

November 6, 1992



Mr. Edward M. Lee, Jr.
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Building 080
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Golden, Colorado 80402-0464

Subject: Rocky Flats Plant Solar Evaporation Ponds Stabilization Project
[WBS 710 PROJECT MANAGEMENT - HALLIBURTON NUS ROCKY FLATS]
RESPONSE TO LOCKHEED REPORT DATED OCTOBER 23, 1992
FINAL REPORT EVALUATION OF THE ROCKY FLATS PLANT
PONDCRETE SAMPLING & ANALYSIS PROGRAM
RF-HED-92-0774

Dear Mr. Lee:

Enclosed is the response to the cited report from Mr. Rich Ninesteel, Project Manager for the Treatability Study.

It is our understanding that the report status has been changed to a draft report.

Please forward this information to the appropriate parties.

Sincerely,

HALLIBURTON NUS ENVIRONMENTAL
CORPORATION



Ted A. Bittner
Project Manager

TAB/jg

Enclosure

cc: D. Dougherty
D. Ferrier
S. Heiman

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RF-HED-92-0774

**RESPONSE TO COMMENTS BY LOCKHEED ENVIRONMENTAL
SYSTEMS AND TECHNOLOGIES COMPANY ON THE
PONDCRETE SAMPLING AND ANALYSIS PROGRAM**

The following responses parallel the outline of comments provided by Lockheed in their October 23, 1992 document titled "Final Report Evaluation of the Rocky Flats Plant Pondcrete Sampling and Analysis Program."

GENERAL COMMENT

1. Lockheed's stated goal for reviewing the Pondcrete Sampling and Analysis Program is as follows:

"The goal of this evaluation exercise is to provide sufficient information to assess the usefulness of HALLIBURTON NUS' results for adequately characterizing and representing the collection of pondcrete at the RFP, as well as additional, unsampled pondcrete at the Nevada Test Site (NTS)."

The pondcrete waste characterization program was designed to support the reprocessing of the billets remaining at the Rocky Flats Plant, not to characterize pondcrete already shipped to NTS. Lockheed's use of the data for a purpose other than its intended use is questionable. The following responses have been focused on the stated use of the data, namely the support of the pondcrete reprocessing project.

2. Based on all data currently available to HALLIBURTON NUS, Pond 207A was the only source of pondcrete.

PROBLEM AND POPULATION IDENTIFICATION

1. Was the sampling problem properly identified and clearly conceptualized?

The reviewer failed to understand that there are three important steps in this project. They include: sampling, analysis, and treatment. The purpose of each of these steps needs to be identified and described. The HALLIBURTON NUS report clearly states the purpose of each step, as quoted by the reviewer, as well as the overall project objective.

The objectives stated in the Sampling and Analysis Plan and the Pondcrete Waste Characterization Report are neither conflicting nor inconsistent. Lockheed has over-analyzed individual statements made in both reports and has apparently failed to understand the stated objectives. HALLIBURTON NUS stands behind the objectives as stated.

2. Was the population of interest properly identified?

It is impossible to relate pondcrete hazardous constituent concentrations to their respective pond locational constituents for the following reasons:

- There are no analytical data for Pond 207A for the time period during which the pondcrete buried at NTS was produced.
- Operational records, if this exists at all, have not been provided to HALLIBURTON NUS. These would be needed to correlate pondcrete data with pond sludge data.
- Many triwalls are missing production data information.

Lockheed has failed to recognize that the source of waste to the original pondcrete process (pond sludge) is different than the source of waste to the reprocessing process (pondcrete billets). Since the HALLIBURTON NUS project is interested only in pondcrete reprocessing, the data were collected only for the purpose of supporting pondcrete reprocessing.

Lockheed also implies that each batch of pondcrete from the original process would represent an individual population, assuming an efficient cement mixing process. This is probably not true, nor is it relevant to pondcrete reprocessing, for the following reasons:

- The original pondcrete process, by all accounts, was not efficient. Inadequate mixing and lack of process control has been identified as major problems during pondcrete production.
- Lack of processing records and missing accumulation date data would make identification of individual batches difficult, if not impossible.
- The triwalls are stored randomly with respect to accumulation date. For the purpose of reprocessing pondcrete, it is not feasible to stage the triwalls by date prior to treatment due to the limited space available, the manpower that would be required, and waste storage regulatory requirements. The only reasonable approach to reprocessing is the development of a stabilization recipe that can operate over a fairly wide range of conditions, and processing the triwalls as they are encountered in the storage tents. Again, the sampling program was developed in support of the overall project engineering.

3. Was the chemical and physical constituency of the population homogeneous or heterogeneous in space or time?

HALLIBURTON NUS did not assume that any chemical and physical characteristics within each waste form are homogeneous temporally or spatially. The HALLIBURTON NUS sampling program was designed based on the knowledge of the heterogeneous properties of each waste form. The purpose of this sampling program was to characterize this heterogeneity within each waste form. The need to employ cluster sampling and to sample each waste form separately, as proposed in the HALLIBURTON NUS sampling program, are all the direct results of considering heterogeneity. On the other hand, identifying the sources of this heterogeneity was not what this sampling program was designed for, because input to the current treatment system is the product of the past operation, and not the past operation itself.

4. Was the form of the data properly identified?

The reviewer failed to understand the difference between a confidence interval of the mean and a tolerance interval of the population. The "97.5% <" values presented in Tables 1-2 and 1-3 of the sampling plan are tolerance limits which were used to estimate the possible highest concentrations in the population. Tolerance limit, as applied, is not sensitive to the underlying distribution of the population. These values were only used in the selection of the most critical chemical regarding the TCLP test, but not in the actual design of the sampling plan. For this purpose, a regular tolerance limit calculation is considered appropriate. Normality was not assumed in the sampling plan design itself.

Correlation structures between all the chemical and physical characteristics analyzed in the WESTON study were developed

and evaluated by HALLIBURTON NUS before the final sampling approach was selected. There was no informative strong correlation identified that can be useful in the sampling plan design. Therefore, they were not presented in the HALLIBURTON NUS Sampling Plan.

SAMPLING PLAN

1. Was the sample size adequate and correctly specified with respect to the sampling problem?

RSD and DQO are separately described in Sections 1.3.2 and 1.3.3. However, they are both required in the equation for estimating the sample size, as clearly shown in Section 1.3.4 of the report. Therefore, the variance of the population and the accuracy requirement are both considered in the sample size determination. This equation is widely used for sample size determination. The reviewer apparently failed to read through the report.

RSD and DQO are simply normalized (divided by the mean) standard deviation and standard error as defined in the report. The purpose of normalization was to make the dimensionless graphical presentation between DQO and sample size as shown in Figures 1-1 and 1-2 in the Sampling and Analysis Plan, the required increase in sample size is very significant to further reduce the DQO. This was explained in Section 1.3.3.

2. Was the sample representative of the population?

HALLIBURTON NUS did not assume homogeneity. The assumption required for cluster sampling was that the underlying distribution of the population is stationary (the distribution of any part of the population is the same as the distribution

of the entire population) as described in Section 1.3.6. The reviewer did not understand the difference between homogeneous and stationary. The cluster sampling scheme was proposed by HALLIBURTON NUS to ensure that samples will be collected from as many different areas as possible and also to consider the access problems of sampling triwalls in the middle of the stacks. Under a homogeneous condition, this would not be necessary.

3. Did the plan employ an appropriate estimator for the population mean?

No response required.

4. Did the plan employ an appropriate estimator for the inherent variability?

As mentioned earlier, HALLIBURTON NUS did not assume normality in the actual sampling size calculation and sampling procedure.

The multiphase procedure was not applied, so it did not need to be explained in detail in the HALLIBURTON NUS report.

HALLIBURTON NUS did have some knowledge about the population. This information was from the Weston study and was utilized in the sampling plan development.

IMPLEMENTATION OF THE SAMPLING PLAN

1. Were the samples properly labeled and identified?

All triwalls were staged and sampled by EG&G Rocky Flats. Sample origin data (Pad, Test, Stack, Triwall Number) are listed in tables A-2 and A-3 of the Pondcrete Waste

Characterization Report. All pondcrete samples sent to HALLIBURTON NUS for analysis were assigned a unique sample number and maintained under chain-of-custody.

2. Was the sampling operation executed faithfully with respect to the sampling plan?

The assertion that "the modifications to the pondcrete sampling plan were lacking in proper justification" is completely false. In fact, Appendix A of the Pondcrete Waste Characterization Report (Summary of Proposed Pondcrete Sample Collection compared with Actual Sample Collection) is devoted entirely to documentation of actual vs. proposed sample locations.

Likewise the assertion that "HALLIBURTON NUS modified their method for sample size determination during the planning phase of the project without proper justification" is also completely false. Section 1.0 of the Sampling and Analysis Plan presents the rationale for the sampling program. This was the only rationale developed, and it was not modified.

Lockheed also asserts that the use of TCLP data for sample size determination was not discussed. The reviewer is referred to page 1-21 of the Sampling and Analysis Plan (Rev. 0) for this discussion.

3. Were departures from the sampling plan justified?

As previously discussed, the assertion that "the original plan included numerous departures from approved methods and procedures" has been shown to be false and unsubstantiated.

ANALYTICAL PLAN AND IMPLEMENTATION

1. Quality assurance of the toxicity characteristic leaching procedure (TCLP).

The Toxicity Characteristic Leaching Procedure (TCLP) used for the analysis of pondcrete was SW 1311 as clearly shown and completely documented in Table 3-1 of the Pondcrete Sampling and Analysis Plan. This method utilized by the HNUS laboratory is the same as the method in 40 CFR 261. The SOP was not provided for this method in the Pondcrete Sampling and Analysis Plan. However, no SOPs were provided for any of the analyses specified in the Sampling and Analysis Plan. It is unclear why the reader can not accept the method used, SW 1311, without an SOP. A copy of the HNUS laboratory SOP for SW 1311 has been included to satisfy the reviewer's need to review the SOP.

2. Analytical validity of the data.

The analysis of the pondcrete stored at the Rocky Flats Plant was completed at the HALLIBURTON NUS Laboratory in Pittsburgh, Pennsylvania, which is approved for use in EPA's Contract Laboratory Program (CLP). The analytical program for the characterization of pondcrete generally followed methods as prescribed in SW-846. The analytical program is presented in detail in Table 2-4 of the Pondcrete Waste Characterization Report, Rev. 0. All analyses prescribed in Table 2-4 with a Data Quality Objective (DQO) Level IV included production of a complete CLP-equivalent data package, and the resulting data were subjected to the data validation process which produces legally defensible data. The data validation utilized the general guidelines set forth by the EPA, and specific guidelines set forth by EPA Region VIII. This process evaluates all quality control data with respect to the method

and produces qualified data. Therefore, the complete data packages need not be included in the Pondcrete Characterization Report. The overall quality and validity of the data is not reduced nor compromised by the exclusion of the complete data packages. The data validation cover letters are included as Appendix C in the Pondcrete Characterization Report. The database presented in Appendix B and all the Tables included in the Pondcrete Waste Characterization Report represent post-validation results.

The assertion that "the sampling QC sample strategy did not account for the appropriate number of duplicate, co-located, and blank samples" is false.

The sampling effort for pondcrete collected field duplicates at a frequency of one duplicate for every twenty samples. For populations with less than twenty samples, one duplicate was collected for every population. A summary of the pondcrete sampling program is presented in Table 3-2 of the Pondcrete Sampling & Analysis Plan. The impact of the field duplicates collected on the data generated has been evaluated during the validation process. The field duplicates are presented and clearly identified in the text and database of the Pondcrete Waste Characterization Report.

During the sampling program for pondcrete, several rinsate blanks were collected. The rinsate blanks were collected at the same frequency as the field duplicates. A complete listing of rinsate blanks collected in the pondcrete sampling program is presented in Table 3-2 of the Pondcrete and Saltcrete Sampling and Analysis Plan. The results from the analysis of the rinsate blanks are not included in the Pondcrete Waste Characterization Report, but can be easily added to the database. However, the impact of all rinsate blank analytical results on the corresponding sample results

has been evaluated during the data validation process. Therefore, the effect on the results presented in the Pondcrete Waste Characterization Report from contamination found in rinsate blanks has been evaluated and qualified accordingly.

Samples were not heated during collection. The basis for the reviewer's comments concerning heating of the samples is not documented, nor is it justified.

3. Inter-analyte Trends and Outliers.

Lockheed asserts that "this bimodal distribution indicates that the population was misspecified." Given the populations defined for pondcrete reprocessing (triwalls and metal containers), the data merely indicate a certain amount of heterogeneity of the data. The presence of positive detections and non-detects in a population of waste analytical data is not unexpected.

RECOMMENDATIONS

The suggestion that the QA procedures can be improved ignores the fact that the samples were maintained under chain-of-custody following sampling, the samples were analyzed by approved methods as per the Sampling and Analysis Plan, CLP-equivalent deliverables were generated for DQO level IV data, and the data were validated using EPA-approved guidelines. The data generated are equivalent to the data generated by EPA under the CLP program and can be considered legally defensible.

The issue of specifying populations has already been discussed. HALLIBURTON NUS totally disagrees with Lockheed's assertion that the populations have been misspecified. Instead, HALLIBURTON NUS believes that Lockheed does not understand the nature of the

reprocessing project and has not fully read the planning documents, resulting in erroneous assertions that permeate the comment document. The only feasible definition of populations to support reprocessing is as specified in the HALLIBURTON NUS reports.

CONCLUSIONS AND DISCUSSION

See the previous paragraph for a discussion of the populations chosen for analysis.

Lockheed states that "It seems apparent that the HALLIBURTON NUS data were gathered in an attempt to determine the causes of poor performance of the pondcrete mixture." This was a minor goal of the project. The specific goals in support of the pondcrete reprocessing project were stated in the HALLIBURTON NUS reports.

