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Idaho Operations Office

Treatability Study Test Plan for Soil Stabilization

INEEL
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Prepared for the
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ABSTRACT

This test plan discusses the objectives and methods of conducting treatability studies on waste material. The wastes are primarily soils containing radionuclides and Resource Conservation and Recovery Act heavy metals, namely mercury. To dispose of these waste soils, the heavy metals must be removed or stabilized such that the final treated form does not leach contaminants above the standards defined by the Environmental Protection Agency in 40 CFR 268.49, "Alternative LDR Treatment Standards for Contaminated Soil."

The treatment method in this treatability study is a Portland cement-based chemical fixation system that stabilizes the heavy metals in a nonleachable form. This study will use actual waste material. The waste samples will be subjected to a matrix of tests wherein the Portland cement will be supplemented with chemical additives and the waste loading. The treated waste samples will be analyzed via the toxicity characteristic leaching procedure and the paint filter test for free liquids to determine if the treated material would meet disposal criteria.

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ACRONYMS

AA	alternative action
ASTM	American Society for Testing and Materials
BFS	blast furnace slag
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CFS	chemical fixation and stabilization
CRP	Community Relations Plan
CWID	CERCLA Waste Inventory Database
DOE-ID	Department of Energy Idaho Operations Office
DQO	data quality objective
DS	decision statement
EPA	Environmental Protection Agency
ERIS	Environmental Restoration Information System
HWMA	Hazardous Waste Management Act
ICDF	INEEL CERCLA Disposal Facility
IDW	investigation-derived waste
IHR	Independent Hazard Review
INEEL	Idaho National Engineering and Environmental Laboratory
INTEC	Idaho Nuclear Technology and Engineering Center
LDR	land disposal restriction
MQO	measurement quality objective
OU	operable unit
PFT	paint filter test
PPE	personal protective equipment
PRD	program requirements document
PSQ	principal study question

QAPjP	Quality Assurance Project Plan
RCRA	Resource Conservation and Recovery Act
RD/RA	remedial design/remedial action
ROD	Record of Decision
SMO	Sample Management Office
SRPA	Snake River Plain Aquifer
SSSTF	Staging, Storage, Sizing, and Treatment Facility
TCLP	toxicity characteristic leaching procedure
TSCA	Toxic Substances Control Act
UTS	Universal Treatment Standard
WAC	Waste Acceptance Criteria
WAG	waste area group
W/C	water-to-cement
WL	waste loading

Treatability Study Test Plan for Soil Stabilization

1. PROJECT DESCRIPTION

1.1 Introduction

The Department of Energy Idaho Operations Office (DOE-ID) authorized a remedial design/remedial action (RD/RA) for the Idaho Nuclear Technology and Engineering Center (INTEC) in accordance with the Waste Area Group (WAG) 3, Operable Unit (OU) 3-13 Record of Decision (ROD) (DOE 1999).

The ROD requires Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) remediation wastes generated within the Idaho National Engineering and Environmental Laboratory (INEEL) boundaries to be removed and disposed of on-Site in the INEEL CERCLA Disposal Facility (ICDF). The ICDF landfill, which is located south of INTEC and next to the existing percolation ponds, is an on-Site, engineered facility meeting DOE O 435.1, Resource Conservation and Recovery Act (RCRA) Subtitle C (42 USC 6921 et seq.), Idaho Hazardous Waste Management Act (HWMA) (HWMA 1983), and the Toxic Substances Control Act (TSCA) landfill design and construction requirements (15 USC 2601 et seq.). The ICDF includes the necessary subsystems and support facilities to provide a complete waste disposal system.

The major components of the ICDF Complex are the disposal cells, an evaporation pond, and the Staging, Storage, Sizing, and Treatment Facility (SSSTF). The disposal cells, including a buffer zone, covers approximately 40 acres, with a disposal capacity of about 510,000 yd^3 (389,900 m^3). The ICDF Complex is designed to provide centralized receiving, inspection, and treatment necessary to stage, store, and treat incoming waste from various INEEL CERCLA remediation sites prior to disposal in the landfill, or shipment off-Site. All ICDF Complex activities shall take place within the WAG 3 area of contamination (AOC) to allow flexibility in managing the consolidation and remediation of wastes without triggering land disposal restrictions (LDRs) and other RCRA requirements, in accordance with the OU 3-13 ROD. Low-level, mixed low-level, hazardous, and limited quantities of TSCA wastes will be treated and/or disposed of at the ICDF. Most of the waste will be contaminated soil, but debris and investigation-derived waste (IDW) will also be included in the waste inventory. Landfill leachate, decontamination water, and water from CERCLA well purging, sampling, and well development activities will also be disposed of in the ICDF evaporation pond.

Only INEEL on-Site CERCLA wastes meeting the Agency-approved Waste Acceptance Criteria (WAC) will be accepted at the ICDF. An important objective of the WAC will be to ensure that hazardous substances disposed in the landfill will not exceed groundwater quality standards in the underlying groundwater aquifer. Acceptance criteria will include restrictions on contaminant concentrations based on groundwater modeling results with the goal of preventing potential future risk to the Snake River Plain Aquifer (SRPA).

