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OPERABLE UNIT 4 TREATABILITY STUDY WORK PLAN FOR THE VITRIFICATION OF RESIDUES FROM SILOS 1, 2, AND 3 MARCH 1992

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OPERABLE UNIT 4 TREATABILITY STUDY WORK PLAN FOR THE VITRIFICATION OF RESIDUES FROM SILOS 1, 2, AND 3

Fernald Environmental Management Project
Fernald, Ohio

PUBLIC ENVIRONMENTAL INFORMATION CENTER
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March 1992

Fernald Office
U.S. DEPARTMENT OF ENERGY

3006

FINAL

Fernald Environmental Management Project

OPERABLE UNIT 4

TREATABILITY STUDY WORK PLAN FOR THE VITRIFICATION OF RESIDUES FROM SILOS 1, 2, AND 3

March 1992

Fernald Office
Department of Energy

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LIST OF ACRONYMS

ARARs	Applicable or Relevant and Appropriate Requirements
ASI/IT	Advanced Sciences, Inc./International Technology Corporation
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
DOE	Department of Energy
DQO	Data Quality Objectives
EPA	Environmental Protection Agency
FEMP	Fernald Environmental Management Project
FFCA	Federal Facilities Compliance Agreement
FS	Feasibility Study
HSL	Hazardous Substance List
mL	milliliters
mrem	millirem
mTCLP	Modified Toxicity Characteristic Leaching Procedure
Pb	Lead
pCi/m^2-s	picoCuries/meter^2 per second
pCi/L	picoCuries/liter
PCi/hr	picoCuries/hour
pCi/g	picoCuries/gram
PCT	Product Consistency Test
PNL	Pacific Northwest Laboratory
Po	Polonium
ppm	parts per million
QA	Quality Assurance
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Program Plan
Ra	Radium
RAO	Remedial Action Objectives
RCRA	Resource Conservation and Recovery Act
RI/FS	Remedial Investigation/Feasibility Study
RME	Reasonable Maximum Exposure
ROD	Record of Decision
TBC	To Be Considered
TCLP	Toxicity Characteristic Leaching Procedure
Th	Thorium
U	Uranium
WEMCO	Westinghouse Environmental Management Company of Ohio

1.0 PROJECT DESCRIPTION

1.1 SITE DESCRIPTION

The Fernald Environmental Management Project (FEMP) is a contractor-managed federal facility once used 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 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. The FEMP has been segregated into five operable units. Operable Unit 4 consists of Silos 1 and 2 (K-65 Silos), Silo 3 (metal oxide silo), the unused Silo 4, and their ancillary structures and the surrounding berms. Operable Unit 4 is located at the western periphery of the site, south of the waste pit area. The Feasibility Study (FS) for Operable Unit 4 is considering remedial actions for the silo structures, for wastes stored in the silos and for contaminants in the surrounding berms.

1.2 BACKGROUND INFORMATION

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 approximately 7,200 cubic yards of residues from 1952 to 1958. Raffinates (residues resulting from uranium solvent extraction) were pumped into the silos as a slurry where the solids would settle. The free liquid was decanted through a series of valves and piping placed at various levels along the height of the silo wall. This procedure, pumping of slurry, followed by the settling and decanting, continued until the waste material was approximately four feet below the top of the vertical wall. Historic analyses of the K-65 silo residues indicate that approximately 11,200 kilograms of uranium (0.71 percent U-235) is present in Silos 1 and 2. Analytical results of residue samples, taken in July 1988, indicated uranium concentrations was 1400 parts per million (ppm) in Silo 1 and 1800 ppm in Silo 2. In addition, the estimated concentration of radium was between 0.13 to 0.21 ppm in the K-65 residues.

In 1989, Westinghouse Environmental Management Company of Ohio (WEMCO) collected residue samples from Silos 1 and 2. The analyses of the samples indicate that the concentration of radium-226 (Ra-226) in Silo 1 ranges from 89,280 picoCuries/gram (pCi/g) to 192,600 pCi/g; in Silo 2 from 657 to 145,300 pCi/g. Thorium-230 (Th-230) concentrations in Silo 1 range from 10,569 to 43,771 pCi/g and from 8,365 to 40,124 pCi/g in Silo 2. The concentration of lead-210 (Pb-210) in Silo 1 ranges from 48,490 to 181,000 pCi/g and from

77,940 to 399,200 in Silo 2. Total uranium concentrations in Silo 1 range from 1,189 to 2,753 ppm and from 137 to 3,717 ppm in Silo 2.

Radon and the elements resulting from its decay (referred to as daughter products, or progeny) are the nuclides of concern from a health and environmental perspective. Radon is known to be emanating from the silos via cracks and at joints. Radon and its daughter products are relatively mobile and capable of migrating through air and water. Due to the probable diffusion of radon into the berms, it is believed that the berms and subsoils contain elevated levels of lead-210 (Pb-210) and polonium-210 (Po-210). Also, 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 underway to confirm the nature and extent of any soil contamination.

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 a dry waste for removal to Silo 3. The material was blown in under pressure to fill Silo 3. Silo 4 was never used and, except for rainwater infiltration, remains empty today.

Silo 3 contains approximately 5,100 cubic yards of calcined residues consisting of silica, uranium (39,600 pounds), a very small amount of radium, thorium, and other metal oxides. Silo 3 is not a significant radon source because of the physical and chemical characteristics of its contents. Also, Silo 3 is not believed to be a source of contamination to the surrounding areas and underlying soils. Nevertheless, Silo 3 must be considered a potential hazard because its contents are radioactive and in its dry powdery state susceptible to airborne dispersal if exposed to wind.

Appendix C contains more detailed information on the radiological, organic, and inorganic constituents of the silo material. However, these results do not fully characterize the contents of Silos 1 and 2. The variability and inconsistency of results from previous sampling efforts and the lack of material from the lower areas of the silos precludes the use of these data for fully characterizing the silos' contents. Therefore, a resampling program was conducted (and completed in August 1991), but analytical results are not available for inclusion into this document. The results will be documented in the Operable Unit 4 Remedial Investigation (RI) Report.

1.3 REMEDIAL ACTION OBJECTIVES

Remedial action objectives (RAOs) are medium-specific cleanup goals for protecting human health and the environment. The RAOs 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

- U.S. EPA guidance for risk to public health from hazardous substances

- Regulatory standards for control of radiation and radioactivity in the environment

The remediation objectives 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 and direct radiation RAOs at a point immediately adjacent to the silos, as well as drinking water RAOs in any perched water that might be encountered directly below the silos. The treatability study goals are given in Section 1.5.

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 required to evaluate the alternatives, eliminate alternatives that are not technically feasible, and aid in the selection of the preferred alternatives(s). Further details of the alternatives are given in Section 2.0.

1.4 TREATABILITY STUDY

1.4.1 Justification

The justification to conduct these tests is provided by EPA in "Guide for Conducting Treatability Studies Under CERCLA." Treatability studies can provide the critical performance and cost information needed to evaluate and select treatment alternatives. The document recommended treatability tests for those substances that do not have standard treatment methods or supporting data in the literature that prove the material of interest can be effectively treated to render it nonhazardous. More explicitly in the case of Operable Unit 4, the purpose of treatment is to render the residues from Silos 1, 2, and 3 nonleachable so that it is not classified as characteristic waste under the Resource Conservation and Recovery Act (RCRA). However, the primary goal of these treatability studies will be to develop a stable waste form with minimal leachability of all contaminants, including radionuclides. Among the studies being conducted is the vitrification of the K-65 and metal oxide materials. These studies will provide information in determining the impacts of the effectiveness of vitrification.

The laboratory testing previously accomplished by Battelle Pacific Northwest Laboratory (PNL) in Richland, Washington provides a basis that the K-65 material has the capabilities to be vitrified. In the vitrification process, the waste material is combined with glass forming reagents, mixed together and placed into a crucible. The crucible and its contents are

MEDIA		REMEDIAL ACTION OBJECTIVES
1. SILO CONTENTS	1-1	For Human Health: Prevent exposures to non-carcinogens which would result in a Hazard index greater than or equal to unity (1), and/or combined risks from exposure to carcinogens greater 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 non-zero MCLGs, that would result in a Hazard Index greater than or equal to unity (1), and/or combined risks from exposure to carcinogens greater 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 greater than or equal to unity (1), and/or combined risks from exposure to carcinogens greater 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 20 pCi/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: Prevent inhalation of/ingestion of/direct contact with soils surrounding the silos which would result in a Hazard Index greater than or equal to unity (1), and/or combined risks from exposure to carcinogens greater 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 non-zero MCLGs, that would result in a Hazard Index greater than or equal to unity (1), and/or combined risks from exposure to carcinogens greater than or equal to 1.0E-04, using 1.0E-06 as the point of departure.
	3-3	Prevent radium and thorium concentrations from exceeding 5 pCi/g in the first 15 cm of soil, and 15 pCi/g at lower depths. Prevent concentrations of other nuclides from exceeding levels that would result in doses greater than 100 mrem/yr.
	3-4	For Environmental Protection: 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: Prevent ingestion of/direct contact with sediment contaminants which would result in a Hazard Index greater than or equal to unity (1), and/or combined risks from exposure to carcinogens greater than or equal to 1.0E-04, using 1.0E-06 as the point of departure.
	4-2	For Environmental Protection: 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: Prevent exposures to non-carcinogens which would result in a Hazard Index greater than or equal to unity (1), and/or combined risks from exposure to carcinogens greater than or equal to 1.0E-04, using 1.0E-06 as the point of departure.
	5-2	For Environmental Protection: Restore surface water to below ambient water quality criteria.
6. GROUNDWATER	6-1	For Human Health: Prevent ingestion of water having contaminant levels greater than the MCLS, non-zero MCLGs, TBCs, or which would result in a Hazard Index greater than or equal to unity (1), and/or combined risks from exposure to carcinogens greater than or equal to 1.0E-04, using 1.0E-06 as the point of departure.
	6-2	For Environmental Protection: Restore groundwater aquifer to contaminant concentration below the MCLs.

FIGURE 1-1.
(CONTINUED)

gradually heated up and brought to its melting temperature. During the heating process, some compounds in the waste will decompose or chemically react, often releasing off-gases. After the waste has been at its melting temperature long enough for the rock, debris, and other large particles to dissolve into the glass phase, the power will be shut off and it will be allowed to cool down.

In order to be able to compare the effectiveness of vitrification to stabilization and metal extraction as treatment options for the remedial alternatives for Silo 3 and the K-65 Silos in the feasibility studies and in the subsequent engineering designs, vitrification tests must be performed on the metal oxide residues (Silo 3 material) and additional data must be obtained for the K-65 residues. It is planned to utilize PNL to accomplish the treatability studies outlined in this document. PNL has extensive experience in conducting vitrification tests and has developed the laboratory bench-scale apparatus and the necessary experimental procedures for meeting quality assurance (QA) requirements.

The objectives of the previous laboratory tests were to determine the quantity and composition of the off-gas (including radon concentration) generated during vitrification of K-65 residue, the radon emanation rate from the vitrified K-65 waste, and the leachability of the vitrified K-65 waste. The test results from the previous laboratory tests have been documented in "Characteristics of Fernald's Silos 1 and 2 Residue Before, During and After Vitrification." Due to unforeseen laboratory conditions, inadvertently, the total volume of the generated off-gas during vitrification and, therefore, the total emanation of radon during vitrification, was not accurately measured. During the bench-scale treatability tests outlined in this document, special effort will be made to obtain accurate measurements of the total volume of the off-gas during vitrification and the emanation of radon during vitrification. The composition of the off-gas generated from the previous laboratory tests and the composition of the condensate from the previous laboratory tests are presented in Table 1-1.

During the previous vitrification tests, the radon emanation rate from the vitrified K-65 waste indicated that the radon concentration, which began at 0 pCi/L, averaged about 4 pCi/L. For the once through open loop system used, this represents an emanation rate of 48 pCi/hr or 1.56 pCi/m^2-s. This is an order of magnitude lower than the EPA limit of 20 pCi/m^2-s (40 CFR Part 61, Subpart Q).

Also, the Toxicity Characteristic Leaching Procedure (TCLP) leachate results from the previous laboratory test for the vitrified K-65 waste are presented in Table 1-2. The results are well below the established TCLP limits.

1.4.2 EPA Treatability Guidance

EPA's "Guide for Conducting Treatability Studies Under CERCLA" outlined a three-tiered approach to conducting treatability studies for a SUPERFUND site. The approach is exhibited in Figure 1-2. The evaluation of remedial alternatives phase of the RI/FS may require as many as three tiers of treatability testing:

TABLE 1-1
Composition of the Vitrification Off-Gas

COMPONENT	VOLUME %
Nitrogen	77.2%
Oxygen	17.1%
Other Ions	3.4%
Water	1.4%
Argon	0.9%
Carbon Dioxide	0.06%
Organic Compounds	None Detected

Condensate Composition

COMPONENT	(mg/L)
Aluminum	<0.1
Antimony	0.04
Arsenic	<0.01
Barium	0.07
Cadmium	<0.01
Calcium	14.1
Chromium	<0.01
Cobalt	0.01
Iron	0.06
Lead	0.09
Magnesium	3.7
Mercury	0.0029
Nickel	0.1
Phosphorus	0.2
Potassium	0.7
Selenium	0.19
Sodium	3.2
Total Uranium	0.011

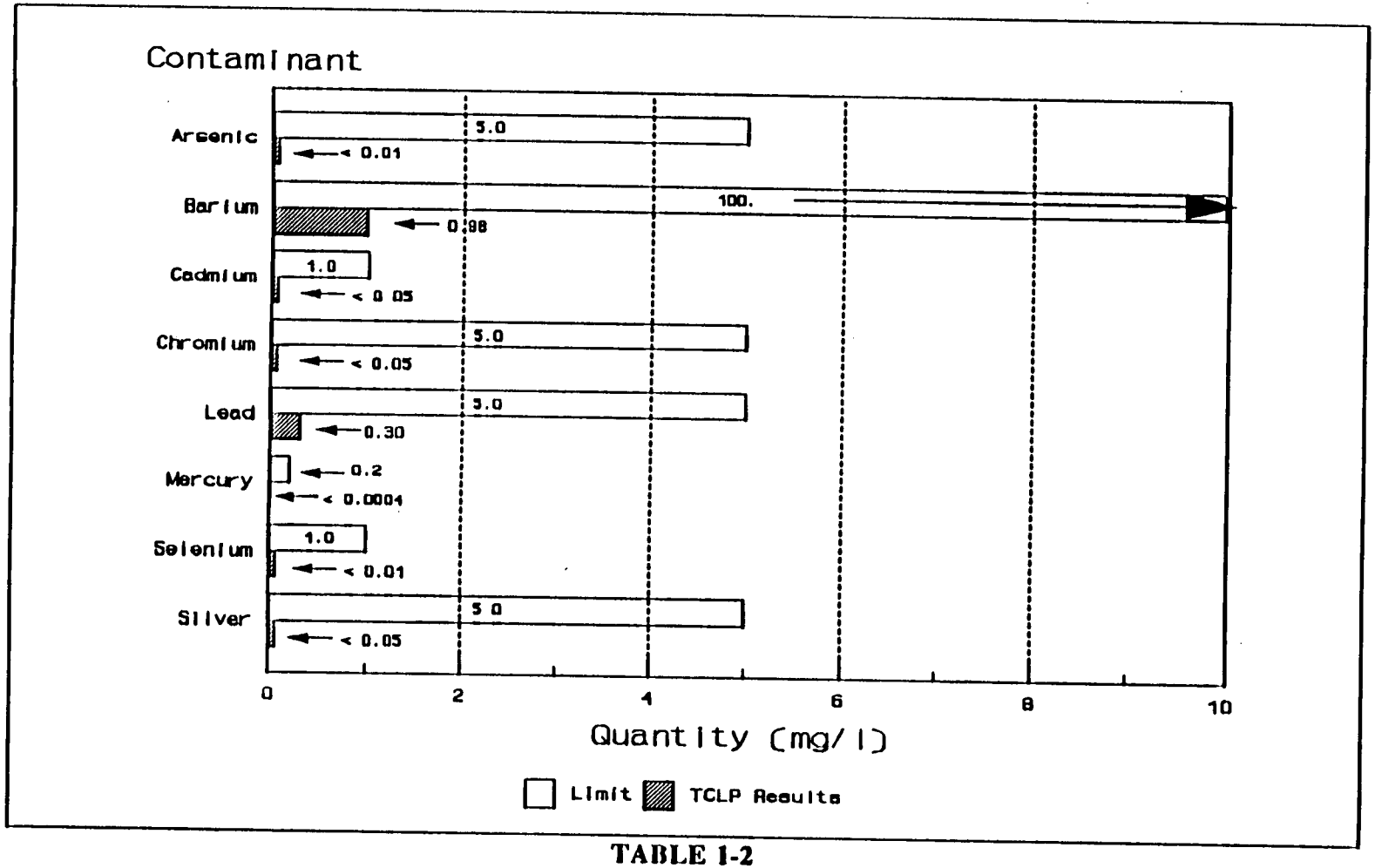

TABLE 1-2
Previous Laboratory Vitrification Tests
TCLP Leachate Results for Vitrified K-65 Material:
Concentration of Metals in Leachate

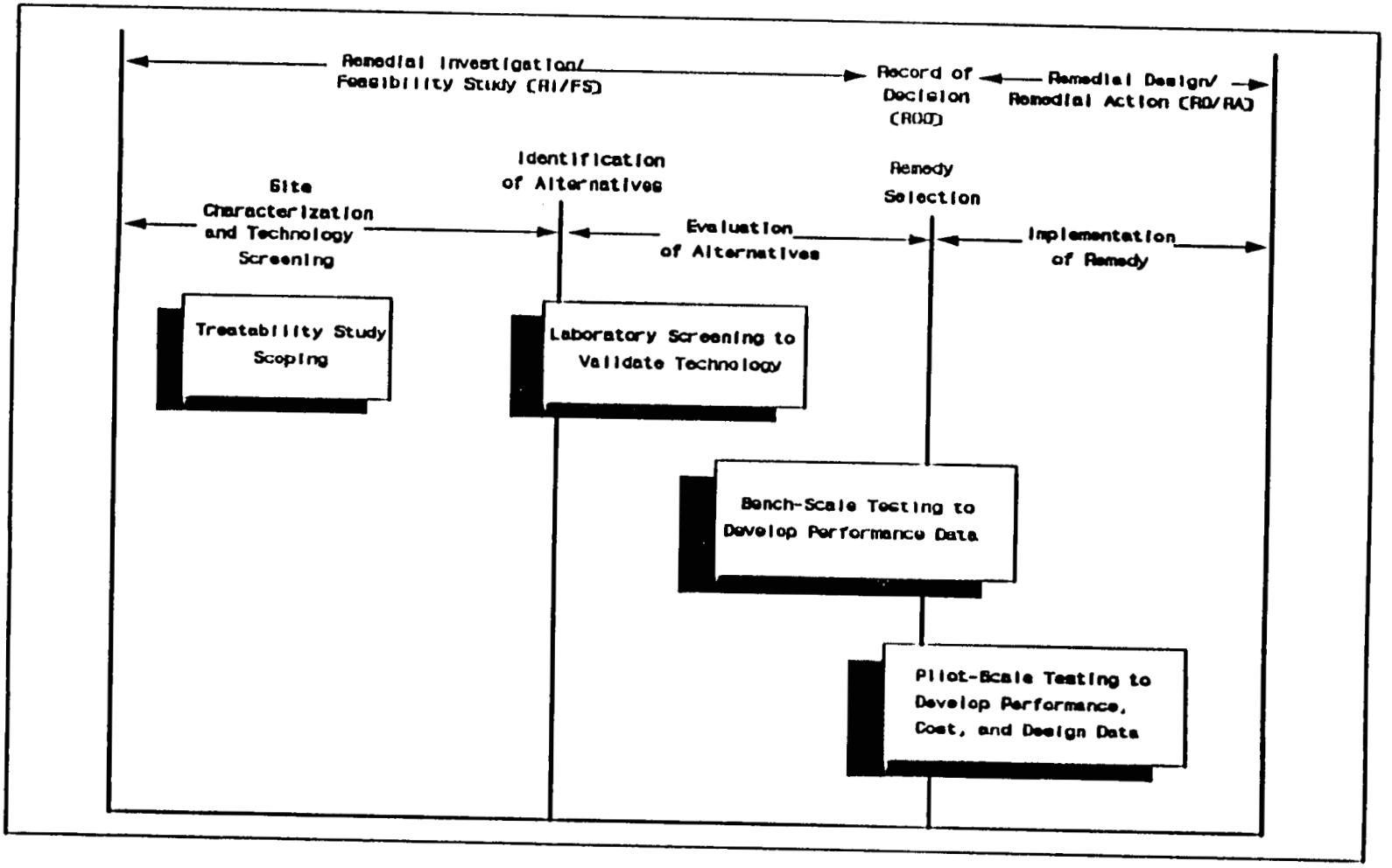

FIGURE 1-2
The Role of Treatability Studies in the RI/FS and RD/RA Process

- Laboratory Screening
- Bench Scale Testing
- Pilot-scale Testing

Laboratory screening and bench-scale testing are usually performed during evaluation of remedial alternatives. Pilot-scale testing is generally accomplished during remedy implementation. Laboratory screening has been performed on the K-65 material (Silos 1 and 2 material) during the previous laboratory testing accomplished by PNL. Additional laboratory screening of the K-65 material will be completed, as described in this work plan, prior to the bench-scale vitrification testing of the K-65 material. Laboratory screening of the metal oxide material (Silo 3 material) will be outlined in this document and will be performed prior to the bench-scale vitrification testing of the metal oxide material. The completion of the RI/FS detail analysis of remedial alternatives will determine if pilot-scale testing of the vitrification treatment option is required. Figure 1-3 illustrates the relationship of the completed, planned, and to be determined Operable Unit 4 vitrification treatability studies to the RI/FS process.

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 Record of Decision (ROD). During the detailed analysis of alternatives, all remedial alternatives are evaluated based on nine RI/FS evaluation criteria.

Results of the treatability studies should address seven of these criteria:

1) Overall protection of human health and the environment
2) Compliance with ARARs
3) Implementability
4) Reduction of toxicity, mobility, or volume
5) Short-term effectiveness
6) Cost
7) Long-term effectiveness

1.4.3 Approach

Treatability studies on the K-65 materials and the metal oxides will be performed as part of the evaluation of remedial alternatives phase of the RI/FS. The vitrification treatability studies described in this work plan and the stabilization and metal extraction treatability studies outlined in the "Treatability Study Work Plan for Operable Unit 4" prepared by Advanced Sciences, Inc./International Technology Corporation (ASI/IT), dated July 1991, will aid in the selection of a remedial alternative that is feasible, implementable, and cost effective.

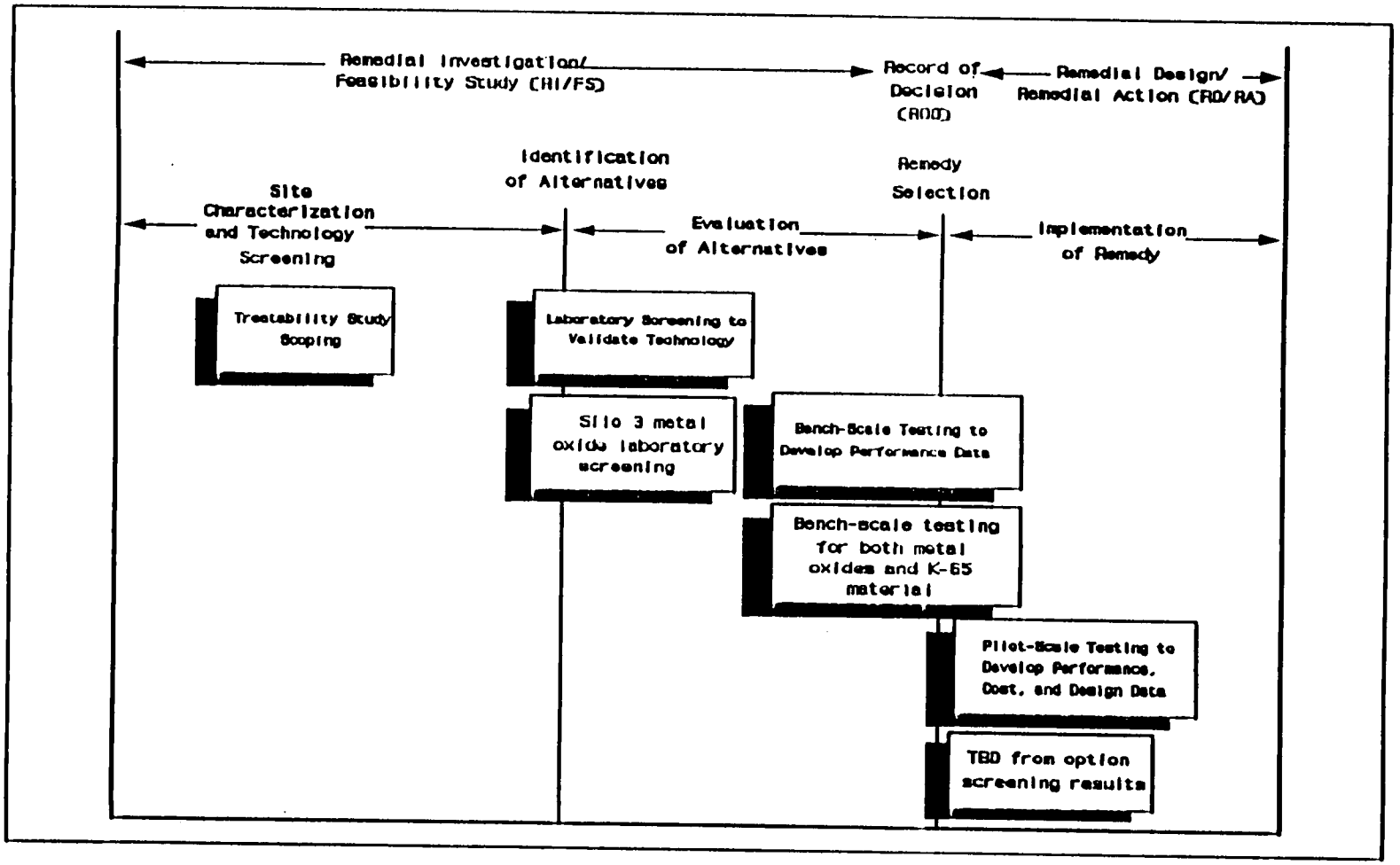

FIGURE 1-3
Relationship of the Operable Unit 4 Vitrification Treatability Studies to the RI/FS

The vitrification treatability studies described in this work plan will involve the vitrification of K-65 material from Silos 1 and 2 and the metal oxide material from Silo 3. Vitrification studies will be performed on the K-65 material by itself, the K-65 material with added Bento-grout, the metal oxide material by itself, and a predetermined mixture of K-65 material and metal oxide material.