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**APPENDIX II - METHOD 1311
 TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)**

1.0 SCOPE AND APPLICATION

- 1.1 The TCLP is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.
- 1.2 If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.
- 1.3 If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be equal to or above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.
- 1.4 If an analysis of extract obtained using a bottle extractor shows that the concentration of any regulated volatile analyte equals or exceeds the regulatory level for that compound, then the waste is hazardous and extraction using the ZHE is not necessary. However, extract from a bottle extractor cannot be used to demonstrate that the concentration of volatile compounds is below the regulatory 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 waste. A special extractor vessel is used when testing for volatile analytes. (See Table 1

Approvals: Lab Operations Mgr. - PAL: *Jane Semani* 11/6/90
 Lab Operations Mgr. - HAL: *Scott Mayo* 11/12/90
 Quality Assurance Coordinator: *Patricia McNamee* 11/5/90

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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

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

4.0 APPARATUS AND MATERIALS

4.1 Agitation Apparatus

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

4.2 Extraction Vessels

4.2.1 Zero-Headspace Extraction Vessel (ZHE)

This device is used only when the waste is being tested for the mobility of volatile analytes (i.e., those listed in Table 1). The ZHE (see Figure 2) allows for liquid/solid separation within the device, and effectively precludes headspace. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel. (See section 4.3.1.) The vessels shall have an internal volume of 500-600 mL and be equipped to accommodate a 90-110 mm filter. The devices contain VITON® O-rings which should be replaced frequently. Suitable ZHE devices known to EPA are identified in Table 3.

¹ VITON® is a registered trademark of DuPont.

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

The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure guard, pressurize the device to 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 procedures (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 to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel.

The extraction bottles may be constructed from various materials, depending on the analytes of interest and the nature of the waste. (See section 4.3.3.) It is recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles, other than polytetrafluoroethylene (PTFE), shall not be used if organics are to be determined. Bottles are available from a number of laboratory suppliers.

4.3 Filtration Devices

NOTE: Filtrations are to be performed in a hood.

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4.3.1 Zero-Headspace Extractor Vessel (ZHE)

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

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

4.3.2 Filter Holder

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

4.3.3 Materials of Construction

Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb waste components. Glass, polytetrafluoroethylene (PTFE), or 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.

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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 are identified in Table 5. Prefilters must not be used. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses with reagent water. (A minimum of 1 L per rinse is recommended.) Glass fiber filter are fragile and should be handled with care.

4.5 pH Meters

The meter should be accurate to within ± 0.05 units at 25°C.

4.6 ZHE Extract Collection Devices

TEDLAR^{®2} bags or glass, stainless steel or PTFE gag-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 60 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.

² TEDLAR[®] is a registered trademark of DuPont.

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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 0.01 grams.)

4.9 Beaker or Erlenmeyer flask, glass, 500 mL

4.10 Watchglass, appropriate diameter to cover beaker or Erlenmeyer flask

5.0 REAGENTS

5.1 Reagent Grade Chemicals

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

Reagent water is defined as water in which an interferent is not observed at or above the methods detection limits of the analyte(s) of interest. For nonvolatile extractions, ASTM Type II water or equivalent meets the definition of reagent water. Reagent water for volatile extractions is generated by passing deionized water through a carbon filter bed containing about 500 grams of activated carbon (Calgon Corporation, Filtrasorb-300 or equivalent).

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

5.4 Nitric acid (1N), HNO₃, made from ACS reagent grade.

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

5.6 Glacial acetic acid, CH₃CH₂OOH, ACS reagent grade.

5.7 Extraction fluid

5.7.1 Extraction Fluid #1: Add 5.7 mL glacial acetic acid to 500 mL of reagent water, add 64.3 mL of 1N NaOH, and dilute to 1 liter. When correctly prepared, the pH of this fluid will be 4.93±0.05. Verify the pH of the fluid each time it is prepared. Discard the fluid if it is not within the stated pH range.

5.7.2 Extraction Fluid #2: Dilute 5.7 mL glacial acetic acid with reagent water to 1 liter. When correctly prepared, the pH of this fluid will be 2.88±0.05. Verify the pH of the fluid each time it is prepared. Discard the fluid if it is not within the stated pH range.

5.8 Analytical Standards:

Analytical standards for matrix spiking cocktails shall be prepared according to the appropriate analytical method.

6.0 SAMPLE PRESERVATION AND HANDLING

6.1 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 procedures. 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.

6.2 Preservatives shall not be added to samples before extraction.

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

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- 6.4 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. Samples are stored at 4°C and opened only immediately prior to extraction.
- 6.5 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 6.3 if precipitation occurs.) Extracts for other analytes are stored at 4°C. Extracts or portions of extracts for volatile organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses.
- 6.6 See section 8.0 for 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. The following characteristics are evaluated:

- Percent Solids (7.1.1)
- Particle Size (7.1.2)
- Appropriate Extraction Fluid (7.1.3)

7.1.1 Does the Sample Contain Significant 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.

If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids) proceed to section 7.1.2 to determine if particle size reduction is required.

If the sample is liquid or multiphasic, perform liquid/solid separation as described below to make a preliminary determination of percent solids. This involves the filtration device described in section 4.3.2.

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7.1.1.1 Liquid/Solid Separation and Determination of Percent (Wet) Solids

- a. Pre-weigh the filter and the container that will receive the filtrate.
- b. Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid-wash the filter when metals are target analytes and this sample aliquot may be used for the TCLP extraction.

NOTE: Acid-washed filters may be used for all non-volatile extractions even when metals are not target analytes.

- c. Weigh out a subsample of the waste (100 gram minimum) and record the weight.
- d. 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 is decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.
- e. 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.

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NOTE: If more than 1% of the mass of the waste material 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 7.1.1.1c 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.

- f. 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 7.1.1.1c, 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.

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- g. Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see section 7.1.1.1a) 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 7.1.1.1c or e.

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

$$\text{Percent solids} = \frac{\text{Weight of solid (section 7.1.1.1a)}}{\text{Total weight of waste (section 7.1.1.1c/7.1.1.1e)}} \times 100$$

- h. If the percent solids determined in section 7.1.1.1g is <0.5%, proceed to section 7.2.2 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.

If the percent solids is $\geq 0.5\%$ and a small amount of the filtrate is entrained in wetting of the filter, proceed to 7.1.1.2, Determination of Percent Dry Solids. Otherwise, proceed to 7.1.2 to determine if particle size reduction is required.

7.1.1.2 Determination of Percent Dry Solids

- a. Remove the solid phase and filter from the filtration apparatus.
- b. Dry the filter and solid phase at $100 \pm 20^\circ\text{C}$ until two successive weighings yield the same value within $\pm 1\%$.

NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. The drying oven must be vented to a hood or other appropriate device.

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- c. Calculate the percent dry solids as follows:

$$\% \text{ dry solids} = \frac{(\text{Weight of dry waste + filter}) - \text{tared weight of filter}}{\text{Initial weight of waste (section 7.1.1.1c/7.1.1.1e)}} \times 100$$

- d. If the percent dry solids is <0.5%, 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 $\geq 0.5\%$, return to 7.1.1.1 and, with a fresh portion of waste, determine whether particle size reduction is necessary (section 7.1.2).

7.1.2 Is Particle Size Reduction Required?

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 per gram ratio 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 7.3.4.)

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.

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7.1.3 Which Extraction Fluid is Appropriate?

7.1.3.1 Nonvolatile Analytes

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:

- a. 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.
- b. 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.
- c. If the pH from the previous step 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.
- d. 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

7.1.3.2 Volatile Analytes

TCLP extraction for volatile constituents uses only extraction fluid #1 (section 5.7.1).

7.1.4 Is a Fresh Sample Aliquot Required for TCLP Extraction?

If the aliquot of the waste used for the preliminary evaluation was determined to be 100% solids, it can be used for the section 7.2 bottle extraction of nonvolatile analytes (assuming at least 100 grams remain)

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and the section 7.3 zero headspace extraction of volatile analytes (assuming at least 25 grams remain). However, if the aliquot was subjected to liquid/solid separation, another aliquot shall be used for the volatile extraction procedure in section 7.3. The aliquot of the waste subjected to liquid/solid separation is appropriate for use for the section 7.2 bottle extraction of nonvolatile analytes if an adequate amount of solid (as determined by section 7.1.1.1g) was obtained. The amount of the solid necessary is dependent upon whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solids remain, proceed directly to section 7.2.10.

7.2 TCLP Bottle Extraction for Nonvolatile Analytes

Refer to section 8.0 for quality assurance requirements.

A minimum sample size of 100 grams (solid and liquid phases) is recommended. A larger sample size will frequently be required, depending on the solids content of the waste sample, 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.

7.2.1 Samples with 100% Solids

If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids), weigh out a subsample of the waste (100 gram minimum) and proceed to section 7.2.3.7.

7.2.2 Samples with <0.5% Solids

When the sample contains <0.5% dry solids, the liquid portion of the waste, after filtration, is defined as the TCLP extract. If extract volume from section 7.1.1.1h is insufficient to support all of the required analyses, filter additional sample to obtain the desired extract volume. Proceed to section 7.2.4.

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7.2.3 Samples with $\geq 0.5\%$ Solids and $< 100\%$ Solids

If an adequate amount of solids was obtained from the preliminary percent solids determination, proceed to 7.2.3.7. Otherwise, proceed as follows.

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

7.2.3.2 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 metals are target analytes. (See section 4.4.)

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

7.2.3.3 Weigh out a subsample of the waste (100 gram minimum) and record the weight. Use the percent solids information obtained in section 7.1.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids must be generated by filtration to support the analyses to be performed on the TCLP extract.

7.2.3.4 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 is decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.2.3.5 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.

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NOTE: If more than 1% of the mass of the waste material 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 7.2.3.3 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.

7.2.3.6 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. Store the liquid phase at 4°C.

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, 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.

7.2.3.7 If particle size reduction of the solid was needed in section 7.1.2, proceed to section 7.2.3.8.

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If particle size reduction was not needed in section 7.1.3, 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 7.2.3.9.

7.2.3.8 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 7.1.2. 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, use a Teflon-coated sieve to avoid contamination of the sample.

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

$$\text{Wt of extra. fluid} = \frac{20 \times \% \text{ solids (sec 7.1.1)} \times \text{wt of waste filtered (sec 7.2.3.3/7.2.3.5)}}{100}$$

Slowly add this amount of appropriate extraction fluid (see section 7.1.3) to the extractor vessel. Close the extractor bottle tightly (Teflon tape can 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 $23 \pm 2^\circ\text{C}$ during the extraction period.

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NOTE: As agitation continues, pressure may build 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.

7.2.3.10 Following the 18 ± 2 hour extraction, allow the sample to settle. 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 7.2.3.5. 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 metals will be determined.

7.2.3.11 Prepare the TCLP extract as follows:

- If the waste contained no initial liquid phase (i.e., is 100% solids), the filtered liquid extracted from the solid material is defined as the TCLP extract. Proceed to section 7.2.4.
- If the filtered liquid extracted from the solid material and the initial liquid phase of the sample are compatible (e.g., multiple phases will not result on combination), combine the filtered liquids. This combined liquid is defined as the TCLP extract. Proceed to section 7.2.4.
- If the initial liquid phase of the sample is not or may not be compatible with the filtered liquid extracted from the solid material, do not combine these liquids. These liquids, collectively defined as the TCLP extract, are analyzed separately and the results combined mathematically, as described in section 7.2.4.3.

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7.2.4 TCLP Extract Preservation and Analysis

7.2.4.1 Following collection of the TCLP extract, measure and record the pH of the extract. 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, 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.

7.2.4.2 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 not hazardous.

Semivolatile analyses are routinely performed at a 1:10 dilution (performed during extraction or analysis) to minimize the affects of the acetic acid matrix on instrument function. To increase sensitivity, semivolatile analyses are performed by GC, with confirmation of positive results by reanalysis on a dissimilar column.

7.2.4.3 If the individual liquid phases were analyzed separately, combine the results mathematically by using a simple volume-weighted average:

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

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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).

7.2.4.4 Analyte concentrations in the TCLP extract, corrected for matrix spike recovery (see Section 8.0), are compared with the levels identified in the appropriate regulations to determine if the waste exhibits the toxicity characteristic.

7.3 TCLP Extraction of Volatile Analytes by Zero Headspace Extraction (ZHE)

Refer to section 8.0 for quality assurance requirements.

Use the ZHE device to obtain TCLP extract for analysis of volatile compounds only. Extracts resulting from the use of the ZHE shall not be used for the determination 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. If necessary to obtain adequate leachate volumes, perform multiple extractions.

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 is done when the sample is cold (4°C) to minimize loss of volatiles.

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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. 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. 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 Determine the amount of sample to be extracted based on the information obtained in section 7.1.1 and the following.

7.3.3.1 Samples with 100% Solids

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 7.3.4.

7.3.3.2 Samples with <5% Dry Solids

If the waste contains <5% dry solids (section 7.1.1.2), 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. A 500 gm aliquot is recommended. Weigh out the aliquot and record the weight.

7.3.3.3 Samples with $\geq 5\%$ Solids and <100% Solids

For wastes containing $\geq 5\%$ and 100% dry solids (section 7.1.1.2), use the percent solids information obtained in section 7.1.1 to determine the optimum sample size to charge into the ZHE. Determine the amount of waste to charge into the ZHE as follows:

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

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

- 7.3.4 If particle size reduction of the solid portion of the waste was required in section 7.1.2, proceed as follows. If particle size reduction was not required in section 7.1.2, proceed to section 7.3.5.

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 7.1.2. 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 or 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 materials. Actual measurement of surface area is not recommended.

When the surface area or particle size has been appropriately altered, proceed to section 7.3.5.

- 7.3.5 Waste slurries are not allowed to stand to permit the solid phase to settle. Do not centrifuge wastes prior to filtration.
- 7.3.6 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

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NOTE: If more than 1% of the mass of the waste material has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in section 7.3.3 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, 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 7.3.10.

7.3.7 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. If solids are less dense than the extraction fluid, invert the device to prevent the filter from becoming clogged. 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.

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7.3.8 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase. Store the liquid phase at 4°C under minimal headspace conditions 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 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 <5% dry solids (see section 7.1.1.2), this filtrate is defined as the TCLP extract and is analyzed directly. Proceed to section 7.3.13.

7.3.9 Determine the weight of extraction fluid #1 to add to the ZHE as follows. Extraction fluid #1 is used in all cases.

$$\text{Wt of extrac. fluid} = \frac{20 \times \% \text{ solids(sec 7.1.1)} \times \text{wt. of waste filtered(sec 7.3.3.2) (7.3.6)}}{100}$$

7.3.10 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.

7.3.10.1 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 must 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.

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7.3.10.2 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.