1.2 Scope of Treatability Plan

The scope of this treatability plan includes the following:

- Provides a standard recipe for the targeted waste sites using a Portland cement-based stabilization system
- Provides the methods of adjusting waste sample loading to produce an acceptable waste product

- Uses the inventory from CFA-04, CPP-92, CPP-98, and CPP-99 as typical wastes requiring treatment (EDF-ER-296)

- Uses the controlling design inventory in EDF-ER-264 ("INEEL CERCLA Disposal Facility Design Inventory") that only has data for total metals (i.e., not toxicity characteristic leaching procedure [TCLP])

- Provides chemical fixation and stabilization (CFS) formulations to treat heavy metals.

The scope does not include

- Regulatory analysis or interpretation

- An attempt to predict success of any CFS formulation presented herein

- Methods to stabilize radionuclides or organic compounds

- An attempt to assess the impact of trace organic compounds on stabilization

- An attempt to assess the representativeness of the sites waste samples, i.e., it is an intrinsic assumption that the generators provide representative samples to the laboratory.

1.3 Background

The wastes that will be processed through the ICDF Complex are identified in the "Waste Inventory Design Basis" (EDF-1540). This inventory was derived from the CERCLA Waste Inventory Database Report (CWID) (DOE-ID 2000), which contains contaminant identification and concentration information derived from available field sample data.

Further analysis of the design inventory has been completed to provide conservative estimates for sites having little or no data (EDF-ER-264). This design inventory included weighted averaging and statistical techniques to provide conservative estimates for metals, organic compounds, and radionuclides. There could also be some liquid wastes that require treatment.

The portions of the waste identified for treatment have been designated as potentially characteristic (toxic) for heavy metals under 40 CFR 261. The hazardous metals identified include barium, mercury, lead, chromium, cadmium, and silver. These wastes also contain low levels of beta-gamma-emitting radionuclide contaminants and some identified alpha-emitting radionuclides. Table 1-1 lists the volumes and waste descriptions for each of the waste streams requiring treatment.

Table 1-1. Designation of wastes for soils stabilization treatment (EDF-ER-264).

Site	Volume[a] (yd^3)	Waste Description	Treatment Method
CFA-04	800	Rocky soil with a small percentage of calcine	Stabilization
CPP-92	1,197	Soil (584 boxes of 2 × 4 × 8 ft plus 5 boxes of 4 × 4 × 8 ft – assume 85% full)	Stabilization
CPP-98	30	Soil (15 boxes of 2 × 4 × 8 ft – assume 85% full)	Stabilization
CPP-99	30	Soil (15 boxes of 2 × 4 × 8 ft – assume 85% full)	Stabilization

a. Actual volumes requiring treatment will be determined in associated RD/RA Work Plan.

After analyzing several treatment process alternatives, a CFS process was chosen as one of the potential methods to treat wastes within the ICDF Complex (EDF-1542). The process will use a cement-based binder that will stabilize the heavy metals and produce a leach-resistant (as determined by the TCLP) waste product. The information in Tables 1-1 and 1-2 are the basis behind treatability studies that will develop CFS formulations. These formulations will be used to treat the waste to meet the ICDF landfill WAC. The treatment process is intended solely for fixation and stabilization of metals and is not considered treatment for radionuclides or organic compounds.

1.4 Characteristic Waste (D0XX) Determination Process

Characteristic waste is a waste that exhibits the properties of ignitability, corrosivity, reactivity, and/or toxicity as defined in 40 CFR 261. Some of the ICDF Complex candidate waste streams potentially exhibit toxicity characteristics. If a TCLP analysis on a representative sample of the waste contains any of the contaminants listed in Table 1 of 40 CFR 261.24 at the concentration equal to or greater than the value listed in that table, then the waste stream is hazardous characteristic waste and the waste must be evaluated for treatment. Waste streams requiring treatment will be treated to the extent that the Universal Treatment Standard (UTS) metals are reduced by 90% or to less than 10 × UTS, whichever is reached first.

Table 1-2 provides a list of the heavy metals for the sites that are within the scope of this test plan. However, this plan is not strictly limited to these sites and can apply to any site waste with characteristic metals. The table also includes the characteristic TCLP values given in 40 CFR 261.24, the soil concentrations from the CWID (DOE-ID 2000), and the target concentrations (i.e., 10 × UTS) for nonwastewater. The shaded sections in Table 1-2 are those metals for which treatment is not required. All TCLP tests on treated wastes metals will be analyzed for all TCLP metals.

1.5 Treatability Study Approach

A Portland cement-based media will be used to treat and stabilize the waste. This treatability plan will use actual waste samples at various waste loadings to determine when the appropriate treatment level has been attained (actually, a threshold test). Performance of the recipe on the waste sample will be based upon TCLP testing, the paint filter test (PFT) as necessary to meet the ICDF landfill WAC. The recipe is intended to provide a moist, nonslab, final waste product similar in physical character to the original soil.

