depending upon the amount of cement added, the final product may be a monolithic solid or may have a crumbly soil-like consistency (EPA 1985). The optimum combination of waste, water, and portland cement will vary with waste type and composition. The minimum water to cement ratio is about 0.40, by weight, for portland cement, but this also depends upon the moisture content of the waste. The addition of too much water may result in free-standing water on the surface of the solidified product, as well as a reduction in its strength and an increase in the permeability of the final product (Conner 1990).

The bulk density of cement-based waste forms varies between 1.25 and 1.75 g/cm$^3$, with water contents ranging from about 15 to 60 percent. The unconfined compressive strength (UCS) varies also, depending upon the mix ratio.

Most products range from 15 to 1000 psi but can be strengthened by other additives. Permeability is influenced by solidification of the waste. The permeability of cement-based waste forms is similar to that of clay (Conner 1990).

The chemical properties of cement-based forms are described in terms of leachability. The interaction of organic and inorganic substances in cement affects the setting and hardening of the cement matrix. Salts of manganese, tin, zinc, copper, and lead tend to reduce the strength of the waste form. Cement solidification can immobilize metals; but if the waste form is subjected to even a mild acidic solution, leaching could take place (EPA 1985). Because of these limitations, portland cement is normally used as a setting agent in combination with other solidification processes.

The cost of the portland cement-based process is low and the equipment for the process is readily available.

F.2.2.2 Portland Cement/Soluble Silicate Processes

The Portland Cement Soluble Silicate (PCSS) process is based on the reactions between soluble silicates and portland cement to produce a solid matrix. This process depends on three different reactions, the first being a rapid reaction between the soluble silicate (such as sodium silicate) and metal ions to produce a low-solubility metal silicate. The second set of reactions occurs between the soluble silicate and portland cement. The third set of reactions occurs among the cement, waste, and water. The soluble silicate functions as a surfactant (keeping retarders such as oil or particulates in suspension), which helps in the setting and hardening of the waste.

By adding soluble silicate to the portland cement, low-solid waste can be solidified without the addition of massive amounts of bulking agents. This is a cost-effective approach, but the water content of the waste form is high, which increases the porosity of the solid. Higher water content also causes reduced strength and higher permeability. The UCS ranges between 15 and 100 psi, but stronger products can be prepared (with the addition of cement). The advantages of this process include relatively low cost and small volume increase; however, the UCS is lower than the 500 psi proposed by the Nuclear Regulatory Commission (NRC 1991).

F.2.2.3 Lime, Fly Ash-Based Process

Combining lime and fly ash with water forms a cementitious material. Initially a noncrystalline gel, which eventually becomes a calcium silicate hydrate, is formed. The reactions that occur are similar to cement-based systems. The reactions are slower however and do not produce the same products as the cement-based system in terms of physical and chemical properties. A problem with the lime/fly ash process is that fly ash is a by-product of coal-burning power plants and its composition depends

upon the type of coal burned and how the plant was operated. Unburned organics in the fly ash can reduce the cementing action by covering reactive surfaces. Also, the lime-based process is not as effective in reducing leachability as the cement-based systems, due in part to its high pH. Much of the lime/fly ash treatment used has been in nonhazardous waste applications.

F.2.2.4 Portland Cement/Fly Ash Process

Portland cement and fly ash have been used in applications for many years. When fly ash is used with cement in an application, the percentage of cement required is reduced significantly. Because fly ash itself is a waste, it is desirable to use it as a component in solidification systems.

Fly ash in portland cement acts as a bulking agent and as a pozzolan. The reaction between the two materials produces a product that may have higher strength than when portland cement is used alone. The fly ash also helps to bind additional water and decrease pH, as well as acting as an adsorbent for metal ions. The greatest disadvantage of this process is the volume increase associated with large additions of fly ash. The range of the fly ash to cement ratio (by weight) is two to four, with total weight increases of 50 to 150 percent. Where increase in volume is not important, the cement/fly ash process is the optimum choice (Conner 1990).