7.3.10.3 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 $22 \pm 3^\circ\text{C}$ during agitation.

7.3.11 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 is compatible with the liquid extracted from the solid material, 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 7.3.7. All extract shall be filtered and collected if the TEDLAR® bag is used, if the extract is multiphase, or if the waste contained an initial liquid phase. (See sections 4.6 and 7.3.1.)

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

7.3.12 If the original waste contained no initial liquid phase (i.e., is 100% solids), the filtered liquid material obtained from section 7.3.11 is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained from section 7.3.11 and the initial liquid phase (section 7.3.7) are collectively defined as the TCLP extract.

7.3.13 Following collection of the TCLP extract, immediately prepare the extract for analysis (i.e., transfer leachate to a VOA vial with minimal agitation) and store with minimal headspace at 4°C until analyzed. If the individual phases are not miscible, determine and record the volume of the individual phases to ±0.5%.

7.3.14 The TCLP extract is analyzed according to the appropriate analytical methods. ZHB leachates are routinely run at a 1:10 dilution to minimize the affects of the acetic acid matrix on the instrument function.

7.3.15 If the individual liquid phases were analyzed separately, combine the results mathematically by using a simple volume-weighted average:

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

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).

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7.3.16 Analyte concentrations in the TCLP extract, corrected for matrix spike recovery (see Section 8.0), are compared with the levels identified in the appropriate regulations to determine if the waste exhibits the toxicity characteristic.

8.0 QUALITY CONTROL REQUIREMENTS

8.1 Leachate Blanks

8.1.1 Bottle Extractions

At a minimum, extract one leachate blank for every 20 samples extracted using extraction fluid #1, and one leachate blank for every 20 samples extracted using extraction fluid #2. These blanks must be analyzed for all of the 40CFR Part 261 parameters. (See Table 1.) If any additional parameters are requested, a leachate blank to be run for these additional parameters only must be extracted with the samples. Use each bottle extraction vessel one time only, then clean thoroughly and discard.

8.1.2 ZHE Extractions

At a minimum, extract one method blank for every 20 extractions performed in an extraction vessel, using extraction fluid #1. These blanks must be analyzed for all of the 40CFR Part 261 parameters. (See Table 1.) If any additional parameters are requested, a method blank to be run for those parameters only must be extracted with the samples.

8.2 Matrix Spikes

A matrix spike shall be performed upon client request for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) from each client site. Note: If the result for a waste type exceeds the regulatory level and the data is being solely to demonstrate that the waste property exceeds the regulatory level, a matrix spike is not required by the EPA. The bias determined from the matrix spike determination is then used to correct the measured values for the client's samples of similar waste type. (See sections 8.2.4 and 8.2.5.)

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8.2.1 Matrix spikes are to be added for all target analytes after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

Mercury will be spiked into a separate aliquot of leachate, as will toxaphene, to avoid plating and coelution problems, respectively.

8.2.2 In most cases, matrix spikes should be added at the concentration shown on Table 6. The spike concentration may be as high as the regulatory level, but may not be less than five times the method 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 will be analyzed for the unspiked sample.

8.2.3 Matrix spike recoveries are calculated by the following formula:

$$\%R (\% \text{ Recovery}) = 100 (X_s - X_u)/K$$

where:

X_s = measured value for the spiked sample

X_u = measured value for the unspiked sample, and

K = known value of the spike in the sample

If the amount of analyte spiked in is $\leq 1/5$ of the amount of analyte in the unspiked leachate, bring this to the Account Executive/Project Manager's attention.

Report the unspiked result and matrix spike recovery for the sample numbers associated with the unspiked sample and matrix spike, respectively.

8.2.4 The client must then correct the unspiked value for analytical bias using the following formula:

$$X_c = 100 (X_u/\%R)$$

where:

X_c = corrected value, and

X_u = measured value of the unspiked sample

8.3 Sample Analysis QC Requirements

TCLP leachates will be analyzed in batches by themselves with the following quality control checks:

- If a batch of leachates includes a leachate blank, an additional method blank need not be run. The blank must be analyzed for all target analytes. However, if there is not a leachate blank in a batch of leachates or the blank was prepared for only a subset of the target analytes, a method blank must be prepared and run.
- Laboratory control standards or surrogate standards must be run. Separate surrogate standard control limits will be calculated for TCLP leachates, based on TCLP recoveries.

Duplicate analysis and matrix spike analysis in addition to the spikes performed in 8.2 are not run with each batch of leachates.

8.4 Holding Times

Samples must undergo TCLP extraction within the following time periods:

	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
Semivolatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360

NA = Not applicable.

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

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TABLE 1
 40CFR PART 261 ANALYTES^a

Compound	CAS No.	Regulatory Level (mg/L)
VOLATILES		
Benzene ¹	71-43-2	0.5
Carbon tetrachloride ¹	56-23-5	0.5
Chlorobenzene ¹	106-90-7	100
Chloroform ¹	67-66-3	6
1,2-Dichloroethane ¹	107-06-2	0.5
1,1-Dichloroethylene ¹	75-35-4	0.7
Methyl ethyl ketone ¹	78-93-3	200
Tetrachloroethylene ¹	127-18-4	0.7
Trichloroethylene ¹	79-01-6	0.5
Vinyl chloride ¹	75-01-4	0.2
SEMIVOLATILES		
o-Cresol	95-48-7	200
m-Cresol	108-39-4	200
p-Cresol	106-44-5	200
1,4-Dichlorobenzene	106-46-7	7.5
2,4-Dinitrotoluene	121-14-2	0.13
Hexachlorobenzene	118-74-1	0.13
Hexachlorobutadiene	87-68-3	0.5
Hexachloroethane	67-72-1	3
Nitrobenzene	98-95-3	2
Pentachlorophenol	87-86-5	100
Pyridine	110-86-1	5
2,4,5-Trichlorophenol	95-95-4	400
2,4,6-Trichlorophenol	88-06-2	2

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TABLE 1
40CFR PART 261 ANALYTES
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Compound	CAS No.	Regulatory Level (mg/L)
PESTICIDES		
Chlordane	57-74-9	0.03
Endrin	72-20-8	0.02
Heptachlor	76-44-8	0.008
Lindane	58-89-9	0.4
Methoxychlor	72-43-5	10
Toxaphene	8001-35-2	0.5
HERBICIDES		
2,4-D	94-75-7	10
2,4,5-TP	93-72-1	1
METALS		
Arsenic	7440-38-2	5
Barium	7440-39-3	100
Cadmium	7440-43-9	1
Chromium	7440-47-3	5
Lead	74-39-92-1	5
Mercury	7439-97-6	0.2
Selenium	7782-49-2	1
Silver	7440-22-4	5

Notes:

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

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TABLE 2
SUITABLE ROTARY AGITATION APPARATUS¹

Company	Location	Model No.
Analytical Testing and Consulting Services, Inc.	Warrington, PA (216) 343-4490	4-vessel (DC20S) 8-vessel (DC20) 12-vessel (DC208)
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	Whitemore Lake, MI (313) 449-4116	10-vessel (01VRE) 5-vessel (5VRE)
Millipore Corporation	Bedford, MA (800) 225-3384	4-ZHE or 4 1-liter bottle extractor (YT30ORAWH)

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

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TABLE 3
SUITABLE ZERO-HEADSPACE EXTRACTOR VESSELS¹

Company	Location	Model No.
Analytical Testing and 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	Whitenoire 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

¹ Any device that meets the specifications listed in section 4.2.1 of the method is acceptable.

² This device uses a 110 mm filter.

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TABLE 4
SUITABLE FILTER HOLDERS¹

Company	Location	Model/Catalogue No.	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	YT30142HS XXK1004700	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.

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TABLE 5
SUITABLE FILTER MEDIA¹

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

¹ Any filter that meets the specifications in section 4.4 of the Method is suitable.

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TABLE 6
TCLP MATRIX SPIKING LEVELS¹

Analyte	Spiking Level
VOLATILES	
Benzene	0.20
Carbon Tetrachloride	0.20
Chlorobenzene	0.20
Chloroform	0.20
1,2-Dichloroethane	0.20
1,1-Dichloroethylene	0.20
Methyl Ethyl Ketone	0.20
Tetrachloroethylene	0.20
Trichloroethylene	0.20
Vinyl Chloride	0.20
SEMIVOLATILES	
o-Cresol	1.0
m/p-Cresol	1.0
1,4-Dichlorobenzene	1.0
2,4-Dinitrotoluene	0.13
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.50
Hexachloroethane	1.0
Nitrobenzene	1.0
Pentachlorophenol	1.0
Pyridine	1.0
2,4,5-Trichlorophenol	1.0
2,4,6-Trichlorophenol	1.0

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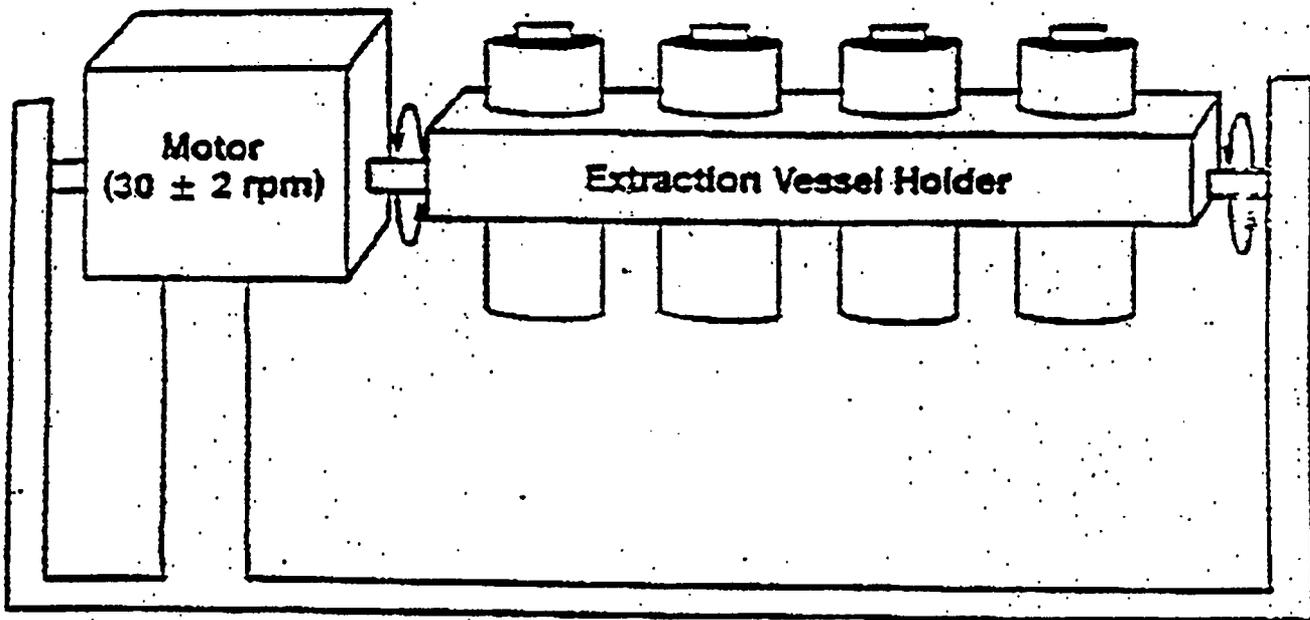
TABLE 6
TCLP MATRIX SPIKING LEVELS¹
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Analyte	Spiking Level
PESTICIDES	
Chlordane	0.0020
Endrin	0.0020
Heptachlor	0.0020
Lindane	0.0020
Methoxychlor	0.0120
Toxaphene*	0.0020
HERBICIDES	
2,4-D	0.0120
2,4,5-TP	0.0030
METALS	
Arsenic	0.20
Barium	20.0
Cadmium	0.50
Chromium	2.0
Lead	5.0
Selenium	1.0
Silver	5.0
Mercury*	0.05

* These analytes must be spiked into separate aliquots of leachate to avoid analytical problems.

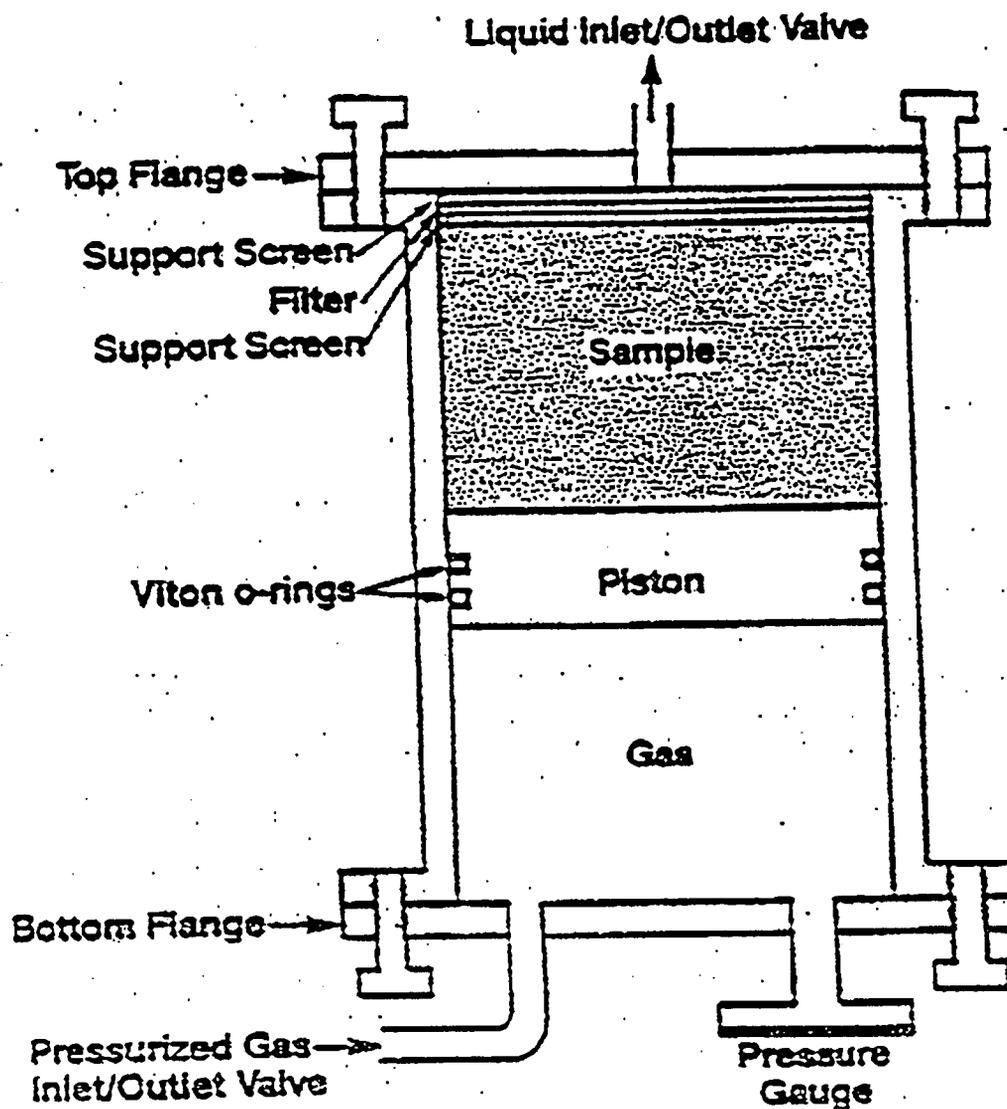
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FIGURE 1
ROTARY AGITATION APPARATUS