As part of the Silos 1 and 2 (K-65) Removal Action, a layer of Bento-grout was applied to the residues contained in Silos 1 and 2. The Bento-grout layer retards the diffusion of radon being produced during the uranium decay sequence. Consequently, the Bento-grout layer with its trapped hazardous and radiological constituents will require the same treatment option as that of the K-65 material. To determine the impacts of this Bento-grout layer, one testing sequence will include Bento-grout added to the K-65 material prior to the vitrification.

The method used to collect residue samples from Silos 1 and 2 was to use the Vibra-Corer Unit. These samples were collected according to the methods and procedures detailed in the "Revised K-65 Silo Sampling and Analysis Plan" issued by ASI/IT on July 15, 1991. PNL will receive samples of the K-65 material from sections "A", "B", and "C" for each Silo 1 and 2 and one composite sample from each of the K-65 Silos. Metal oxide material from Silo 3 is available from cores archived during previous sampling operations. PNL will receive composite material from Silo 3 for use in the bench-scale tests for the metal oxide material.

Prior to performing vitrification testing on the metal oxide material, laboratory screening of the metal oxide material will be accomplished to determine the optimum glass forming material(s) to be added to the metal oxide material during vitrification. Also using existing K-65 material, tests will be conducted on the off-gas collection system and the radon adsorption system to optimize the bench-scale design of these systems.

1.4.4 Verification of Results

After a successful test run of the following vitrification sequences; K-65 material, K-65 material/Bento-grout mixture, and metal oxide material/K-65 material mixture, analyses will be conducted on the vitrified residues to determine the leachability of hazardous constituents, the leachability of radionuclides and the radon emanation of the vitrified residues. These tests will include the standard EPA TCLP protocols, radionuclide analyses per the Quality Assurance Project Plan (QAPP) approved by the U.S. EPA as part of the RI/FS Work Plan, and determining the radon emanation with appropriate instrumentation. Utilizing the laboratory equipment and instrumentation available, PNL will monitor the vitrified K-65 waste to determine the radon emanation (at 7 days and 30 days). Determining the radon emanation will not be performed for Sequence C vitrified material. After a successful test run, PNL will send duplicate samples of the vitrified waste from each vitrification sequence to an independent laboratory for the TCLP analysis as established in the QAPP approved by the U.S. EPA as part of the RI/FS Work Plan.

Also, for each vitrification sequence, the condensate collected from separating the moisture from the off-gas will be sent to an independent laboratory. The condensate will then be analyzed for: general water quality parameters, Hazardous Substance List (HSL) parameters, gross alpha, gross beta, gamma spectral analysis, and the following radionuclides:

- Radon-219*
- Radium-223*
- Radium-224
- Radium-226
- Radium-228
- Actinium-227
- Protactinium-231
- Polonium-210
- Lead-210
- Lead-211*
- Lead-214*
- Bismuth-214*
- Isotopic Thorium
- Isotopic Uranium
- Total Uranium

* Added at the direction of the Ohio EPA.

The analysis of the liquid effluent will aid in determining the required treatment or evaporation of the liquid. This data will aid in implementation of the design for the vitrification treatment option for the preferred alternative.

Information obtained for the identified vitrification treatability studies will aid in estimating the cost of implementation of the vitrification treatment option of remedial Alternatives 6 and 7 for the Silos 1 and 2, the vitrification treatment option of remedial Alternatives 3 and 4 for Silo 3, and the vitrification treatment of the leaching/stabilization of the contaminant separation stage of Alternatives 8 and 9 for the Silos 1 and 2.

1.5 GOALS OF THE TREATABILITY STUDY

The primary goal of the treatability study is to support remedy selection during the feasibility study (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 vitrifying them into an altered material. These data will be compared to

preliminary remediation goals, toxic constituent regulatory limits (TCLP limits), and site background concentrations to determine if attainment of any or all of these goals is feasible using the vitrification technology. These quantitative goals are developed in Section 3.0, which outlines the treatability study's specific performance objectives.

2.0 REMEDIAL TECHNOLOGY DESCRIPTION

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

The vitrification technology considered in the following alternatives consists of heating the residues to sufficient temperatures to induce the formation of glass-like mass. The resulting vitreous solid will have a reduced volume, be less likely to leach hazardous and radioactive components, and have a greatly reduced radon emanation rate. The vitrified material would be well suited for long-term disposal.

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 mitigative measures. However, it does include the installation of long-term monitoring equipment and the implementation of a monitoring program.

Alternative 1A - Nonremoval and Silo 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 walls are among the technologies considered for this alternative.

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

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

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

This alternative for Silos 1 and 2 calls for the removal and conventional stabilization or vitrification of the silo contents before on-property disposal in an engineered disposal facility.

This alternative includes siio demolition and disposal of the debris. Figure 2-1 is a flow diagram of Alternative 6.

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

This alternative is identical to Alternative 6 except that the material would be packaged for shipment to an approved off-site disposal facility. A flow diagram for Alternative 7 is provided in Figure 2-2.

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

This alternative 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. A potential 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 Seeley (1977), Mound Laboratories (1951), and Battelle (1981). Lead, barium, copper, and other metals will also be dissolved in the extraction fluid. Following this leaching stage, the remaining solids will enter a solid/liquid separation stage, and the leachate containing the radioactive and hazardous materials will be sent to a precipitation stage. This precipitation stage will add selected anions to yield a radioactive/hazardous precipitate to be solidified or stabilized for disposal. With the successful leaching process, the raffinate residues remaining after the acid or EDTA leaching processed will be disposed of as a nonhazardous waste. A flow diagram of this alternative is presented in Figure 2-3.

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

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

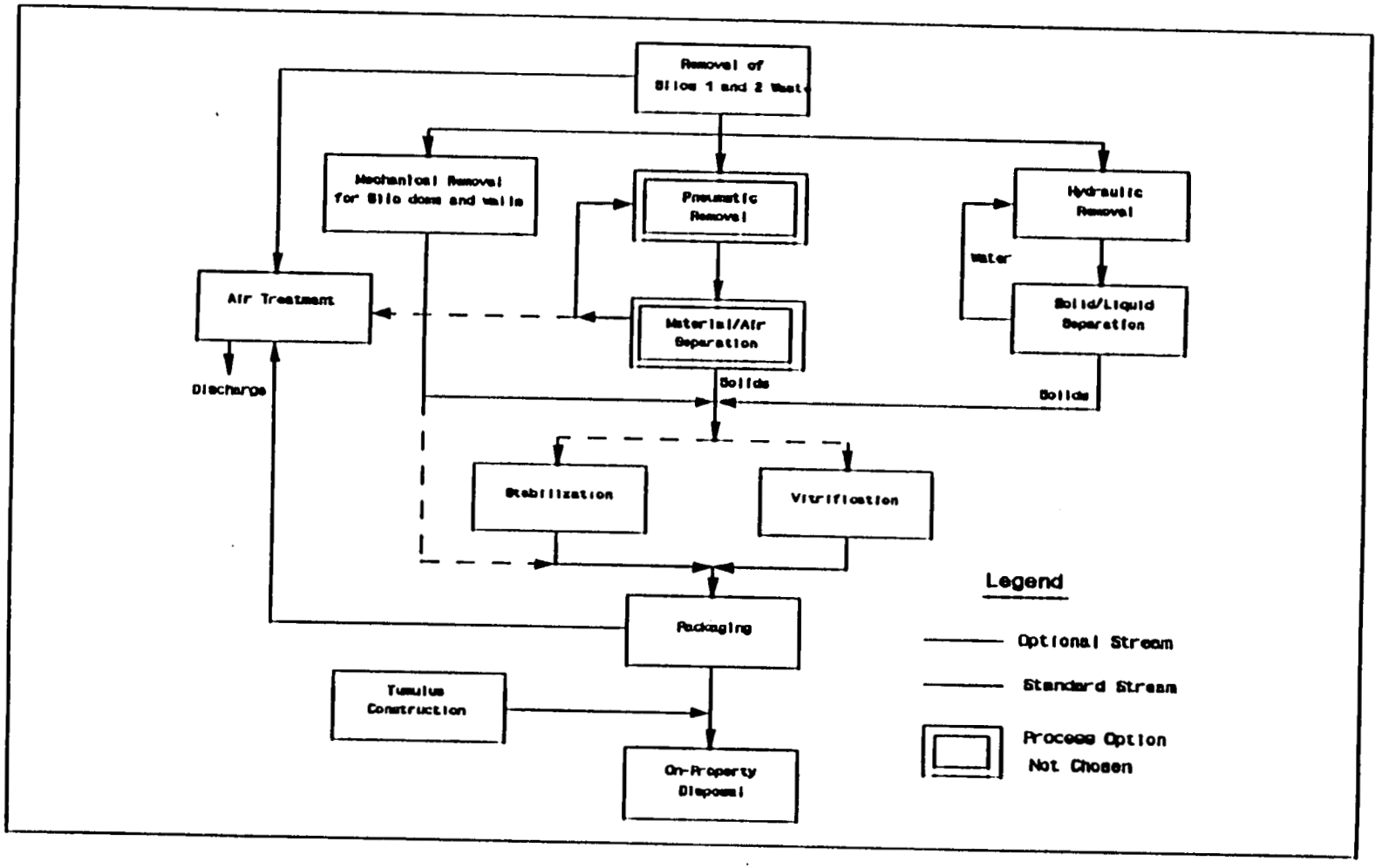

FIGURE 2-1
ALTERNATIVE 6: REMOVAL, TREATMENT, ON-PROPERTY DISPOSAL

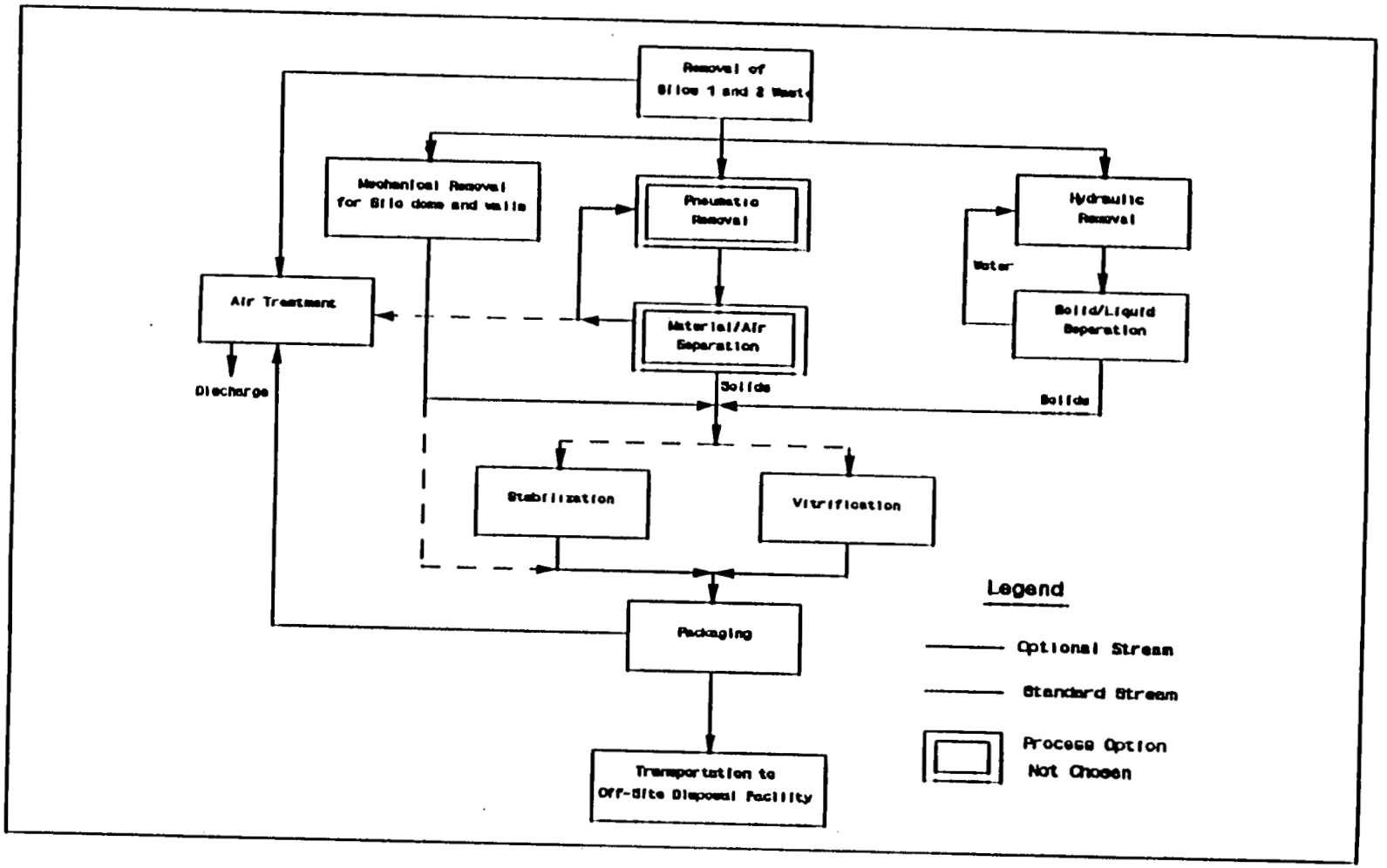

FIGURE 2-2
ALTERNATIVE 7: REMOVAL, TREATMENT, OFF-SITE DISPOSAL

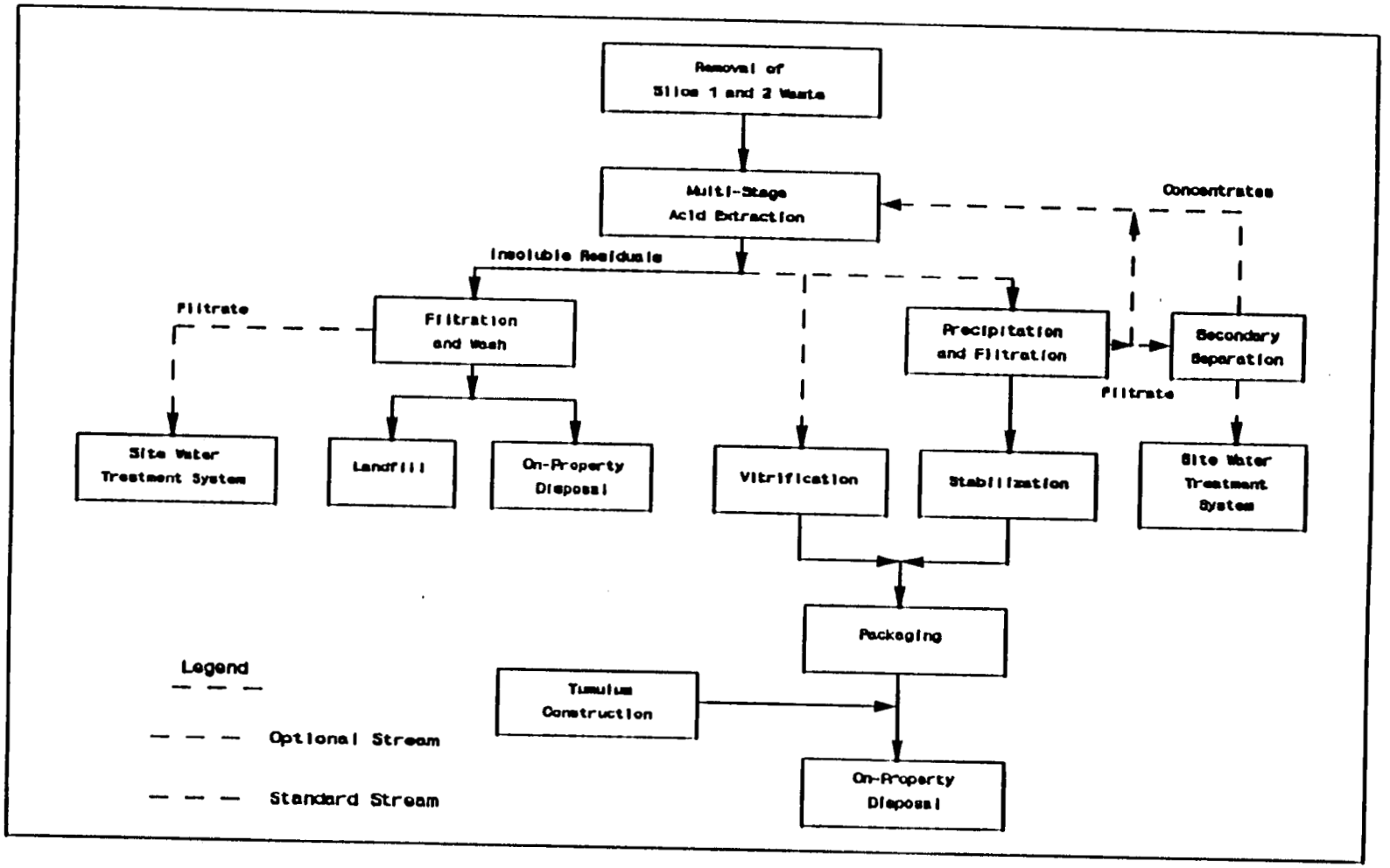

FIGURE 2-3
ALTERNATIVE 8: REMOVAL, CONTAMINANT SEPARATION, ON-PROPERTY DISPOSAL

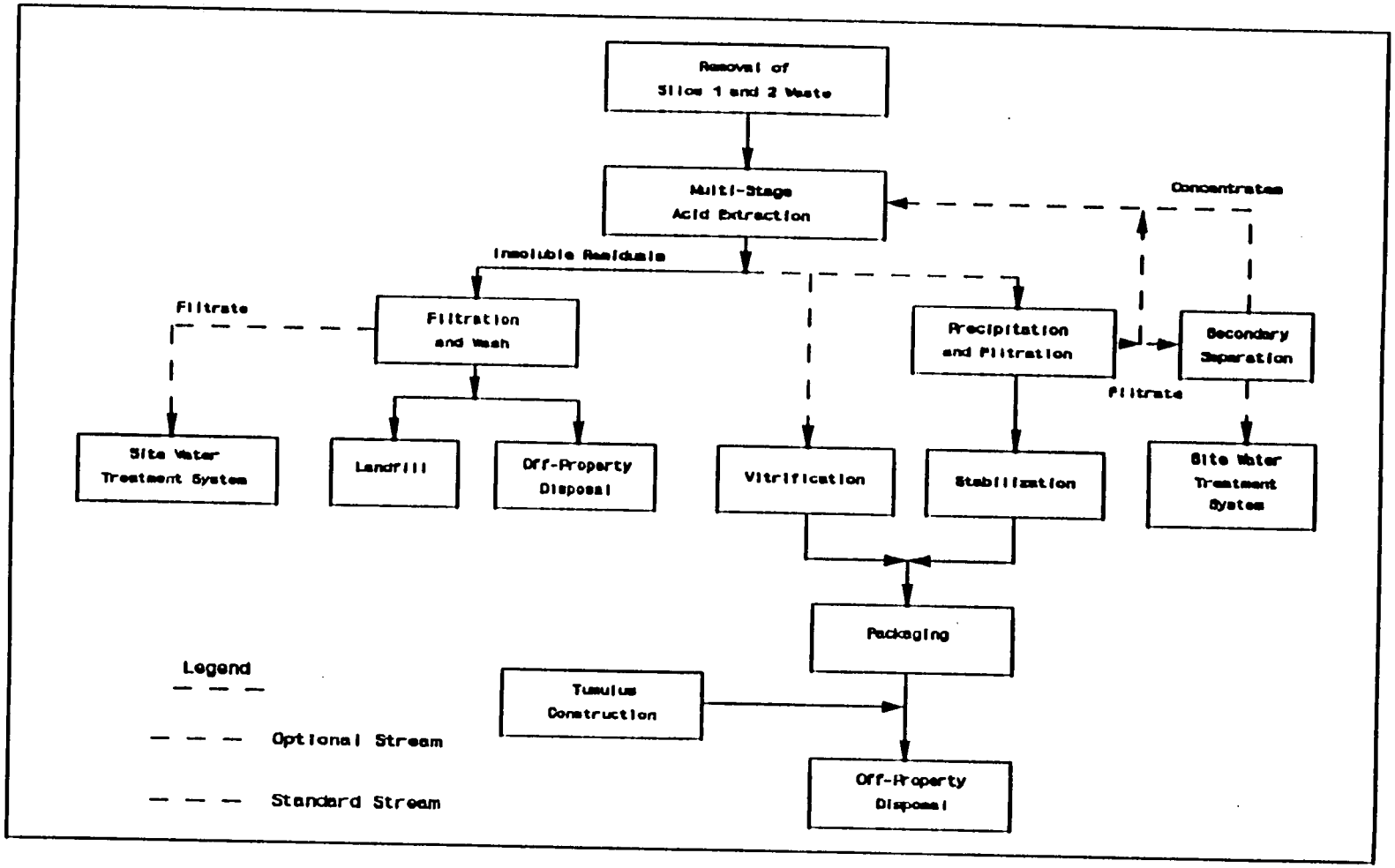

FIGURE 2-4
ALTERNATIVE 9: REMOVAL, CONTAMINANT SEPARATION, OFF-SITE DISPOSAL

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 implementation of the monitoring program are included.

Alternative 1B - Nonremoval and 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 walls are among the technologies considered for this alternative.

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

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 the K-65 silos 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 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.

Four alternatives for the three silos are considered non-viable. These alternatives are the "No Action" alternatives, OA (K-65 Silos) and OB (Silo 3); Alternative 2B "Nonremoval, In Situ

Stabilization, and Cap," (Silo 3); and Alternative 5 "Removal and Replacement in Rehabilitated Silo 3" Alternative 5.

The treatability tests described in this work plan will provide data for evaluating the performance of the remedial alternatives for both the K-65 residues (Silos 1 and 2) and the metal oxide waste stored in Silo 3. The treatability testing will be conducted to determine the effectiveness and long-term stability of; the vitrification treatment option of remedial Alternatives 6 and 7 for the Silos 1 and 2, the vitrification treatment option of remedial Alternatives 3 and 4 for Silo 3, and the vitrification treatment of the leaching/stabilization of the contaminant separation stage of Alternatives 8 and 9 for the Silos 1 and 2.

3.0 TEST AND DATA QUALITY OBJECTIVES

The purpose of the proposed vitrification tests is to obtain quantitative data for assessing the performance of the process in support of the RI/FS. The operational and performance information resulting from the proposed bench-scale test will permit more accurate full-scale cost and schedule estimates than those that can be made from laboratory screening information. The bench-scale tests will also provide information to configure and size unit operation for pilot scale testing.

3.1 PERFORMANCE OBJECTIVES AND DESIRED DATA

Specific test objectives have been identified so that the performance of this process can be readily compared to other remediation technologies under investigation. Leachability and volume reduction are two aspects on which this technology will be evaluated. The objectives of the test, both laboratory screening and bench-scale testing, are listed below:

Laboratory Screening

- To determine the chemical inorganic composition of samples from both K-65 material and the metal oxides
- To determine the anions present in both primary waste streams (K-65 and metal oxide materials)
- To determine the concentration of radioactive isotopes in both primary waste streams
- To measure radon emanation of untreated K-65 material
- To measure the bulk density of untreated wastes
- To determine the percent moisture of untreated wastes
- To measure the specific gravity of untreated wastes

Bench-Scale Testing

- To determine the composition of the off-gas
- To determine the radon emanation during vitrification of the K-65 material
- To measure the radon emanation from the vitrified K-65 material

- To ensure the final waste product meets the leaching criteria established in 40 CFR 261.24 by performing the TCLP on final waste products
- To evaluate the volume reduction potential of the vitrification process for the two primary waste streams
- To determine the chemical composition of the condensate
- To measure the specific gravity of vitrified waste

3.2 DATA QUALITY OBJECTIVES

The establishment of data quality objectives (DQOs) is part of the process that defines the data quality needs of the project. The implementation of an appropriate quality assurance/ quality control (QA/QC) program is required to ensure that data of known and documented quality are generated. Establishment of the DQOs will determine the level of QA/QC required for the treatability testing and analysis. DQO analytical levels are defined in EPA's guidance document "Guide for Conducting Treatability Studies Under CERCLA" (EPA/540/2-89-058). Table 3-1 summarizes the DQO levels. A list of tests and associated DQOs is delineated in Table 3-2.

3.3 PROCEDURES

The following procedures specific to the Operable Unit 4 vitrification treatability studies have been developed and are included in Appendix D:

- Physical properties of the untreated waste
- Radon emanation from untreated K-65 waste
- Radon emanation during vitrification of K-65 material
- Radon emanation from treated K-65 waste
- Modified TCLP for Arsenic, Barium, Cadmium, Chromium, Lead, Mercury, Selenium, and Silver
- Method for determining volume reduction
- Method for determining gamma dose rates of vitrified waste
- Product Consistency Test (PCT)

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/AC steps employed. Data typically reported in concentration ranges.
Level III	
Type of analysis	Organics/inorganics performed in an off-site analytical laboratory. May or may not use CLP procedures. Laboratory may or may not be a CLP laboratory.
Limitations	Tentative compound identification in some cases.
Data Quality	Detection limits similar to CLP. Rigorous QA/QC.
Level IV	
Type of analysis	Hazardous Substances List (HSL) organics/inorganics by GC/MS, AA, ICP. Low parts-per-billion detection limits.
Limitations	Tentative identification of non-HSL parameters. Validation of laboratory results may take several weeks.
Data Quality	Goal is data of known quality. Rigorous QA/QC.
Level V	
Type of analysis	Analysis by nonstandard methods.
Limitations	May require method development or modification. Method-specific detection limits. Will probably require special lead time.
Data Quality	Method-specific.