In a pure water-cement system, the permeability is essentially zero at a water to cement ratio of 0.32. The water to cement ratio can be increased when a bulking agent such as fly ash is added to the process.

Several vendors use the cement/fly ash process and many studies have been performed. One such program was performed on waste at the Idaho National Engineering Laboratory (INEL).

F.2.2.5 Kiln Dust and Fly Ash based Process

Kiln dust and fly ash have been used in several solidification projects. They function primarily as adsorbents or bulking agents. The kiln dusts are highly alkaline, which gives them the ability to remove free water by hydration of calcium oxide to calcium hydroxide. This process can produce hard, strong solids that continue to harden with time. The actual setting reactions of the kiln dust and fly ash are pozzolanic and resemble those of portland cement. A limitation of the use of these materials is that they contain significant amounts of metals, which leach at levels above regulatory standards. These materials are available, and their costs are low compared to portland cement. The cost of these materials however has been increasing; if the trend continues, they could be replaced by more expensive but more efficient reagents (Conner 1990).

F.2.2.6 Polyethylene Process

Brookhaven National Laboratory (BNL) has also developed a process for the solidification of salt wastes, incinerator ash, and ion-exchange resins in polyethylene. Although the most common solidification agents used in solidification of low-level radioactive waste (LLW) are portland cement, bitumen, and thermo settling polymers, operational difficulties such as incompatibility with waste constituents, low loading efficiency, premature setting, or formulation of solidified products with poor performance properties have been observed with these materials (Franz 1987).

The choice of polyethylene as an improved solidification agent was based on such considerations as compatibility with waste, solidification efficiency, material properties, availability of materials, economic feasibility, and ease of processibility. Because the solidification process is not dependent upon complex chemical reactions as it is in the case of hydraulic cements and thermosetting polymers, the processing is simplified and solidification of the waste is ensured.

Polyethylene is a thermoplastic organic polymer of crystalline-amorphous structure formed through the polymerization of ethylene gas. At elevated temperatures thermoplastic polymers change from a hard material to a rubbery flowable liquid. On cooling, the polymers revert to their original form.

Polyethylene is resistent to most acids, bases, and organics normally encountered in waste streams. The superior mechanical properties of polyethylene (i.e., compressive strength) allow higher waste loading than normally can be incorporated into other materials such as cement or bitumen, without compromising the integrity of the waste form.

Some of the more important factors that affect the properties of polyethylene are density, molecular weight, molecular weight distribution, melt index, and cross linking. Low-density polyethylene (0.910 to 0.925 g/cm$^3$. The process parameters investigated included temperature, pressure, mixing kinetics, and volumetric efficiency. In general, polyethylenes with a density of 0.924 g/cm$^3$ and melt indices of 35.0 to 55.0 g/10 minutes were able to incorporate greater quantities of waste. In the case of the incinerator ash, the maximum amount of waste was 40 weight percent (dry) that represents the maximum amount of waste that can be incorporated to form a monolithic solid. For the determination of the release of radionuclides through leach tests, radioactive tracers were added to the incinerator ash. The radioisotopes used were cobalt-60, strontium-85, and cesium-137 because these are the radionuclides of greatest concern in low-level wastes. Results of this study indicated a clear dependence of leachability upon increased waste loadings for all three isotopes for the incinerator ash samples. With increased waste loading, the average leaching of the radioisotopes decreased. Results of the polyethylene studies indicate that polyethylene is a viable solidification agent for various types of low-level waste (Franz 1987).

F.2.2.7 Magnesium-based Cement

The magnesium-based cement technology discussed here is one developed by Envirotite Incorporated (ETI). ETI literature states that approximately 65 percent of the stabilization products marketed use portland cement or a mixture of portland cement and catalysts. ETI identified only three corporations that used magnesium-based cements for stabilization. Magnesium-based cements have been formulated and perfected to possess physical properties similar to ceramics. The ETI literature also states that due to the improved qualities of magnesium cement, it can meet more disposal needs than other stabilization products and offer some unique properties significantly different than those provided through the use of portland cement (ETI 1991).