Other recipes and/or reagent types may be used to improve the performance as long as the test objectives are met. While the Portland cement-based system is the baseline CFS formulation, other systems that can be shown to meet the objectives may be substituted.

Table 1-2. Target metals for stabilization.

Metal	EPA HW No.	Characteristic Level (TCLP) (mg/L)	Characteristic Level (20 × Rule) (mg/kg)	Soil Concentrations (mg/kg)[a]			Treatment Standard 10 × UTS (mg/L) [40 CFR 268.49]
				CFA-04[b]	CPP-92	CPP-98/99	
Antimony (Sb)	—	—	—	2.2	NA	ND	11.5
Arsenic (As)	D004	5.0	100	8.9	4.7	4.7	50
Barium (Ba)	D005	100	2,000	300	NA	71	210
Beryllium (Be)	—	—	—	0.83	NA	0.40	12.2
Cadmium (Cd)	D006	1.0	20	1.6	2.8	0.32	1.1
Chromium (Cr)	D007	5.0	100	46	30	12	6.0
Lead (Pb)	D008	5.0	100	21	28	6.8	7.5
Mercury (Hg)	D009	0.2	4.0	58	4.6	0.1	0.25
Nickel (Ni)	—	—	—	65	20	14	110
Selenium (Se)	D010	1.0	20	0.99	0.41	0.8	57
Silver (Ag)	D011	5.0	100	9.9	NA	0.28	1.4
Thallium (Tl)	—	—	—	0.31	NA	NA	2.0
Vanadium (V)	—	—	—	35	23	16	16
Zinc (Zn)	—	—	—	86	NA	45	43

Notes

Shading indicates that the metal was below 10 x Universal Treatment Standard (UTS) at each site and requires no treatment

ND – Not Detected

a. Soil concentrations were extracted from the ICDF Design Inventory (EDF-ER-264)

b. CFA-04 has been confirmed as characteristic for mercury via TCLP analysis.

2. TEST OBJECTIVES

This study will use actual waste samples to prepare test batches for TCLP testing. The objective is to verify that the CFS formulation will stabilize the soils to meet the LDR concentrations (UTS). The test objective is not intended to provide optimization as the amount of soils requiring treatment for the minimum treatment option is relatively small. Therefore, the expense required for optimization is not justified.

The current criteria for disposal requires that the treated waste meet the ICDF landfill WAC, which will include the following criteria:

- Land disposal restrictions for hazardous waste soils must be met as defined in 40 CFR 268.49
- No free liquid may be exhibited as determined by the paint filter test
- Though not a requirement, it is desirable to provide a non-monolithic, friable waste product.

Portland cement will be used as the primary binding agent for treating the waste. Admixtures, including flyash, blast furnace slag (BFS), and free sulfide, will be used in a set CFS formulation. Sulfide will be used as necessary to meet criteria listed above.

Secondary objectives of this study relate to implementing this treatment on a large scale. It is desired for the waste product to remain in a nonslab form suitable for direct exhumation from the treatment site. The concept is that treated waste will be moved from the treatment facility and placed directly into the landfill following verification that the treated waste meets the ICDF landfill WAC and the Landfill Compaction/Subsidence Study (EDF-ER-267). A friable solid material would allow simple material handling for personnel and minimize subsidence in the landfill.

3. EXPERIMENTAL DESIGN AND PROCEDURES

3.1 Data Quality Objectives

The seven-step data quality objective (DQO) process (EPA 1994) was employed to develop test plan quality objectives. A summary of the DQOs is provided in Appendix A. The data quality requirements are based on the ability of the CFS system to stabilize the RCRA metals. This includes soils from the sites shown in Table 1-1. Ultimately, the treatability study will be used to determine when sufficient reagent is added so that stabilized waste can meet the acceptance criteria of the ICDF. For the treatability study testing, the analyses of the stabilized waste samples will consist of

- Paint filter test
- TCLP for UTS metals (40 CFR 268.49).

The results of the DQO process in Appendix A are that

- If the TCLP fails at the waste loading (WL), then the WL is reduced and a verification will be performed to ensure that stabilization is providing an additional benefit beyond dilution. For example, a nonlinear decrease in constituent concentration vs. waste loading would indicate effective stabilization.
- If the TCLP fails at all WLs, then the TCLP trending is examined and the formula changed.
- The TCLP passes if for all metals (M), M $<10 \times$ UTS (40 CFR 268.49).

3.2 Test Design

3.2.1 Introduction

This section will describe stabilization chemistry, the number of samples, recipes, and the number of test iterations that will be conducted. The test design and strategy will attempt to minimize sampling and analysis, while providing a baseline CFS formulation by the varying of the WL. The strategy is to conduct tests on actual wastes to determine CFS formulations that bind all contaminants of concern.

3.2.2 CFS Chemistry

Stabilization processes use chemically reactive formulations that, together with water and other components, form stable solids. Stable, in this case, means that the solids are physically stable under normal or expected environmental conditions and will not revert to the original liquid, semi-liquid, or unstable solid state (Conner 1990).