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FIGURE 2
ZERO-HEADSPACE EXTRACTOR (ZHE)



PRELIMINARY (11/6/92)
RESPONSE TO COMMENTS BY DOE EM-453
ON THE PONDCRETE WASTE CHARACTERIZATION REPORT

CRITICAL COMMENT

The following responses should clarify the logic used to select the number of pondcrete samples needed to characterize the population of billets:

- The sampling rationale presented in the Pondcrete Waste Characterization Report was a condensed version of the rationale presented in the Sampling and Analysis Plan for Pondcrete and Saltcrete, Combined Deliverable Number 211B, 211C, 221B, and 221C (HALLIBURTON NUS, October 1991). The reviewer is referred to the pertinent sections of this document for a better understanding of the sampling rationale (see attachment).
- As stated in the Sampling and Analysis Plan, previous pondcrete data was statistically analyzed to determine the relative standard deviations (RSDs) and the possible worst case concentrations (97.5% upper tolerance limits) of selected parameters. The RSD is a measurement of the variability of a parameter in a population. The sample size required to characterize a population to a specified confidence level is related to the variability of the population. For example, a population with higher variability would require more samples to characterize it than a population with lower variability for the same level of confidence.

The main objective of the waste characterization program was to support the development of a successful stabilization recipe, not to characterize every single chemical constituent to the same level of confidence regardless of its importance to the project.

The data showed that some parameters had relatively high RSDs. However, not all parameters are of equal importance as they relate to the development of stabilization recipes. More specifically, many parameters that had high RSDs were not present at concentrations that were of concern, either from a regulatory standpoint or a chemical engineering standpoint for cement stabilization. Therefore, it was not a sound engineering approach to base the sampling program on parameters that had little relevance to the problem. Instead, it was determined that the most likely mode of failure of the stabilization mixes would involve the leaching of hazardous constituents, using the TCLP test, above regulatory standards, in this case the applicable Land Disposal Restrictions (LDRs). The leachate concentrations were conservatively estimated for key parameters and then compared to the parameter's LDR standard. This comparison allowed an evaluation of which parameters had the highest probability of exceeding their respective standards. These parameters would therefore be the most important for the development of a successful stabilization recipe.

For both triwalls and metal containers, cadmium had the highest leachate/standard ratio, by at least an order of magnitude over the next contaminant. Cadmium was thus chosen as the parameter whose statistical data would be used to determine the number of samples to characterize the populations of pondcrete. All other contaminants were not present at concentrations that were of regulatory or engineering concern. If a contaminant of lesser

concern but higher RSD was used to estimate the number of samples for pondcrete characterization, excess samples would have been required, with no benefit to the project.

SPECIFIC COMMENTS

1. The first goal stated in Section 1.3 was to "characterize the two population forms of pondcrete." This statement can be expanded to include an assessment of regulatory compliance, including the LDR standards applicable to the waste.
- 2,3. Please see the response to the CRITICAL COMMENT and the section of the Sampling and Analysis Plan appended to this memorandum.
4. HALLIBURTON NUS was not provided with production data, therefore it is not possible to accurately determine whether the production dates of the sampled billets are proportional to the rate of production. This problem is further exacerbated by the lack of accumulation date data for some of the billets.
5. This comment is acknowledged. However, the two sections were presented in the order in which they appear because the laboratory analysis (Section 2.3.1) precedes data validation (Section 2.4). As a compromise, we propose adding references to Section 2.4 in Section 2.3.1 where definitions are required.
6. The commenter reviewed the Internal Draft of the subject report. The sentence referenced in this comment no longer appears in Revision 0, which was issued in September 1992. Section 4.2 in Revision 0 discusses the salt content of the two pondcrete populations.
7. The referenced statement from the Internal Draft report no longer appears in Revision 0. The average data do not show a significant difference between the two populations of pondcrete for moisture (gravimetric) or cement constituents (calcium, iron, aluminum). However, the chemical data are not capable assessing the efficiency of the mixing and cement hydration, both of which are key to producing a stable waste form. It should also be noted that both populations of pondcrete were apparently produced with a high water/cement ratio, indicating that all pondcrete was probably deficient in cement content.
8. Section 4.2 has been modified in Revision 0. The data indicate that the two populations are similar based on comparison of average data for key parameters. Since the regulatory concerns were already discussed in Section 4.1, and considering the similarities presented in Section 4.2, the statement concerning methanol is reasonable.
9. The triwall sample that exceeded the LDR standard for amenable cyanide was PC-11500-T-D, which was produced on April 7, 1988. It should be noted that the duplicate of this sample did not exceed the LDR standard. The nearest triwall sample was PC-12503-T, which was produced April 18, 1988. This sample, as with all the other triwall samples, did not exceed the LDR standard for amenable cyanide. The method specified by SW846 shows that the triwall population as a whole does not exceed the LDR standard, and that no further sampling is required.

1.0. PROJECT DESCRIPTION

This plan describes the requirements for sampling several waste forms located at the Rocky Flats Plant in support of the Solar Pond/Pondcrete Stabilization project being conducted by HALLIBURTON NUS Environmental Corporation (HALLIBURTON NUS). The waste forms of concern are as follows:

- Solar pond sludge and water
- Pondcrete tri-walls
- Saltcrete tri-walls
- Clarifier sludge and water
- Evaporator bottoms (not currently available)

This Sampling and Analysis Plan will only address the Pondcrete and Saltcrete. The remaining waste sources are addressed in the Waste Sampling and Analysis Plan for Pond Sludge and Water, and Clarifier Sludge and Water (HALLIBURTON NUS, 1991).

The following sections contain descriptions of the waste forms to be sampled, the scope of sampling activities, and sampling strategy and rationale.

1.1 Site Description

1.1.1 Pondcrete

Department of Energy (DOE)/Rockwell began phasing out use of the solar evaporation ponds in the early 1980's because of environmental concern. The plan for cleanup of the ponds was to drain and treat the liquid waste and to mix the pond sediments/sludges with cement. The resulting solidified material known as Pondcrete was to be disposed of at DOE's Nevada Test site (NTS).

Clean out of the largest surface impoundment (Pond 207A) began in 1985 with a pugmill process. The sludge from the bottom of the pond was pumped to a clarifier where it was allowed to settle out before being pumped to the pugmill. Cement was added to the sludge and mixed to a desired consistency by paddles attached to the auger shaft. The Pondcrete mixture was then fed through a chute into lined tri-walls. Improper mixing of cement and sludge resulted in some Pondcrete blocks that did not solidify properly or crumbled and cracked during storage pursuant to disposal at NTS.

Since the discovery of the Pondcrete problems in May 1988, DOE has not cleaned up any additional sediment from the solar ponds. Approximately 2,000 Pondcrete blocks had already been buried at NTS prior to the discovery of the problems. Since that time, 8,666 blocks have been inspected, approved, repackaged, and shipped to the test site for storage; and 8,031 blocks are awaiting remixing and repackaging. Substantial additional work remains to be done to clean up the ponds.

The Pondcrete blocks awaiting reprocessing are currently being stored in tension membrane structures (tents) on the 750 and 904 pad areas. Approximately 2550 of the Pondcrete blocks failed to solidify properly and the tri-wall containers are being stored in metal containers.

Pondcrete is a mixture of cement and sludge material generated from evaporating wastewater and is very high in salts, primarily calcium and potassium salts, with some sodium salts. Pondcrete has been sampled and analyzed several times for numerous compounds and parameters. The following provides a brief description of the chemical characterization of Pondcrete (Rockwell International, 1989).

Volatiles

Only five volatile compounds registered above detection limits (ADL) in any of the Pondcrete samples analyzed. Information on those analytes are summarized as follows:

Volatile Analyte	Number of ADL Readings	Average of ADL Readings	Range of ADL Readings
Methylene Chloride	3* of 30	16.9 ppb	7.3 to 35 ppb
Acetone	20 of 30	39.7 ppb	11 to 180 ppb
2-Butanone	9 of 30	16.7 ppb	12 to 23 ppb
Tetrachloroethene	10 of 30	20.2 ppb	5 to 73 ppb
1,1,2,2-Tetrachloroethane	1 of 30	160.0 ppb	----

- * A series of three other samples indicated very high methylene chloride concentrations but were not included in the ADL readings shown because of very high concentrations in the blank also.

Semivolatiles

Only four semivolatile compounds registered above detection limits in any of the Pondcrete samples analyzed. Information on those analytes is summarized as follows:

Semivolatile Analyte	Number of ADL Readings	Average of ADL Readings (ppb)	Range of ADL Readings (ppb)
2-Nitroaniline	1 of 30	970	----
Di-n-Butyl phthalate	1 of 30	590	----
Fluoranthene	8 of 30	722	374-1,683
Bis(2-ethylhexyl)phthalate	12 of 30	4,497	152-14,949

Pesticides/PCBs

Of the two samples tested, all concentrations were below detection limits for the pesticide/PCB analytes.

Metals

Total metal analysis was performed on six Pondcrete samples. The results are summarized as follows:

Metals	Average Concentration (ppm)	Range of Concentrations (ppm)
Aluminum	27,330	16,820-33,400
Arsenic	8.98	4.11-24.6
Barium	600	205-2,000
Beryllium	54	1.16-77.6
Cadmium	390	8.16-590
Calcium	371,280	243,300-577,180
Chromium	278	176-420
Cobalt	30.9	20.9-33.8
Copper	155	23.4-236
Iron	13,620	9,730-17,620
Lead	29.6	2.38-43.0
Magnesium	5,670	1,210-7,680
Manganese	2,090	804-6,910
Mercury	1.43	<0.02-2.32
Nickel	116	57.4-156
Potassium	157,840	9,470-329,300
Silver	13.4	6.63-23.4
Sodium	26,090	1,580-53,230
Vanadium	43.6	28.8-62.7
Zinc	113	62.1-210
Percent Solids	67.8%	44.4%-94%

Cyanides

Analyses for total cyanide and cyanide amenable to chlorination were performed on samples taken from five different blocks of Pondcrete. A duplicate sample was taken from one of the blocks, therefore a total of six samples were analyzed. The results are summarized as follows:

Analysis	Average Concentration (ppm)	Range of Concentrations (ppm)
Total Cyanide	9.65	7.14-12.1
Amenable Cyanide	7.41	4.05-9.90

Radiochemistry

Radiochemistry analyses were performed on five Pondcrete samples. The results are summarized as follows:

Analysis	Average Concentration (pCi/g)	Range of Concentrations (pCi/g)
Gross Alpha	2,400	1,700-3,800
Gross Beta	38	12-53
Pu-239	750	130-1,800
Am-241	1,000	690-1,600
U-233, U-234	44	33-60
U-238	48	40-66
Tritium	1.7 pCi/mL	1.5-2.1 pCi/ml

Toxic Characteristic Leaching Procedure (TCLP)

- TCLP Volatiles.** Three Pondcrete samples were subjected to the TCLP and analyzed for 21 volatile compounds. These are the volatile compounds that appear in 40 CFR 268.41, Table CCWE (Constituent Concentrations Waste Extract), for F001 through F005 spent solvents. Only three constituents were observed at concentrations above the detection levels and in each case, this occurred in only one out of three results. The three compounds and their single concentration above detection levels are as follows:

Compound	TCLP Concentration (ppb)
1,1,1-Trichloroethane	8
Tetrachloroethane	5
Toluene	60

It should be noted that toluene was also detected in the blank at 23 ppb.

- **TCLP Semivolatiles.** The same three Pondcrete samples were analyzed for the semivolatile compounds that also appear on the Table CCWE for F001, F002, F003, and F005 spent solvents. None of the four compounds considered (cyclohexane, 1,2-dichlorobenzene, pyridene, and 2-nitropropane) were observed at concentrations above detection.

In addition to the TCLP, the Pondcrete was also tested for ignitability, corrosivity, reactivity, and EP toxic metals. Pondcrete did not test positive for ignitability, corrosivity, or reactivity. Only one EP toxic metal tested positive for the Pondcrete samples. The Pondcrete was found to be toxic for cadmium in eight of the 26 samples. In four of the eight readings, the average was 16.4 mg/l, with a range of 1.5 mg/l to 42 mg/l. The EP toxicity standard for cadmium is 1 mg/l.

Applicable EPA Hazardous Waste Numbers

The Pondcrete waste has its origin in a collection of wastewaters coming from approximately 30 different buildings, most of those

with multiple contributing streams. The applicable EPA hazardous waste numbers for Pondcrete are as follows:

Hazardous Waste Number	Description
D006	Toxic for cadmium
F001	Spent halogenated solvents used in degreasing
F002	Spent halogenated solvents
F003	Spent nonhalogenated solvents
F005	Spent nonhalogenated solvents
F006	Wastewater treatment sludges from electroplating operations
F007	Spent cyanide plating bath solutions from electroplating
F009	Spent stripping and cleaning bath solutions from electroplating operations where cyanides are used

1.1.2 Saltcrete

Saltcrete is generated by solidifying the nitrate salt residue from an evaporation process at the Liquid Waste Treatment Facility in Building 374. In very simplified terms, the 374 wastewater treatment operation can be broken into three processes. Depending on its radiological contamination and point of origin, wastewater can go straight into any one of the three treatment process; however, inside the facility, the processes are interrelated. The three basic processes are:

- (1) Evaporation
- (2) Flocculation/precipitation
- (3) Sludge dewatering

The flocculation/precipitation activity is designed for the removal of radioactive material. The settled sludge from this process goes to the sludge handling step and the overflow goes to the evaporator. The evaporator receives less contaminated wastewater directly. The residue or concentrated salt solution from the

evaporator is mixed with cement to immobilize particulates and remove the oxidizer and corrosive characteristics of the salt and/or concentrated salt solutions. The resulting waste form is referred to as Saltcrete (Rockwell International, 1989).