ETI provides the following table to show the comparison of portland cement versus magnesium cement:

| Standards for Comparison | Portland Cement | Magnesium Cement |
|---|---|---|
| Compressive strength | hard | very hard |
| Finished surface | smooth | glass-like |
| Acid resistance | mild reaction | no reaction |
| Free water | visible | not visible |
| Miscibility in oil | no | yes |

The magnesium-based cement offered by ETI are CERAMAG-S1 and CERAMAG-L1.

CERAMAG-S1

CERAMAG-S1 is a magnesium-based concrete specifically formulated to stabilize hazardous wastes present in solid matrices such as clay, dirt, sand, gravel, ash, and sludge. CERAMAG-S1 reduces TCLP values less than regulatory limits for a wide variety of inorganic and organic wastes. Stabilized products meet applicable land disposal restriction (LDR) treatment standards.

CERAMAG-L1

CERAMAG-L1 is also a magnesium-based concrete specifically formulated to stabilize hazardous waste present in liquid matrices including acids, caustic, solutions of inorganic wastes, solutions of organic wastes, and petroleum products. CERAMAG-L1 reduces TCLP values less than regulatory

limits for a wide variety of inorganic and organic wastes. Stabilized products meet applicable LDR treatment standards.

The performance data by ETI for the magnesium-based concrete indicate that there would be no free-standing water in the stabilized product that the UCS would be far greater than the 500 psi UCS quoted by the Nuclear Regulatory Commission technical position paper (NRC 1991). Specific data from a particular site was not provided but the chemical characteristics of the stabilized waste provided by ETI indicate that TCLP values for organic and inorganics are below regulatory limits.

F.2.2.8 Modified Sulfur Cement Encapsulation

Modified sulfur cement is a thermoplastic material that can be easily melted, combined with waste components in a homogeneous mixture, and cooled to form a solid monolithic waste form. Compared with portland cements, sulfur cement has several advantages. For example, no chemical reactions are required for solidification, eliminating the possibility that elements in the waste can interfere with setting and thereby limit the range of waste materials that can be encapsulated successfully. Sulfur concrete compressive and tensile strengths twice those of comparable portland concretes have been achieved, and full strength is attained in several hours rather than weeks. Sulfur concretes are resistant to attack by most acids and salts, e.g., sulfates that can severely degrade hydraulic cement have little or no effect on the integrity of sulfur cement (Kalb 1991).

As a result of defense and research activities the U.S. Department of Defense (DOD) generates a broad range of waste types, including hazardous/radioactive waste, one of which is incinerator ash. In an effort to develop new methods of stabilizing/solidifying mixed wastes generated at DOE facilities, work is being performed at BNL to encapsulate incinerator fly ash waste.

The incinerator fly ash in this study are generated in the Waste Experimental Reduction Facility (WERF) at INEL. This fly ash contains a total of 40 pCi/g of activity consisting of fission products (Cs-137) and activation products (Co-57 and Sb-125). The ash was analyzed for 12 elements and the results are shown below:

Elemental Composition of INEL
Incinerator Fly Ash

| Element | Weight Percentage |
|---|---|
| Zinc | 36.0 |
| Lead | 7.5 |
| Sodium | 5.5 |
| Potassium | 2.8 |
| Calcium | 0.8 |
| Copper | 0.7 |
| Iron | 0.5 |
| Cadmium | 0.2 |
| Chromium | BDL* |
| Barium | BDL |
| Silver | BDL |
| Nickel | BDL |

*Below detection limits (<0.05 wt. percent)

The incinerator fly ash contains zinc, lead, sodium compounds, and highly soluble metal chloride salts that creates an acidic environment in the presence of moisture. The presence of these element and compounds have been shown to impede or interfere with cement solidification by reducing the ultimate mechanical strength of the waste form, by causing cracking and could greatly increase the mobility of contaminants (Kalb 1991).