Chemical fixation and stabilization systems not only solidify the waste by chemical means, but also insolubilize, immobilize, encapsulate, destroy, sorb, or otherwise interact with selected waste components. The purpose of these systems is to produce solids that are nonhazardous, or less hazardous, than the original waste. The goal for the treatment unit soil stabilization is to sorb, insolubilize, and immobilize the UTS metals. This will be referred to as CFS.

Treatment formulations for this work will include the following reagents: Portland cement Type I, blast furnace slag (BFS), Class F flyash, and sodium sulfide. These will be used in a single CFS formulation with variation in the sulfide to match equivalents of the metals of the particular waste.

Portland cement is made up of four main compounds: tricalcium silicate ($3CaO \bullet SiO_2$), dicalcium silicate ($2CaO \bullet SiO_2$), tricalcium aluminate ($3CaO \bullet Al_2O_3$), and a tetra-calcium aluminoferrite ($4CaO \bullet Al_2O_3 \bullet Fe_2O_3$). In an abbreviated notation differing from the normal atomic symbols, these compounds are designated as C_3S, C_2S, C_3A, and C_3AF, where C stands for calcium oxide (lime), S for silica, A for alumina, and F for iron oxide. Small amounts of uncombined lime and magnesia also are present, along with alkalis and minor amounts of other elements. The composition of Portland cements falls within the range of 60–67% lime, 19–25% silica, 3–8% alumina, and 0.3–6% iron oxide together with 1–3% sulphur trioxide, derived mainly from the added gypsum, 0.5–5% magnesia, and 0.3–1.3% alkalis. Titanium oxide is usually present to the extent of 0.1–0.4%. Manganese oxide is usually present only in small amounts except when BFS is used as a raw material. Then it may rise to 1% giving the cement a brownish tinge rather than the normal grey color. A typical mineral composition of American Society for Testing and Materials (ASTM) Type I Portland cement is shown in Table 3-1.

Table 3-1. Mineral composition of Type I Portland cement (typical).

Component	Weight %
$3CaO \bullet SiO_2$	45
$2CaO \bullet SiO_2$	27
$3CaO \bullet Al_2O_3$	11
$4CaO \bullet Al_2O_3 \bullet Fe_2O_3$	8
Free CaO	5
$CaSO_4$	3.1

Flyash has a typical composition as shown in Table 3-2. The shape, fineness, particle-size distribution, density, and composition of flyash particles influence the properties of end use products. Flyash produced at different power plants or at one plant with different coal sources may have different colors. In addition, particle size and shape characteristics of flyash are dependent upon the source and uniformity of the coal, the degree of pulverization before burning, and the type of collection system used. Rapid cooling of the ash from the molten state as it leaves the flame causes flyash to be predominantly noncrystalline (glassy) with minor amounts of crystalline constituents, such as mullite, quartz, magnetite (or ferrel spinel), and hematite. Other constituents which may be present in high-calcium flyash include periclase, anhydrite, lime, alkali sulfate, melilite, merwinite, nepheline, sodalite, C_3S, and C_2A.

Table 3-2. Typical flyash composition (wt %).

Component	Class F	Class C
SiO_2	35	35
Al_2O_3	20	20
Fe_2O_3	6	6
$SiO_2 + Al_2O_3 + Fe_2O_3$	70 min	50 min
SO_3	5 max	5 max
CaO	5	15
MgO	5 max	5 max
H_2O	3 max	3 max
Alkali as Na_2O	1.5 max	1.5 max

Table 3-3 lists the typical chemical composition of BFS. The chemical compositions shown are in general applicable to all types of slag. When ground to the proper fineness, the chemical composition and glassy (noncrystalline) nature of vitrified slags are such that, when combined with water, they react to form cementitious hydration products. The magnitude of these cementitious reactions depends upon the chemical composition, glass content, and fineness of the slag. The chemical reaction between BFS and water is slow, but it is greatly enhanced by the presence of calcium hydroxide, alkali, and gypsum ($CaSO_4$).

Table 3-3. Typical composition of BFS (wt %).

Constituent	Mean %	Range %
Calcium oxide (CaO)	39	34-43
Silicon dioxide (SiO_2)	36	27-38
Aluminum oxide (Al_2O_3)	10	7-12
Magnesium oxide (MgO)	12	7-15
Iron (FeO or Fe_2O_3)	0.5	0.2-1.6
Manganese oxide (MnO)	0.44	0.15-0.76
Sulfur (S)	1.4	1-1.9

Because of these cementitious properties, BFS can be used as a supplementary cementitious material either by premixing the slag with Portland cement to produce a blended cement (during the cement production process) or by adding the slag to Portland cement as a mineral admixture. BFS is mildly alkaline and exhibits a pH in solution in the range of 8–10. Although BFS contains a small component of elemental sulfur (1–2%), the leachate tends to be slightly alkaline and does not present a corrosion risk to steel in pilings or to steel embedded in concrete made with BFS cement or aggregates.