TABLE 3-1
SUMMARY OF ANALYTICAL LEVELS

TEST	DQO/COMMENT	DQO LEVEL
LABORATORY SCREENING		
Chemical Inorganic composition by Inductively Coupled Plasma (ICP)	Prior to vitrification tests, samples of both K-65 material and metal oxides will be analyzed to determine their inorganic components. This information will be used to predict the type and quantity of glass forming agents required.	II
Anion composition by Inductively Coupled Plasma Mass Spectrometer (ICPMS)	Anion composition analysis will give a basis from which to predict some general off-gas characteristics.	II
Radioactive isotope composition by Radioisotopic Analysis	Determining the quantity of some pertinent isotopes will aid in predicting radon concentration in the off-gas.	II
Physical Property Determination (percent moisture, bulk density, and specific gravity)	Determining the physical properties of the untreated wastes will aid in predicting vitrification process parameters.	V
Radon emanation from untreated K-65 material	This information will be used as a comparison to the radon emanation from the vitrified material.	V
BENCH-SCALE TESTING		
Elemental composition of the off-gas by Mass Spectrometric Gas Analysis	Characterization of the off-gas is important in determining the best additives for vitrifying that waste stream.	II
Radon emanation during vitrification	Determining the amount of radon contained in the off-gas is critical in designing the off-gas treatment system for full-scale implementation.	II
Measurement of the radon emanation from the vitrified product	This quantity will be used to evaluate the effectiveness of vitrification in reducing radon emanation.	II
Modified Toxicity Characteristic Leaching Procedure (mTCLP)	The MTCLP will be used as a preliminary test to determine if the vitrified product should be accepted as a satisfactory test. A sample from the product that passes this test will be sent to an independent lab for the full TCLP.	V
Full TCLP	To determine if the vitrified product meets the TCLP criteria. This test will provide data for the FS risk assessment calculations.	III
Volume reduction	To quantify vitrification's ability to reduce the volume of waste requiring disposal.	V

TABLE 3-2
ANALYTICAL TESTS AND ASSOCIATED DQO LEVELS

4.0 EXPERIMENTAL DESIGN AND PROCEDURES

4.1 LABORATORY SCREENING

Laboratory screening will be performed on the metal oxide material from Silo 3 and the K-65 material from Silos 1 and 2. The laboratory screening will be used to establish the validity of vitrifying the metal oxide material. This screening study will yield data that will be used as indicators of the vitrification technology to meet performance goals and will identify parameters for investigation during the bench-scale testing outlined in Sections 4.2.2 through 4.2.5.

Laboratory screening of the metal oxide material will involve analytical tests to determine the chemical inorganic and anion composition of the metal oxide material. Table 4-1 lists the elements that will be included in the inorganic analysis of the metal oxide material. The results of the analysis will be expressed in weight percent (wt%) as oxides. Table 4-2 specifies the chemicals to be included in the analyses for anions in the metal oxide material. The analytes listed in Tables 4-1 and 4-2 are the specific elements that affect the feasibility of the vitrification technology as determined by PNL. Also during the laboratory screening of the metal oxide material, a radioisotopic analysis will be conducted on the metal oxide material that will be used in the bench-scale vitrification testing to determine the radionuclide isotopic content of the material. The isotopes that will be identified during laboratory screening are as follows:

- Actinium-227
- Protactinium-231
- Lead-210
- Lead-211*
- Lead-214*
- Bismuth-214*
- Polonium-210
- Radium-223*
- Radium-224
- Radium-226
- Radium-228
- Radon-219*
- Thorium-228
- Thorium-230
- Thorium-232
- Uranium-234
- Uranium-235/236
- Uranium-238

* Added at the direction of the Ohio EPA

During the previous laboratory tests on the K-65 residue material that was supplied to PNL, analytical tests were performed to determine the chemical inorganic composition of the K-65 residue. The sample material provided to PNL for these previous laboratory tests was understood not to be representative of the material in Silos 1 and 2. Since this sample was from the 1989 sampling effort, it is understood that most of this sample came from Zone A. Current vitrification samples are identified as Zone A, B, and C, as well as a composite and are, therefore, considered representative. Therefore, the laboratory screening tests to be executed per this work plan will include analyses to determine the chemical inorganic composition of the K-65 material to be supplied to PNL, which includes samples from sections "A", "B", and "C" for each Silo 1 and 2 (six separate samples). PNL will be required to extract material from each of the samples provided to form a composite sample for each silo. Each of these composite samples will be analyzed for chemical inorganic composition of the material. Table 4-1 specifies the elements that will be included in the inorganic analysis of the K-65 material.

During the previous laboratory test on the K-65 material, the analytical tests did not include determining the anion composition of the K-65 material. The laboratory screening tests to be executed per this work plan will also include analytical tests to determine the anion composition of the K-65 material to be supplied to PNL. Each sample from sections "A", "B", and "C" for each Silo 1 and 2 and the composite sample formed from the samples provided will be analyzed for anion composition. Table 4-2 specifies the chemicals to be included in the analyses for anions in the K-65 material.

Also, during the previous laboratory tests on the K-65 residue material, a radioisotopic analysis was conducted to determine the isotopic content of the material. Table 4-3 represents the isotopic content of the K-65 material that was vitrified during the previous laboratory tests.

A radioisotopic analysis will also be conducted on the K-65 material samples from sections "A", "B", and "C" for Silo 1 and 2 and the composite sample for each of the K-65 Silos that will be vitrified as outlined in this work plan. The isotopes that will be identified during laboratory screening are as follows:

- Polonium-210
- Actinium-227
- Protactinium-231
- Lead-210
- Lead-211*
- Lead-214*
- Bismuth-214*
- Radium-223*
- Radium-224
- Radium-226
- Radium-228

- Radon-219*
- Thorium-228
- Thorium-230
- Thorium-232
- Uranium-234
- Uranium-235/236
- Uranium-238

* Added at the direction of the Ohio EPA

Also, during the previous laboratory tests on the K-65 residue material, radon emanation was determined on the untreated K-65 material. Radon emanation from the untreated K-65 material will be determined for the composite sample for each of the K-65 Silo composite samples that will be vitrified as outlined in this work plan.

TABLE 4-1
Laboratory Screening

Inorganic Chemical Analyses of Metal Oxide Material
and K-65 Material:
(weight % as oxides)

Silica	Copper
Lead	Cerium
Iron	Vanadium
Barium	Lanthanum
Aluminum	Uranium
Calcium	Manganese
Magnesium	Zirconium
Sodium	Neodymium
Phosphorus	Strontium
Titanium	Beryllium
Potassium	Thorium
Nickel	Tin
Cobalt	Selenium
Molybdenum	Zinc
Chromium	Chlorine
Sulfur	Fluorine

TABLE 4-2
Laboratory Screening

Characterization of Anions in Metal Oxide Material
and K-65 Material

Sulfate
Sulfide
Sulfite
Chloride
Nitrate
Carbonate

TABLE 4-3
Isotopic Content of K-65 Material
from Previous Laboratory Testing

ISOTOPE	ACTIVITY (nCi/g)
Th-230	264.7
Ra-226	479.4
Pb-214	297.8
Bi-214	280.0
Pb-210	338.2
Pa-231	29.8
Th-227	21.1
Ra-223	22.4
Rn-219	20.8
Pb-211	65

4.2 BENCH-SCALE TESTING

The bench-scale tests are designed to verify whether the alternatives which include vitrification as a treatment option described in Section 2 can meet the performance goals established by the ARARs. These tests will provide a quantitative evaluation of the performance of the vitrification treatment option as well as minimal cost and design information. The general objectives of the following tests are to: determine the composition of the off-gas generated during vitrification, the radon emanation rate from the vitrified K-65 material, and the leachability of the vitrified material. Quantifying the amount of radon during vitrification of the K-65 material will be determined by utilizing an open system, therefore, it will not be required to determine the quantity of off-gas generated. Figure 4-1 illustrates the equipment flow diagram for the open system.

The first test run for each of the Sequences involving K-65 material will be performed utilizing the open system equipment set-up. This is to enable continuous monitoring of the radon emanation during the vitrification process. The second test run for each of the Sequences involving K-65 material will be performed utilizing the closed system equipment set-up. This is to allow for sufficient collection of off-gas to determine off-gas chemical composition. A closed system will be used to ensure a representative sample of the off-gas will be collected. Figure 4-2 illustrates the equipment flow diagram for the closed system.

Prior to performing the testing identified in the following sections, the K-65 material and metal oxide material will be separated into batches. The bench-scale vitrification tests will be conducted by batch operations. The material will not be dried or sieved to remove rocks and other extraneous items as done in the previous vitrification testing. A batch of material from each testing sequence will be processed through the entire bench-scale system. Tests using specific components of the bench-scale system will be required to determine ideal melting temperatures for the various blends of material in Test Sequence B (K-65 material/Bento-grout) and Test Sequence D (K-65 material/Silo 3 material). These tests may also be required to determine ideal melting temperatures for Test Sequence C. These tests, if required, will utilize approximately 100 grams of material per test. Also, open system tests using specific components of the complete system may be performed for Test Sequence C (Silo 3 material) to determine process parameters prior to performing a complete bench-scale system test. Table 4-4 outlines the vitrification tests and identifies the type of material for each testing sequence. The amount of material listed on Table 4-4 for each test sequence is the estimated quantity of material required to complete each test sequence. This estimate will determine the amount of K-65 material and Silo 3 material to be shipped to PNL in support of the vitrification testing. The batch material will be melted in a 4-inch diameter by 12-inch tall (about 2.5 liters) inconel crucible, or relative equal, in a bench-scale furnace. The following data will be recorded for each batch of material tested (the specific data to be recorded for each test is included in Section 8.0):

TABLE 4-4
Operable Unit 4
Vitrification Tests for K-65 Material and Metal Oxide Material

SEQUENCE	TEST	TYPE OF MATERIAL	APPROX. AMOUNT OF MATERIAL	DESCRIPTION
0		K-65	0 kg	Off-gas collection/radon adsorption system testing using 900 grams of material at PNL. Remaining tests will use modified system.
A	1	K-65	1.0 kg	K-65 material and glass forming reagents based on previous tests and chem. composition analyses.
A	2	K-65	1.0 kg	Duplicate of Test 1.
B	3	K-65	.5 kg	Test to determine the influence of Bento-grout on the vitrification of K-65 material. 50/50 ratio is max. on removal of material from Silos 1 and 2.
		Bento-grout	.5 kg	
B	4	K-65	.5 kg	Duplicate of Test 3.
		Bento-grout	.5 kg	
C	5	Silo 3	1.0 kg	Initial trial run of metal oxide to glass forming reagents. Ratio determined during the laboratory screening.
C	6	Silo 3	1.0 kg	If Test 5 results are within specified bounds, this test will be a duplicate of Test 5. Or if initial ratio is revised per Test 5 results, Test 7 will be required.
C	7	Silo 3	1.0 kg	Duplicate of Test 6, if required.
D	8	K-65	.7 kg	Initial trial run of 70/30 ratio to determine characteristics on vitrified product of mixing waste streams.
		Silo 3	.3 kg	
D	9	K-65	.5 kg (min)	If Test 8 results are within specified bounds, this test will be a duplicate of Test 8. Or if initial ratio is revised per Test 8 results, Test 10 will be required.
		Silo 3	.5 kg (min)	
D	10	K-65	.5 kg (min)	Duplicate of Test 9, if required.
		Silo 3	.5 kg (min)	
	Total estimated amount of K-65 sample required: 12.0 kg Total estimated amount of Silo 3 metal oxides required: 9.5 kg			

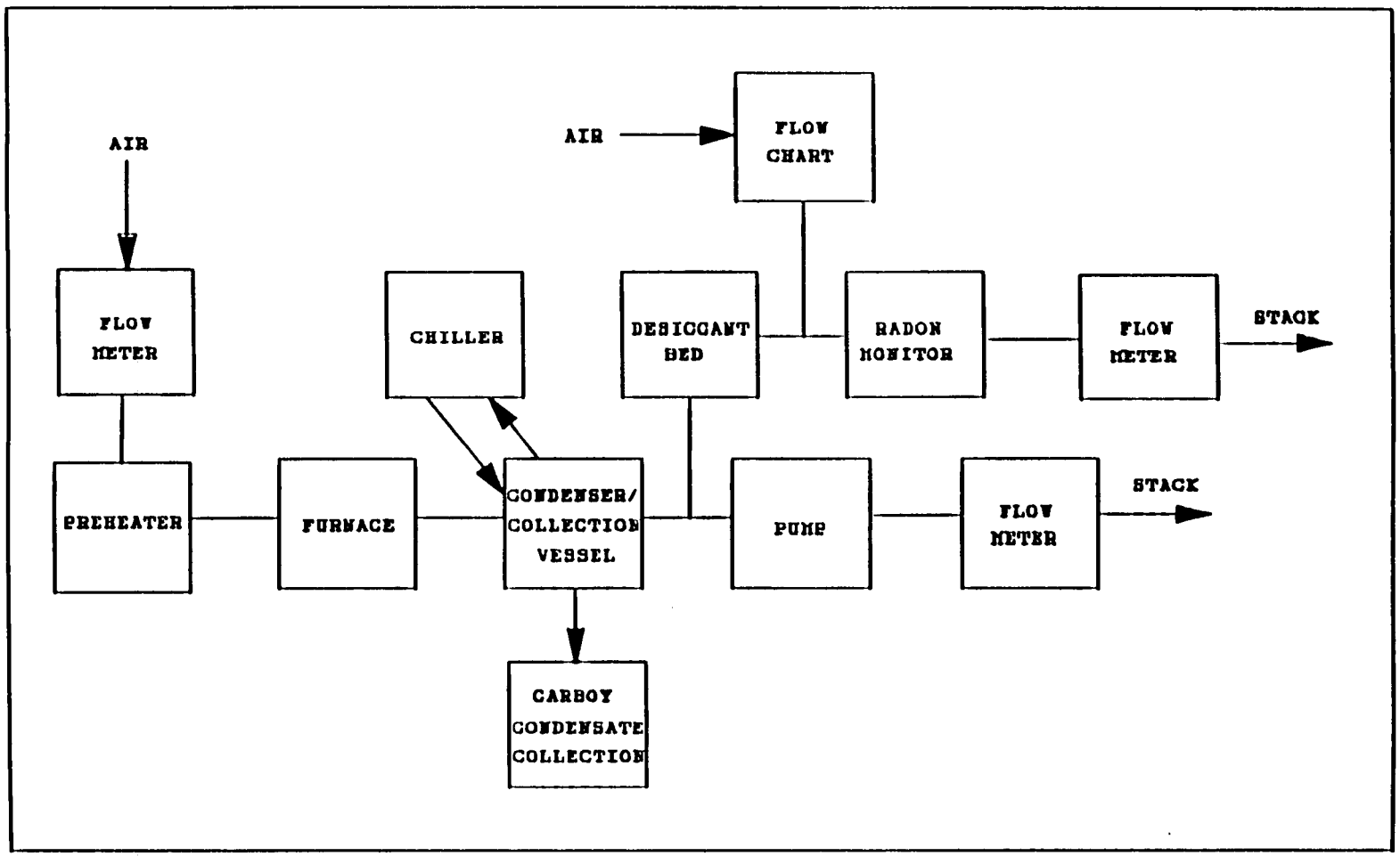

FIGURE 4-1
Equipment Flow Diagram Open System
Operable Unit 4 Vitrification Treatability Studies

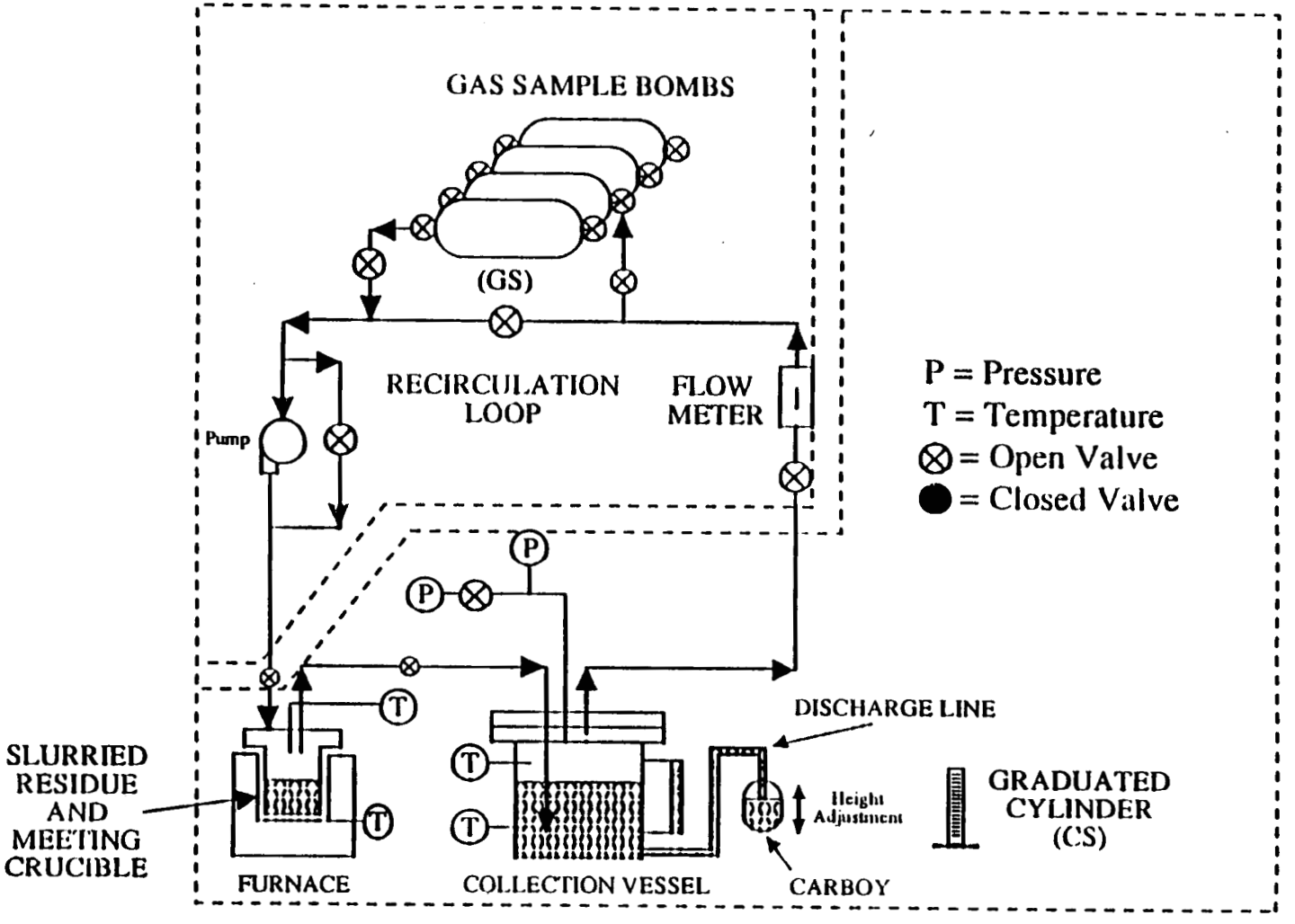

FIGURE 4-2
Equipment Flow Diagram Closed System
(Off-Gas Collection System)
Operable Unit 4 Vitrification Treatability Studies

- The chemical composition of the collected off-gas; 3006
- The resulting vitrified K-65 material from each batch will be monitored for radon emanation;
- The leachability of the vitrified material, including TCLP and radionuclides; and
- The composition of the condensate will be determined as defined in Section 1.4.4.

After a successful test run from each vitrification sequence, PNL will send samples of the vitrified residues from both the successful test run and the duplicate test run to an independent laboratory for the TCLP analysis and PCT as established in the QAPP approved as part of the RI/FS Work Plan. The Decision Analysis Tree for Vitrification Treatability Studies for Silos 1, 2 and 3 Material is shown on Figure 4-3.

4.2.1 Preliminary Off-gas Testing

Based on results of the previous vitrification testing from the used off-gas collection system and radon adsorption system, PNL has evaluated the results of the previous vitrification testing. It was determined that the modifications best suited to radon measurement would be a continuous measurement of radon in the off-gas stream as opposed to a determination of the radon content based on collection of the total quantity of off-gas. Therefore, the total volume of off-gas will not be measured.

Laboratory equipment designed for the continuous monitoring of radon in the off-gas will be used to verify the methodology in determining the amount of radon generated for the identified vitrification sequences. This preliminary testing of the off-gas stream may require several trail test runs to determine the exact equipment set-up of the open system. The material to be used during this testing will be approximately 900 grams of untreated K-65 material previously shipped to PNL that was not used during the previous vitrification tests on the K-65 material.

All subsequent sequence tests to determine the radon emanation during vitrification will use an open system equipment set-up which was determined by the preliminary testing of the off-gas.

4.2.2 Sequence A - Vitrification of K-65 Material

The batch of material for the first vitrification test (Test 1) will consist of a minimum of 1000 grams (1 kg) of K-65 material and the identified flux agents based on the previous vitrification testing and laboratory screening results. It is estimated that approximately 10-15% (by weight) of NaOH as a flux will be added to the K-65 material to form a

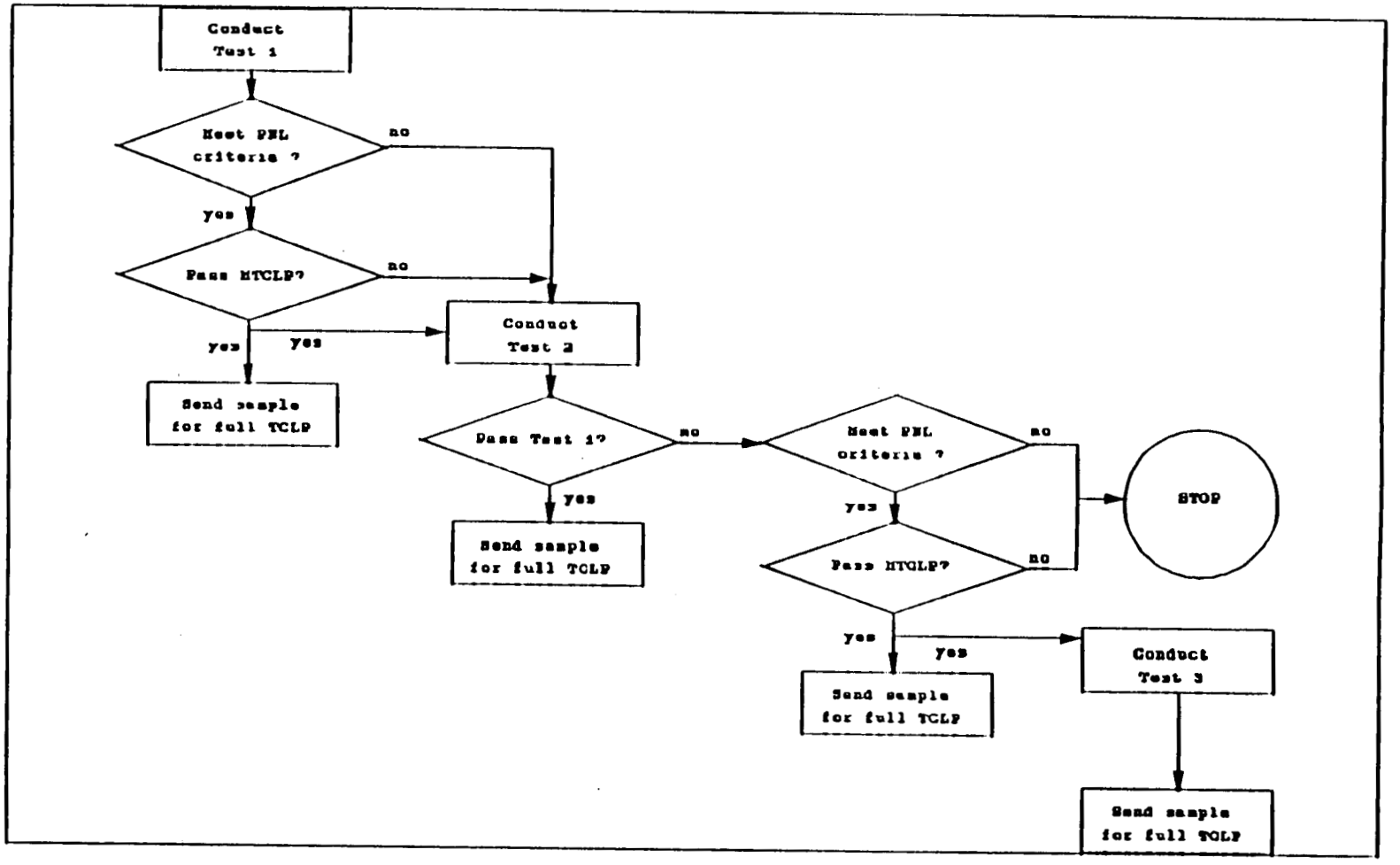

FIGURE 4-3
Decision Analysis Tree for Vitrification Treatability Studies
for Silos 1, 2 and 3 Material

silicate glass. Previous vitrification testing utilized NaOH as a flux for the K-65 material. Previous testing of the K-65 material indicated that the K-65 material contains sufficient glass forming elements such that additional glass forming reagents are not required. Particular attention will be given to the continuous measurement of the radon concentration of the off-gas. Samples of the vitrified waste will be analyzed for TCLP concentration of metals in the leachate. Test 2 will duplicate Test 1 to verify the results. Test 2 will use the closed system equipment set-up. Duplicating or triplicating each successful test for performing bench-scale tests is recommended by the EPA "Guide for Conducting Treatability Studies Under CERCLA."