As stated above, however, modified sulfur cement is resistant to attack by acids and salts.

The modified sulfur cement is a thermoplastic material that means that thermal input is required for processing. Also, when the sulfur cement is mixed with dry waste materials, a thick paste is formed. Therefore, a mixing system would be required to mix the waste and binder to form a homogeneous mixture. Several mixing systems were investigated and based on the processing requirements of modified sulfur cement/waste combinations, a double planetary orbital mixer was chosen as the most appropriate system.

Formulation and process development work was concluded to determine the limits and ease of processibility, while at the same time producing waste forms that conform to regulatory criteria.

Maximum waste loadings were determined by first processing at waste loading above the limits of workability (i.e., extremely dry mixtures that yielded friable products with little structural integrity) and then adding additional increments of modified sulfur cement until acceptable workability and product integrity were achieved. Reported waste loadings represent weight percent of dry ash, after all residual moisture has been removed. Using this procedure, a maximum waste loading of 55 weight percent INEL incinerator fly ash was determined. Due to its low pH and high chloride content, the maximum waste loading using portland cement achieved at INEL was 16 weight percent (Kalb 1991).

Among the tests conducted on the waste forms were compressive strength and leachability to provide information on structural integrity and waste form behavior in a disposal environment. Modified sulfur cement is a brittle material and tends to shatter under axial compressive load.

Compressive strength testing of waste form specimens containing 40 and 55 weight percent INEL fly ash encapsulated in modified sulfur cement were compared with modified sulfur cement specimens containing no waste. The results indicated that compressive strength were not highly dependent upon waste loading (4053 psi to 40 weight percent ash and 4118 psi at 55 weight percent ash) "but both waste loadings displayed more than two times greater strength than the binder material alone (1800 psi)."

The INEL incinerator ash and samples of encapsulated ash at various waste loadings were tested using both the Extraction Procedure-Toxicity (EP Tox) and TCLP.

The TCLP leachate data from the INEL incinerator ash show that cadmium and lead were present in concentration well above the EPA allowable limits for each chemical. The TCLP leachate from waste encapsulated in plan modified sulfur indicated that cadmium and lead above the allowable limits. (Leachate concentrations for encapsulated waste samples tested by the EP Tox method were found to be considerably lower, which demonstrates the conservative nature of the TCLP test.)

Based on results of scoping experiments and other considerations, sodium sulfide was selected as an additive to further reduce mobility of toxic heavy metals in the incinerator ash and to comply with EPA TCLP hazardous waste concentration limits. Sodium sulfide reacts with the toxic metals salts to form metal sulfides of extremely low solubility. Sodium sulfide has been used extensively in the related field of waste watertreatment, and has been identified as an effective treatment technology by EPA. A ratio of sodium sulfide/fly ash of 0.175 was used based on the results of an experiment to determine the effectiveness of this additive on cadmium mobility under EPA leaching conditions. Optimization of INEL incinerator fly ash waste loading with added sodium sulfide (while maintaining

additive/ash ratio constant) yielded a maximum waste loading of 43 weight percent fly ash, 49.5 weight percent modified sulfur cement, and 7.5 weight percent sodium sulfide (Kalb 1991).

By using the optimal INEL incinerator ash with sodium chloride in modified sulfur cement, 2.7 times more incinerator ash can be used per drum (55 gallon) than when using Portland cement as the binder. INEL incinerator ash is difficult to stabilize using ordinary portland cement mixtures and the waste loading is limited to 16 weight percent. Modified sulfur cement is not susceptible to interference from the high concentrations of zinc, lead, sodium, and chloride as portland cement. The waste loading is increased significantly using modified sulfur cement. A process demonstration using production-scale equipment to encapsulate the incinerator fly ash in modified cement is being planned in conjunction with INEL.