The basic hydration chemical reactions that occur in cement reactions include

$$3CaO \bullet Al_2O_3 + 6H_2O \rightarrow 3CaO \bullet Al_2O_3 \bullet 6H_2O \quad \Delta H = 207\, cal/g$$

$$3CaO \bullet Al_2O_3 + 3CaSO_4 + 32H_2O \rightarrow (CaO)_6 \bullet Al_2O_3 \bullet (SO_4)_3 \bullet 32H_2O \quad \Delta H = 347\, cal/g$$

$$2(3CaO \bullet SiO_2) + 6H_2O \rightarrow 3CaO \bullet 2SiO_2 \bullet 3H_2O + 3(CaO \bullet H_2O) \quad \Delta H = 120\, cal/g$$

$$2(2CaO \bullet SiO_2) + 4H_2O \rightarrow 3CaO \bullet 2SiO_2 \bullet 3H_2O + CaO \bullet H_2O \quad \Delta H = 62\, cal/g$$

$$CaO + H_2O \rightarrow CaO \bullet H_2O \quad \Delta H = 279\, cal/g\,.$$

Proposed cement reactions with waste metals include addition, substitution, formation of new compounds, and multiple mechanisms. The addition and substitution reactions are shown below (using lead as an example metal):

$$xCaO \bullet SiO_2 \bullet H_2O + Pb = Pb \bullet xCaO \bullet SiO_2 \bullet H_2O$$

$$xCaO \bullet SiO_2 \bullet H_2O + xPb = xPbO \bullet SiO_2 \bullet H_2O + xCa\,.$$

Sulfides have been used for many years to insolubilize heavy metals. The CFS formulation provided depends on sulfide to insolubilize mercury and silver and to reduce chromium VI (Cr^{+6}) to chromium III (Cr^{+3}) if any is present in the soils. The following equations show the reactions and the solubility products (K_{sp}) from Langes (Dean 1985) for some of the targeted metals. The K_{sp} is used as an indication of how soluble a chemical is: a large number indicates high solubility and, hence, low bonding; a small number indicates low solubility and strong, covalent-type bonding.

$$Cd^{+2} + S^{-2} = CdS \qquad K_{sp} = 8.0x10^{-27}$$

$$Pb^{+2} + S^{-2} = PbS \qquad K_{sp} = 8.0x10^{-28}$$

$$Hg^{+2} + S^{-2} = HgS \qquad K_{sp} = 1.6x10^{-52}$$

$$2Ag^{+} + S^{-2} = Ag_2S \qquad K_{sp} = 6.3x10^{-50}$$

The K_{sp}s shown above are all very low and indicate that little metal would dissolve or be in solution and hence, nonleachable. Of course, to obtain this, good mixing and contacting are required. Differing from the cementitious reactions, free sulfide penetrates the soil pores and reacts with the metals weakly adsorbed to soils by London and other weak forces. The resulting bonded metal-sulfide molecule is stabilized via the strongest chemical force known (i.e., covalent bonding) with a comparatively long-range energy-distance function (Huheey, Keiter, and Keiter 1993). This is a much stronger bond than the type of bonding that occurs in cementitious reactions that rely on hydrogen and other weak bonding mechanisms.

The usage of sulfides in waste stabilization normally requires a restriction of an excess of 20% to prevent forming soluble anionic mercury compounds (Connor 1990). However, in soils there is normally a high demand and free sulfide has a short half-life. Therefore, there should be no particular concern for overdosing sulfide by a factor of two.

3.2.3 Soils

Soil type may impact treatability study results as soils may contain a diverse combination of clay, silt, sand, rock, and natural organic compounds (e.g., humus). The interaction of metals within the soil matrix is complex and difficult to predict. It is known that naturally occurring clays have the ability to provide adsorption and weak ion exchange sites for metals including lead, mercury, and the other contaminants of concern. Additionally, these metals may be dispersed on sites throughout the porous structure within the clay. For cement to stabilize the hazardous metals, these metals must be desorbed from the clay and participate in the cement hydration reactions (cementitious reactions). However, it is questionable whether desorption will take place into the alkaline environment provided by cement. Subjecting this clay, with its sorbed metals, to the acidic solution of the TCLP would likely result in leaching of these metals. Evidence to support this argument includes results from treatability studies conducted on INEEL mixed wastes (Gering 1993), wherein high-clay-content wastes leached hazardous metals at higher than expected levels.

Although soils may be classified as high clay content, they may have a significant fraction of other soil material such as sand or silt. This is demonstrated in Figure 3-1 where clay soils may actually contain in excess of 40% non-clay material.