4.2.3 Sequence B - Vitrification of K-65 Material Mixed with Bento-grout

As part of the Silos 1 and 2 (K-65) Removal Action, a layer of Bento-grout was applied to the residues contained in Silos 1 and 2. The Bento-grout layer retards the diffusion of radon produced during the decay of radium-226. Consequently, the Bento-grout layer, with its trapped hazardous and radiological constituents, will require the same treatment option as that of the K-65 material. To determine the impacts of this Bento-grout layer, testing Sequence B will include Bento-grout added to the K-65 material prior to the vitrification process. It is estimated that approximately 10-15% (by weight) of NaOH as a flux will be added to the K-65 material/Bentonite mix to form a silicate glass. Testing the K-65 material/Bento-grout mix will determine the influence of Bento-grout on the vitrified product. Preliminary tests may be performed to determine ideal melting temperatures, the suitable glass forming reagents and the blend of K-65 material to Bento-grout.

The initial test run (Test 3) will involve a mix ratio by mass of K-65 material to Bento-grout. The initial mix ratio by mass will be based on the maximum amount of Bento-grout that possibly could be in the waste stream upon removal of the top layer of material from Silos 1 and 2 and the results of the preliminary open system tests.

Test 4 will duplicate Test 3 to verify the results.

4.2.4 Sequence C - Vitrification of Metal Oxide Material

Sequence C tests will determine if it is possible to obtain an acceptable vitrified metal oxide product. The specific glass forming reagents that are required will be calculated based on the results of the laboratory screening of the metal oxide material. It is anticipated that glass forming reagents such a silica, iron, aluminum, boron, and calcium will be added to the metal oxide material to form a phosphate glass. Preliminary tests will be performed using the Silo 3 material to determine the initial process parameters prior to performing a complete system test to vitrify the Silo 3 materials. If the identified test for Sequence C is in compliance with the PNL specific criteria for vitrification, the vitrified product will be analyzed for

leachability by PNL or their subcontractor by conducting a modified TCLP[1] (mTCLP) on the vitrified material.

PNL specific criteria for vitrification will be PNL's best engineering judgment of the vitrification process parameters. PNL specific criteria is based on approximately 30 years of experience in performing vitrification studies. The PNL specific criteria would include items such as:

- Melting temperature to form glass would indicate the comparability with the vitrification
- Presence of spinels, crystals, or other observable non-homogeneities would indicate an inadequate waste form
- Possible slag (e.g., sulfate layer) on top of the vitrified waste form would indicate possible incompatibility with the vitrification process
- Presence of heavy metals, sulfides, etc. on bottom of the melted material would indicate possible incompatibility with the vitrification process

A successful test run will be defined as meeting the PNL specific criteria and the leachability of the heavy metals are within regulatory requirements as determined by the modified TCLP.

After further review of the 1989 sampling data for the metal oxide material included in the Draft Operable Unit 4 Remedial Investigation Report, it has been found that due to the sulfur content of the material, vitrification may not be a feasible treatment technology. If vitrification is determined not to be a feasible alternative, no further testing will be performed after the laboratory screening of the Silo 3 material.

If the initial test run, Test 5, is compliant with the PNL specific criteria for vitrification and results of the mTCLP comply with 40 CFR 261.24g, Test 5 will be considered a successful test run and a sample of the vitrified product will be sent to the independent laboratory as established in the QAPP. Test 6 will be performed as a duplicate test of Test 5 and a sample of the vitrified product also sent to the independent laboratory as established in the QAPP.

1

[1]The modified TCLP, as it applies to the identified vitrification tests, is defined as analysis of the vitrified product for leachability of the following heavy metals; Arsenic, Barium, Cadmium, Chromium, Lead, Mercury, Selenium, and Silver. Based on the available EP toxicity data (Buelt, 1989) from the previous vitrification test, all of the heavy metals from the EP toxicity list, with the exception of lead were below the regulatory limits.

If the initial test run, Test 5, is compliant with the PNL specific criteria for vitrification or the vitrified product from the Test 5 does not meet the TCLP leach rates limits, the appropriate modifications based on the results of Test 5 will be made for conducting Test 6. The modifications could involve revising the glass forming reagents or altering the vitrification process parameters, or modifying the bench-scale equipment set-up. Modification of the process parameters will be a major factor in determining whether the same process facility could be utilized for the vitrification of the K-65 material and the metal oxide material.

If required, the appropriate modifications will be made and Test 6 will be performed. If Test 6 is compliant with the PNL specific criteria for vitrification and the mTCLP results meets TCLP leach rates limits, Test 6 will be considered a successful test run and a sample of the vitrified product will be sent to the independent laboratory as established in the QAPP. Test 7 will be performed as a duplicate test of Test 6 and a sample of the vitrified product also sent to the independent laboratory as established in the QAPP.

There is a possibility that based on the results of Test 5, 6 and 7, PNL will determine that vitrification of the metal oxide material is not a technically feasible treatment option for the remediation of the Silo 3 material.

4.2.5 Sequence D - Vitrification of Metal Oxide Material Mixed with K-65 Material

The mixture of Silo 3 and K-65 material has been proposed for the purpose of reducing costs of remediation. The specific glass forming reagents and the amounts that are required will be calculated based on the laboratory screening of the metal oxide material and the K-65 residues. It is not known nor is it assumed that the mixture of K-65 and Silo 3 material will easily vitrify. If an incompatibility of this mixture is determined during laboratory screening tests, further studies will not be conducted (Bench-scale testing Sequence D will not be conducted).

Sequence D tests will determine if it is possible to obtain an acceptable vitrified product by mixing the K-65 material and the metal oxide material. The initial test run of Sequence D (Test 9) will involve a 70/30 mix ratio[2] by mass of K-65 material to metal oxide material. The 70/30 mix ratio by mass is based on the estimated total mass of the two primary Operable Unit 4 waste streams: the K-65 material and the metal oxide material. To conduct Sequence D tests, the specific glass forming reagents and possible flux material, will be calculated using the results of the laboratory screening of the metal oxide material and results from Sequence A and C tests. If the identified test for Sequence D is compliant with the PNL specific criteria for vitrification, the vitrified product will be analyzed for leachability by PNL or their subcontractor by conducting a modified TCLP on the vitrified material.

[2]Unless determined otherwise after reviewing the laboratory screening analytical results.

If the initial test run, Test 8, is compliant with the PNL specific criteria for vitrification and the mTCLP results meets the TCLP leach rate limits, Test 8 will be considered a successful test run and a sample of the vitrified product will be sent to the independent laboratory as established in the QAPP. Test 9 will be performed as a duplicate test of Test 8 and a sample of the vitrified product also sent to the independent laboratory as established in the QAPP.

If the initial test run, Test 8, is not satisfactory, the appropriate modifications based on the results of Test 8 will be made for conducting Test 9. The modifications could involve revising the glass forming reagents or altering the vitrification process parameters or modifying the bench-scale equipment set-up. Results of these tests will be a major factor in determining whether the same process facility could be utilized for the vitrification of the K-65 material and the metal oxide material.

If required, the appropriate modifications will be made and Test 9 will be performed. If Test 9 is satisfactory, this test will be considered a successful test run and a sample of the vitrified product will be sent to the independent laboratory as established in the QAPP. Test 10 will be performed as a duplicate test of Test 9 and a sample of the vitrified product will also be sent to the independent laboratory as established in the QAPP.

There is a possibility that based on the technical results of Test 8, 9, and 10, a determination by PNL will be made that vitrification of the identified waste streams is not a technically feasible treatment option for the remedial alternatives for Operable Unit 4.

5.0 EQUIPMENT AND MATERIALS

Table 5-1 provides a preliminary list of equipment and materials required to complete the bench-scale tests. All the items listed, in addition to those identified by PNL, will be provided by PNL.

TABLE 5-1
EQUIPMENT AND MATERIALS

- Air Heater
- Furnace
- Furnace Temperature Controller
- Thermowells and Thermocouples
- Moisture Analyzer
- Scale (Weight Measurement)
- Flow Meters
- Crucibles with Sealing Flanges and Unique Label
- Gas Sample Bombs
- Oil-Less Vacuum Pump
- Chiller
- Condenser/Collection Vessel
- Seal Materials for Crucibles, Collection Vessel, etc.
- Glass Forming Reagents
- Carboys, Sample Bottles, Beakers, and Other Common Lab Equipment
- Activated Carbon Cartridges
- Desiccant Beds
- Radon Monitors
- Computer with Printer
- Any Other Equipment that Becomes Necessary to Perform the Work

6.0 SAMPLING AND ANALYSIS

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The sampling and analysis plan for the acquisition of residue samples for Silos 1 and 2 is contained in the "Implementation Plan for the K-65 and Metal Oxide Sampling Project at the Feed Materials Production Center, Fernald, Ohio," Addendum-SAP, October 10, 1990.

7.0 DATA MANAGEMENT

The collection and preparation of silo residues for shipment to PNL shall be according to procedures developed by ASI/IT and WEMCO as contained in the "Implementation Plan for the K-65 and Metal Oxide Sampling Project at the Feed Materials Production Center, Fernald, Ohio," Addendum-SAP, October 10, 1990. The vitrification data will be acquired in accordance with the PNL Vitrification QA Plan WTC-060 as presented in Appendix A. PNL shall provide a records-turnover-package which contains all raw data generated during the vitrification project, all calculations performed, plus all QA documentation specified in the above mentioned QA Project Plan.

Laboratory notebooks will be used for this project. All laboratory notebooks are uniquely numbered and permanently bound with sequentially numbered pages. The notebook will be a project-specific notebook which will be assigned to the individuals working on the project. All daily laboratory activities associated with the project will be recorded in the project-specific notebooks.

All records management and reporting for the TCLP analyses performed on the vitrified material will follow standard QA/QC protocol in the QAPP and Volume 4 on the RI/FS Work Plan.

8.0 DATA ANALYSIS AND INTERPRETATION

8.1 EFFECTIVENESS OF WASTE FORM

The results of the TCLP in determining the leach rates of the vitrified material will be used to evaluate the long-term effectiveness of each sequence of testing. The concentrations of radioactive and hazardous constituents in the leachate will be used as input into the geochemical models described in the RI/FS Work Plan 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, and the resulting risks to human health and the environment.

8.2 LABORATORY SCREENING

8.2.1 Metal Oxide Material From Silo 3

The following data will be presented in tabular form for the metal oxide material provided to PNL:

- General description of the waste
- Chemical inorganic composition as listed in Table 4-1
- Anion composition as listed in Table 4-2
- Radionuclide isotopic content as listed in Table 4-3
- Physical characteristics: percent moisture, bulk density, specific gravity

8.2.2 K-65 Material From Silos 1 and 2

The following data will be present in tabular form for each of the six samples provided to PNL from section "A", "B", and "C" of each Silo 1 and 2 and the composite samples for each K-65 Silo PNL made from the samples provided (8 sets of data will be provided):

- General description of the waste
- Chemical inorganic composition as listed in Table 4-1
- Anion composition as listed in Table 4-2
- Radionuclide isotopic content as listed in Table 4-3
- Physical characteristics: percent moisture, bulk density, specific gravity
- Radon emanation of composites of untreated sample material

8.3 BENCH-SCALE TESTS

The following data will be presented for the bench-scale vitrification Sequence A tests:

- Formula of glass forming reagents and weights
- Percent moisture versus percent solids content of the glass forming reagents
- Amount of water added to form a 45% moisture content slurry
- Weight of K-65 waste material
- Size of furnace/crucible
- Temperature of furnace
- Heating time of sample (elapsed time vs temperature)
- Electrical conductivity of molten material
- Viscosity as a function of temperature for molten material
- Composition of off-gas from vitrification
- Radon released during vitrification
- Composition of condensate
- Radon released from vitrified waste
- Specific gravity of vitrified waste
- TCLP leachate results for metals from vitrified waste
- TCLP results from vitrified waste
- Radionuclide leachate results from vitrified waste
- Gamma dose rates of vitrified waste

The following data will be presented for the bench-scale vitrification Sequence B tests:

- Formula of glass forming reagents and weights
- Percent moisture vs percent solids content of glass forming reagents
- Amount of water added to form a 45% moisture content slurry
- Weight of K-65 waste material
- Dry weight of Bento-grout
- Bento-grout slurry composition
- Physical characteristics of K-65 material/Bento-grout mix: percent moisture, bulk density
- Size of furnace/crucible
- Temperature of furnace
- Heating time of sample (elapsed time vs temperature)
- Electrical conductivity of molten material
- Viscosity as a function of temperature for molten material
- Composition of off-gas from vitrification
- Radon released during vitrification
- Composition of condensate
- Radon released from vitrified waste
- Specific gravity of vitrified waste
- TCLP leachate results for metals from vitrified waste
- TCLP results from vitrified waste
- Radionuclide leachate results from vitrified waste
- Gamma dose rates of vitrified waste

The following data will be presented for the bench-scale vitrification Sequence C tests:

- Formula of glass forming reagents and weights
- Amount of water added to form a 45% moisture content slurry
- Percent moisture vs percent solids content of glass forming reagents
- Weight of metal oxide material
- Size of furnace/crucible
- Temperature of furnace
- Heating time of sample (elapsed time vs temperature)
- Electrical conductivity of molten material
- Viscosity as a function of temperature for molten material
- Composition of off-gas from vitrification
- Composition of condensate
- Specific gravity of vitrified waste
- TCLP leachate results for metals from vitrified waste
- TCLP results from vitrified waste
- Radionuclide leachate results from vitrified waste
- Gamma dose rate of vitrified waste

The following data will be presented for the bench-scale vitrification Sequence D tests:

- Formula of glass forming reagents and weights
- Percent moisture vs percent solids content of glass
- Amount of water added to form a 45% moisture content slurry forming reagents
- Weight of K-65 waste material
- Weight of metal oxide material
- Temperature of furnace
- Size of furnace/crucible
- Heating time of sample (elapsed time vs temperature)
- Electrical conductivity of molten material
- Viscosity as a function of temperature for molten material
- Volume of off-gas from vitrification
- Composition of off-gas from vitrification
- Radon released during vitrification
- Composition of condensate
- Radon released from vitrified waste
- Specific gravity of vitrified waste
- TCLP leachate results for metals from vitrified waste
- TCLP results from vitrified waste
- Radionuclide leachate results from vitrified waste
- Gamma dose rate of vitrified waste

9.0 HEALTH AND SAFETY

PNL will conduct the vitrification studies outlined in this work plan in accordance with the applicable OSHA requirements thereby ensuring worker protection in the workplace. The Waste Technology Center component of Battelle Northwest is responsible for vitrification studies at PNL. The Safety Plan for the Waste Technology Center is found in Appendix B.

10.0 RESIDUALS MANAGEMENT

The vitrified residues and any untreated K-65 material and Silo 3 material will be returned to the FEMP for disposal. All other contact laboratory waste material generated as part of the vitrification treatability testing must be disposed of by the appropriate laboratory.

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

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

The client will be responsible for proper transport or shipment of all material to be returned to the FEMP for disposal unless otherwise specified in the written contract.

11.0 REPORTS

An interim draft report will be prepared by PNL personnel and transmitted to WEMCO within 45 calendar days, or no later than November 9, 1992, of completing the laboratory screening and the bench-scale tests. This report will present the data identified in Section 8 and detail the vitrification process employed, along with any problems. The report will be generated utilizing Section 3.12 of the "Guide for Conducting Treatability Studies Under CERCLA". The interim draft report will be reviewed by WEMCO, and PNL personnel will incorporate the WEMCO comments and submit a final report to WEMCO on or before December 30, 1992. This final report will be reviewed internally by WEMCO, ASI, and DOE prior to final submittal to the U.S. EPA. The results of the leachate from the TCLP analyses performed per the QAPP will be incorporated into the report when available.

12.0 COMMUNITY RELATIONS

The vitrification treatability study for Silos 1, 2, and 3 material and community information and involvement activities are required in the CERCLA process. Community Relations activities shall be conducted; to support treatability studies for Operable Unit 4 to explain the role of treatability studies in the RI/FS, and to raise the public's confidence in cleanup alternatives and technologies identified in the alternatives screening/analysis process and in the preferred alternative for this operable unit. The Treatability Study Community Relations activities for Operable Unit 4 will comply with the Community Relations Plan (CRP) -- Remedial Investigation/Feasibility Study and Removal Actions at the U.S. Department of Energy Feed Materials Production Center (now called Fernald Environmental Management Project), 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 per 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. The meetings shall focus on operable unit updates, removal actions, major RI/FS documents, and other appropriate topics. During the July 1991 community meeting, an initial discussion of treatability was held to make the community aware of treatability studies underway.

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

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

Key milestones in treatability studies have been identified through negotiations for the Amended CERCLA Consent Agreement and are included in the schedule in Figure 14-1. These milestones include:

- Submittal of this Work Plan to the DOE and U.S. EPA
- U.S. EPA approval of this Work Plan
- Treatability Testing
- Submittal of Treatability Testing Report

The progress of these key milestones will be reported to the community through the above mentioned presentations and publications.

13.0 MANAGEMENT AND STAFFING

Personnel involved in the management of the overall RI/FS process include: J. R. Craig, DOE Project Director; R. B. Allen, DOE Operable Unit 4 Manager; John Wood, ASI/IT Project Director; D. J. Carr, WEMCO RI/FS Contract Technical Monitor; Susan Rhyne, Acting ASI/IT Operable Unit 4 Manager; and D. A. Nixon, WEMCO Operable Unit 4 Manager.

The principal parties included in the management of the Operable Unit 4 Vitrification Treatability Study are DOE Fernald, WEMCO, ASI/IT, PNL, and Parsons. Personnel involved in the specific management of the Operable Unit 4 Vitrification Treatability Study include: R. B. Allen, DOE Operable Unit 4 Manager; D. A. Nixon, WEMCO Operable Unit 4 Manager; L. A. Heckendorn, WEMCO Operable Unit 4 Program Engineer; C. C. Chapman, PNL, Manager of Operable Unit 4 Vitrification testing; and D. A. Janke, PNL, responsible for WEMCO Operable Unit 4 Vitrification testing and reporting.

14.0 OPERABLE UNIT 4 VITRIFICATION TREATABILITY STUDY SCHEDULE

Figure 14-1 includes the schedule of activities required to complete the Operable Unit 4 treatability studies for vitrification treatability studies for Silos 1, 2, and 3 material. The schedule of activities in Figure 14-1 are part of the RI/FS schedules that were agreed to between the U.S. DOE and the U.S. EPA during negotiations of the Amended CERCLA Consent Agreement.

REM DUR	EARLY START	EARLY FINISH
0	17JUN91A	30JUL91A
0	31JUL91A	31JUL91A
0	1AUG91A	29AUG91A
0	30AUG91A	2OCT91A
0	14SEP91A	10OCT91A
0	3OCT91A	3OCT91A
0	4OCT91A	21OCT91A
0	22OCT91A	11NOV91A
0	12NOV91A	12NOV91A
0	13NOV91A	13NOV91A
0	14NOV91A	30DEC91A
0	31DEC91A	24JAN92A
0	28JAN92A	29JAN92A
0	29JAN92A	29JAN92A
0	30JAN92A	11MAR92A
127	14FEB92A	8SEP92
18	12MAR92A	8APR92
1	9APR92	9APR92
10	9SEP92	22SEP92
21	23SEP92	21OCT92
1	22OCT92	22OCT92
21	23OCT92	20NOV92
15	23NOV92	11DEC92
1	14DEC92	14DEC92
10	15DEC92	28DEC92

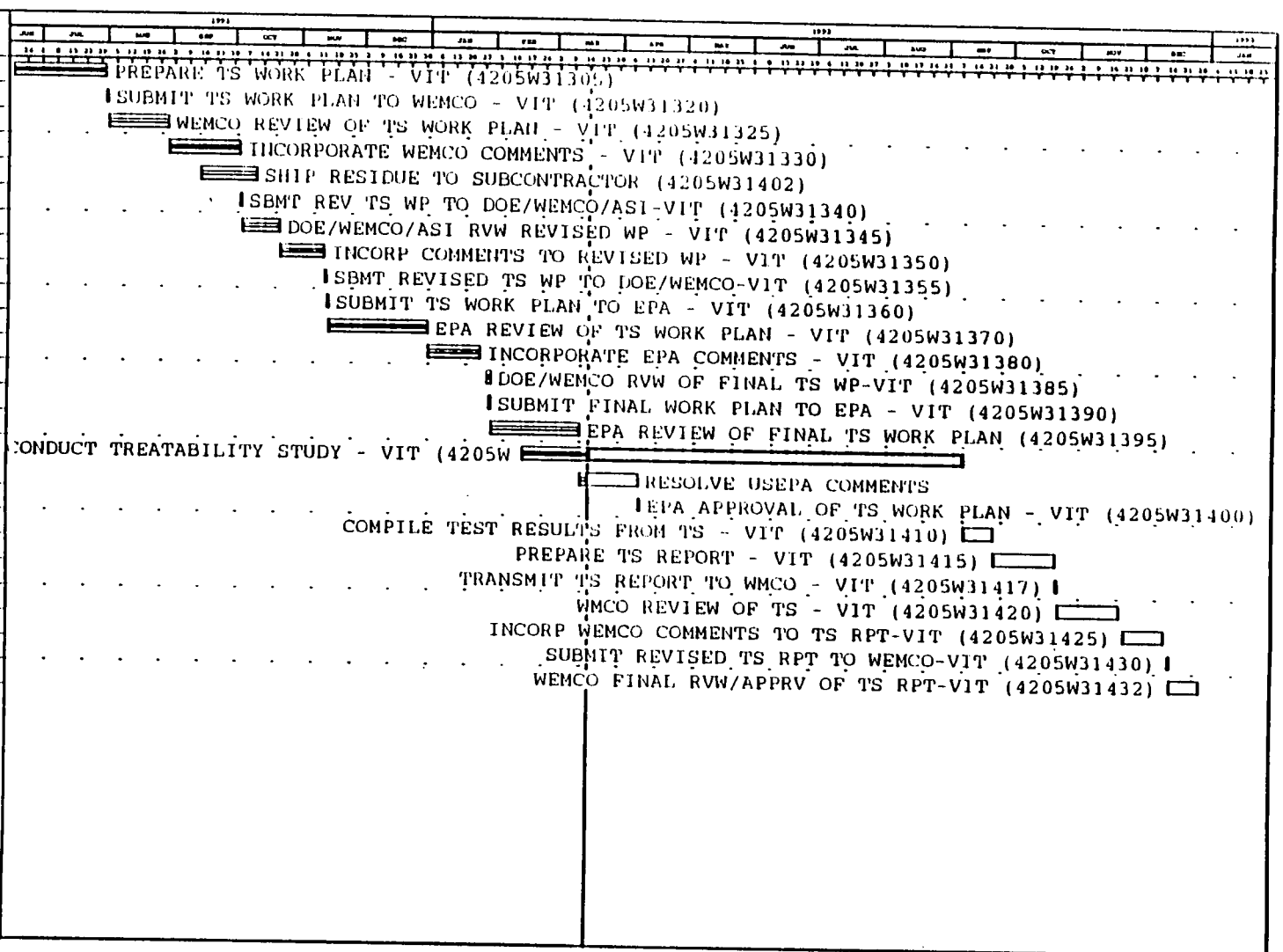

FIGURE 14-1
Operable Unit 4 Vitrification Treatability Study Schedule

REFERENCES

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Chapman, C. C., and Janke, D. S., "Characteristics of Fernald's K-65 Residue Before, During and After Vitrification," February 1991, prepared by Battelle Pacific Northwest Laboratories, Richland Washington for the U.S. Department of Energy.

APPENDIX A

PNL PROJECT QA PLAN

PNL-MA-70 QA PLAN

QA Plan No. WTC-060 Rev. 0
Issue Date
Page 1 of 4

PROJECT IMPACT LEVEL II

TITLE: Vitrification Tests on K-65 Residue Material

SCOPE:

a: Characterize and determine the amount of the off gases evolved during vitrification of the K-65 sample (WBS-02).

b: Define the rate of release of radon and other radioactive products from the solid glass product (WBS-02).

CLIENT: Westinghouse Materials Company of Ohio (WMCO)

AUTHORIZING DOCUMENT: WMCO Purchase Order No. 412337-00; Project No. 16611

QA REQUIREMENT SPECIFICATION(S):

[x] ANSI/ASME NQA-1 as delineated in PNL-MA-70

[ ] Other

Impact Level II WBS element activities shall comply with the applicable requirements, as appropriate for the work being performed, in Parts 1 and 3 of PNL-MA-70. Impact Level III activities shall comply with the GPS Standards located in Part 2 of PNL-MA-70. This QA Plan also identifies client QA requirements, if applicable, and any client imposed exclusions or limitations to PNL procedure requirements. If other quality-related activities are later performed, the appropriate PNL-MA-70 requirements and procedures shall be applied, unless specifically excluded.

CONCURRENCES AND APPROVAL:

CC Chapman — Date 8-2-90
Cognizant Manager (Concurrence)

JW Smith/KR Martin — Date Aug 3 1990
Quality Engineering (Concurrence)

HC Burkholder — Date 8-2-90
Line Manager (Approval)

QA PROGRAM/ORGANIZATION:

The PNL Quality Assurance Program conforms to the requirements of NQA-1 as interpreted by parts 1 and 3 of PNL-MA-70, Quality Assurance Manual. This QA Plan applies only the project Impact Level II WBS Elements, as noted in Exhibit A. WBS elements identified as Impact Level III shall comply with the requirements of PNL-MA-70, part 2. The project organization with key personnel is shown below.

Department Manager: JL McElroy
Project Manager: CC Chapman
Task Leader: DS Janke
Test Plan/Procedure Author: LD Anderson, DS Janke
Test Results Evaluator: CC Chapman, DS Janke, LD Anderson
M&TE Custodian: DS Janke
Records Custodian: DS Janke
Quality Engineer: KR Martin

IMPACT LEVEL:

This QA Plan has been assigned an overall Impact Level of II.

SPECIAL CLIENT REQUIREMENTS:

A. Covered by Part(s) 1 and/or 3 of PNL-MA-70

Client Requirements	Where Covered
- Test Plans shall be planned and executed per PAP-70-1101. Data Sheets incorporated in test plans shall be used to record pertinent test data, and will be traceable to the project LRS.	
- Evaluation of test results shall be reviewed and approved through independent technical reviews per PAP-70-604.	
- Measuring and test equipment shall be calibrated in accordance with PAP-70-1201.	
- All sample material will be treated (tested) and returned to WMCO (per WCO instructions in a vitrified form (except small portions used to determine material composition or to determine the appropriate glass forming additives). Materials will be controlled per PAP-70-601.	