### F.2.3 LITERATURE SEARCH

A literature search was conducted to determine whether the performance of stabilization/solidification have been sufficiently documented on similar wastes and the number of times the technology has been used.

The literature search for Operable Unit 4 involved calling various laboratories that have been involved in stabilization/solidification and reviewing various other available literature. Those laboratories contacted were the INEL, Oak Ridge National Laboratory (ORNL), and BNL.

#### F.2.3.1 INEL Literature

INEL representatives were contacted and they indicated that published information on stabilization/solidification is not available because none has been performed. INEL however provided the name of a private company, Halliburton-NUS Environmental Company, with whom they had worked with previously. The contact person at Halliburton indicated he had performed work for the Savannah River Plant using stabilization/solidification; however, he did not know how to get the report. He further stated that a lot of this type information is difficult to obtain because it is proprietary. During the course of the conversation, he also stated that it is his experience that a treatability study would be needed to indicate the type of inhibitors present in the waste. Although a complete analysis of the raw waste may be performed, sometimes those compounds that inhibit the stabilization/solidification process are not found until the treatability testing is done.

#### F.2.3.2 ORNL Literature

ORNL was also contacted. ORNL provided a list of reports, which provided remedial techniques for various waste sites at ORNL. A review of the list and of some reports indicate that they do not provide information with regards to ex situ stabilization/solidification.

F.2.3.3 BNL Literature

BNL also provided a list of references that used stabilization/solidification methods to treat various wastes. The two methods identified by BNL were the modified sulfur cement and polyethylene solidification processes and are discussed in Sections 2.6 and 2.8.

The results of the analysis performed on the solidified products produced by the two methods, indicate that both methods are viable for solidification agents for low-level waste. The portland cement/fly ash process however is the chosen method for solidifying Operable Unit 4 wastes. Therefore, the results offered by the sulfur cement encapsulation and solidification using polyethylene is not relevant for comparison to portland cement/fly ash method.

F.2.3.4 Soliditech, Incorporated Literature

The literature search also included a paper presented at the Forum of Innovative Hazardous Treatment Technologies by Soliditech, Incorporated. The paper described the Soliditech process, which is a mixing process based on the use of pozzolans or cement and various additives that enhance the ability of the mixture to incorporate organic compounds into the matrix and reduce the potential for these compounds to leach from the solidified product.

The Soliditech process solidifies wastes by use of URRICHEM (a proprietary chemical reagent, U.S. patent pending), additives, pozzolanic solids, and water. The proportions of reagent, additives, and pozzolan are optimized for each particular waste requiring treatment. The solidified material displays properties of excellent unconfined compressive strength, high stability, and a rigid texture similar to that of concrete (Brassow 1989).

Three different waste streams were treated as part of the demonstration, which included a soil contaminated with oily sludge, a filter media with a high percentage of hydrocarbons and an oily tank bottom sludge. The latter stream was co-treated with the filter media during the demonstration.

Untreated waste samples were collected for each test parameter from each of the three waste streams. These samples were analyzed for total chemical constituents, physical characteristics and the amount of solubles removed by leaching/extractions. The results allow a direct comparison of physical and chemical properties between the treated and untreated waste and a determination of effectiveness of the treatment process (Brassow 1989). The information presented below is from the results of Brassow 1989.

Untreated waste -- Untreated waste from the site consisted of contaminated soil, filter cake, and filter cake/oily sludge. These wastes contained 2.8 to 17 percent oil and grease, with relatively low levels of other organic compounds. PCB (Aroclors 1242 and 1260) concentra-

tions ranged from 28 to 43 mg/g; arsenic concentrations from 14 to 94 mg/kg; lead concentrations ranged from 650 to 2470 mg/kg; and zinc concentrations from 26 to 151 mg/kg.