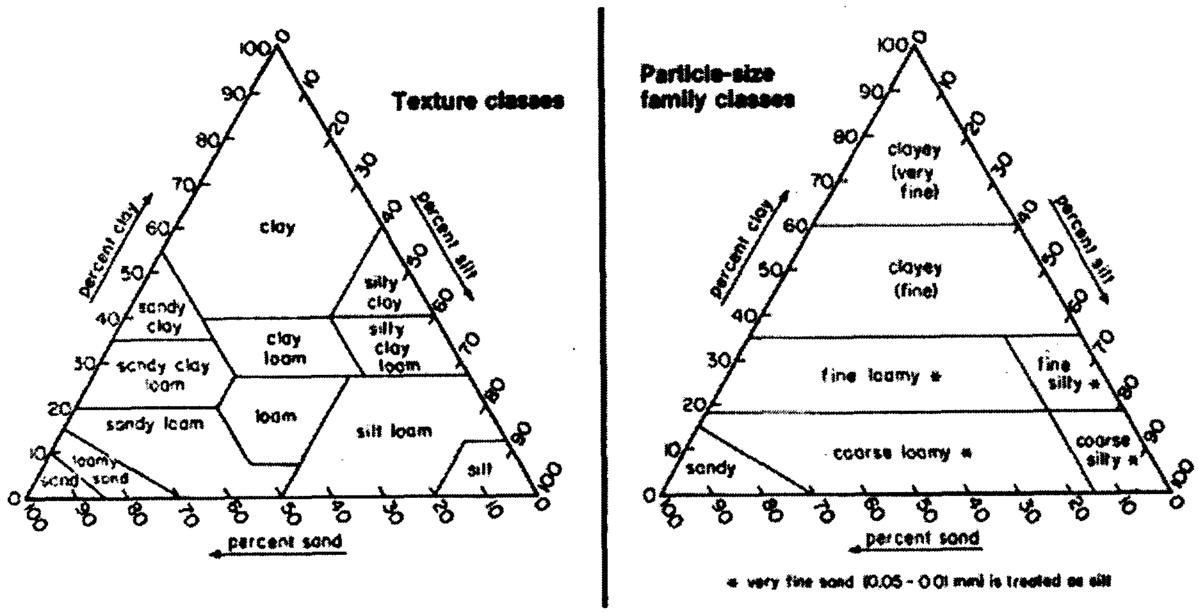

Figure 3-1. Triangular diagrams depicting soil classification.

3.3 Waste Sample Testing

The TCLP will be conducted on the Portland cement/reagent stabilized soil samples in accordance with SW-846 Method 1311 (EPA 1986a). The success criterion will be if the TCLP on the treated waste samples is less than the UTS. The reagents chosen are based on those known to be effective for the metals present (Conner 1990).

The BFS is used to help form insoluble metal sulfides as the slag contains a small fraction of available sulfur. Soluble sulfide, added as sodium sulfide, is used as a treatment for mercury and produces insoluble or sparingly soluble compounds with other toxic metals. Also, it may be required to include an organic fixating additive to prevent organic compounds from interfering with reactions. The reagents consist of the following:

- Type ASTM I Portland cement
- Class F flyash
- BFS
- Na_2S for fixation and stabilization of metals.

3.3.1 Waste Loading

The waste loading (WL) is defined (dry basis) as

$$WL_{dry} \equiv \frac{wt.waste}{wt.waste + wt.reagent}$$

and on a wet basis as

$$WL_{wet} \equiv \frac{wt.waste}{wt.waste + wt.reagent + water\ added}.$$

The basic plan is to vary WL until the CFS formulation is sufficient to ensure the TCLP results are less than 10 × UTS. Starting at a high WL of 95%, the CFS formulation is applied to a composite waste sample. If the TCLP results are greater than the treatment standard, then the WL is reduced until the TCLP results meet the treatment standard. Obviously, this can only continue for so long, at which time undesirable, nonfriable solids result. It is not known what WL this occurs at but is estimated to be WL ≤90. Clay soils have been stabilized to form friable solids in the 90-96% range (Adaska 2001). A nonfriable, monolithic material is a potential occurrence, especially at the lower WLs.

3.3.2 Sulfide Additive

The amount of sulfide addition needs to be controlled. Over-dosing can lead to soluble, anionic speciation of mercury. For mercury, it is recommended to not exceed 1.2 times the stoichiometric amount of sulfide (Conner 1990) in a well-mixed system. Therefore, the amount of sulfide needed for fixating Hg for CFA-04 is

$$\frac{58\,mg/kg}{200mg/mmole} * 1.2 * 78mg/mmole = 27\ mg\,Na_2S/kg.$$

However, note that lead, chromium, barium, silver, and cadmium also compete for sulfide. Calcium may also form compounds with sulfide under the proposed treatment scheme. Though calcium is soluble, it is less soluble than Na_2S. Assuming this reagent will also be consumed by the other toxic metals, additional amounts are needed within each formulation. Based on the design inventory composition given in Table 1-2, Table 3-4 was constructed to show the minimum sulfide (mg/kg) required, accounting for reaction stoichiometry of each of the target sites. Due to nonideal mixing and the likelihood of other sulfide sinks, it is recommended that the Na_2S dose be increased to 100% excess. This will be accounted for in CFS formulations that follow. While this provides a design basis, the TCLP of untreated soil augmented by a sulfide demand test for the soils will be conducted to determine the exact amount required.

Table 3-4. Sulfide usage by site, mg/kg of soil.