B. Not covered by Part(s) 1 and/or 3 of PNL-MA-70

- Review and Approval of Test Plan by WMCO personnel

C. Client required exclusions or limitations of procedure applicability

- None

OTHER REQUIREMENTS, DIRECTION OR PLANNING:

a) Procurement of an appropriate radon monitor per PAP-70-401.

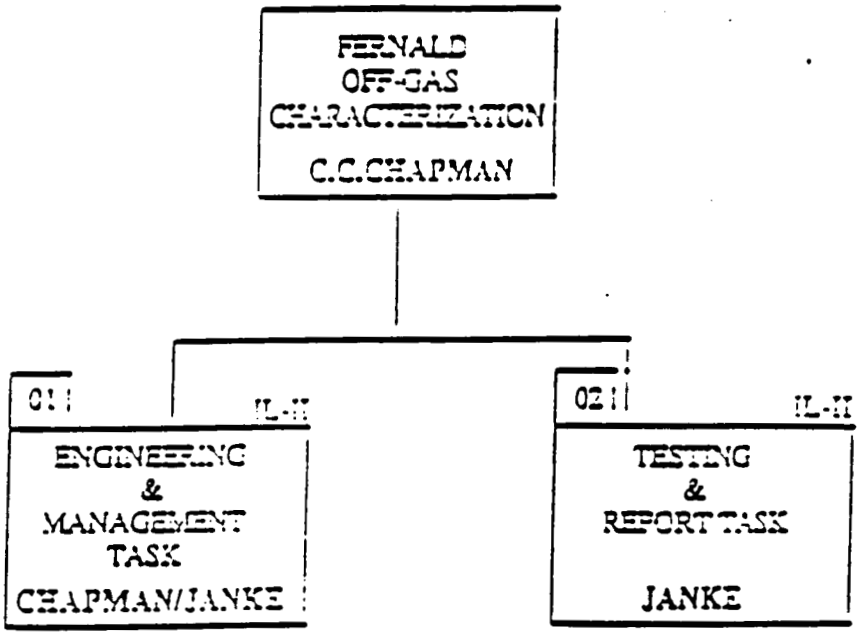
b) There are no known activities which require qualified and certified inspection personnel per PAP-70-203.

c) There are no known controlled processes or special processes to be performed within the scope of this QA plan per PAP-70-901 or PAP-70-902.

d) Records will be indexed and maintained per PAP-70-1701. Records will be designated for single storage and nonpermanent. Records will be maintained by assigned records custodian(s) until turnover to Building 712. Records turnover, including generic records, will be within 90 days of the termination of the project. The cognizant QE will be required to review and concur with the completed project RIDS form. Each record generated will have a activity identifier task number, subtask number, and file classification in the upper right hand corner.

e) Procedures, plans, or instructions will be in accordance with PAP-70-1101 as applicable.

Exhibit A


FERNALD
OFF-GAS
CHARACTERIZATION
C.C.CHAPMAN
01
IL-II
ENGINEERING
&
MANAGEMENT
TASK
CHAPMAN/JANKE
02
IL-II
TESTING
&
REPORT TASK
JANKE

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Analytical Request Form

To: _____ Date: _____ WP/WO Number: _____

Requested by: _____ (name) _____ (signature) _____ (phone) _____ (mail stop)

Analysis Requested:

Identification Numbers:

Material Description:

Special Storage or Handling Requirements: ____ None, ____ Other:

Disposal of Samples: ___ Discard, ___ Return, ___ Other:

Requested Reports/Additional Instructions:

QA Requirements: Impact Level: I II (indicate level)
____ SOW number
____ Other:

Results must be signed and dated by the analyst and reviewer, identifing the measuring and test equipment and the procedure used (including revision).

_____
QP Representative sign/date

QP Representative approval required only for the first ARF in a series for internal work. Approval not required for external work.

To the best of my knowledge, this work was accomplished in accordance with the requirements of this Analytical Request Form:

By: _____ Date: _____
Responsible Analyst or Group Manager

(Return this form or a copy to the requestor).

The report/data furnished has been reviewed and to the best of my knowledge complies with the above request.

By: _____ Date: _____
Requestor

This is a recommended format. Other forms may be used if they provide information which is equivalent to that required by this exhibit.

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Statement of Work

SOW number and revision: (may use the work order or work package number)

Title: type of service, performed by, for what project or organization

Review by: (QP Representative) Date: _____

Concurred by: (Cognizant Scientist/Engineer) Date: _____

Approved by: (Recipient/Person Performing Work) Date: _____

Note: The following is intended as an example only. Each SOW will be written to fit the scope of the work involved.

A. Scope of Work

Include the following:

1. A complete description of the technical work authorized by the SOW including schedule and cost requirements as appropriate. Where necessary, technical requirements shall be specified by reference to drawings, specifications, codes, standards, procedures or instructions.

B. QA Requirements

Include the following:

1. The Impact Level

2. For internal organizations any specific guidance related to the PNL-MA-70 Administrative Procedures.

3. When applicable for other Hanford Contractors, a subsection detailing the applicable QA requirements.

4. A statement that work is to be conducted in accordance with the Service Organizations's activity QA Plan if appropriate. Note, the SOW may act as the QA Plan.

5. A statement allowing Impact Level III scoping studies or preparation activities prior to performing the Impact Level I or II service if necessary to develop methods, procedures, etc.

6. A statement (when applicable) that individual units of work will be (e.g., an Analytical Request Form) transmitted to the recipient. If applicable, identify documentation that will accompany each unit of work.

7. Identification, by name and/or title of personnel who may authorize subsequent changes to the SOW, or related documents.

8. A statement that no subcontracting of the work shall be allowed without prior approval of the cognizant engineer and a QP Representative.

9. A statement requiring QP Representative surveillance prior to or during the performance of the service if appropriate. Identify hold points if appropriate.

C. Reports

1. Define what is to be reported and/or provided and by what date.

2. State that reported results are to be in writing and provided to the individual requesting the work.

3. State that the reported results are to reference the SOW number and are to include the dated signature of the person responsible for the work.

D. Records

1. For work performed by PNL organizations, identify what supporting records are to be submitted to the person requesting the work. Alternately, the organization performing the work may maintain the supporting records in accordance with PAP-70-1701. In either case, specify what records will be maintained and by whom. Examples of supporting records may include:

   - indoctrination and training records
   - calibration records
   - technical procedures
   - raw data including instrument printout
   - other documents required by the applicable PNL-MA-70 Administrative Procedures (unless included as part of the reported results)
   - nonconformance or deficiency reports.

2. For SOWs for other Hanford contractors, identify the records and the retention requirements that apply to the person or organization providing the service. As an alternate, specify the records to be transferred to the requestor at the completion of the work.

APPENDIX B

WASTE TECHNOLOGY CENTER SAFETY PLAN

WASTE TECHNOLOGY CENTER

SAFETY PLAN

March 1991

Approved: _____
J. L. McElroy, Manager
Waste Technology Center

PACIFIC NORTHWEST LABORATORY
RICHLAND, WASHINGTON 99352

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WASTE TECHNOLOGY CENTER
SAFETY PLAN

BACKGROUND

Implementation of the safety aspects of the WTC Environment, Safety Health Plan is described in this plan. Many existing safety requirements incorporated into the plan by reference. Primary emphasis is placed on motivation and attitude, as safety performance can best be improved by frequent and visible management attention to safety.

OBJECTIVES

The objectives of this Safety Plan are to:

- assist in providing and maintaining a safe working environment for Center Staff
- clearly define Center safety policy and the responsibilities of WTC staff concerning safety matters
- motivate all staff members to a high level of safety consciousness
- assist in ensuring good communication between staff members and management on safety matters
- provide an annotated list of sources of safety guidance

CENTER SAFETY POLICY

The Waste Technology Center's safety policy can be summarized in two words: SAFETY FIRST. It is the duty of every staff member to follow established procedures and to continually evaluate potential risks associated with any activity. The individual is not to rely solely on others to define how to perform a job, but must evaluate the directions received and proceed in accordance with safe practices. Staff members will not knowingly engage in unsafe practices--regardless of who instructed them to do so. It is the duty and right of each staff member to know the health aspects (hazards) of the working environment and not to undertake any work that is perceived to be unsafe. Work will be halted until the individual's concern is addressed. The individual is not to restart that work until the situation is evaluated and action is taken.

No individual or manager will take lightly or dismiss the safety concerns of another. No staff member will be ordered to do a task he/she feels might be unsafe. Such intimidation tactics or dismissals of concerns will be reported to the cognizant line manager and project manager or task leader. Resolution will be achieved by working simultaneously through both lines of responsibility, with escalation to higher management levels, if needed, to achieve prompt resolution.

Policies and responsibilities assigned in applicable PNL and WTC specific safety guidance (see Appendix A) will be fulfilled. Because of the wide range of activities within the Center and new activities that are continually being initiated. Flexibility is required within the Center and within individual organizational components. Safety is not "ensured" by adopting unnecessary rules and procedures. The only uniform inviolable policy is--SAFETY FIRST.

Management recognition will be given for demonstrating on the job safety. The safety record, attitude, and awareness of each individual will be evaluated in the yearly Staff Development Review process. Appropriate SDR safety goals will be included in each SDR. Line managers will periodically review the safety performance of their staff during routine and special activities to verify that safe practices are being employed.

The Department Manager will periodically review the safety of each project. If a project has a number of mishaps, it may be caused by excessive emphasis placed on schedules and budgets at the expense of safety. Such a situation shall be corrected immediately.

It is anticipated that this plan will be fully integrated into the WTC Environment, Safety and Health (ES&H) Plan for 1992.

Center Safety Goals

The following six general safety goals are identified for the WTC for CY-1991:

1. Zero Loss Time Accidents

2. Skin contaminations will show a reduction over CY-1990.

3. No reportable occurrences classified as an Emergency or Unusual Occurrence

4. No vehicular accidents

5. First Aid cases will show a reduction over CY-1990.

6. No occurrences or audit deficiencies resulting from the lack of safety training or an appropriate training plan

The following eight specific safety related goals are identified for CY-1991:

1. ES&H training requirements for each WTC staff will be documented by May 14, 1991.

2. ES&H qualifications for each staff position will be documented by April 15, 1991.

3. Scheduled safety meetings will be completed on a timely basis (by December 15, 1991).

4. A management inspection schedule to evaluate and monitor ES&H performance for the facilities operated by the WTC will be completed by April 1, 1991.

5. WTC facility ES&H oversight inspections will be conducted and documented by December 15, 1991.

6. ES&H goals will be included for each Staff in the CY-1991 performance appraisals (by March 31, 1991).

7. By December 31, 1991, complete an evaluation of the performance of all staff in the area of ES&H.

8. Implement an evaluation, control, and decontamination program. (By June 1, 1991)

GENERAL IMPLEMENTATION REQUIREMENTS

Training

Staff assigned to work in laboratory areas will receive training for the specific facility, equipment, and hazards associated with the work they are to perform. Line (Department) Managers are responsible for ensuring that staff receive the necessary safety training. Documentation and status of safety training for staff members is maintained by the PNL Training Coordinator. Duplicate records to provide easy day-to-day reference will be maintained by the Department Manager. Attendance at safety training meetings and should also be documented.

Job Specific Safety Training--

Job-specific safety training requirements apply to both on site and off site research facilities under the control of the WTC. The Department Training Plan will address the job-specific training requirements of assigned staff. Typical areas that the plan might address are: radiation work considerations, facility/equipment SOPs, hazardous chemical handling, crane operation, pollution prevention, and waste minimization.

Training on PNL/WTC Safety Policies/Requirements/Procedures--

Under the direction of the Department Manager, the designated Organizational Safety Representative or other qualified individuals will review general safety policies and procedures with each new staff member. The review will include the information listed under the employee orientation in PNL-MA-43, Section 1, as well as the information contained in the WTC Environment.

Safety and Health Plan and this Plan. This orientation should be accomplished and documented within 1 week of the arrival of new staff. The Hazard Materials Custodian will, on request, provide an orientation on the Chemical and Waste Management Plan, Waste Minimization and Pollution Prevention Plan, and PNL-MA-30, Facility Operational Controls, and Industrial Hygiene Plan.

Emergency Preparedness Training--

Every year and upon initial hire/building reassignment, each staff member should obtain the latest copy of his/her assigned building "Procedures for Emergencies." Annual retraining on the building emergency procedures will, at a minimum, address emergency alarms, procedures, staging areas, and location of fire alarm pull boxes and fire extinguishers. For WTC components occupying space in facilities managed by another contractor or DOE, WTC staff will comply with the requirements of the respective building safety plan/emergency procedures regarding training relating to emergency response.

Vehicle and Office Safety Training--

Vehicle operations, office safety and housekeeping will be periodically reviewed with all staff members.

Safety Meetings--

Documented safety meetings for all WTC staff will be held periodically to carry out appropriate safety training. The minimum frequency of safety meetings for each WTC organizational component is defined in Appendix C. Safety meetings may be part of a periodic organizational staff meeting.

Occurrence Reporting and Investigating

Prompt and accurate reporting of incidents is the basis for early resolution and recovery. After first attempting to stabilize and control any off-normal event, the WTC staff should then contact the immediate line manager. (Call the single point of contact (SPC) on 375-2400 if the line manager is not available.) The line manager will take appropriate additional action. Investigation and reporting shall be in accordance with the requirements of PNL-MA-7, Off-Normal Event Reporting System. Temporary and permanent corrective action for off-normal events will be developed by WTC line management in consultation with appropriate support organizations.

Inspections

Department Managers should inspect all offices and assigned work areas on a periodic basis. Appendix C provides a general schedule of safety related inspections.

SPECIFIC IMPLEMENTATION RESPONSIBILITIES

Building upon the above general guidelines, the roles of WTC management and staff in implementing the WTC Safety Plan are defined below. Appendix C provides a matrix outlining Center management responsibility for safety inspection and safety meetings.

Center Manager:

The WTC Manager has overall responsibility for implementation of all safety programs within the Center, for proper execution of all safety assignments delegated to others within the Center, and for coordinating multi-center facility and ONE reporting activities in the 324 Building. This responsibility is primarily fulfilled by specific delegation, together with personal involvement in evaluating the effectiveness of the Center safety program. Assuring that all staff members achieve a high level of safety awareness and performance is a major area of concern to the Center Manager. Specific responsibilities include:

- Selectively participates in WTC-wide safety inspections including a quarterly audit of compliance to the 324 Building Operational Safety Requirements (OSRs).
- Maintains an open door policy for safety concern and ensures prompt corrective action in response to these concerns, inspections or occurrence reports.
- Ensures that resources are available to Department Managers to implement necessary safety measures.
- Serves on the Center Safety Committee. Ensures that concerns expressed by the committee are promptly addressed.
- Serves as the Lead Facility Manager for the 324 Building and implements provisions of ACT Now Directive 90-7 pertaining to reporting of Off-Normal Events.

Operations Manager:

The Operations Manager has oversight responsibility to the Center Manager for implementation of the ES&H program. Specific responsibilities include:

- Assists in conducting safety inspections of Center facilities and coordinates Center Manager involvement in planned inspections.
- Chairs the WTC Safety Committee. Ensures that safety suggestions/concerns are brought up for discussion in committee meetings.
- Assists, on request, departments with their training plans and safety meetings agendas.
- Provides support to WTC staff thorough the Environmental Compliance Manager, Industrial Hygiene Specialist, NEPA Representative, and Hazardous Material Custodian for chemical and waste management, waste minimization, industrial hygiene, and OSHA compliance activities.
- Reviews all Center staff radiation exposures
- Acts by assignment as a 324 Building Facility Manager.
- Maintains an open door policy concerning safety issues.

Department Manager:

Department Managers are responsible for duties assigned 1) through this Plan, 2) through guidance documents listed in Appendix A (especially PNL-MA-42 and PNL-MA-43), and 3) by the Center Manager. As with the Center Manager, many of these responsibilities are fulfilled through delegation supplemented by significant personal involvement. Specific responsibilities include:

- Reviews and approves a Preliminary Safety Review and Risk Assessment for each project.
- Reviews and approves the Project Management Plan (PMP) or equivalent document for each project and assures that any unusual safety issues are appropriately addressed.
- Ensures that appropriate safety documentation, (e.g., Safety Analysis Report (SAR), Operational Readiness Plan (ORP), Safety Evaluation Document (SED), etc., is in place before beginning work and ensures that all work is carried out in compliance with these requirements.
- Ensures that Laboratory Safety has been made aware of any proposed project that involves a significant safety issue.
- Acts by assignment as a Facility Manager for the 324 Building.
- Interfaces with Building Manager concerning significant changes in programmatic requirements that may impact the building emergency procedures or have significant safety implications to the facility, occupants, equipment, or environment.
- Approves safety related procedures and other operational documents as required.
- Maintains an open-door policy for safety issues and resolves safety issues promptly.
- Reports safety related inspection findings to the WTC Compliance Tracking System. Sets specific schedules for resolving findings and ensures prompt follow up action.
- Ensures that ONE action is timely and thorough and communicates the results of safety audits/inspections and ONEs to the staff.
- Ensures that a Laboratory Manager and Monitor are assigned to each work area.
- Ensures that planned organizational safety meetings are held.
- Visits all department-responsible facilities on site at least bimonthly to verify that safe practices are being employed and to identify and evaluate hazards in the work place. Assures that staff are informed about all hazards associated with their activities.
- Conducts, with the Building Manager, quarterly, documented safety inspections of facilities occupied by the department.
- Appoints a Departmental Organizational Safety Representative from D7W10, D7W20, D7W30.
- Ensures that staff exposure to ionizing radiation and nonradiological hazards is maintained as low as reasonably achievable (ALARA) and that appropriate dosimetry is being used. Assures that the ALARA concept is covered at safety meetings at least annually (D7W20 and D7W30).
- Maintains a current Training Plan addressing the safety training requirements and training status for all assigned staff and assures that all staff receive the appropriate training to perform the assigned work.

- Ensures that new center staff and staff assigned from other organizations receive timely and appropriate safety orientation.

The Department Manager has shutdown authority for operations and activities that pose immediate threat to safe operations.

Group Leader

Group Leader safety responsibilities are focussed on day-to-day operations. Specific responsibilities include:

- Conducts frequent walk-through inspections of work areas.
- Ensures that work is carried out in compliance with all procedures Operational Safety Requirements or other limits and controls necessary to assure safe operations.
- Maintains an open-door policy for safety issues.
- Identifies and evaluates hazards in the work place. Ensure that staff are informed about hazards associated with these tasks.
- Confirms that assigned operating space/space facilities are appropriately posted with emergency information.
- Confirms that staff assigned to work on the project have the required training for the job.
- Confirms that all safety related procedures are approved and posted. where appropriate, before initiation of the work.

The Group Leader has shutdown authority for operations and activities that pose immediate threat to safe operations.

Project Manager or Task Leader

A successful project requires that the Project Manager be concerned about the safety of those working on the project. This requires care in up front planning; and during the project. day-to-day attention to safety in the work place. The Project Manager should know what the safety related issues are with respect to the project and what has and is being done to address them. Specific responsibilities include the following:

- Conducts frequent walk-through inspections of work areas.
- Prepares the preliminary safety review and risk assessment documents for a project and informs Laboratory Safety if the project will generate solid. gaseous. or liquid effluent.
- Prepares and obtains the necessary approvals for safe operating procedures to support the project.
- Ensures that the PMP describes all significant safety issues in the project.
- Identifies job-specific training required for the work and assure that it is complete and properly documented before initiating work.
- Confirms that all physical controls are in place prior to initiation of work.
- Initiates Operational Readiness Reviews (ORR's) if required.
- Resolves promptly safety concerns expressed by project staff.

- Preplans for project safety, including such items as: waste disposal during and after the project; adequacy of facilities to safely house the project (ventilation, fire protection, sewers, etc.); and personnel exposure where hazardous materials are involved.
- Regularly monitors project activities to ensure that all safety issues are addressed and procedures and requirements are complied with.
- Maintains an open door policy for safety issues.

The Project Manager or Task Leader has shutdown authority for operations and activities that pose immediate threat to continued safe operations.

Laboratory Manager

A laboratory manager is designated for each lab. This individual is typically a line manager and is responsible for ensuring that the laboratory/facility has a safe working environment and that those working in the facility have addressed all safety issues related to the work to be performed. The Laboratory Manager delegates day-to-day operating responsibility and authority to the Laboratory Monitor. See Appendix B for a list of assigned Laboratory Managers and Monitors. Specific Laboratory Manager responsibilities are as follows:

- Coordinates external audits and surveillances of the laboratory.
- Ensures that any deficiencies noted during safety audits are addressed promptly and correctly.
- Ensures that the laboratory has a qualified Laboratory Monitor.
- Exercises shutdown authority for the laboratory if unsafe conditions are noted.
- Ensures issues have been satisfactorily resolved before authorizing start up of a laboratory that has been shutdown for safety reasons.

The Laboratory Manager has shutdown authority for operations and activities within the laboratory that pose immediate threat to the laboratory's continued safe operation and to its occupants.

Laboratory Monitor

The Laboratory Monitor is assigned by the Department Manager and will be familiar with the laboratory equipment, ongoing processes, and utilities that are available in the laboratory. The assigned Laboratory Monitor, acting under the direction of the Laboratory Manager, has the responsibility and authority to ensure safety within a specific laboratory or work area. Duties and responsibilities of the Laboratory Monitor are as follows:

- Takes the lead, consistent with Hazardous Material Custodian (HMC) guidance, to maintain a current inventory of the chemicals and other materials under their cognizance.
- Posts and maintains current emergency notification listings by the main entry door of the laboratory consistent with PNL-wide guidelines.
- Acts as the principal contact for proposed or ongoing laboratory work and coordinates facility and maintenance activities. Ensures that new

work to be introduced into the laboratory or work area is consistant with the intended use of the work area.

- Resolves with responsible staff any deficiencies noted by safety and housekeeping inspections and ensures issues have been satisfactorily resolved before authorizing start up of a laboratory that has been shut down for safety reasons.
- Is familiar with any job specific training required for work in the laboratory and ensures that all staff working with equipment, chemicals, materials, and generated waste in the laboratory have received the necessary job-specific training.
- Ensures that all staff assigned to work in the laboratory maintain their work area in a safe and orderly manner.
- Monitors work and ensures that it is performed consistant with procedures and requirements of the laboratory.
- Coordinates audits of assigned space.

The Laboratory Monitor has shutdown authority for operations and activities within the laboratory that pose immediate threat to the laboratory's continued safe operation and to its occupants and also the authority to obtain assistance in housekeeping.

Individual Contributor

The attitude and commitment of the individual to safety is the crucial element to assure that activities are performed safely. The individual contributor is expected to take the initiative in ensuring that he/she is prepared to undertake work that involves a hazard and that a continued attention to safe practices and procedures is maintained throughout the project. Specific responsibilities include the following:

- Participates in the preparation of safety documentation, procedures, etc.
- Completes job specific safety training before initiating work.
- Refers any safety inquiries from the public or news media to Press Relations and WTC line management.
- Maintains own exposure to radiological and nonradiological hazards to levels consistent with ALARA.
- Is familiar with the PMP and performs assigned work safely and responsibly according to applicable limits, PNL Manuals, SOPs, JHBs (or JSAs), RWPs, CSSs, etc.
- Observes the local safety program when visiting or working off site.
- Informs the immediate line manager and project manager or task leader about any safety concerns.
- Provides safety guidance for, and reviews safety performance of less experienced personnel working near you.
- Ensures good housekeeping in the work area and that equipment is maintained and properly identified.
- Reports any off-normal events to his/her immediate line manager. For emergencies or where the line manager is not immediately available, contacts 375-2400.

Organizational Safety Representative:

Organizational Safety Representatives are assigned from and represent the following WTC organizational components:

- D7W10, 11, 12, & 13. (Represented by one individual)
- D7W20, D7W30.

The specific duties and responsibilities of the Organizational Safety Representative are as follows:

- Represents WTC organizational components on the WTC Safety Committee.
- Keeps informed of current safety morale, awareness and implementation/concerns within organization(s) represented.
- Provides feedback to the Department Manager and staff regarding safety issues.
- Assists management on inspections and corrective actions.
- Under the direction of the Department Manager, provides a safety orientation to all new center staff.

Center Safety Committee:

The Center Safety Committee provides a forum to address specific safety issues. It meets approximately every 2 months to discuss grassroots-level safety morale, safety awareness, and specific safety concerns within the Center. The Committee is chartered to facilitate two-way communications about safety throughout the Center.

The roles of the Environmental Compliance Manager, Hazardous Materials Custodian, and Industrial Hygiene Representative are described in the Waste Technology Center Chemical and Waste Management Plan.

PRINCIPAL SAFETY INTERFACES

The Facilities Management, Technical Services, and Laboratory Safety Departments all provide resources necessary to support line management in carrying out their safety responsibilities. These resources provide information on safety criteria, training methods, and inspection techniques. They are also charged with performing safety appraisals and audits and for follow-up to ensure that corrective actions are completed.

The Laboratory Safety Department (see Appendix D) is responsible for 1) assisting line management by serving as a technical safety resource and 2) conducting independent safety reviews and audits. These responsibilities include:

- Accident prevention (formal audits and appraisals, walk-through inspections, exercising "Stop Work" authority).
- General safety education and counseling (orientation for new staff members; conveying information on standards, requirements and

regulations; and conveying information on general safety and "lessons learned" from off-normal events either within or outside PNL).

- Occurrence reporting and investigation (determining classification of accidents, managing the Unusual Occurrence Reporting system, serving as technical experts during investigations).

The Building Manager is an immediate resource in maintaining safe operations. A key responsibility of the Building Managers (Facilities Management Department) is to identify potential safety problems through daily monitoring of activities and to communicate areas of concern to line management. Routine housekeeping functions, status change in experiments under way in the assigned facilities, and the start up of new operations are all monitored by the Building Managers.

Building Managers function as extensions of line management to ensure management awareness of any potential impacts on the safety of the facility. Their principal safety responsibility is to report directly to line management any unusual circumstances that might relate to the safety of that facility. Act Now Directive 90-2 defines the interaction of line and facility management in operation of building facilities and equipment.