The wastewater now going to Building 374 includes that which previously went to the evaporation ponds from which Pondcrete was generated. Therefore, in general terms, the waste streams contributing to the formation of Saltcrete are similar to those identified for Pondcrete. Multiple sources/activities are involved (about 30 different buildings), generating wastewater with both radiological and hazardous chemical contaminants. The major distribution of wastewaters have radiological contamination below a specified level and are sent directly to the evaporator. Some of the processes generating wastewaters that are of particular concern from a RCRA standpoint include:

- (1) Various laboratory activities
- (2) Electroplating operations which include the use of cyanides
- (3) Metal machining/manufacturing including cleaning/degreasing with solvents
- (4) Acid and caustic cleaning/rinsing solutions

The analytical results from various sampling events are described in the following paragraphs.

Volatiles

Only six volatile compounds registered above detection limits in any of the 18 Saltcrete samples analyzed. Information on those analytes is summarized as follows:

Analyte	Number of ADL Readings	Average of ADL Readings	Range of ADL Readings
Acetone	15 of 15 ¹	168 ppb	89-380 ppb
2-Butanone	15 of 15 ¹	39 ppb	21-70 ppb
Benzene	1 of 15 ¹	26 ppb	----
Methylene chloride ²	2 of 18	14 ppb	7.7-20 ppb
Tetrachloroethene	2 of 18	7 ppb	6-8 ppb
Toluene	15 of 15 ¹	22 ppb	5.1-51 ppb

¹ The Appendix III volatile analyses of samples taken August 1988 did not include these compounds; hence only 15 readings.

² The volatile analyses of samples taken in August 1988 were all positive for this analyte, but because method and extract blanks were also positive at similar values, these values were not included as ADL readings.

Semivolatiles

Only three semivolatile compounds were detected above detection limits in any of the 18 Saltcrete samples analyzed. It should be noted that the semivolatile analyses of samples taken in August 1988 did not include any of the analytes observed ADL; therefore, the total number of readings is only shown as 15. Information on those analytes is summarized as follows:

Analyte	Number of ADL Readings	Average of ADL Readings	Range of ADL Readings
4-Chloro-3-methyl phenol	1 of 15	660 ppb	----
Butyl benzyl phthalate	1 of 15	3,530 ppb	----
Bis(2-ethylhexyl)phthalate	1 of 15	4,156	----

Metals

Total metal analyses were performed on only one Saltcrete sample. The results are as follows:

Metal	Concentration (ppm)
Aluminum	11,520
Antimony	<6.96
Arsenic	4.04
Barium	160
Beryllium	0.70
Cadmium	4.30
Calcium	182,390
Chromium	117
Cobalt	19.8
Copper	17.9
Iron	14,290
Lead	3.55
Magnesium	2,860
Manganese	606
Mercury	<0.02
Nickel	<0.02
Potassium	30.4
Selenium	<0.58
Silver	8.94
Sodium	4,870
Thallium	<1.16
Vanadium	38.3
Zinc	61.5

Cyanide

Analyses for total cyanide and cyanide amenable to chlorination were performed on samples taken from four different blocks of Saltcrete. A duplicate sample was taken from one of the blocks; therefore, a total of five samples were analyzed. The results are summarized as follows:

Analysis	Average Concentration (ppm)	Range of Concentrations (ppm)
Total Cyanide	15.5	12.6-18.5
Amenable Cyanide	13.2	6.2-18.2

Radiochemistry

Radiochemistry analyses were also performed on only a single Saltcrete sample. The results are as follows:

Analysis	Concentration (pCi/g)*
Gross Alpha	240 +/- 60
Gross Beta	170 +/- 60
Pu-239	160 +/- 10
Am-241	88 +/- 4
U-233, U-234	25 +/- 10
U-238	88 +/- 18
Tritium	1.3 +/- 0.3 (pCi/mL)

*Plus or minus (+/-) values indicate the 95 percent confidence range for the reported values.

RCRA Characteristics

TCLP analysis was conducted on three Saltcrete samples for volatiles, acids, and methanol (i.e., the compounds associated with F001 through F005 wastes). Acetone and methylene chloride were detected in the low ppb range (10 to 25 ppb) for the extract, however these compounds were also in the extract blank. Methyl isobutyl ketone, 2-butanone, and toluene had estimated readings below the detection limit of 10, 10, and 5 ppb, respectively.

EP Toxic Metals analysis were taken on 13 samples in April 1988. All analytical results for EP Toxic Metals except lead were below the following detection limits:

Metal	Detection Limit (ppm)	EP Toxicity Limit
Arsenic	0.10	5.0
Barium	1.0	100.0
Cadmium	0.05	1.0
Chromium	0.5	5.0
Lead	0.5	5.0
Mercury	0.005	0.2
Selenium	0.1	1.0
Silver	5.0	5.0

Lead was observed in a single sample at a concentration at the detection limit (0.5 ppm). Two samples taken within one month of each other in 1986 provided variable information. The first provided positive readings for five metals while the second had less than detectable for all eight metals. These results are summarized as follows:

Metal	Concentration (ppm)	
	1st 1986 Sample	2nd 1986 Sample
Barium	0.30	<20.0
Cadmium	0.092	<0.2
Chromium	2.99	<1.0
Lead	0.33	<1.0
Silver	0.050	<1.0

Saltcrete was also tested for ignitability, corrosivity and reactivity. The solidified material did not test positive for any of the above RCRA characteristics. However, Saltcrete that is not solidified would be considered ignitable and corrosive.

Applicable EPA Hazardous Waste Numbers

The applicable listed EPA waste numbers for Saltcrete are as follows:

Hazardous Waste Number	Description
F001	Spent halogenated solvents used in degreasing
F002	Spent halogenated solvents
F003	Spent nonhalogenated solvents
F005	Spent nonhalogenated solvents
F006	Wastewater treatment sludges from electroplating operations
F007	Spent cyanide plating bath solutions from electroplating
F009	Spent stripping and cleaning bath solutions from electroplating operations where cyanides are used

1.2 Scope of Work

The following waste forms will be sampled in support of the Solar Pond/Pondcrete Stabilization Project:

- Pondcrete
- Saltcrete

The Pondcrete is segregated into two subgroups for sampling. The tri-walls are considered one group and the tri-walls in the metal containers will be the second group.

Saltcrete is divided into three subgroups. The tri-walls are one subgroup, tri-walls in metal containers are a subgroup, and 1/2 crates are a subgroup.

The purpose of the sampling effort is to obtain a sufficient number of samples to characterize each waste form. Specific goals of the waste characterization effort are as follows:

- To develop an analytical profile of each waste form such that, within a specified statistical confidence limit, each waste form can be characterized as a single population.
- To determine specific analytes that are known or suspected to be deleterious to cement chemistry reactions.
- To develop analytical values for specific analytes such that the capture efficiency of the final waste/cement formulations can be evaluated.
- To determine selected physical characteristics of the samples collected.

Additionally, samples of each waste form will be collected for treatability studies which will be conducted at the HALLIBURTON NUS laboratory in Pittsburgh, Pennsylvania. Details of the proposed treatability study are included in the Treatability Study Work Plan (HALLIBURTON NUS, 1991).

Table 1-1 provides a summary of the number of billets that will be sampled and the associated volumes that will be collected to accomplish the goals of the Sampling and Analysis Plan for waste characterization.

1.3 Sampling Strategy and Rationale

1.3.1 Introduction

In general, the goal of a sampling program is to collect a small but informative portion of the population being investigated. A representative sample is a sample that can be expected to adequately reflect the properties of interest of the entire media being sampled. As an integral part of the waste characterization and treatability studies, the objective of the sampling program of

**TABLE 1-1
VOLUME OF MEDIA TO BE COLLECTED DURING PONDCRETE AND SALTCRETE SAMPLING
ROCKY FLATS FACILITY**

TABLE 1-1					TABLE A			
WASTE SOURCE	LOCATION	TOTAL DISCRETE SAMPLE COLLECTED VOLUME PER BILLET	TOTAL DISCRETE VOLUME SHIPPED TO HALLIBURTON MWS FOR CHARACTERIZATION	TOTAL DISCRETE VOLUME TO BE COMPOSITED FOR TREATABILITY STUDY	TOTAL VOLUME OF COMPOSITE SAMPLE	VOLUME OF COMPOSITE SAMPLE TO BE STORED AT ROCKY FLATS	VOLUME OF COMPOSITE SAMPLE TO BE SHIPPED TO HALLIBURTON MWS FOR TREATABILITY STUDY	VOLUME OF COMPOSITE SAMPLE TO BE SHIPPED TO HALLIBURTON MWS FOR CHARACTERIZATION
Pondcrete	Tri-walls (16)	3.5 gal	2 gal	1.5 gal	24 gal	12 gal	10 gal	2 gal
Pondcrete	Tri-walls in Metals (40)	3 gal	2 gal	1 gal	24 gal	12 gal	10 gal	2 gal
Saltcrete	Tri-walls (42)	3.6 gal	2 gal	1.6 gal	64 gal	32 gal	30 gal	2 gal
Saltcrete	Tri-walls in Metals (6)	13 gal	2 gal	11 gal	64 gal	32 gal	30 gal	2 gal
Saltcrete	1/2 crates (12)	8 gal	2 gal	6 gal	64 gal	32 gal	30 gal	2 gal

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this project is to obtain representative samples from each major waste source for specified field measurements and laboratory analyses. These samples will provide an evaluation of the physical and chemical properties of the waste, as they directly apply to the development of stabilization process formulations and the design of process equipment.

The waste forms considered for this sampling event include Pondcrete and Saltcrete. Because of the different nature of these waste forms and the availability of previous information, samples will be taken using different approaches and will be analyzed separately. Therefore, sampling strategies need to be developed based on specific conditions to assure that the samples collected will give an accurate representation of each waste source.

To achieve the sampling objective, fundamental statistical concepts will be utilized where possible to develop sampling strategies to address the following issues:

- How many samples to take
- How to choose the sample
- How to estimate a population mean
- How to characterize the uncertainty in the estimate

1.3.2 Presurvey Estimate of Relative Standard Deviation

Formal sequential (multiphase) procedures are available which can guarantee, under certain conditions, achieving a prespecified boundary on the sampling error without previous knowledge of the population. Because only one sampling run will be conducted for this project, application of statistical formulas to determine the number of samples requires that previously obtained information on the population under consideration be available for evaluation. The previous sampling results will be utilized to provide rough

estimates for relative standard deviations (RSDs) of a waste form. The RSD is estimated to be the ratio between the sample standard deviation and the sample mean, for each parameter to be analyzed in each waste form. Any analysis error incurred in making observations on sample units was negligible for the existing data.

The importance of a RSD, which is a measurement of the variability of a parameter in the population, for determining the sample size for each waste form is clear. The sample size required to characterize a population to a specified confidence level is related to the variability of the population. The sample size required to characterize populations which have relatively low variability would, in most cases, be expected to be smaller than those required to characterize a population in which the variability is higher for the same level of confidence.

1.3.2.1 Relative Standard Deviation of Existing Data

Pondcrete tri-walls and Pondcrete in metal containers were sampled for chemical and geotechnical data (Weston 1991). Fourteen samples of the Pondcrete tri-walls were collected and five Pondcrete samples from the metal containers were collected.

Summarized in Tables 1-2 and 1-3 are analytical results and general statistics for selected parameters for Pondcrete samples previously taken from tri-walls and metals, respectively. These parameters were selected based on their importance to the design of the development of stabilization process. Sample averages, standard deviations, relative standard deviations, and the 97.5% upper limit of each parameter's possible range were calculated.