	CFA-04	CPP-92	CPP-98	CPP-99
Barium	170.43	0.00	0.00	0.00
Cadmium	1.11	1.94	0.22	0.22
Chromium	69.00	45.00	18.00	18.00
Lead	7.91	10.54	2.56	2.56
Mercury	22.62	1.79	0.00	0.00
Silver	3.58	0.00	0.10	0.10
Total Na_2S	274.64	59.28	20.89	20.89

3.3.3 Water Addition

Water is a crucial component of treatment methods based on hydraulic binders. It facilitates aqueous phase stabilization reactions, promotes more intimate mixing between waste particles and stabilization reagents, and provides adequate waters of hydration that are required for the hydrated cementitious species that form during stabilization.

The amount of water from the soil available for cement reactions is dependent on pore volume and bulk density (also the hydrophillic propensity of cementitious material and silt/clay) and is equal to the quantity:

$$water\ content = \frac{\varepsilon * fraction\ Saturated * 1kg / L}{\rho_B}$$

$$\varepsilon = 1 - \frac{\rho_B}{\rho_p}$$

where

ρ_B	=	Soil bulk density (kg/L)
ρ_P	=	Soil particle density (kg/L).

For the actual testing, relatively dry, as is, soil will be used with water added to meet the water-to-cement (W/C) ratio required and workability of the wet CFS formulation. The total amount of water impacts permeability, the amount of unreacted cement, air voids, and bleed water. The amount of water used varies in cement reactions. Additional water will be required for homogenization, mixing, and reagent dispersion. Care must be taken to prevent bleed water (excess water that separates from the waste product). While water is not a controlled parameter, the amounts will be measured.

3.3.4 Paint Filter Testing

Stabilized samples will be tested with Method 9095A, the paint filter test (EPA 1986b).

3.3.5 Waste Composition

The waste will consist of the materials, volumes, and concentrations from Tables 1-1 and 1-2.

3.3.6 CFS Formulations

The CFS formulations to be used in the treatability studies are shown in Table 3-5. The formulation is the same for each site except that the sulfide will differ. Basically, the formulation is based on a Portland cement:flyash ratio of 6:1 from EDF-1542, Stabilization Treatment Process Selection. Table 3-4 provides the stoichiometric amount of sulfide required to bond with all of the target metals. In Table 3-5, this amount is doubled to obtain 100% excess. As part of the test plan, the TCLP of the untreated soil, possibly augmented by sulfide demand, will provide the actual amounts of sulfide. While Table 3-5 provides approximate magnitudes for the sulfide demand, the TCLP/sulfide demand will provide the sulfide needed.

Table 3-5. CFS formulations based on dry basis.

WL	Soil (g)	Portland Cement (g)	Flyash (g)	BFS (g)	Na_2S[a] (mg) CFA-04	CPP-92	CPP-98, 99	Water (g)
95	100	4	0.67	0.4	55	12	4.2	1.28
85	100	14	2.33	1.4	55	12	4.2	4.48
75	100	26	4.33	2.6	55	12	4.2	8.32
65	100	42	7.00	4.2	55	12	4.2	13.44

a. Stoichiometric amount × 2 to obtain 100% excess.

3.3.7 Test Plan Strategy

The overall object of the tests is to obtain a single baseline recipe that delivers a waste product that meets the ICDF landfill WAC. This will include meeting LDRs for hazardous metals and passing the paint filter test. Additionally, a friable waste product is required to allow easier materials handling during full-scale operations.

The strategy is to vary the WL downward as shown in Table 3-5 until the site's waste sample passes the TCLP. If the baseline recipe fails, the waste loading will be reduced.

The plan is represented by the flowchart in Figure 3-2. The sample will be stabilized in accordance with the formula for 95% WL shown in Table 3-5. Following stabilization, the sample will be analyzed using the TCLP and the paint filter test will be conducted. If the TCLP fails, the next lower WL from Table 3-5 is used. If the TCLP passes but the paint filter test fails, the water added will need to be reduced followed by the TCLP at the same WL.

A determination to show that dilution is not the controlling factor will be conducted on those treatability studies where the waste loading is less than or equal to 50% of the treated waste.

3.3.8 Liquids

As previously discussed, there may be aqueous liquids that require treatment likely containing heavy metals and radionuclides. Liquids that cannot be used in the treatment systems or disposed directly to the evaporation pond will be stored for off-Site shipment to an appropriate facility. Those liquids designated for treatment will be used as makeup/addition water in the stabilization process. The treatment would be by either adding Na_2S to a mix tank prior to adding to the stabilization process or adjusting the dry mixture and adding the liquid directly. In either case, the test plan for liquids is to mimic the planned treatment method. The generator will provide the liquid analytical results.