The Laboratory Preparedness function within the Technical Services Department provides emergency planning, operational readiness review (ORR) management, and event management support; coordinates the Safety Review Council activity; and coordinates major accident investigations.

APPENDIX A

SOURCES OF SAFETY GUIDANCE

PNL Environment, Safety and Health Plan Issued by W. R. Wiley
Provides overall guidance for safety within PNL, stressing line management responsibility.

Management Guide 11.2 Safety, Summarizes PNL policies and responsibilities in assuring a safe working environment.

PNL-MA-6 Radiation Protection. Establishes basic radiation protection standards applicable to all PNL work with radioactive materials or radiation-generating devices. Designed to minimize radiation exposures of personnel and releases of radioactive material to the environment.

PNL-MA-7 Off-Normal Event Reporting System. Provides guidelines for reporting of all off-normal events, including unusual occurrences. Describes overall system, notifications, investigation, reporting, and recovery.

PNL-MA-8 Waste Management and Environmental Compliance. Presents procedures and requirements related to the handling and storage of radioactive and/or nonradioactive hazardous waste materials.

PNL-MA-25 Criticality Safety. Describes requirements for preventing accidental criticality in the handling, storage, and use of fissionable materials.

PNL-MA-42 Manager's Guide to Safety. Provides a concise summary of the information managers need to know or be aware of in establishing and maintaining safety programs. In addition, the "Guide" contains a list of the primary contacts in Laboratory Safety who can answer safety-related questions.

PNL-MA-43 Health and Safety Management. Gives guidance for industrial safety within PNL. Includes 29 chapters an Appendices on a variety of Safety related topics.

PNL-MA-50 Facility Operational Controls. Provides guidelines for integrating PNL facility operations and the individual operating occupant groups to ensure that: 1) the individual operations are each conducted in an effective, safe, secure and environmentally acceptable manner; 2) several individual operations are mutually compatible; 3) the facility systems (e.g., exhaust ventilation systems) are designed and operated to provide the necessary capacity and capability to support the needs of the individual operations and ensure the safe, secure, and environmentally acceptable operation of the combined operations. Defines

APPENDIX B

WTC STAFF ASSIGNMENTS FOR CHEMICAL AND WASTE MANAGEMENT

324 Building/Facility Common Space - WTC Center Manager -- J. L. McElroy
Environmental Compliance Manager -- C. M. Andersen
324 Building/Facility - HAZARDOUS MATERIAL CUSTODIAN -- C. M. Andersen

SPACE	LABORATORY MONITOR	LABORATORY MANAGER
EDL-101	Tom Brouns	Harry Burkholder
EDL-102	Chris Chapman	Harry Burkholder
Module 1.	Dan Janke	Harry Burkholder
2, 3	Dan Janke	Harry Burkholder
4.	Tom Brouns	Harry Burkholder
5.	Bill Heath	Harry Burkholder
6.	Matt Cooper	Harry Burkholder
7, 8	Joe Perez	Harry Burkholder
11	Dan Janke	Harry Burkholder
12, 14	Greg Whyatt	Chuck Allen
13	Dan Janke	Harry Burkholder
15, 16, 17, 18	Joe Perez	Harry Burkholder
Tank Pit	Joe Perez	Harry Burkholder
Lab 115	Tom Brouns	Harry Burkholder
Lab 207/Lab 208	Tom Powell	Harry Burkholder
Lab 210	Mike Elliott	Harry Burkholder
Lab 212	Greg Whyatt	Chuck Allen
Room 145, 147	Jim Jarrett	Chuck Allen
Room 146	Dan Janke	Harry Burkholder
Room 18	Jim Jarrett	Chuck Allen
Room 308A	Cheryl Thornhill	Chuck Allen
Chem Makeup Room	Jeff Surma	Chuck Allen
Head Tank Room	Jeff Surma	Chuck Allen
371/372 Warehouses	Cameron Andersen	Don Knowlton
324 Building Yard	Mike Puescher	Lynn Ebernardt
High-Bay	Gary Ketner	Chuck Allen
Cask Handling	Jim Jarrett	Chuck Allen
Truck Lock	Jim Jarrett	Chuck Allen
324 Bldg. North LLW Storage	Jim Jarrett	Chuck Allen
ISV Site	Jim Jeffs/Tom Powell	Harry Burkholder
D Cell	Cheryl Thornhill	Chuck Allen
A, B Cells	Jim Jarrett	Chuck Allen
C Cell	Jeff Surma	Chuck Allen
Sky Park	Cameron Andersen	Don Knowlton

Updated 3/22/91:Lab-Mntr.Asn

APPENDIX C

MATRIX OF SPECIFIC WTC INSPECTION AND SAFETY MEETING RESPONSIBILITIES

Org. Code	Documented Facility Inspections Freq per yr. Lab.*	Office	Safety Meetings Freq per yr.	Responsible Manager
7W00	2	1	1	JL McElroy
7W10		1	1	GW McNair
7W20	4	1	4	CR Allen
7W22	4	1	4	HC Burkholder

* These inspections are conducted with the Building Manager. Not included here are bimonthly walk through inspections of all experimental work areas by the Department Manager.

Primary Contacts in Laboratory Safety

Glenn Hoenes, Manager, 376-1137

3006

Occupational & Radiological Safety
Ben Wright, 375-1634

Fire Protection
Andy Minster, 376-4938

Occupational Safety
Bob Gough, 375-1886

Industrial Hygiene
Monty Rosson, 376-3037

Radiological Engineering

Safety & Risk Assessment
Donna Lunn, 376-1331

Hazardous Waste Site Health & Safety
Jim Moran, 375-3353

Injury or Illness Investigation
Pam Myers, 375-0918

ALARA
Cheryl Colen, 375-0[illegible]

Criticality Safety
Lee Davenport, 376-3333

Design Review, Mod Permits, & Nuclear Processes
Russ Richmond, 376-1887

Fire Extinguishers
Doug Clark, 375-2853

Explosives & Flammable Liquids
Scott Allen, 375-1042

Motor Vehicle Safety
Doug Wright, 375-4792

MSDSs & Chemical Hygiene
Rick Johnson, 375-1535

Rad. Air Emissions
Monte Sula, 375-0605

Respirator Protection
[illegible]

Waste Management & Environmental Compliance
Bill Blackburn, 376-[illegible]

NEPA Reviews
David Gunnison, 376-3732

Hazardous Material Shipping/Transportation
John Taylor, 376-1736

Regulatory Analysis
Harold Tilden, 376-0499

Oils, PCBs, & USTs
Brian Day, 376-3835

Compliance Inspections
Mike McCoy, 376-1483

Environmental Permits
Harold Tilden, 376-0499

Treatability Permits
Mike McCoy, 376-1483

Spill Reporting
Dennis Kluger, 376-4088

Waste Minimization
Eric Harris, 376-3631

Mixed Waste
Kevin Selby, 376-7[illegible]

TRU Waste Disposal
Kyle Webster, 376-1587

Low-Level Radioactive Waste and Radioactive Liquid Waste Disposal
Bruce Killian, 376-3158

Liquid or Airborne Releases
Dennis Kluger, 376-4088

RCRA & Hazardous Waste Disposal
Glenn Thornton, 375-7688

Waste Designation
Gregg Barter-Bailey, 375-4[illegible]

Radiation Protection
Dave Rigby, 376-3087

Field Dosimetry Services
Mary Ann Hennyer, 376-3533

Medical Scheduling
Stacy Berg, 376-3645

Radiation Instrumentation
Andy Milleham, 376-0942

RPT Supervisions

300 Area
Ron Schmoke, 376-4703

308, 324, 325 Bldgs.
Jerry Allen, 376-3502

331 Bldg., 3000, & Outer Areas
Joe Hunsperm, 376-3155

Safety Education
Jeane Hobbs, 376-1631

Female Rad Worker Concerns
Jean Buck, 376-3771

Variances
Jerry Weston, 376-1929

Emergency Assistance
375-2400

Alarm Review
375-2343

Employee Concerns Hotline
375-3999

APPENDIX C

SILOS 1, 2 AND 3
RADIOLOGICAL AND CHEMICAL CONSTITUENTS

TABLE C-1

CHARACTERISTICS OF THE K-65 RESIDUES STORED AT THE FEMP

	Silos 1 and 2				Silo 3
Characteristics	Vitro (1952)	Litz[a] (1974)	NLO[a] (1980)	Gill (1988)	DOE (1987)
Physical					
Dry Weight (kg)	1.59×10^6	--	8.79×10^6	--	--
Volume (m^3)	3,155	--	5,522	--	3,902
Density (kg/m^3)	1,179	--	--	--	--
Water content (%)	30	--	--	--	--
Radiological	(ppm)	(ppm)	(ppm)	(ppm)	(kg)
Raduim	0.3	0.28-0.36	0.2	0.13-0.21	0.015[b]
Uranium	2,110	1,800-3,200	600	1,400-1,800	18,000
Total thorium	--	--	--	301-322	--
Chemical					
Carbonates + Sulfates (%)	20				
Quartz (%)	25				
Muscovite clay (%)	60				

[a]As reported by Dettore et al., 1981.
[b]Assumes all radium in K-65 residues is Ra-226 with specific activity of 0.988 Ci/g.
Note: Data validation is currently in progress.

TABLE C-2

RADIONUCLIDE CONCENTRATIONS IN THE SILOS
(1989 Sampling Program)

SILO 1

Nuclide (pCi/g)	S1NE1A	S1NE1B	S1NE1C	S1SE1	S1SE2	S1SW1	S1N
Th-228	ND	ND	ND	ND	ND	ND	ND
Th-230	21,412	39,693	30,751	10,569	20,848	40,818	43,7
Th-232	ND	ND	ND	ND	ND	ND	766
Ra-226	108,100	192,600	166,400	116,800	89,280	181,200	163,3
Ra-228	ND	ND	ND	ND	ND	ND	ND
Pb-210	181,100	83,110	77,460	71,920	48,980	69,480	54,35
U-234	815	326	622	663	814	594	897
U-235/236	ND	ND	ND	ND	56	ND	50
U-238	920	398	610	545	758	532	687
U-Total (ppm)	2753	1189	1831	1633	2280	1602	2066

SILO 2

Nuclide (pCi/g)	S2SW1	S2NW1	S2NE2	S2SW2	S2NE1	S2NW2
Th-228	ND	ND	ND	411	ND	638
Th-230	31,825	32,784	8365	29,716	40,124	25,391
Th-232	ND	ND	ND	851	ND	ND
Ra-226	145,300	61,780	657	104,900	65,520	68,310
Ra-228	ND	ND	ND	ND	ND	ND
Pb-210	141,900	145,200	87,930	77,940	150,700	399,200
U-234	859	1107	974	121	848	1404
U-235/236	ND	74	47	ND	36	70
U-238	661	1069	874	46	814	1240
U-Total (ppm)	1972	3210	2620	137	2437	3717

ND = Not Detected
Note: Data validation is currently in progress.

TABLE C-3

RADIONUCLIDE CONCENTRATIONS IN THE SILOS
(1989 Sampling Program)

SILO 3

Nuclide (pCi/g)	# 21	# 22	# 23	# 24	# 25	# 26
Ac-227	523	416	234	1363	534	706
Pa-231	521	401	266	NA	556	889
Th-228	907	ND	554	ND	459	859
Th-230	41,911	33,881	21,010	71,650	40,968	41,555
Th-232	1451	ND	815	911	411	ND
Ra-224	453	451	64	213	295	335
Ra-226	2589	2192	467	6435	3073	1862
Ra-228	525	559	82	ND	392	441
Pb-210	2437	2221	454	6427	2493	1910
U-234	1935	1618	348	1524	1467	1910
U-235/236	152	117	ND	127	54	76
U-238	2043	1649	320	1600	1392	1860
U-Total (ppm)	4040	4305	738	2595	3064	4554

SILO 3

Nuclide (pCi/g)	# 27	# 28	# 29	# 30	# 33
Ac-227	421	412	443	773	566
Pa-231	458	NA	564	931	431
Th-228	ND	996	537	ND	949
Th-230	53,227	63,649	61,190	68,759	65,488
Th-232	ND	755	672	581	672
Ra-224	370	106	137	449	313
Ra-226	1518	3702	4169	2240	4451
Ra-228	325	ND	117	360	415
Pb-210	1084	2589	3553	1942	3674
U-234	1317	1052	1843	1643	1600
U-235/236	80	42	158	75	118
U-238	1243	994	1951	1574	1878
U-Total	2740	1463	1114	4050	3854

NA = Not Analyzed
ND = Not Detected

Note: Data validation is currently in progress.

TABLE C-4

ORGANICS CONCENTRATIONS IN THE SILOS

CONTAMINANT	Silo 1	Silo 2	Silo 3
VOLATILE ORGANICS ANALYSIS DATA (ppb)			
Methylene Chloride	840 - 4100	1100 - 6300	1000 - 2800
Acetone	140 - 5300	ND - 1600	3400 - 12000
Chloroform	480 - 1500	660- 1300	560 - 810
2-Butanone	7100 - 21000	7800 - 15000	9700 - 16000
4-Methyl-2-Pentanone	ND - 1400	ND - 2700	ND
Toluene	ND - 430	ND - 250	180 - 6800
Trichloroethane	ND	ND - 120	ND
Chloromethane	ND	ND	ND - 140
Styrene	ND - 350	ND - 200	ND
Total Xylenes	ND	ND - 200	ND
SEMIVOLATILE ORGANICS ANALYSIS DATA (ppb)			
Bis(2-Ethylhexyl)Phthalate	93 - 6000	ND - 560	ND - 40
Di-n-Octyl Phthalate	ND - 820	ND	ND
PESTICIDE ORGANICS ANALYSIS DATA (ppb)			
Aroclor-1248	ND - 8000	ND	ND
Aroclor-1254	1100 - 14000	420 - 6000	ND

ND = Not Detected
Note: Data validation is currently in progress.

TABLE C-5

INORGANICS CONCENTRATIONS IN THE SILOS
(1989 Sampling Program)

Contaminant (ppm)	Silo 1	Silo 2	Silo 3
Aluminum	60.4- 1430	464 - 2570	10800 - 23700
Antimony	ND	ND - 7.2	ND
Arsenic	14.7 - 68.4	57.5 - 1960	532 - 6380
Barium	1970 - 7860	89.2 - 8370	118 - 332
Beryllium	0.88 - 2.8	0.66 - 6.0	10.0 - 39.9
Cadmium	2.1 - 8.0	3.4 - 19.1	21.5 - 204
Calcium	2150 - 5700	2430 - 301000	21300 - 39900
Chromium	21.0 - 165	12.9 - 68.8	139 - 560
Cobalt	349 - 1260	6.2 - 2430	ND - 3520
Copper	122 - 473	ND - 1790	1610 - 7060
Iron	4340 - 75100	4010 - 37800	13900 - 67600
Lead	35800 - 85100	153 - 29800	646 - 4430
Magnesium	1500 - 6020	1520 - 8740	38200 - 80900
Manganese	33.5 - 257	74.2 - 403	2420 - 6500
Mercury	0.23 - 2.8	ND - 2.3	ND - 0.69
Nickel	629 - 2580	14.6 - 2200	1200 - 6170
Potassium	158 - 492	37.8 - 289	1300 - 22800
Selenium	106 - 180	ND - 118	101 - 349
Silver	5.0 - 23.3	ND - 22.8	9.2 - 23.8
Sodium	360 - 13100	226 - 4070	22900 - 51700
Thallium	ND - 0.52	ND - 1.4	3.1 - 73.9
Vanadium	72.2 - 240	21.9 - 214	418 - 4550
Zinc	14.4 - 212	11.2 - 154	301 - 672
Cyanide	0.52 - 4.4	ND - 4.5	ND

ND = Not Detected
Note: Data validation is currently in progress

TABLE C-6

EP TOXIC METALS RANGE OF VALUES FOR K-65 AND METAL OXIDE SILOS
(1989 Sampling Program)

Analyte	Silo 1	Silo 2	Silo 3	Maximum Allowable Concentration
Arsenic (ppm)	ND - 0.484	0.163 - 0.592	ND - 41.5	5.0
Barium (ppm)	0.079 - 14.5	0.095 - 2.62	0.020 - 0.156	100
Cadmium (ppm)	ND - 0.100	0.017 - 0.278	0.108 - 6.32	1.0
Chromium (ppm)	0.020 - 0.964	ND - 1.02	0.336 - 11.9	5.0
Lead (ppm)	0.159 - 904	0.155 - 714	ND - 1.01	5.0
Selenium (ppm)	0.217 - 0.997	0.240 - 1.56	0.92 - 11.7	1.0
Silver (ppm)	ND - 0.121	ND - 0.213	ND - 0.032	5.0
Mercury (ppm)	ND	ND	ND - 0.003	0.2

ND = Not Detected
Note: Data validation is currently in progress.

TABLE C-7

GEOTECHNICAL ANALYTICAL RESULTS
(1989 Sampling Plan)

Sample ID	Color	Water Content (%)	Specific Gravity	Liquid Limit	Plastic Limit	Plasticity Index	200 Sieve (Percent Finer)
S1-NE-1A	Dark Brown	50.7	3.19	55.2	50.0	5.2	72.7
S1-NE-1C	Light Brown	71.5	2.74	70.3	66.6	3.7	71.5
S1-SE-2T	Sandy Brown	31.9	3.37	NP	NP	NP	43.9
S1-Compos.	NA	22.8	2.58	NP	NP	NP	54.5
S2-NW-1A	Brown	25.9	2.87	NP	NP	NP	39.8
S2-NE-2BT	White	21.8	2.59	NP	NP	NP	51.9
S2-SW-1A	Black	73.5	3.11	NP	NP	NP	63.3
S2-Compo	NA	34.2	2.78	NP	NP	NP	38.1
S3-NW-1A	Reddish Brown	7.4	2.35	NP	NP	NP	93.2
S3-NW-1C	Brown	3.7	2.08	NP	NP	NP	93.9
S3-SE-1A	Reddish Brown	10.2	2.58	NP	NP	NP	90.0
S3-SE-1C	Dark Brown	6.3	2.29	NP	NP	NP	92.9
S3-Compo	NA	3.8	2.75	NP	NP	NP	87.8

NA = Not Applicable
NP = Non-Plastic
Note: Data validation is in progress

APPENDIX D

VITRIFICATION TREATABILITY STUDY PROCEDURES AND METHODS

PNL PROCEDURES

Fernald OU-4 Vitrification Treatability Study

General Procedures

1. Representative samples will be obtained by standard laboratory techniques such as coning and quartering or rolling and quartering.

2. Sample size will be sufficient to give the required precision of measurements.

3. Adequacy of sampling method and size will be indicated by the reproducibility of results.

4. All determinations will be done in triplicate.

5. All measurements for determination of physical properties will be done on a laboratory balance accurate to ±0.001g (all weight measurements to be within ±0.01g).

Procedure for Determination of Moisture Content

1. Measure and record the mass of the sample to be dried.
2. Cover and place in the drying oven at 105 °C until a constant weight is reached.
3. Measure and record the mass of the dry sample.
4. *% moisture = 100% * (mass initial - mass final)/mass initial*

Procedure for Determination of Bulk Density

For solids:

1. Tare a 25 mL graduated cylinder.
2. Pour the solid into the graduated cylinder, being careful to avoid the formation of air pockets.
3. Tap the sides of the cylinder until no further settling is obvious.
4. Measure and record the volume of the solid material.
5. Measure and record the mass of material added to the cylinder.
6. *bulk density = mass sample/volume of settled solid*

For glass:

The bulk density of the glass is assumed to be equal to the apparent density. This assumption is valid if the glass monolith contains negligible open pore volume. The apparent density determination is described in the following procedure.

Procedure for Determination of Apparent Density

Note: The apparent density will be equal to the true density if the closed pore volume of the material sampled is negligible.

For soils:

1. Tare a 10 mL volumetric flask.

2. Add about 5 g of solid material to the flask and measure and record the mass.

3. Fill with water to the volumetric mark, mixing sufficiently to ensure complete wetting of the soils and elimination of any entrapped air bubbles. Measure and record the mass.

4. Determine the mass of water added by difference and convert to volume.

5. *apparent density = mass sample/(volume of flask - volume water added)*

For glass:

If the glass sample is broken into small pieces, the above method can be used. For large pieces, use the following method. This method measures the buoyant force exerted on the submerged sample. The buoyant force is equal to the weight of water displaced by the sample and the basket. After correcting for the buoyant force due to the basket, the weight of water displace, and thus the sample volume can be determined.

1. Measure and record the mass of the sample to be measured.

2. Place the sample in a basket and immerse the sample completely in a beaker of water which is tared on a balance.

3. Record the mass shown by the balance.

4. Immerse the basket alone to the same point as in step 3 and record the mass shown by the balance.

5. *apparent density = mass sample * density of water/(mass from step 3 - mass from step 4)*

Procedure for Determination of Radon Emanation from Waste and Glass

This procedure is the same for both the untreated and vitrified waste except for the slight preparation required for the untreated sample.

1. Prepare the samples for measurement. The untreated waste will be placed in the test crucible and evenly distributed. The vitrified waste will be measured as is in the test crucible after cooling.

2. Measure the cross sectional area of the crucible at the surface of the material.

3. Place the sample in the airtight can designed for the radon emanation measurements. This can is airtight except for two ports which will allow a controlled flow of air through the can.

4. Measure and record the background reading from the radon monitor.

5. Pull air through the can at a constant flow rate. Measure and record the flow.

6. Monitor the radon concentration until steady state is reached. Record the steady state concentration.

7. *Radon emanation rate ($pCi/m^2 \cdot s$) = Steady state concentration * Flow rate / cross sectional area*

Procedure for Determination of Radon Emanation During Vitrification

The general procedure will be to draw a slipstream from the quenched off-gas, dry the slipstream on a desiccant bed, dilute it with an air bleed, and pass the dry, diluted stream through the radon monitor. Refer to the figure below for the simple flowsheet of the off-gas monitoring.

1. Begin with all flowmeters in the system closed and all pumps off.

2. Place the test crucible and sample in the furnace and seal the furnace.

3. Open the flowmeter on the air inlet to the furnace (F4) to allow air to freely enter.

4. Turn on the radon monitor and measure and record the background reading.

5. Open the flowmeter on the radon monitor outlet stream (F1) and adjust until the desired flow is passing through the radon monitor.

6. Open the flow meter on the air bleed into the radon monitor stream (F2) and adjust until the air bleed is the desired percentage of flow through the monitor.

7. Turn on the vacuum pump in the main off-gas line and adjust the flowmeter in the main off-gas line (F3) until the desired total flow is passing through the furnace as measured at the furnace inlet (F4).

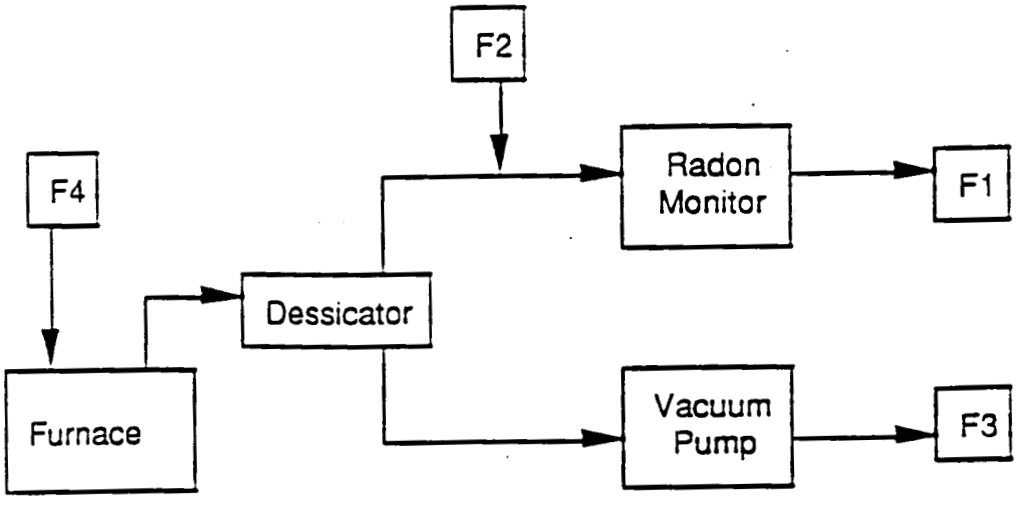
8. Check the flows on all of the lines and make adjustments as necessary to obtain the desired flows.

9. Begin vitrification. Periodically check the flows in each of the streams. Monitor and record the radon concentration.

10. Calculate the radon emanation as a function of time.

*Radon emanation (pCi/min) = Measured concentration * [F1/(F1-F2)] * (F3+F1-F2)*

The first flow term corrects the diluted concentration to the actual concentration in the off-gas, and the second term is the total off-gas flow.



Procedure for Performing Modified TCLP

The attached document describes the procedure (Method 1311) to be used by the laboratory performing the TCLP analyses. A flowsheet/logic diagram for the procedure is given on pages 1311-34 and 1131-35. The analysis will require a 100 g sample of glass. Analysis will be performed for metals only, as no organics remain in the glass after processing. The metals analyzed for will include arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver.

METHOD 1311

TOXICITY CHARACTERISTIC LEACHING PROCEDURE

1.0 SCOPE AND APPLICATION

1.1 The TCLP is designed to determine the mobility of both organic a inorganic analytes present in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individu analytes are not present in the waste, or that they are present but at such lo concentrations that the appropriate regulatory levels could not possibly b exceeded, the TCLP need not be run.

1.3 If an analysis of any one of the liquid fractions of the TCL extract indicates that a regulated compound is present at such hig concentrations that, even after accounting for dilution from the other fraction of the extract, the concentration would be above the regulatory level for tha compound, then the waste is hazardous and it is not necessary to analyze th remaining fractions of the extract.

1.4 If an analysis of extract obtained using a bottle extractor show that the concentration of any regulated volatile analyte exceeds the regulator level for that compound, then the waste is hazardous and extraction using the ZH is not necessary. However, extract from a bottle extractor cannot be used to demonstrate that the concentration of volatile compounds is below the regulator level.