TABLE 1-2
GENERAL STATISTICS AND ANALYTICAL RESULTS FOR TRI-WALLS
ROCKY FLATS FACILITY

SAMPLE ID	ACETONE (ug/kg)	2-BUTANONE (ug/kg)	CADMIUM (mg/kg)	CHROMIUM (mg/kg)	LEAD (mg/kg)	NICKEL (mg/kg)	MERCURY (mg/kg)	BORON (ug/l)	POTASSIUM (ug/l)	SODIUM (ug/l)
904-01	5700	1900	4630	2450	445.0	782	16.00	167	276000	441000
904-02	5100	1800	3190	1580	273.0	498	10.90	2640	3180000	5400000
904-03	4400	1500	3020	1720	393.0	549	11.10	50	236000	395000
904-04	6000	2300	2580	1460	71.3	468	11.90	761	3200000	5110000
904-05	4400	910	2570	1370	144.0	449	8.70	50	2730000	5120000
904-06	5800	1700	3380	1670	189.0	515	14.60	1340	2060000	2370000
904-07	3100	770	2250	1300	136.0	403	9.30	50	2890000	5480000
750-01	2900	710	3400	1990	189.0	649	0.79	50	3060000	5440000
750-02	4800	1600	1650	988	224.0	310	11.40	594	2720000	4250000
750-03	5800	1800	1940	1070	158.0	344	9.10	50	2640000	4640000
750-04	5000	1400	2810	1500	254.0	493	9.80	187	2720000	4210000
750-05	3900	900	2760	1580	243.0	545	14.10	50	2530000	4660000
750-06	3400	810	1420	824	167.0	252	9.60	50	2570000	4690000
750-07	5200	1700	2560	1340	243.0	412	9.70	50	221000	324000
AVERAGE	4678.6	1414.3	2725.7	1488.7	223.5	476.4	10.5	434.9	2216642.9	3752142.9
S.D.	1034.9	505.2	813.3	413.2	99.6	136.2	3.6	740.7	1106916.5	1982824.7
R.S.D.	0.221	0.357	0.298	0.278	0.446	0.286	0.341	1.703	0.499	0.528
97.5% <	6707.0	2404.5	4319.7	2298.5	418.7	743.4	17.5	1886.6	4386199.3	7638479.2

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TABLE 1-2
GENERAL STATISTICS AND ANALYTICAL RESULTS FOR TRI-HALLS
ROCKY FLATS FACILITY
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SAMPLE ID	CYANIDE (mg/kg)	SULFATE (mg/l)	NITRATE (mg/l)	TOC (mg/kg)	MOISTURE (%)	PASS #200 (%)	NATURAL MOISTURE (%)	PENETROMETER (T/sq.ft)	TOTAL CALCIUM (mg/kg)	TOTAL CALCIUM (ug/l)	SILVER (mg/kg)	ARSENIC (mg/kg)
904-01	96.3	1360.0	11000	15100	63.9	76.5	181.4	0.00	337000	2500	147.0	7.60
904-02	93.1	1770.0	11700	21900	59.6	86.0	180.4	0.00	202000	7350	92.3	6.00
904-03	123.0	1190.0	9820	52600	69.8	93.0	229.1	0.00	235000	2500	103.0	11.10
904-04	49.7	1160.0	10700	21100	63.5	77.4	181.1	0.00	167000	7780	96.0	2.75
904-05	43.8	952.0	12300	23600	61.5	89.3	162.6	1.70	337000	28500	81.2	12.50
904-06	107.0	1060.0	5040	11100	61.5	60.9	159.0	0.00	139000	6270	102.0	4.90
904-07	43.4	1440.0	13100	16900	59.5	93.2	161.3	1.70	247000	23200	77.1	11.20
750-01	48.4	1190.0	11000	8640	61.1	86.4	158.1	1.30	328000	21400	119.0	2.55
750-02	36.8	1310.0	8080	17800	64.4	80.0	204.0	0.00	125000	12500	64.2	6.80
750-03	58.2	1260.0	9380	15100	64.1	86.7	196.0	0.75	189000	21900	55.9	5.90
750-04	29.4	1330.0	9330	13700	54.9	54.1	140.9	0.00	188000	12500	2.2	4.70
750-05	50.4	363.0	10400	21300	60.5	87.2	153.7	1.50	332000	44000	93.1	5.10
750-06	52.4	822.0	11100	18500	61.5	73.6	163.5	3.00	142000	27400	59.8	2.15
750-07	40.1	1320.0	7210	18900	61.8	81.9	165.2	0.00	180000	2500	79.2	6.50
AVERAGE	62.3	1180.5	10011.4	19731.4	62.0	80.4	176.0	0.71	224857.1	15735.7	83.7	6.4
S.D.	29.5	325.4	2122.9	10359.3	3.3	11.4	23.3	0.97	78739.0	12382.9	33.6	3.3
R.S.D.	0.474	0.276	0.212	0.525	0.054	0.142	0.134	1.364	0.350	0.787	0.402	0.507
97.5% <	120.1	1818.2	14172.3	40035.7	68.5	102.9	219.6	2.6	379185.5	40006.1	149.6	12.8

Source: Weston 1991

Data reported in mg/kg and µg/kg are concentrations in the solid waste.

Data reported in mg/l and µg/l are concentrations in the waste leachate.

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 PONDLEITE SAMPLING & ANALYSIS PLAN
 SALTLEITE SAMPLING & ANALYSIS PLAN

TABLE 1-3
 GENERAL STATISTICS AND ANALYTICAL RESULTS FOR METALS
 ROCKY FLATS FACILITY

SAMPLE ID	ACETONE (ug/kg)	2-BUTANONE (ug/kg)	CADMIUM (mg/kg)	CHROMIUM (mg/kg)	LEAD (mg/kg)	NICKEL (mg/kg)	MERCURY (mg/kg)	BORON (ug/l)	POTASSIUM (ug/l)	SODIUM (ug/l)	CYANIDE (mg/kg)
750-08	6200	2200	2420	1160	298.0	381	11.60	137	2330000	3590000	33.9
750-09	2900	840	858	396	371.0	136	6.70	50	2300000	3930000	38/5
750-10	2900	560	2100	1030	223.0	432	8.40	1880	2760000	4420000	77.1
750-11	690	65	3850	1970	423.0	647	13.90	421	2390000	2650000	38.6
750-12	770	55	1850	1040	303.0	338	11.60	616	2690000	4400000	33.2
AVERAGE	2692.0	744.0	2215.6	1119.2	323.6	386.8	10.4	620.8	2494000.0	3798000.0	44.1
S.D.	2241.4	880.2	1084.3	561.9	76.4	183.8	2.9	739.2	214779.0	729362.7	18.6
R.S.D.	0.833	1.183	0.489	0.502	0.236	0.475	0.274	1.191	0.086	0.192	.422
97.5X <	7085.1	2469.1	4340.9	2220.5	473.3	747.0	16.1	2069.5	2914966.8	5227551.0	80.5

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SAMPLE ID	SULFATE (mg/l)	NITRATE (mg/l)	TOC (mg/kg)	MOISTURE (%)	PASS #200 (%)	MOISTURE (%)	PENETROMETER (T/ft ²)	TOTAL CALCIUM (mg/kg)	TOTAL CALCIUM (ug/l)	SILVER (mg/kg)	ARSENIC (mg/kg)
750-08	1160.0	8010	22000	56.1	79.6	131.3	1.25	185000	21400	73.4	8.2
750-09	79.9	7760	7680	62.2	60.6	148.8	4.50	110000	129000	29.5	18.1
750-10	1270.0	11400	16600	58.3	79.3	135.7	1.25	166000	9310	102.0	1.85
750-11	1210.0	4500	23800	56.4	61.3	61.5	2.50	224000	11200	124.0	8.8
750-12	1360.0	10000	23100	57.9	77.7	137.7	1.25	159000	27300	69.6	8.2
AVERAGE	1016.0	8334.0	18636.0	58.2	71.7	123.2	2.2	168800.0	39642.0	79.7	9.0
S.D.	528.6	2612.9	6747.0	2.4	9.8	34.5	1.4	41445.1	50494.9	35.8	5.8
R.S.D.	0.520	0.314	0.362	0.042	0.137	0.280	0.661	0.246	1.274	0.449	0.644
97.5X <	2052.0	13455.4	31860.2	63.0	91.0	190.9	4.9	250032.5	138611.9	149.8	20.4

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Source: Weston, 1991
 Data reported in mg/kg and ug/kg are concentrations in the solid waste.
 Data reported in mg/l and ug/l are concentrations in the waste leachate.

As shown in the tables, some parameters have very high RSDs; however, the final result of the stabilization process may not be as sensitive to those parameters when compared to other considerations. Instead of using the highest RSD to set the sample size, it was determined that a more practical approach is to use the parameters that will be most likely to cause failure of the TCLP tests after the stabilization process. A conservative estimate of constituent concentration in the waste extract (CCWE) was made based on a 60% dilution (based on weight) of Pondcrete during stabilization (assumes a baseline 3:1:1 mixing ratio between Pondcrete material, cement, and water in the stabilization process), a 20:1 dilution during the standard TCLP test, and assuming 100% of the contaminant will leach. The following equation was used to estimate the leachate concentrations of each contaminant after the stabilization process:

$$CCWE(mg/l) = C_s(mg/kg) \times \frac{3}{5} \times \frac{1}{20}$$

Where: CCWE (Constituent Concentrations in Waste Extract) is the leachate concentration and C_s is the 97.5% upper limit of possible original concentration.

The estimated CCWEs were then compared with the regulatory level for each regulated contaminant, respectively. Table 1-4 summarizes these results. In both tri-walls and metals, cadmium has the highest CCWE/standard ratio and will be used to determine the data quality objectives for sampling.

1.3.3 Data Quality Objectives for the Sampling

Data Quality Objectives (DQOs) are statements that provide the critical definitions of confidence required in drawing conclusions from the entire project data. These objectives determine the degree of total variability (uncertainty or error) that can be tolerated in the data. As both sampling and analysis error contribute to the overall uncertainty of data, these limits of

TABLE 1-4
 COMPARISON OF ESTIMATED CCWEs WITH REGULATORY LEVEL
 PONDCRETE
 ROCKY FLATS FACILITY

TRI-WALL

SAMPLE ID	ACETONE (ug/kg)	2-BUTANONE (ug/kg)	CADMIUM (mg/kg)	CHROMIUM (mg/kg)	LEAD (mg/kg)	NICKEL (mg/kg)	MERCURY (mg/kg)	SILVER (mg/kg)	ARSENIC (mg/kg)
904-01	5700	1900	4630	2450	445.0	782	16.00	147	7.60
904-02	5100	1800	3190	1580	273.0	498	10.90	92.3	6.00
904-03	4400	1500	3020	1720	393.0	549	11.10	103	11.10
904-04	6000	2300	2580	1460	71.3	468	11.90	96	2.75
904-05	4400	910	2570	1370	144.0	449	8.70	81.2	12.50
904-06	5800	1700	3380	1670	189.0	515	14.60	102	4.90
904-07	3100	770	2250	1300	136.0	403	9.30	77.1	11.20
750-01	2900	710	3400	1990	189.0	649	0.79	119	2.55
750-02	4800	1600	1650	988	224.0	310	11.40	64.2	6.80
750-03	5800	1800	1940	1070	158.0	344	9.10	55.9	5.90
750-04	5000	1400	2810	1500	254.0	493	9.80	2.2	4.70
750-05	3900	900	2760	1580	243.0	545	14.10	93.1	5.10
750-06	3400	810	1420	824	167.0	252	9.60	59.8	2.15
750-07	5200	1700	2560	1360	243.0	412	9.70	79.2	6.50
AVERAGE	4678.6	1414.3	2725.7	1488.7	223.5	476.4	10.5	83.7	6.4
S.D.	1034.9	505.2	813.3	413.2	99.6	136.2	3.6	33.6	3.3
R.S.D.	0.221	0.357	0.298	0.278	0.446	0.286	0.341	0.402	0.507
97.5% <	6707.0	2404.5	4319.7	2298.5	418.7	743.4	17.5	149.6	12.8
EST. CCWE	201.2	72.1	129.6	69.0	12.6	22.3	0.5	4.5	0.4
STANDARD	590.0	750.0	0.066	5.2	0.51	0.32	0.2	0.072	5.0
RATIO	0.34	0.10	1963.50	13.26	24.63	69.69	2.63	62.35	0.08

Estimated CCWE assumes 100 percent leaching of constituent, 60 percent dilution during stabilization, and 20:1 dilution in TCLP test.

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 SALTICRETE SAMPLING & ANALYSIS PLAN

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 PONDCRETE SAMPLING & ANALYSIS PLAN
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TABLE 1-4
 COMPARISON OF ESTIMATED CCWE WITH REGULATORY LEVEL
 PONDCRETE
 ROCKY FLATS FACILITY
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METAL

SAMPLE ID	ACETONE (ug/kg)	2-BUTANONE (ug/kg)	CADMIUM (mg/kg)	CHROMIUM (mg/kg)	LEAD (mg/kg)	NICKEL (mg/kg)	MERCURY (mg/kg)	SILVER (mg/kg)	ARSENIC (mg/kg)
750-08	6200	2200	2420	1160	298.0	381	11.60	73.4	8.2
750-09	2900	840	858	396	371.0	136	6.70	29.5	18.1
750-10	2900	560	2100	1030	223.0	432	8.40	102	1.85
750-11	690	65	3850	1970	423.0	647	13.90	124	8.8
750-12	770	55	1850	1040	303.0	338	11.60	69.6	8.2
AVERAGE	2692.0	744.0	2215.6	1119.2	323.6	386.8	10.4	79.7	9.0
S.D.	2241.4	880.2	1084.3	561.9	76.4	183.8	2.9	35.8	5.8
R.S.D.	0.833	1.183	0.489	0.502	0.236	0.475	0.274	0.449	0.644
97.5% <	7085.1	2469.1	4340.9	2220.5	473.3	747.0	16.1	149.8	20.4
EST. CCWE	212.6	74.1	130.2	66.6	14.2	22.4	0.5	4.5	0.6
STANDARD	590	750	0.066	5.2	0.51	0.32	0.20	0.072	5
RATIO	0.36	0.10	1973.14	12.81	27.84	70.03	2.41	62.43	

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variability must be incorporated into the sampling and analysis plan and achieved with detailed sampling and analysis protocols.

The standard error estimates and confidence intervals presented for the sampling strategy will reflect only uncertainty due to sampling error, that is, the error associated with the fact that only a sample, rather than the whole population, is observed. This assumes that the sample is representative of the entire waste form.

By defining the sampling DQOs separately from the overall project DQOs, the sampling protocols can be developed using simple statistical concepts to achieve the specified quantitative standards for sampling errors. DQOs for sampling in each waste form will be defined as relative percent error, i.e., the magnitude of tolerable sampling error is expressed in relative terms as a percent of the quantity to be estimated. An initial value of the sampling DQO is selected as 15% error of the sample mean. This percent error was selected because the number of samples required to achieve this DQO is reasonable based on schedule and cost. Additionally, a greater number of samples does not decrease the DQO error in a significant manner until a very large sample population is selected (i.e., large increases in sample size results in small decreases in DQO error). Further discussion is provided in Section 1.3.4.1.

1.3.4 Determination of Sample Size

As mentioned earlier, statistical approaches will be used to determine the sample size required to generate data which satisfy the specified sampling DQO.

For random sampling of a finite population, the formula for standard error of the estimator of population mean specifies a relationship between sample size n and the uncertainty of the estimation (Wadsworth, 1990). This relationship can be used to

determine the sample size required to obtain an estimate with a desired level of precision. Given a DQO, expressed in a relative percent error, and the estimated RSD of the population, under 95 percent confidence limit the sample size can be determined as

$$n = n_0 / [1 + (n_0/N)]$$

Where:

$$n_0 = \left(\frac{z \cdot RSD}{DQO} \right)^2$$

N is the population size (total number of billets in a waste form), and z is the 2.5 percent quantile from Student's t Distribution with n-1 degree of freedom.

This equation results in collecting more samples than what would be required to be collected using the equations recommended in SW-846; therefore, this methodology will exceed the minimum sampling requirements of SW-846. This approach was taken to satisfy the technical needs and better quantify the uncertainties of the project.