Treated Waste -- The Soliditech stabilization process produced solidified waste with high structural stability and low permeability. UCS values ranged from 392 to 856 psi. Permeability values ranged from $8.9 \times 10^{-9}$ to $4.5 \times 10^{-7}$ cm/s. Because of the cementitious additives in the Soliditech process, pH values of the solidified wastes ranged from 11.7 to 12.0. Arsenic concentrations ranged from 28 to 92 mg/kg; lead concentrations from 480 to 850 mg/kg; zinc concentrations from 23 to 95 mg/kg; and PCB (Aroclors 1242 and 1260) concentrations from approximately 15 to 41 mg/kg. Low concentrations of phenol and p-cresol were found in solidified filter cake and filter cake/oily waste samples. These compounds were not detected in the untreated wastes.

Extract of Untreated Waste -- Arsenic, lead, and zinc were found in EP, TCLP, and BET extracts of the untreated wastes. No PCBs were detected in the TCLP extracts of the untreated wastes. Total concentrations of up to 1.3 mg/L of volatile organic compounds and up to 0.38 mg/L of semivolatile organic compounds were detected in TCLP extract of the untreated waste. Oil and grease concentrations of 1.4 to 1.9 mg/L were detected in the TCLP extract of the untreated waste. Untreated wastes could not be tested by ANS 16.1.

Extract of Treated Waste -- Significantly reduced amounts of metals were detected in the TCLP, EP, BET, and ANS 16.1 extracts of the treated waste. No PCBs or volatile organic compounds were detected in the TCLP extract of the treated waste. Phenol, p-cresol, o-cresol, and 2,4-dimethylphenol were detected in the post-treatment TCLP waste extracts. Oil and grease concentrations of 2.4 to 12.0 mg/L were detected in the TCLP extracts.

The range of UCS and low permeabilities verify the solidification objective.

The change in volume ranged from 0 to 60 percent but the median appeared to be less than 30 percent. This is an important parameter when estimating disposal volume of treated waste and this level is probably an acceptable increase now (Brassow 1989).

## F.3.0 SOLIDIFICATION PROCESS CHOSEN FOR OPERABLE UNIT 4

Section 2.0 contains descriptions of the various types of stabilization/solidification methods and their associated advantages or disadvantages. As a result of reviewing these methods, the portland cement/fly ash process is the technology that has been chosen to solidify the waste in Operable Unit 4.

The modified sulfur cement encapsulation method, which appears to be a viable technology but data results from other studies using this method are not documented, to verify its success rate. Also, the use of the modified sulfur cement requires the use of an additive, such as sodium sulfide, to reduce the mobility of toxic metals. The results from the laboratory study for modified sulfur indicates that is a better binder than portland cement in that the modified sulfur cement would have higher waste

loadings than the portland cement. Studies using portland cement/fly ash have however been performed is pozzolonic and acts as an adsorbent for metal ions. Therefore, by using portland cement/fly ash, an existing waste can be used as resource to aid in treating other wastes at the site.

## F.4.0 CONCLUSIONS AND RECOMMENDATIONS

The purpose of this additional literature search was to provide additional justification for choosing stabilization/solidification using a portland cement/fly ash mixture as the treatment process option.

One of the main criteria to determine whether a treatability study is required is to determine from a literature search whether sufficient documentation of results exist for the treatment method being proposed.

Based on the results of this literature search, it can be concluded that sufficient documentation of results of stabilization/solidification of wastes similar to Operable Unit 4 is not available. Therefore, the treatability study for Operable Unit should be conducted.

# APPENDIX F
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U.S. Nuclear Regulatory Commission, 1991, "Technical Position on Waste Form (Revision 1)," prepared for the NRC Office of Nuclear Safety and Safeguards by the Low-Level Waste Management Branch, Division of Low-Level Waste Management and Decommissioning, NRC, Washington, DC.