2.0 SUMMARY OF METHOD

2.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the TCLP extract.

2.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A special extractor vessel is used when testing for volatile analytes (see Table 1 for a list of volatile compounds). Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

3.1 Potential interferences that may be encountered during analysis discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus: The agitation apparatus must be capable rotating the extraction vessel in an end-over-end fashion (see Figure 1) 30 ± 2 rpm. Suitable devices known to EPA are identified in Table 2.

4.2 Extraction Vessels

4.2.1 Zero-Headspace Extraction Vessel (ZHE). This device for use only when the waste is being tested for the mobility of volati analytes (i.e., those listed in Table 1). The ZHE (depicted in Figure allows for liquid/solid separation within the device, and effective precludes headspace. This type of vessel allows for initial liquid/sol separation, extraction, and final extract filtration without opening t vessel (see Section 4.3.1). The vessels shall have an internal volume 500-600 mL, and be equipped to accommodate a 90-110 mm filter. The devic contain VITON®[1] O-rings which should be replaced frequently. Suitable ZI devices known to EPA are identified in Table 3.

For the ZHE to be acceptable for use, the piston within the ZI should be able to be moved with approximately 15 psi or less. If it take more pressure to move the piston, the O-rings in the device should b replaced. If this does not solve the problem, the ZHE is unacceptable fo TCLP analyses and the manufacturer should be contacted.

The ZHE should be checked for leaks after every extraction. If th device contains a built-in pressure gauge, pressurize the device t 50 psi, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psi, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

Some ZHEs use gas pressure to actuate the ZHE piston, while others use mechanical pressure (see Table 3). Whereas the volatiles procedure (see Section 7.3) refers to pounds per square inch (psi), for the mechanically actuated piston, the pressure applied is measured in torque-inch-pounds. Refer to the manufacturer's instructions as to the proper conversion.

4.2.2 Bottle Extraction Vessel. When the waste is being evaluated using the nonvolatile extraction, a jar with sufficient capacity

[1] VITON® is a trademark of Du Pont.

to hold the sample and the extraction fluid is needed. Headspace allowed in this vessel.

The extraction bottles may be constructed from various material depending on the analytes to be analyzed and the nature of the waste (s Section 4.3.3). It is recommended that borosilicate glass bottles be us instead of other types of glass, especially when inorganics are concern. Plastic bottles, other than polytetrafluoroethylene, shall n be used if organics are to be investigated. Bottles are available from number of laboratory suppliers. When this type of extraction vessel used, the filtration device discussed in Section 4.3.2 is used for initi liquid/solid separation and final extract filtration.

4.3 Filtration Devices: It is recommended that all filtrations performed in a hood.

4.3.1 Zero-Headspace Extractor Vessel (ZHE): When the waste evaluated for volatiles, the zero-headspace extraction vessel described Section 4.2.1 is used for filtration. The device shall be capable supporting and keeping in place the glass fiber filter and be able withstand the pressure needed to accomplish separation (50 psi).

Note: When it is suspected that the glass fiber filter has been ruptured, in-line glass fiber filter may be used to filter the material within th ZHE.

4.3.2 Filter Holder: When the waste is evaluated for other tha volatile analytes, any filter holder capable of supporting a glass fibe filter and able to withstand the pressure needed to accomplish separatio may be used. Suitable filter holders range from simple vacuum units t relatively complex systems capable of exerting pressures of up to 50 ps or more. The type of filter holder used depends on the properties of th material to be filtered (see Section 4.3.3). These devices shall have minimum internal volume of 300 mL and be equipped to accommodate a minimu filter size of 47 mm (filter holders having an internal capacity of 1.5 or greater, and equipped to accommodate a 142 mm diameter filter, ar recommended). Vacuum filtration can only be used for wastes with lo solids content (<10%) and for highly granular, liquid-containing wastes All other types of wastes should be filtered using positive pressur filtration. Suitable filter holders known to EPA are shown in Table 4.

4.3.3 Materials of Construction: Extraction vessels an filtration devices shall be made of inert materials which will not leac or absorb waste components. Glass, polytetrafluoroethylene (PTFE), o type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components. Devices made of high density polyethylene (HDPE), polypropylene (PP), or polyvinyl chloride (PVC) may be used only when evaluating the mobility of metals. Borosilicate glass bottles are recommended for use over other types of glass bottles, especially when inorganics are analytes of concern.

4.4 Filters: Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to

0.8 µm. or equivalent. Filters known to EPA which meet these specifications ar identified in Table 5. Pre-filters must not be used. When evaluating th mobility of metals, filters shall be acid-washed prior to use by rinsing with 1 nitric acid followed by three consecutive rinses with deionized distilled wate (a minimum of 1 L per rinse is recommended). Glass fiber filters are fragile anc should be handled with care.

4.5 pH Meters: The meter should be accurate to ± 0.05 units at 25°C.

4.6 ZHE Extract Collection Devices: TEDLAR®[2] bags or glass, stainless steel or PTFE gas-tight syringes are used to collect the initial liquid phase and the final extract of the waste when using the ZHE device. The devices listed are recommended for use under the following conditions:

4.6.1 If a waste contains an aqueous liquid phase or if a waste does not contain a significant amount of nonaqueous liquid (i.e., <1% of total waste), the TEDLAR® bag or a 600 mL syringe should be used to collect and combine the initial liquid and solid extract.

4.6.2 If a waste contains a significant amount of nonaqueous liquid in the initial liquid phase (i.e., >1% of total waste), the syringe or the TEDLAR® bag may be used for both the initial solid/liquid separation and the final extract filtration. However, analysts should use one or the other, not both.

4.6.3 If the waste contains no initial liquid phase (is 100% solid) or has no significant solid phase (is 100% liquid), either the TEDLAR® bag or the syringe may be used. If the syringe is used, discard the first 5 mL of liquid expressed from the device. The remaining aliquots are used for analysis.

4.7 ZHE Extraction Fluid Transfer Devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or peristaltic pump, a gas tight syringe, pressure filtration unit (see Section 4.3.2), or other ZHE device).

4.8 Laboratory Balance: Any laboratory balance accurate to within ± 0.01 grams may be used (all weight measurements are to be within ± 0.1 grams).

4.9 Beaker or Erlenmeyer flask, glass, 500 mL.

4.10 Watchglass, appropriate diameter to cover beaker or Erlenmeyer flask.

4.11 Magnetic stirrer.

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[2] TEDLAR® is a registered trademark of Du Pont.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent water. All references to reagent water in this method refer to one of the following, as appropriate:

5.2.1 Inorganic Analytes: Water which is generated by any method which would achieve the performance standards for ASTM Type II water. The analyte(s) of concern must be no higher than the highest of either (1) the detection limit, or (2) five percent of the regulatory level for that analyte, or (3) five percent of the measured concentration in the sample.

5.2.2 Volatile Analytes: Water in which an interferant is not observed at the method detection limit of the compounds of interest. Organic-free water can be generated by passing tap water through a carbon filter bed containing about 1 lb. of activated carbon. A water purification system may be used to generate organic-free deionized water. Organic-free water may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the temperature at 90°C, bubble a contaminant-free inert gas through the water for 1 hour. The analyte(s) of concern must be no higher than the highest of either (1) the detection limit, or (2) five percent of the regulatory level for that analyte, or (3) five percent of the measured concentration in the sample.

5.2.3 Semivolatile Analytes: Water in which an interferant is not observed at the method detection limit of the compounds of interest. Organic-free water can be generated by passing tap water through a carbon filter bed containing about 1 lb. of activated carbon. A water purification system may be used to generate organic-free deionized water. The analyte(s) of concern must be no higher than the highest of either (1) the detection limit, or (2) five percent of the regulatory level for that analyte, or (3) five percent of the measured concentration in the sample.

5.3 Hydrochloric acid (1N), HCl, made from ACS reagent grade.

5.4 Nitric acid (1N), HNO_3, made from ACS reagent grade.

5.5 Sodium hydroxide (1N), NaOH, made from ACS reagent grade.

5.6 Glacial acetic acid, CH_3CH_2OOH, ACS reagent grade.

5.7 Extraction fluid.

5.7.1 Extraction fluid # 1: Add 5.7 mL glacial CH_3CH_2OOH to 500 mL of reagent water (See Section 5.2), add 64.3 mL of 1N NaOH, and

dilute to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05.

5.7.2 Extraction fluid # 2: Dilute 5.7 mL glacial CH_3CH_2OOH with reagent water (See Section 5.2) to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05.

Note: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

5.8 Analytical standards shall be prepared according to the appropriate analytical method.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples shall be collected using an appropriate sampling plan.

6.2 The TCLP may place requirements on the minimal size of the field sample, depending upon the physical state or states of the waste and the analytes of concern. An aliquot is needed for preliminary evaluation of which extraction fluid is to be used for the nonvolatile analyte extraction procedure. Another aliquot may be needed to actually conduct the nonvolatile extraction (see Section 1.4 concerning the use of this extract for volatile organics). If volatile organics are of concern, another aliquot may be needed. Quality control measures may require additional aliquots. Further, it is always wise to collect more sample just in case something goes wrong with the initial attempt to conduct the test.

6.3 Preservatives shall not be added to samples before extraction.

6.4 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

6.5 When the waste is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon-lined septum capped vials and stored at 4°C. Samples should be opened only immediately prior to extraction).

6.6 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH < 2, unless precipitation occurs (see Section 7.2.14 if precipitation occurs). Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. See Section 8.0 (QA requirements) for acceptable sample and extract holding times.

7.0 PROCEDURE

7.1 Preliminary Evaluations

Perform preliminary TCLP evaluations on a minimum 100 gram aliquot of waste. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of the percent solids (Section 7.1.1); (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration (Section 7.1.2); (3) determination of whether the solid portion of the waste requires particle size reduction (Section 7.1.3); and (4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste (Section 7.1.4).

7.1.1 Preliminary determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

7.1.1.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids) proceed to Section 7.1.3.

7.1.1.2 If the sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device described in Section 4.3.2 and is outlined in Sections 7.1.1.3 through 7.1.1.9.

7.1.1.3 Pre-weigh the filter and the container that will receive the filtrate.

7.1.1.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

7.1.1.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight.

7.1.1.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.1.7 Quantitatively transfer the waste sample to the filter holder (liquid and solid phases). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature then allow the sample to warm up to room temperature in the device before filtering.

Note: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus,

determine the weight of this residue and subtract it from the sample weight determined in Section 7.1.1.5 to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.1.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

Note: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Section 7.1.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.1.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Section 7.1.1.3) from the total weight of the filtrate-filled container. Determine the weight of the solid phase of the waste sample by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in Section 7.1.1.5 or 7.1.1.7.

Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid (Section 7.1.1.9)}}{\text{Total weight of waste (Section 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2 If the percent solids determined in Section 7.1.1.9 is equal to or greater than 0.5%, then proceed either to Section 7.1.3 to determine whether the solid material requires particle size reduction or to Section 7.1.2.1 if it is noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids determined in Section 7.1.1.9 is less than 0.5%, then proceed to Section 7.2.9 if the nonvolatile TCLP is to be performed and to Section 7.3 with a fresh portion of the waste if the volatile TCLP is to be performed.

7.1.2.1 Remove the solid phase and filter from the filtration apparatus.

7.1.2.2 Dry the filter and solid phase at 100 ± 20°C until two successive weighing yield the same value within ± 1%. Record the final weight.

Note: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

7.1.2.3 Calculate the percent dry solids as follows:

$$\text{Percent dry solids} = \frac{\text{(Wt. of dry waste + filter) - tared wt. of filter}}{\text{Initial wt. of waste (Section 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2.4 If the percent dry solids is less than 0.5%, then proceed to Section 7.2.9 if the nonvolatile TCLP is to be performed, and to Section 7.3 if the volatile TCLP is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the nonvolatile TCLP is to be performed, return to the beginning of this Section (7.1) and, with a fresh portion of waste, determine whether particle size reduction is necessary (Section 7.1.3) and determine the appropriate extraction fluid (Section 7.1.4). If only the volatile TCLP is to be performed, see the note in Section 7.1.4.

7.1.3 Determination of whether the waste requires particle size reduction (particle size is reduced during this step): Using the solid portion of the waste, evaluate the solid for particle size. Particle size reduction is required, unless the solid has a surface area per gram of material equal to or greater than 3.1 cm^2, or is smaller than 1 cm in its narrowest dimension (i.e., is capable of passing through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. If the solids are prepared for organic volatiles extraction, special precautions must be taken (see Section 7.3.6).

Note: Surface area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, sample specific methods would need to be developed and employed to measure the surface area. Such methodology is currently not available.

7.1.4 Determination of appropriate extraction fluid: If the solid content of the waste is greater than or equal to 0.5% and if the sample will be extracted for nonvolatile constituents (Section 7.2),

determine the appropriate fluid (Section 5.7) for the nonvolatiles extraction as follows:

Note: TCLP extraction for volatile constituents uses only extraction fluid #1 (Section 5.7.1). Therefore, if TCLP extraction for nonvolatiles is not required, proceed to Section 7.3.

7.1.4.1 Weigh out a small subsample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 500 mL beaker or Erlenmeyer flask.

7.1.4.2 Add 96.5 mL of reagent water to the beaker, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use extraction fluid #1. Proceed to Section 7.2.

7.1.4.3 If the pH from Section 7.1.4.2 is >5.0, add 3.5 mL 1N HCl, slurry briefly, cover with a watchglass, heat to 50°C, and hold at 50°C for 10 minutes.

7.1.4.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2. Proceed to Section 7.2.

7.1.5 If the aliquot of the waste used for the preliminary evaluation (Sections 7.1.1 - 7.1.4) was determined to be 100% solid at Section 7.1.1.1, then it can be used for the Section 7.2 extraction (assuming at least 100 grams remain), and the Section 7.3 extraction (assuming at least 25 grams remain). If the aliquot was subjected to the procedure in Section 7.1.1.7, then another aliquot shall be used for the volatile extraction procedure in Section 7.3. The aliquot of the waste subjected to the procedure in Section 7.1.1.7 might be appropriate for use for the Section 7.2 extraction if an adequate amount of solid (as determined by Section 7.1.1.9) was obtained. The amount of solid necessary is dependent upon whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solid remains, proceed to Section 7.2.10 of the nonvolatile TCLP extraction.

7.2 Procedure When Volatiles are not Involved

A minimum sample size of 100 grams (solid and liquid phases) is recommended. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample (percent solids, See Section 7.1.1), whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required. If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis.

7.2.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solid, see Section 7.1.1), weigh out a subsample of the waste (100 gram minimum) and proceed to Section 7.2.9.

7.2.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in Section 4.3.2 and is outlined in Sections 7.2.3 to 7.2.8.

7.2.3 Pre-weigh the container that will receive the filtrate.

7.2.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (see Section 4.4).

Note: Acid washed filters may be used for all nonvolatile extractions even when metals are not of concern.

7.2.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight. If the waste contains <0.5% dry solids (Section 7.1.2), the liquid portion of the waste, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the TCLP extract. For wastes containing >0.5% dry solids (Sections 7.1.1 or 7.1.2), use the percent solids information obtained in Section 7.1.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the TCLP extract.

7.2.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the waste is centrifuged, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.2.7 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder (see Section 4.3.2). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

Note: If waste material (>1% of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section 7.2.5, to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the

pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2 minute period), stop the filtration.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.2.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase may now be either analyzed (See Section 7.2.12) or stored at 4°C until time of analysis.

Note: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Section 7.2.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.2.9 If the waste contains <0.5% dry solids (see Section 7.1.2), proceed to Section 7.2.13. If the waste contains >0.5% dry solids (see Section 7.1.1 or 7.1.2), and if particle size reduction of the solid was needed in Section 7.1.3, proceed to Section 7.2.10. If the waste as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to Section 7.2.11.

7.2.10 Prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 7.1.3. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

Note: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon coated sieve should be used to avoid contamination of the sample.

7.2.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Section 7.1.1)} \times \text{weight of waste filtered (Section 7.2.5 or 7.2.7)}}{100}$$

Slowly add this amount of appropriate extraction fluid (see Section 7.1.4) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction takes place) shall be maintained at 22 ± 3°C during the extraction period.

Note: As agitation continues, pressure may build up within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

7.2.12 Following the 18 ± 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Section 7.2.7. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (see Section 4.4) if evaluating the mobility of metals.

7.2.13 Prepare the TCLP extract as follows:

7.2.13.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.2.12 is defined as the TCLP extract. Proceed to Section 7.2.14.

7.2.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Section 7.2.12 with the initial liquid phase of the waste obtained in Section 7.2.7. This combined liquid is defined as the TCLP extract. Proceed to Section 7.2.14.

7.2.13.3 If the initial liquid phase of the waste, as obtained from Section 7.2.7, is not or may not be compatible with the filtered liquid resulting from Section 7.2.12, do not combine these liquids. Analyze these liquids, collectively defined as the TCLP extract, and combine the results mathematically, as described in Section 7.2.14.

7.2.14 Following collection of the TCLP extract, the pH of the extract should be recorded. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4°C) until analyzed. The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested

extracts alone cannot be used to demonstrate that the waste is n hazardous. If the individual phases are to be analyzed separatel determine the volume of the individual phases (to ± 0.5%), conduct t appropriate analyses, and combine the results mathematically by using simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)\ (C_1) + (V_2)\ (C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L)
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.2.15 Compare the analyte concentrations in the TCLP extrac with the levels identified in the appropriate regulations. Refer t Section 8.0 for quality assurance requirements.

7.3 Procedure When Volatiles are Involved

Use the ZHE device to obtain TCLP extract for analysis of volatil compounds only. Extract resulting from the use of the ZHE shall not be used tc evaluate the mobility of nonvolatile analytes (e.g., metals, pesticides, etc.).

The ZHE device has approximately a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid (defined as that fraction of a sample from which no additional liquid may be forced out by an applied pressure of 50 psi), due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

Charge the ZHE with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4°C) to minimize loss of volatiles.

7.3.1 Pre-weigh the (evacuated) filtrate collection container (See Section 4.6) and set aside. If using a TEDLAR® bag, express all liquid from the ZHE device into the bag, whether for the initial or final liquid/solid separation, and take an aliquot from the liquid in the bag for analysis. The containers listed in Section 4.6 are recommended for use under the conditions stated in Sections 4.6.1 - 4.6.3.

7.3.2 Place the ZHE piston within the body of the ZHE (it may be helpful first to moisten the piston O-rings slightly with extraction

fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Section 7.3, Section 7.1.1 and/or 7.1.2). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

7.3.3 If the waste is 100% solid (see Section 7.1.1), weigh out a subsample (25 gram maximum) of the waste, record weight, and proceed to Section 7.3.5.

7.3.4 If the waste contains < 0.5% dry solids (Section 7.1.2), the liquid portion of waste, after filtration, is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For wastes containing ≥ 0.5% dry solids (Sections 7.1.1 and/or 7.1.2), use the percent solids information obtained in Section 7.1.1 to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

7.3.4.1 For wastes containing < 0.5% solids (see Section 7.1.1), weigh out a 500 gram subsample of waste and record the weight.

7.3.4.2 For wastes containing ≥ 0.5% solids (see Section 7.1.1), determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge ZHE} = \frac{25}{\text{percent solids (Section 7.1.1)}} \times 100$$

Weigh out a subsample of the waste of the appropriate size and record the weight.

7.3.5 If particle size reduction of the solid portion of the waste was required in Section 7.1.3, proceed to Section 7.3.6. If particle size reduction was not required in Section 7.1.3, proceed to Section 7.3.7.

7.3.6 Prepare the waste for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size as described in Section 7.1.3. Wastes and appropriate reduction equipment should be refrigerated, if possible, to 4°C prior to particle size reduction. The means used to effect particle size reduction must not generate heat in and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

Note: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative. Surface area requirements are

meant for filamentous (e.g., paper, cloth) and similar waste material: Actual measurement of surface area is not recommended.

When the surface area or particle size has been appropriatel altered, proceed to Section 7.3.7.

7.3.7 Waste slurries need not be allowed to stand to permit th solid phase to settle. Do not centrifuge wastes prior to filtration.

7.3.8 Quantitatively transfer the entire sample (liquid an solid phases) quickly to the ZHE. Secure the filter and support screen onto the top flange of the device and secure the top flange to the ZH body in accordance with the manufacturer's instructions. Tighten all ZH fittings and place the device in the vertical position (gas inlet/outle flange on the bottom). Do not attach the extract collection device to th top plate.

Note: If waste material (>1% of original sample weight) has obviously adhered t the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined ir Section 7.3.4 to determine the weight of the waste sample that will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. If the waste is 100% solid (see Section 7.1.1), slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the device and proceed to Section 7.3.12.

7.3.9 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psi to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2 minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.3.10 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

Note: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the TCLP extraction as a solid.

If the original waste contained <0.5% dry solids (see Section 7.1.2), this filtrate is defined as the TCLP extract and is analyzed directly. Proceed to Section 7.3.15.

7.3.11 The liquid phase may now be either analyzed immediately (See Sections 7.3.13 through 7.3.15) or stored at 4°C under minimal headspace conditions until time of analysis. Determine the weight of extraction fluid #1 to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Section 7.1.1)} \times \text{weight of waste filtered (Section 7.3.4 or 7.3.8)}}{100}$$

7.3.12 The following Sections detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases (See Section 5.7).

7.3.12.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

7.3.12.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psi (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed.

7.3.12.3 Place the ZHE in the rotary agitation apparatus (if it is not already there) and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in

which extraction occurs) shall be maintained at 23 ± 2°C during agitation.

7.3.13 Following the 18 ± 2 hour agitation period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Check the ZHE for leaking as specified in Section 4.2.1, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR® bag) holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container. Filter through the glass fiber filter, using the ZHE device as discussed in Section 7.3.9. All extract shall be filtered and collected if the TEDLAR® bag is used, if the extract is multiphasic, or if the waste contained an initial liquid phase (see Sections 4.6 and 7.3.1).

Note: An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

7.3.14 If the original waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.3.13 is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained from Section 7.3.13 and the initial liquid phase (Section 7.3.9) are collectively defined as the TCLP extract.

7.3.15 Following collection of the TCLP extract, immediately prepare the extract for analysis and store with minimal headspace at 4°C until analyzed. Analyze the TCLP extract according to the appropriate analytical methods. If the individual phases are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)\ (C_1) + (V_2)\ (C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phases (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.3.16 Compare the analyte concentrations in the TCLP extra with the levels identified in the appropriate regulations. Refer Section 8.0 for quality assurance requirements.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for all required quality control procedures A minimum of one blank (using the same extraction fluid as used for the samples for every 20 extractions that have been conducted in an extraction vessel.

8.2 A matrix spike shall be performed for each waste type (e.g. wastewater treatment sludge, contaminated soil, etc.) unless the result exceed the regulatory level and the data are being used solely to demonstrate that th waste property exceeds the regulatory level. As a minimum, follow the matri spike addition guidance provided in each analytical method.

8.2.1 Matrix spikes are to be added after filtration of the TCL. extract and before preservation. Matrix spikes should not be added prio to TCLP extraction of the sample.

8.2.2 In most cases, matrix spikes should be added at concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be not less than five times the methoc detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of TCLP extract as that which was analyzed for the unspiked sample.

8.2.3 The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration in the TCLP extract when the recovery of the matrix spike is below the expected analytical method performance.

8.3 All quality control measures described in the appropriate analytical methods shall be followed.

8.4 Samples must undergo TCLP extraction within the following time periods:

SAMPLE MAXIMUM HOLDING TIMES (days)

	From: Field Collection To: TCLP extraction	From: TCLP extraction To: Preparative extraction	From: Preparative extraction To: determinative analysis	Total Elapsed Time
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360
NA = Not Applicable				

If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

9.0 METHOD PERFORMANCE

9.1 Ruggedness. Two ruggedness studies have been performed to determine the effect of various perturbations on specific elements of the TCLP protocol. Ruggedness testing determines the sensitivity of small procedural variations which might be expected to occur during routine laboratory application.

9.1.1 Metals - The following conditions were used when leaching a waste for metals analysis:

Varying Conditions	
Liquid/Solid ratio	19:1 vs. 21:1
Extraction time	16 hours vs. 18 hours
Headspace	20% vs. 60%
Buffer #2 acidity	190 meq vs. 210 meq
Acid-washed filters	yes vs. no

Filter type	0.7 μm glass fiber vs. 0.45 μm vs. polycarbonate
Bottle type	borosilicate vs. flint glass

Of the seven method variations examined, acidity of the extraction fluid had the greatest impact on the results. Four of 13 metals from an API separator sludge/electroplating waste (API/EW) mixture and two of three metals from an ammonia lime still bottom waste were extracted at higher levels by the more acidic buffer. Because of the sensitivity to pH changes, the method requires that the extraction fluids be prepared so that the final pH is within ± 0.05 units as specified.

9.1.2 Volatile Organic Compounds - The following conditions were used when leaching a waste for VOC analysis:

Varying Conditions	
Liquid/Solid ratio	19:1 vs. 21:1
Headspace	0% vs. 5%
Buffer #1 acidity	60 meq vs. 80 meq
Method of storing extract	Syringe vs. Tedlar® bag
Aliquotting	yes vs. no
Pressure behind piston	0 psi vs. 20 psi

None of the parameters had a significant effect on the results of the ruggedness test.

9.2 Precision. Many TCLP precision (reproducibility) studies have been performed, and have shown that, in general, the precision of the TCLP is comparable to or exceeds that of the EP toxicity test and that method precision is adequate. One of the more significant contributions to poor precision appears to be related to sample homogeneity and inter-laboratory variation (due to the nature of waste materials).

9.2.1 Metals - The results of a multi-laboratory study are shown in Table 6, and indicate that a single analysis of a waste may not be adequate for waste characterization and identification requirements.

9.2.2 Semi-Volatile Organic Compounds - The results of two studies are shown in Tables 7 and 8. Single laboratory precision was excellent with greater than 90 percent of the results exhibiting an RSD less than 25 percent. Over 85 percent of all individual compounds in the multi-laboratory study fell in the RSD range of 20 - 120 percent. Both studies concluded that the TCLP provides adequate precision. It was also determined that the high acetate content of the extraction fluid did not

present problems (i.e., column degradation of the gas chromatograph) f the analytical conditions used.