1.3.4.1 Pondcrete Sample Size

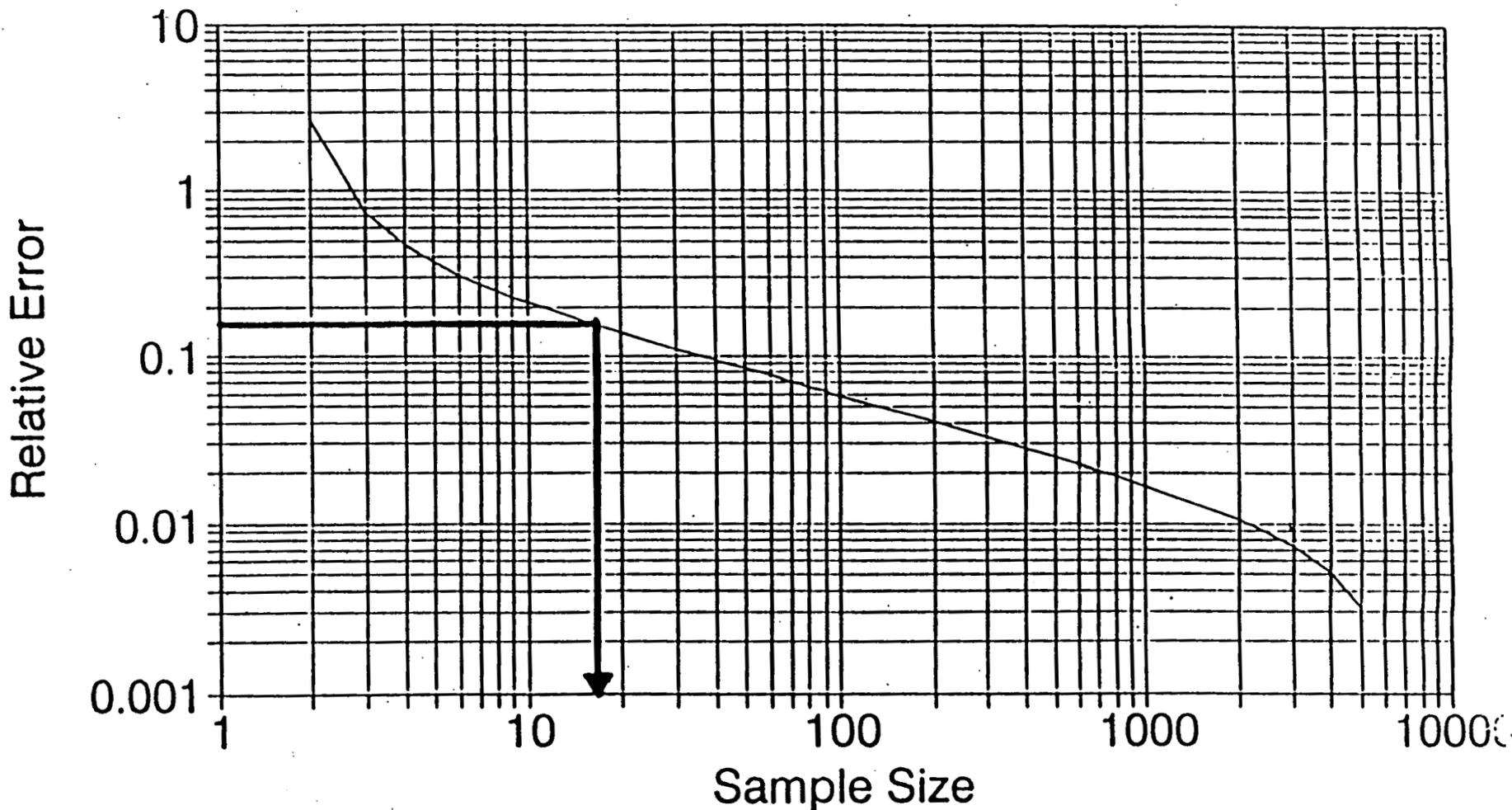
As mentioned previously, cadmium was determined to be the parameter which may cause the most concern for the TCLP analysis of the solidified product. Therefore, the RSDs calculated for cadmium were used to determine the required sample sizes for Pondcrete tri-walls and for Pondcrete in metal containers. Figures 1-1 and 1-2 were developed, based on formulas described earlier, to assist the selection of sample sizes based on RSDs of cadmium and the total numbers of billets for tri-walls and metals, respectively.

TRIWALL (TOTAL OF 5806 BOXES)

SAMPLE SIZE BASED ON RSD OF CADMIUM

DELIVERABLE 2118, 211C, 2218, 221C
 PONDICHE SAMPUNG & ANALYSIS PLAN
 SALTICETE SAMPUNG & ANALYSIS PLAN

1-26



REVISION 0
 October 24, 1991

— RSD = 0.298

Figure 1-1
 Comparison of Relative Error
 to Sample Size (Triwalls)

METAL (TOTAL OF 2293 BOXES) SAMPLE SIZE BASED ON RSD OF CADMIUM

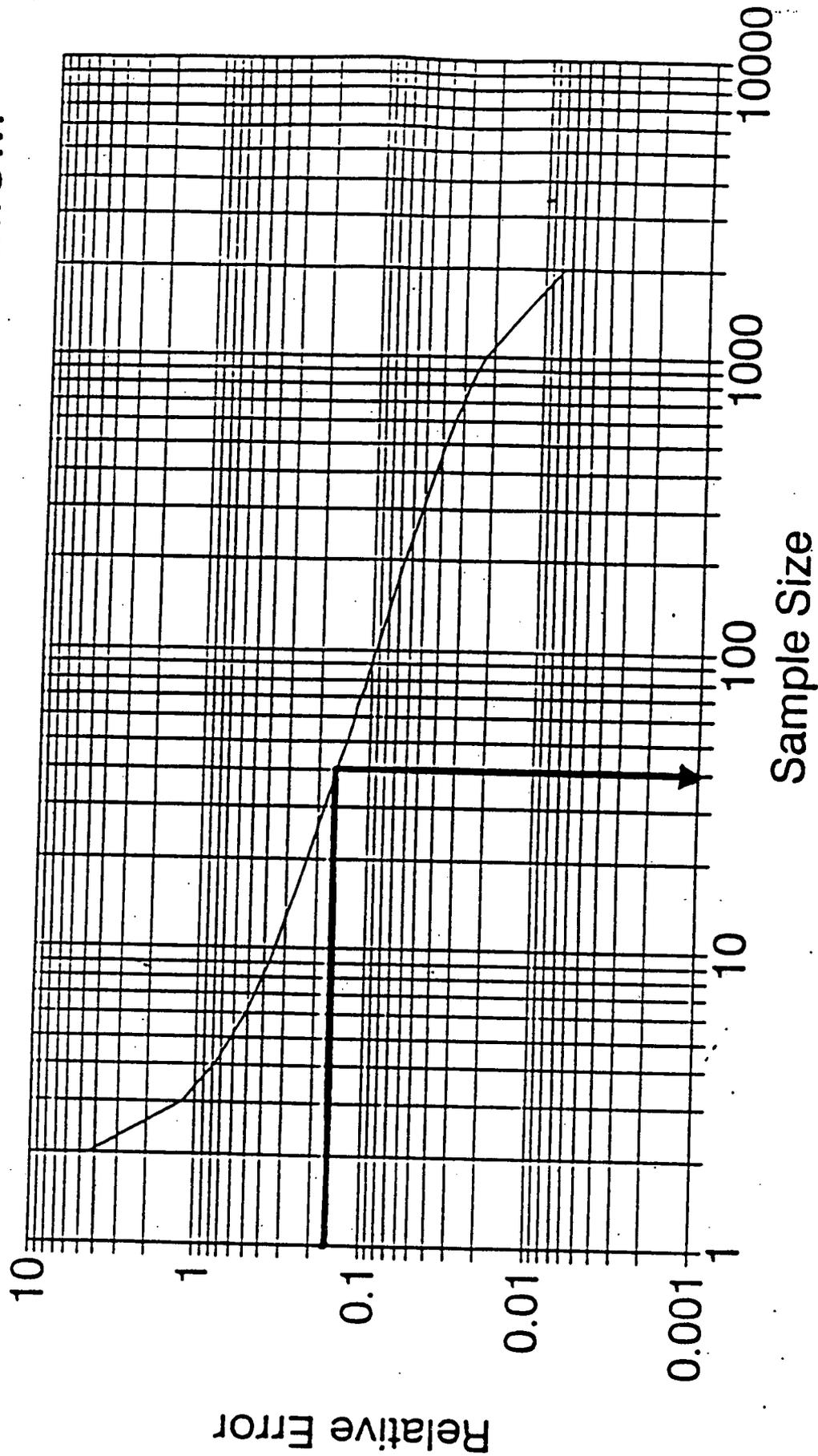


Figure 1-2

Comparison of Relative Error
to Sample Size (Metals)

— RSD = 0.489

As shown in the Figures, using a relative error of 15%, the required number of samples for tri-walls and metals are 16 and 40, respectively. As can be seen in Figures 1-1 and 1-2, relatively large changes in sample sizes are required to decrease the DQO error.

1.3.4.2 Saltcrete Sample Size

Statistical concepts based on previous analytical data are not applicable to the Saltcrete billets because existing data for critical parameters (metals, anions) do not exist. Because of the lack of data for Saltcrete, uncertainties exist for the chemical composition of Saltcrete. Therefore, it will be assumed the Saltcrete is more variable than Pondcrete and will require more samples per total population size to accommodate potentially larger variances.

Because the schedule is relatively short for this project, only one sampling round will be conducted for Saltcrete. Therefore, sample size must be as large as possible to ensure that quality data is obtained to adequately characterize the Saltcrete. Sixty samples is considered to be the maximum sample size that can be accommodated because of the short schedule. Sixty samples is believed to be sufficiently large to account for variances in the Saltcrete. Essentially, 60 samples for Saltcrete, compared to 56 samples for Pondcrete, represents almost three times as many samples when compared to a total population of 2,936 and 8,099, respectively.

The 60 samples for the total population of Saltcrete will be subdivided into three groups. The three subgroups will be tri-walls, 1/2 crates, and tri-walls in metal containers. The number of samples for each subgroup were determined by proportioning the total number of samples between the three subgroups based on the total number of billets (i.e., tri-walls, 1/2 crates, or tri-walls

in metal containers). The number of samples per subgroup were also proportionally divided between the 904 Pad and the 750 Pad. This method provided the following number of samples for each subgroup:

	<u>750 Pad</u>	<u>904 Pad</u>
Tri-walls	14	28
1/2 Crates	12	0
Metal	2	4

1.3.5 Uses of Statistical Computer Package

SYSTAT/SYGRAPH 5.0 with DESIGN and SAMPLE modules, a comprehensive statistics, graphics, and data management package for IBM-PC compatibles, will be utilized throughout the entire sampling task. This computer package can perform tasks ranging from simple statistical calculations to design of highly complex sampling that use stratification, clustering, and variable probabilities. The use of a statistic computer package saves time, reduces possible human errors and produce high quality graphic outputs.

1.3.6 Sample Design for Pondcrete and Saltcrete

Because of the way the waste containers are stored (i.e., large stacks in separated tents), it is desired to have a representative sample consisting of containers from every storage area and layer of stacks without moving too many containers. Given the relatively small size of sample (i.e., approximately 60 each for Pondcrete and Saltcrete), a simple random sampling approach clearly cannot assure that samples are selected from the middle of a stack or from only some of the tents. Therefore, the predetermined numbers of waste samples required for the treatability study were selected by a systematic sampling approach. This approach was designed to obtain samples from every portion of the waste storage areas/layers but also consider the accessibility of selected containers.

The number of samples required to achieve the DQO was initially developed by assuming a simple random sampling approach would be used. The following assumptions were considered thereby allowing the same number of samples to be used with a more controlled sampling approach:

- There is no statistically significant spatial patterns of the waste characteristic in a layer of any stack (i.e., the underlying probability distributions of the characteristics is stationary within a layer).
- The waste characteristics are independent of the different layers in a stack.
- The waste characteristics are independent of stacks in a tent and between tents or laydown areas.

The first assumption allows containers to be selected from corners or outsides of a stack so it is not necessary to move many container in order to take a container in the middle of a stack. With the second assumption, containers of all layers at a selected location in a stack can be collected simultaneously. The third assumption supports using tents and stacks as two levels of sampling clusters in the multi-stage cluster sampling approach.

1.3.6.1 Pondcrete Sampling

The multi-stage cluster sampling approach was accomplished in the following steps for Pondcrete:

- Develop maps showing the layout of the two waste storage pads, locations of stacks in the tents and outside laydown areas, and detailed drawings of positions of every container in a stack. All the maps and drawings are included in Appendix A and B.

- Identify the waste type and form for every container on the drawings.
- Verify the numbers of containers against the list of inventory.
- Select and mark potential containers or groups of containers to be sampled from each stack considering the size of the stack and the accessibility of the containers. These groups are located at corners or outsides of a stack and consist of overlaying containers from each layer of the stack.
- A random number table was used to generate random choices whenever a cluster (tent or stack) or waste container selection was performed in the following steps.
- The Pondcrete Tri-wall samples were selected from tents that contain mostly this type of waste (i.e., tents 9, 10, 11 in 904 Pad and tents 3, 4, 5 in 750 Pad). One stack was selected from a tent first, then one group of containers among the previously determined potential sampling groups of this stack was chosen. This procedure was repeated for each tent listed above. Overall, 16 Pondcrete Tri-walls were selected.
- The Pondcrete metal containers were located in one outside area and three stacks in Tent 9 on the 904 Pad and 27 double-layer rows in the south and north laydown areas on the 750 Pad. Each metal container usually has three Pondcrete Tri-walls inside. The sampling was performed by selecting stacks or rows then containers similar to the Tri-wall sampling. Overall, 14 metal containers with 42 Pondcrete samples were selected.

1.3.6.2 Saltcrete Sampling

The multi-stage cluster sampling approach was accomplished in the following steps for Saltcrete:

- No previous information was available to determine specific numbers of samples required for each container type to achieve a given DQO. Therefore, the total Saltcrete sample number (i.e., 60) was divided among waste storage areas and the three different waste-types by using simple proportions. The following table shows the numbers of samples to be collected from each waste-form on each pad.