# APPENDIX G

## APPLICABLE MSDSs

chemists helping chemists in research & industry

**aldrich chemical co.**

P.O. Box 355, Milwaukee, Wisconsin 53201 USA • (414) 273-3850

2247

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

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., Inc.
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Telephone (07329) 87-0
Telex 714838 Aldri D
FAX (07329) 87-39

FORM 200 REV. 3-87

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.

M A T E R I A L  S A F E T Y  D A T A  S H E E T  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.

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

## SECTION I - PRODUCT IDENTIFICATION

RODUCT NAME: NITRIC ACID
OMMON SYNONYMS: HYDROGEN NITRATE; AZOTIC ACID
HEMICAL FAMILY: INORGANIC ACIDS
ORMULA: HNO3
ORMULA WT.: 63.01
AS NO.: 7697-37-2
IOSH/RTECS NO.: QU5775000
RODUCT USE: LABORATORY REAGENT
RODUCT CODES: 4801,9597,5113,9602,5371,9598,9605,9600,9616,6901,9606,9601

## PRECAUTIONARY LABELING

AKER SAF-T-DATA* SYSTEM

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

ABORATORY PROTECTIVE EQUIPMENT

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

U.S. PRECAUTIONARY LABELING

POISON DANGER

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

CONTINUED ON PAGE: 2

## 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.

CONTINUED ON PAGE: 3

## SECTION IV - FIRE AND EXPLOSION HAZARD DATA

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

UTOIGNITION TEMPERATURE: N/A

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

IRE EXTINGUISHING MEDIA
USE WATER SPRAY.

PECIAL 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.

NUSUAL 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.

OXIC GASES PRODUCED
OXIDES OF NITROGEN, HYDROGEN

XPLOSION DATA-SENSITIVITY TO MECHANICAL IMPACT
NONE IDENTIFIED.

XPLOSION DATA-SENSITIVITY TO STATIC DISCHARGE
NONE IDENTIFIED.

## SECTION V - HEALTH HAZARD DATA

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

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

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

CONTINUED ON PAGE: 4

## SECTION V - HEALTH HAZARD DATA (CONTINUED)

XICITY OF COMPONENTS

| | | |
|---|---|---|
| TRAPERITONEAL MOUSE LD50 FOR WATER | 190 | G/KG |
| TRAVENOUS MOUSE LD50 FOR WATER | 25 | G/KG |

RCINOGENICITY: NTP: NO IARC: NO Z LIST: NO OSHA REG: NO

RCINOGENICITY
NONE IDENTIFIED.

PRODUCTIVE EFFECTS
NONE IDENTIFIED.

FECTS 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

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

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

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

CONTINUED ON PAGE: 5

## SECTION V - HEALTH HAZARD DATA (CONTINUED)

ERGENCY 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

TE: YES CHRONIC: YES FLAMMABILITY: YES PRESSURE: NO REACTIVITY: NO

REMELY HAZARDOUS SUBSTANCE: YES CONTAINS NITRIC ACID (RQ = 1,000 LBS, TPQ = 1,000 LBS)
CLA HAZARDOUS SUBSTANCE: YES CONTAINS NITRIC ACID (RQ = 1000 LBS)
IC CHEMICALS: YES CONTAINS NITRIC ACID
GENERIC CLASS: C16
A INVENTORY: YES

## SECTION VI - REACTIVITY DATA

BILITY: STABLE HAZARDOUS POLYMERIZATION: WILL NOT OCCUR

DITIONS TO AVOID: HEAT, LIGHT, MOISTURE

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

OMPOSITION PRODUCTS: OXIDES OF NITROGEN, HYDROGEN

CONTINUED ON PAGE: 6

## 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.

CONTINUED ON PAGE: 7

## SECTION X - TRANSPORTATION DATA AND ADDITIONAL INFORMATION

OMESTIC (D.O.T.)

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

NTERNATIONAL (I.M.O.)

ROPER SHIPPING NAME: NITRIC ACID
AZARD 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

CONTINUED ON PAGE: 8

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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
D 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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