9.2.3 Volatile Organic Compounds - Eleven laboratori participated in a collaborative study of the use of the ZHE with two was types which were fortified with a mixture of VOCs. The results of t collaborative study are shown in Table 9. Precision results for VOCs te to occur over a considerable range. However, the range and mean R compared very closely to the same collaborative study metals results Table 6. Blackburn and Show concluded that at the 95% level significance: 1) recoveries among laboratories were statistical similar, 2) recoveries did not vary significantly between the two sampi types, and 3) each laboratory showed the same pattern of recovery for eac of the two samples.

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Table 1.
Volatile Analytes[1,2]

Compound	CAS No.
Acetone	67-64-1
Benzene	71-43-2
n-Butyl alcohol	71-36-3
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethylene	75-35-4
Ethyl acetate	141-78-6
Ethyl benzene	100-41-4
Ethyl ether	60-29-7
Isobutanol	78-83-1
Methanol	67-56-1
Methylene chloride	75-09-2
Methyl ethyl ketone	78-93-3
Methyl isobutyl ketone	108-10-1
Tetrachloroethylene	127-18-4
Toluene	108-88-3
1,1,1,-Trichloroethane	71-55-6
Trichloroethylene	79-01-6
Trichlorofluoromethane	75-69-4
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1
Vinyl chloride	75-01-4
Xylene	1330-20-7

[1] When testing for any or all of these analytes, the zero-headspace extractor vessel shall be used instead of the bottle extractor.

[2] Benzene, carbon tetrachloride, chlorobenzene, chloroform, 1,2-dichloroethane, 1,1-dichloroethylene, methyl ethyl ketone, tetrachloroethylene, and vinyl chloride are toxicity characteristic constituents.

Table 2.
Suitable Rotary Agitation Apparatus[1]

Company	Location	Model No.
Analytical Testing and Consulting Services, Inc.	Warrington, PA (215) 343-4490	4-vessel extractor (DC20S 8-vessel extractor (DC20) 12-vessel extractor (DC20B
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	2-vessel (3740-2); 4-vessel (3740-4); 6-vessel (3740-6); 8-vessel (3740-8); 12-vessel (3740-12); 24-vessel (3740-24)
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	8-vessel (08-00-00) 4-vessel (04-00-00)
IRA Machine Shop and Laboratory	Santurce, PR (809) 752-4004	8-vessel (011001)
Lars Lande Manufacturing	Whitmore Lake, MI (313) 449-4116	10-vessel (10VRE) 5-vessel (5VRE)
Millipore Corp.	Bedford, MA (800) 225-3384	4-ZHE or 4 1-liter bottle extractor (YT300RAHW) 4-vessel (SD1M352C0)

[1] Any device that rotates the extraction vessel in an end-over-end fashion at 30 ± 2 rpm is acceptable.

Table 3.
Suitable Zero-Headspace Extractor Vessels[1]

Company	Location	Model No.
Analytical Testing & Consulting Services, Inc.	Warrington, PA (215) 343-4490	C102, Mechanical Pressure Device
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	3745-ZHE, Gas Pressure Device
Lars Lande Manufacturing[2]	Whitmore Lake, MI (313) 449-4116	ZHE-11, Gas Pressure Device
Millipore Corporation	Bedford, MA (800) 225-3384	YT30090HW, Gas Pressure Device
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	VOLA-TOX1, Gas Pressure Device
Gelman Science	Ann Arbor, MI (800) 521-1520	15400 Gas Pressure Device

[1] Any device that meets the specifications listed in Section 4.2.1 of the method is suitable.

[2] This device uses a 110 mm filter.

Table 4.
Suitable Filter Holders[1]

Company	Location	Model/ Catalogue #	Size
Nucleopore Corporation	Pleasanton, CA (800) 882-7711	425910 410400	142 mm 47 mm
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	302400 311400	142 mm 47 mm
Millipore Corporation	Bedford, MA (800) 225-3384	YT30142HW XX1004700	142 mm 47 mm

[1] Any device capable of separating the liquid from the solid phase of the waste is suitable, providing that it is chemically compatible with the waste and the constituents to be analyzed. Plastic devices (not listed above) may be used when only inorganic analytes are of concern. The 142 mm size filter holder is recommended.

Table 5.
Suitable Filter Media[1]

Company	Location	Model	Pore Size (μm)
Millipore Corporation	Bedford, MA (800) 225-3384	AP40	0.7
Nucleopore Corporation	Pleasanton, CA (415) 463-2530	211625	0.7
Whatman Laboratory Products, Inc.	Clifton, NJ (201) 773-5800	GFF	0.7
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	GF75	0.7
Gelman Science	Ann Arbor, MI (800) 521-1520	66256 (90mm) 66257 (142mm)	0.7

[1] Any filter that meets the specifications in Section 4.4 of the Method is suitable.

Table 6. Multi-Laboratory TCLP Metals, Precision

Waste	Extraction Fluid	Metal	$\bar{X}$	S	%RSD
Ammonia Lime Still Bottoms	#1	Cadmium	0.053	0.031	60
	#2		0.023	0.017	76
	#1	Chromium	0.015	0.0014	93
	#2		0.0032	0.0037	118
	#1	Lead	0.0030	0.0027	90
	#2		0.0032	0.0028	87
API/EW Mixture	#1	Cadmium	0.0046	0.0028	61
	#2		0.0005	0.0004	77
	#1	Chromium	0.0561	0.0227	40
	#2		0.105	0.018	17
	#1	Lead	0.0031	0.0031	100
	#2		0.0124	0.0136	110
Fossil Fuel Fly Ash	#1	Cadmium	0.080	0.069	86
	#2		0.093	0.067	72
	#1	Chromium	0.017	0.014	85
	#2		0.070	0.040	57
	#1	Lead	0.0087	0.0074	85
	#2		0.0457	0.0083	18
				%RSD Range = 17 - 118	
				Mean %RSD = 74	

Note: $\bar{X}$ = Mean results from 6 - 12 different laboratories
Units = mg/L
Extraction Fluid #1 = pH 4.9
#2 = pH 2.9

Table 7. Single-Laboratory Semi-Volatiles, Precision

Waste	Compound	Extraction Fluid	$\bar{X}$	S	%RSD
Ammonia Lime Still Bottoms	Phenol	#1	19000	2230	11.6
		#2	19400	929	4.8
	2-Methylphenol	#1	2000	297	14.9
		#2	1860	52.9	2.8
	4-Methylphenol	#1	7940	1380	17.4
		#2	7490	200	2.7
	2,4-Dimethylphenol	#1	321	46.8	14.6
		#2	307	45.8	14.9
	Naphthalene	#1	3920	413	10.5
		#2	3827	176	4.6
	2-Methylnaphthalene	#1	290	44.8	15.5
		#2	273	19.3	7.1
	Dibenzofuran	#1	187	22.7	12.1
		#2	187	7.2	3.9
	Acenaphthylene	#1	703	89.2	12.7
		#2	663	20.1	3.0
	Fluorene	#1	151	17.6	11.7
		#2	156	2.1	1.3
	Phenanthrene	#1	241	22.7	9.4
		#2	243	7.9	3.3
	Anthracene	#1	33.2	6.19	18.6
		#2	34.6	1.55	4.5
	Fluoranthrene	#1	25.3	1.8	7.1
		#2	26.0	1.8	7.1
API/EW Mixture	Phenol	#1	40.7	13.5	33.0
		#2	19.0	1.76	9.3
	2,4-Dimethylphenol	#1	33.0	9.35	28.3
		#2	43.3	8.61	19.9
	Naphthalene	#1	185	29.4	15.8
		#2	165	24.8	15.0
	2-Methylnaphthalene	#1	265	61.2	23.1
		#2	200	18.9	9.5
				%RSD Range = 1 - 33 Mean %RSD = 12	

Note: Units = µg/L
Extractions were performed in triplicate
All results were at least 2x the detection limit
Extraction Fluid #1 = pH 4.9
#2 = pH 2.9

Table 8. Multi-Laboratory Semi-Volatiles, Precision

Waste	Compound	Extraction Fluid	$\bar{X}$	S	%RSI
Ammonia Lime Still Bottoms (A)	BNAs	#1	10043	7680	76.!
		#2	10376	6552	63.:
API/EW Mixture (B)	BNAs	#1	1624	675	41.6
		#2	2074	1463	70.5
Fossil Fuel Fly Ash (C)	BNAs	#1	750	175	23.4
		#2	739	342	46.3
					Mean %RSD = 54

Note: Units = µg/L
$\bar{X}$ = Mean results from 3 - 10 labs
Extraction Fluid #1 = pH 4.9
#2 = pH 2.9

%RSD Range for Individual Compounds

A, #1	0 - 113
A, #2	28 - 108
B, #1	20 - 156
B, #2	49 - 128
C, #1	36 - 143
C, #2	61 - 164

Table 9. Multi-Laboratory (11 Labs) VOCs. Precision

Waste	Compound	$\bar{X}$	S	%RSD
Mine Tailings	Vinyl chloride	6.36	6.36	100
	Methylene chloride	12.1	11.8	98
	Carbon disulfide	5.57	2.83	51
	1,1-Dichloroethene	21.9	27.7	127
	1,1-Dichloroethane	31.4	25.4	81
	Chloroform	46.6	29.2	63
	1,2-Dichloroethane	47.8	33.6	70
	2-Butanone	43.5	36.9	85
	1,1,1-Trichloroethane	20.9	20.9	100
	Carbon tetrachloride	12.0	8.2	68
	Trichloroethene	24.7	21.2	86
	1,1,2-Trichloroethene	19.6	10.9	56
	Benzene	37.9	28.7	76
	1,1,2,2-Tetrachloroethane	34.9	25.6	73
	Toluene	29.3	11.2	38
	Chlorobenzene	35.6	19.3	54
	Ethylbenzene	4.27	2.80	66
	Trichlorofluoromethane	3.82	4.40	115
	Acrylonitrile	76.7	110.8	144
Ammonia Lime Still Bottoms	Vinyl chloride	5.00	4.71	94
	Methylene chloride	14.3	13.1	92
	Carbon disulfide	3.37	2.07	61
	1,1-Dichloroethene	52.1	38.8	75
	1,1-Dichloroethane	52.8	25.6	49
	Chloroform	64.7	28.4	44
	1,2-Dichloroethane	43.1	31.5	73
	2-Butanone	59.0	39.6	67
	1,1,1-Trichloroethane	53.6	40.9	76
	Carbon tetrachloride	7.10	6.1	86
	Trichloroethene	57.3	34.2	60
	1,1,2-Trichloroethene	6.7	4.7	70
	Benzene	61.3	26.8	44
	1,1,2,2-Tetrachloroethane	3.16	2.1	66
	Toluene	69.0	18.5	27
	Chlorobenzene	71.8	12.0	17
	Ethylbenzene	3.70	2.2	58
	Trichlorofluoromethane	4.05	4.8	119
	Acrylonitrile	29.4	34.8	118
		%RSD Range = 17 - 144 Mean %RSD = 75		

Note: Units = µg/L

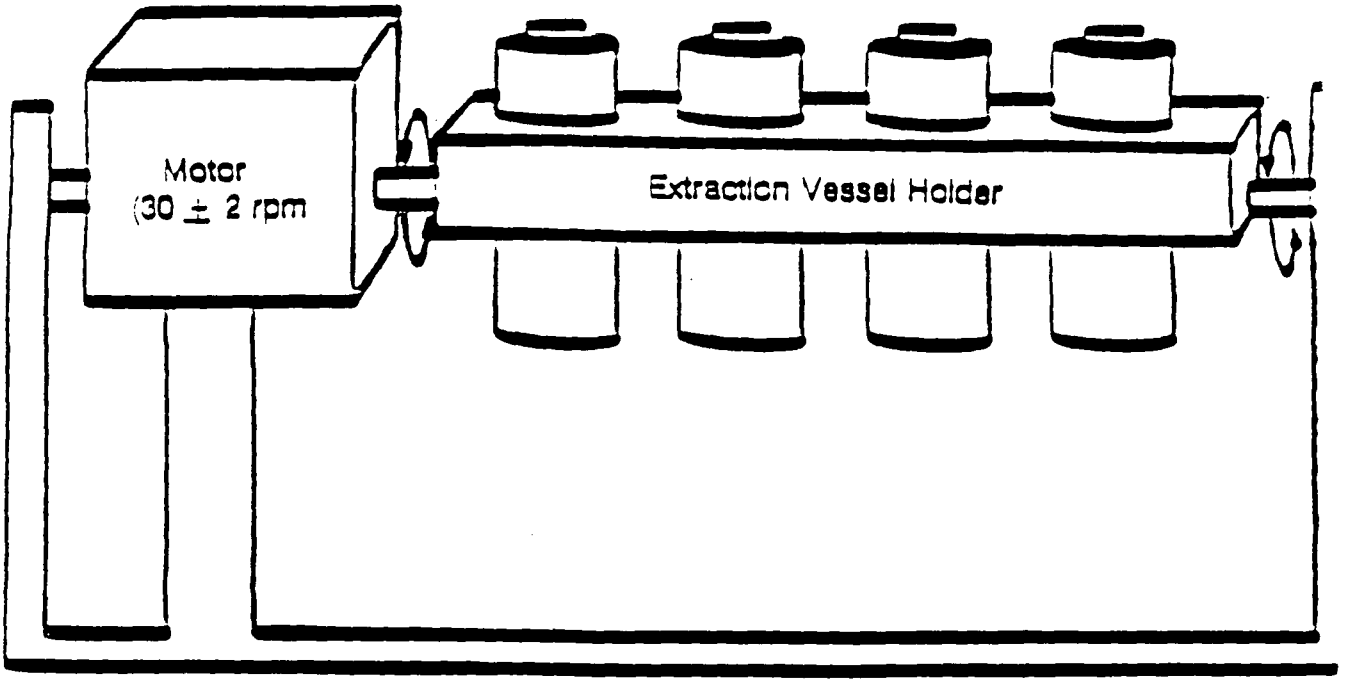


Figure 1. Rotary Agitation Apparatus

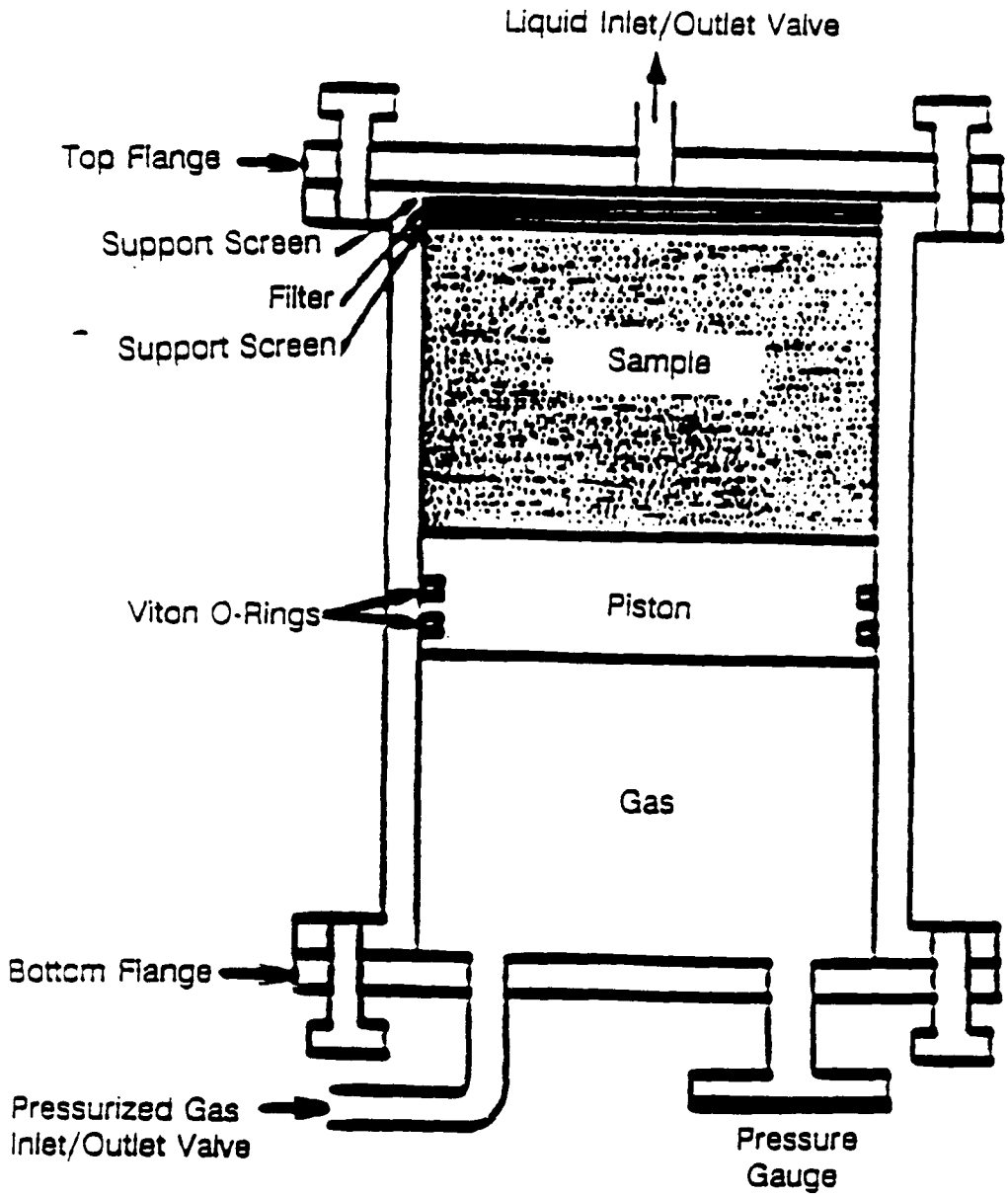
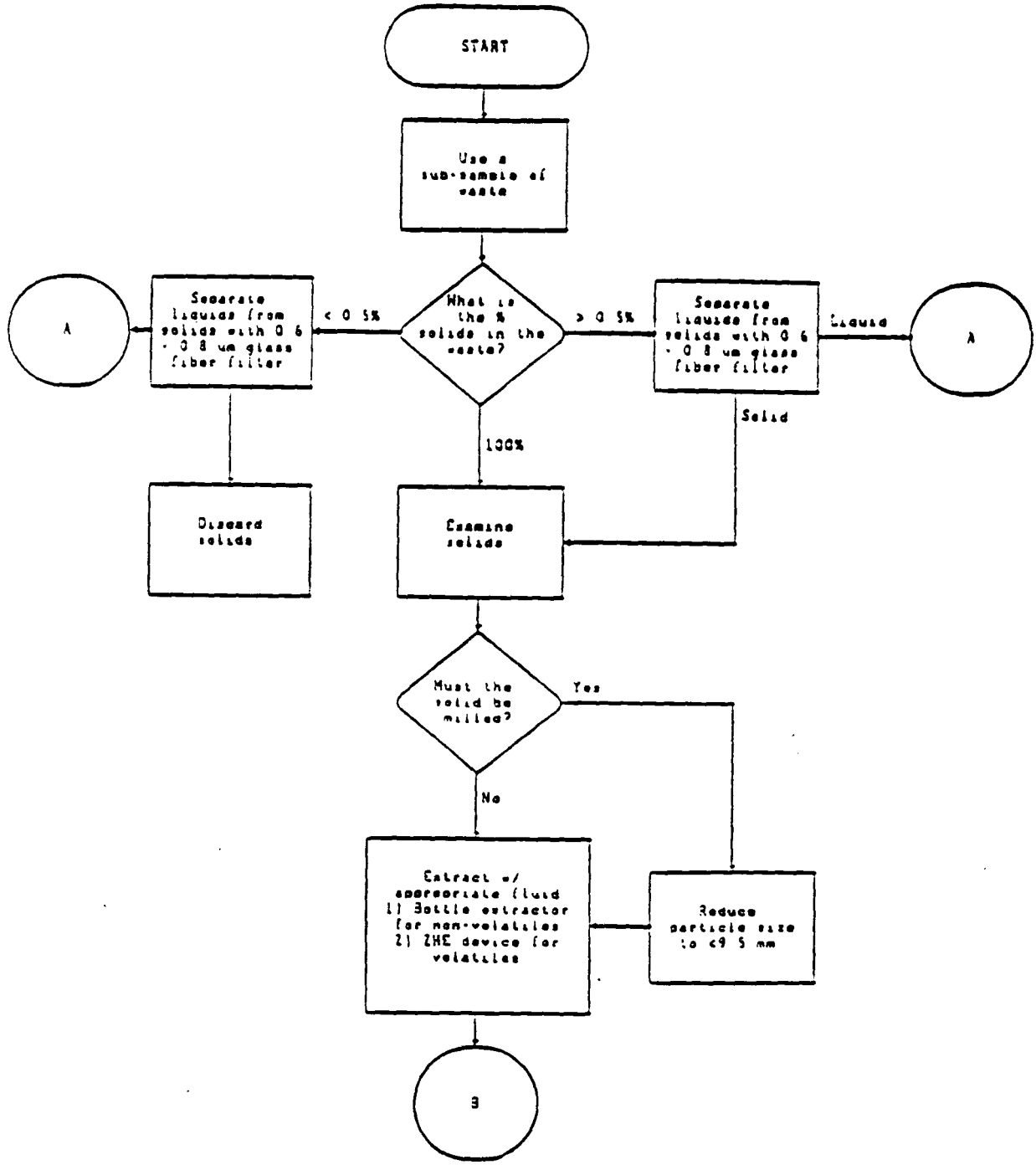


Figure 2. Zero-Headspace Extractor (ZHE)

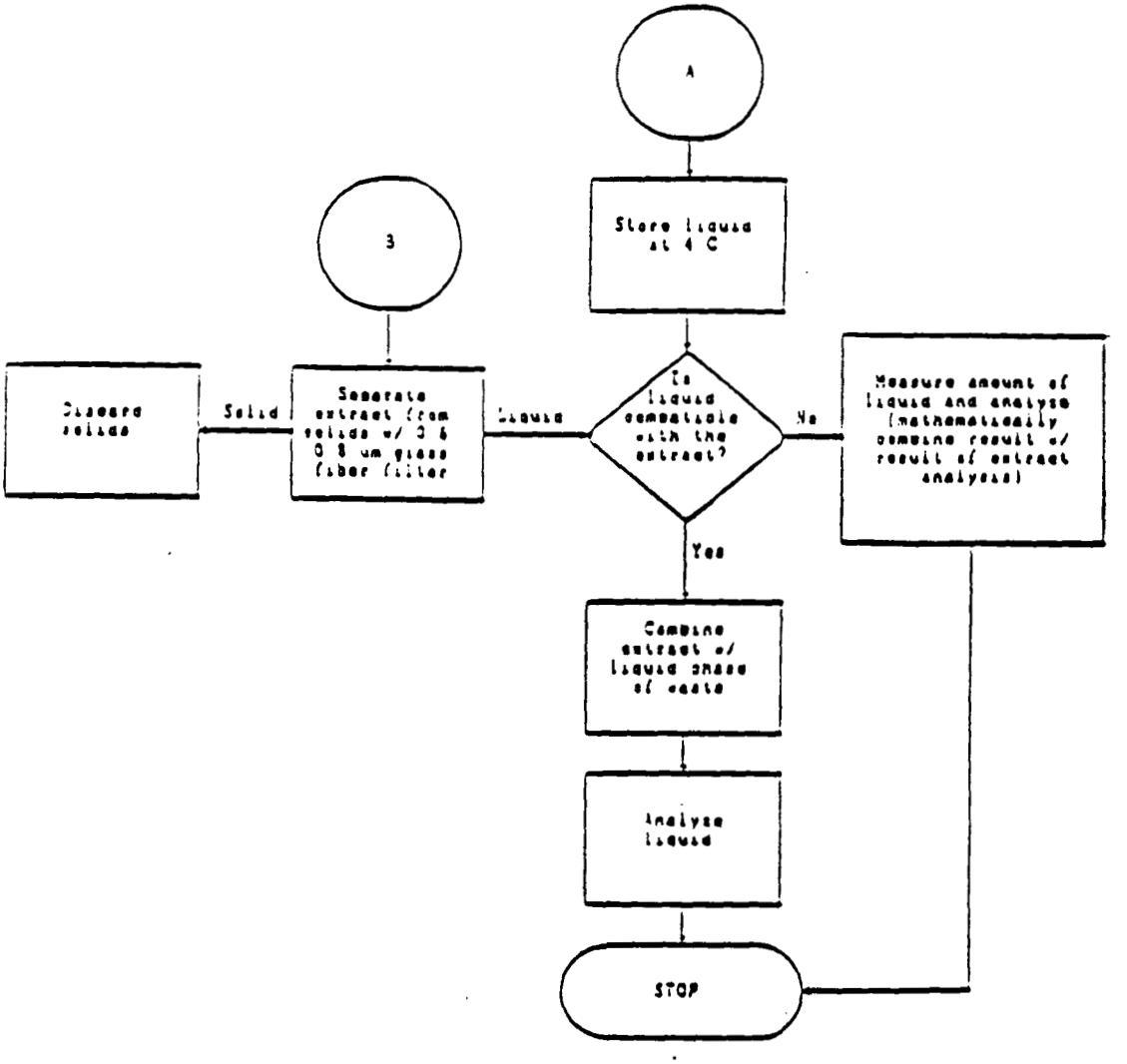
METHOD 1311

TOXICITY CHARACTERISTIC LEACHATE PROCEDURE



METHOD 1311 (CONTINUED)

TOXICITY CHARACTERISTIC LEACHATE PROCEDURE



Procedure for Determination of Volume Reduction

1. Determine the bulk density of the initial sample.
2. Determine the apparent density of the final glass.
3. *volume reduction = 100% * (1 - initial density/final density)*

Procedure for Determination of Gamma Dose Rates of Vitrified Wastes

1. Record the approximate dimensions of the vitrified waste block.

2. Measure and record the gamma dose rate at contact, 25, and 50 cm from the melt crucible along the centerline.

3. Repeat step 2, except measure radially outward at the half height of the vitrified mass.

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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