Waste-forms	750 Pad	904 Pad
Tri-wall	14/771	28/1544
Half-Crate	12/675	0/0
Metal	2/102	4/210

NOTE: Sample number/total Saltcrete number

- The Saltcrete Tri-wall samples were selected from tent 8 in 904 Pad and tents 2 and 6 in 750 Pad. Similarly, stacks were selected first, then followed by sample groups. Overall, 42 Tri-wall samples were selected.
- The Saltcrete half-crates were stored in the south laydown area in 750 Pad. Following the stack then sample group procedure, 12 half-crates were selected.
- The Saltcrete metal containers usually have two Saltcrete billets in one container. These containers were located in two outside areas and one stack in tent 8 in 904 Pad and two stacks in the south laydown area in 750 Pad. Overall, 3

metal containers with 6 Saltcrete samples were selected following the stack then container procedure.

- The times that these selected -Saltcrete samples were produced was identified and a histogram (Figure 1-3) developed to determine the variation of the production over time.

Figure 1-3 was produced after EG&G personnel located the Saltcrete billets that were selected for sampling and determined their dates of production. Of the 42 Tri-walls selected, 18 had production dates that were accessible to EG&G personnel. These Tri-walls are evenly distributed with time over the period in which Tri-walls were produced. The remaining unknown Tri-walls shown in Figure 1-3 may be characterized with time during sampling or possibly after analytical results are obtained.

Of the 24 unknown Tri-walls, 15 Tri-walls may have a production date on a side of the Tri-wall that cannot be observed because it is adjacent to another Tri-wall. During Tri-wall mobilization the Tri-walls will be examined to see if any production dates are visible.

The remaining 9 unknown Tri-walls have serial numbers but no production date on the outer packaging. If possible, these Tri-wall's production dates could be determined from historical production log books. If this is not possible, then upon receiving the analytical data from characterization, this information will be compared to the data from the Tri-walls with known production dates to determine if any correlations exist.

The half-crates that have known production dates are evenly distributed with time. Two half-crates do not have production dates which is not considered to be a significant concern.

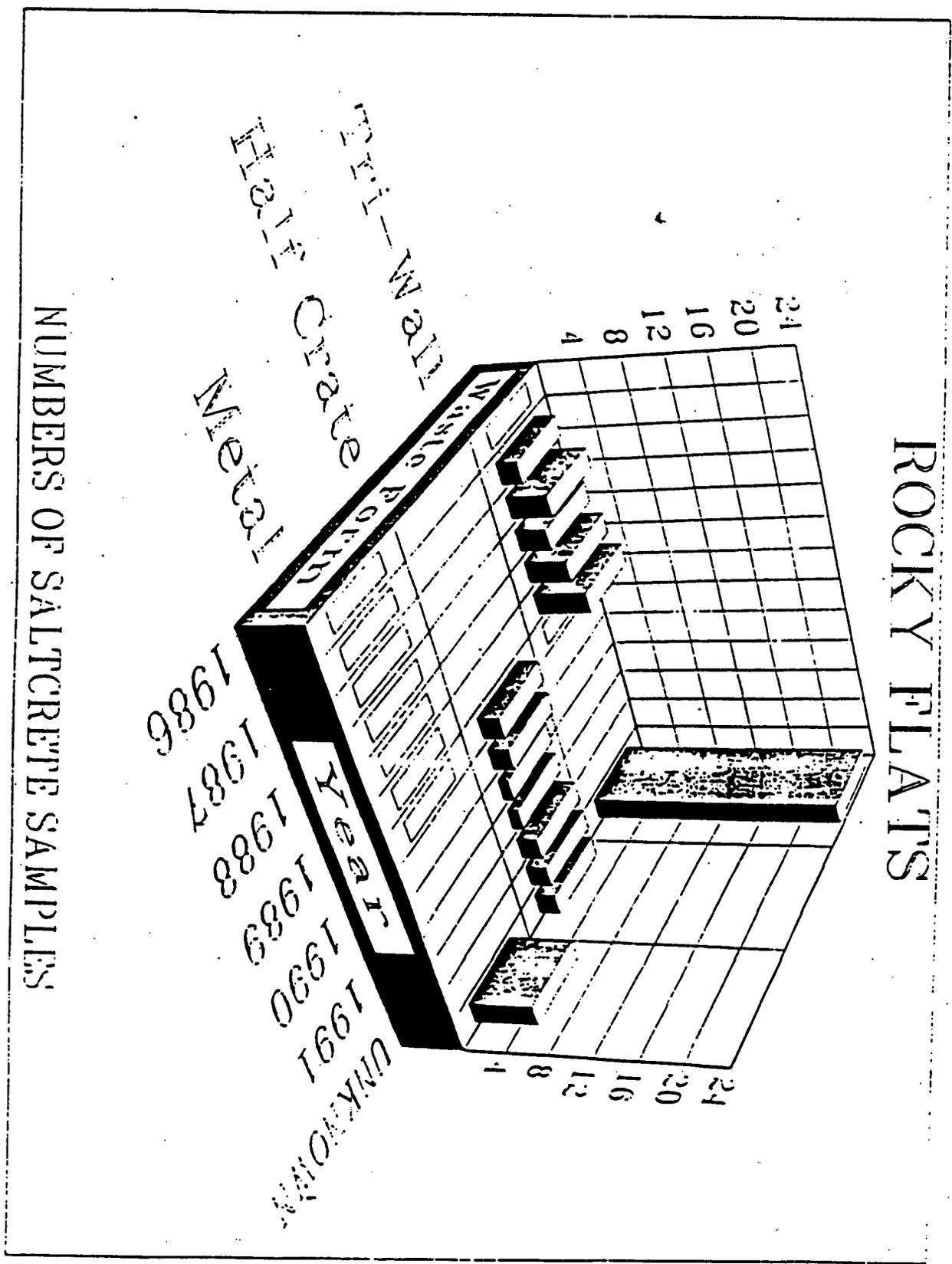


FIGURE 1-3

HISTOGRAM OF WASTE FORMS
FROM

YEARS 1986 TO 1991

None of the three metal containers selected for sampling have known production dates. However, because the billets in metal containers are a subset of the Tri-walls, this may not be a major concern. When the metal containers are opened during sampling, production dates may be distinguishable on the Tri-wall packing. When analytical data is obtained for the Tri-walls in the metal containers, it will be compared to the data for the Tri-walls.

Appendices A and B illustrate the locations of all the selected samples. When the selected samples are against the wall and not easily accessed, optional (alternative) sampling locations are identified.

1.4 Sample Analyses and Rationale

The purpose for conducting this sampling and analysis program is to provide input to the solidification formula development and is required to address various regulatory concerns, mainly the Land Disposal Restrictions (LDRs) from 40 CFR 268 and 49 CFR packaging and shipping requirements. A brief overview of the LDRs is provided below.

1.4.1 Land Disposal Restrictions

The land disposal restriction (LDR) requirements (40 CFR 268) apply to all hazardous wastes as designated by the U.S. EPA. The LDR regulations specify treatment standards that must be met prior to land disposal of hazardous waste. Treatment standards are expressed as a concentration limit in an extract of the waste, as a concentration limit in the waste, or as a specified technology. If a technology is not specified, any method of treatment may be used as long as the treatment standard is met.

The EPA Hazardous Waste Numbers associated with the Pondcrete and Saltcrete waste are F001, F002, F003, F005, F006, F007, and F009.

EPA Waste Code D006 is also appropriate for Pondcrete. The LDR treatment standards for these waste codes are provided in Table 1-5. Most treatment standards are expressed as concentration in the waste extract (mg/l), and some are expressed as concentration in the waste. If two treatment standards are given for a particular constituent (i.e., cadmium), the lowest value applies. It should be noted that for F005 waste, the treatment standard for 2-nitropropane and 2-ethoxyethanol is incineration. That is, if the solvent used was 2-nitropropane or 2-ethoxyethanol, then the waste must be incinerated or a variance from the treatment standard could be sought. However, these chemicals are not anticipated to be present based on process knowledge. If they are found to be present, EG&G shall be notified immediately.

The treatment standards for mixed (hazardous/radioactive) waste are whatever is specified for the corresponding nonradioactive hazardous waste. There are a few exceptions to this; however, they do not apply to the waste sources associated with this project.

In summary, the treated Saltcrete and Pondcrete, after solidification, must meet the treatment standards in Table 1-5 prior to land disposal. Therefore, the raw (untreated) waste should be analyzed for the constituents presented in Table 1-5. If any of these constituents are not present in the untreated waste, or are present below the LDR treatment standard concentration limits, there is no need to further analyze the solidified (treated) waste for such constituents.

1.4.2 Specific Analyses and Rationale

Analysis for the LDR-regulated organics will be conducted on each waste source. This analysis is being conducted to determine if the total amount of each compound in the waste sources is sufficiently low such that, when considering the TCLP procedure, the maximum possible leachate concentration is below the regulatory values in

TABLE 1-5

**LDR TREATMENT STANDARDS
PONDCRETE AND SALTCRETE
ROCKY FLATS FACILITY**

Regulated Hazardous Constituent	LDR Treatment Standard (Nonwastewaters)			
	F001-F003	F005	F006, F007, F009	D006
Acetone	0.59 mg/l ⁽¹⁾	0.59 mg/l	NA	NA
n-butyl alcohol	5.0 mg/l	5.0 mg/l	NA	NA
Carbon disulfide	4.81 mg/l	4.81 mg/l	NA	NA
Carbon tetrachloride	0.96 mg/l	0.96 mg/l	NA	NA
Chlorobenzene	0.05 mg/l	0.05 mg/l	NA	NA
Cresols and Cresylic Acid	0.75 mg/l	0.75 mg/l	NA	NA
Cyclohexanone	0.75 mg/l	0.75 mg/l	NA	NA
1,2-dichlorobenzene	0.125 mg/l	0.125 mg/l	NA	NA
Ethyl acetate	0.75 mg/l	0.75 mg/l	NA	NA
Ethyl benzene	0.053 mg/l	0.053 mg/l	NA	NA
Ethyl ether	0.75 mg/l	0.75 mg/l	NA	NA
Isobutanol	5.0 mg/l	5.0 mg/l	NA	NA
Methanol	0.75 mg/l	0.75 mg/l	NA	NA
Methylene chloride	0.96 mg/l	0.96 mg/l	NA	NA
Methyl ethyl ketone	0.75 mg/l	0.75 mg/l	NA	NA
Methyl isobutyl ketone	0.33 mg/l	0.33 mg/l	NA	NA
Nitrobenzene	0.125 mg/l	0.125 mg/l	NA	NA
Pyridine	0.33 mg/l	0.33 mg/l	NA	NA
Tetrachloroethene	0.05 mg/l	0.05 mg/l	NA	NA
Toluene	0.33 mg/l	0.33 mg/l	NA	NA
1,1,1-trichloroethane	0.41 mg/l	0.41 mg/l	NA	NA
1,1,2-trichloro-1,2,2-trifluoroethane	0.96 mg/l	0.96 mg/l	NA	NA
Trichloroethene	0.091 mg/l	0.091 mg/l	NA	NA
Trichlorotrifluoromethane	0.96 mg/l	0.96 mg/l	NA	NA
Xylene	0.15 mg/l	0.15 mg/l	NA	NA
1,1,2-trichloroethane	7.6 mg/kg ⁽²⁾	7.6 mg/kg	NA	NA
Benzene	3.7 mg/kg	3.7 mg/kg	NA	NA
2-nitropropane	NA	Incineration ⁽³⁾	NA	NA
2-ethoxyethanol	NA	Incineration ⁽³⁾	NA	NA
Cyanides (total)	NA	NA	590 mg/kg	NA
Cyanides (amenable)	NA	NA	30 mg/kg	NA
Cadmium	NA	NA	0.066 mg/l	1.0 mg/l
Chromium (total)	NA	NA	5.2 mg/l	NA
Lead	NA	NA	0.51 mg/l	NA
Nickel	NA	NA	0.32 mg/l	NA
Silver	NA	NA	0.072 mg/l	NA

(1) mg/l - concentration in waste extract

(2) mg/kg - concentration in waste

(3) Specified treatment technology

40 CFR 261, Subpart C for toxicity characteristic and values in 40 CFR 268, Subpart D, Land Disposal Restrictions. Therefore, depending on the analysis results, the analysis of the stabilized waste for certain compounds may not be required prior to final disposal.

Metals analysis will be conducted for both waste forms and will include those metals regulated by 40 CFR 261.24 (toxicity characteristic), plus nickel and boron. Total metal content and TCLP analysis will be performed for each parameter. The toxicity characteristic metals will be analyzed for regulatory purposes; nickel will be analyzed because it is a constituent of F006-type wastes (electroplating wastes), which is applicable to the waste forms, and boron will be analyzed because it can interfere with cement chemistry. Cyanide will be analyzed because it is a constituent of F006-type wastes.

Both Pondcrete and Saltcrete will be analyzed for ammonia and total organic carbon. Both of these parameters, depending on their concentrations, can affect cement chemistry.

The wastes will be analyzed for alkalinity, potassium, calcium, magnesium, and sodium. These parameters will provide input to developing the waste/cement formulation.

An ASTM (D3987-85) leach test will be conducted on the Saltcrete and Pondcrete. The leachate will be analyzed for phosphate, sulfate, nitrate, chloride, and total dissolved solids. This analysis will determine the amount, if any, of these compounds that will redissolve. Additionally, these compounds can affect the cement chemistry.

Gross alpha and gross beta will be analyzed on each waste form to characterize the activity level of the waste.

Several geotechnical parameters will be analyzed to characterize the physical condition of the solid waste. Percent moisture, bulk density, and specific gravity are common physical parameters for characterization of the waste source. The Blaine fineness test provides an indication of the fineness of the material based on the permeability of air. The Atterberg limits will provide an indication of the plasticity of the material. Particle size analysis will determine the distribution of the material size and the swell test will determine if dry material will expand when exposed to water. Disaggregation testing will determine if the material will dissolve when exposed to water.

Unconfined compressive strength will provide an estimate of the waste's current strength. Also, comparisons with other chemical parameters may be possible to develop correlations that will indicate if a particular parameter affects strength. Cement content will provide a rough estimate of the ratio of cement to waste. Petrographic analysis will provide qualitative analysis of the current structure of Pondcrete and Saltcrete. Information pertaining to mixing, unhydrated cement, and cement formation can be provided by petrographic analysis.