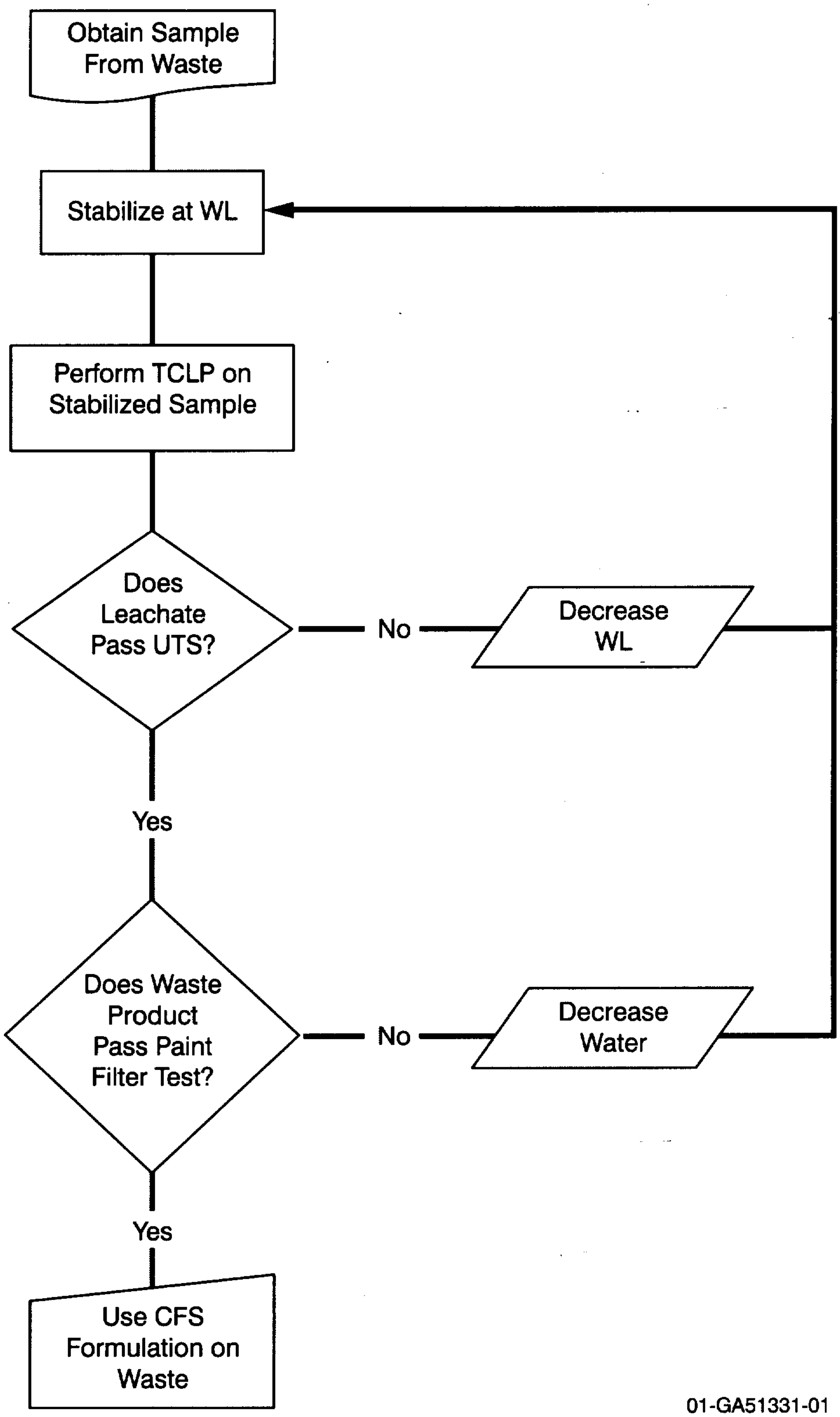

Figure 3-2. Test plan strategy flowchart.

3.4 Quality Assurance and Quality Control

The quality assurance/quality control (QA/QC) requirements for this project are established in the following environmental restoration plan:

- *Quality Assurance Project Plan for Waste Area Groups 1, 2, 3, 4, 5, 6, 7, 10 and Inactive Sites* (DOE-ID 2002).

Field logbooks, COC forms, Environmental Restoration Information System (ERIS) data files, and data limitations and validation reports will be rigorously controlled, as outlined in Section 10 of DOE-ID (2003a). Definitive data (EPA 1988) will be produced including all TCLP and paint filter analyses results. These analytical tests will be performed in accordance with the applicable EPA reference methods. Quality control samples must meet the minimum requirements stated in the Quality Assurance Project Plan (QAPjP) (DOE-ID 2002). The analytical data packages submitted will be validated to validation level "B," as defined by the QAPjP.

4. EQUIPMENT AND MATERIALS

4.1 Waste Samples

The generators will provide TCLP analysis results if they have already obtained them. The generators will provide an approximate 2-kg sample to the testing laboratory. This sample needs to be biased toward the high COCs and in the most difficult waste form to treat (e.g., sludge). The samples will be taken at the waste generator facilities and transported to the laboratory (unidentified at this time) for water content, stabilization, paint filter test, and TCLP testing. TCLPs will be performed on a portion of the untreated samples. The waste samples will be stabilized with the Portland cement-based CFS systems described in Section 3 and the TCLP performed on the stabilized sample batch. The sulfide required will be based on double the TCLP and augmented by the sulfide demand as required.

Laboratory subsampling quality is required to ensure minimization of bias. For the analytical process to produce reliable data for decision-making purposes, the errors associated with laboratory subsampling must be understood and addressed (Ramsey and Suggs 2001). There are two types of errors:

- Fundamental error that results from compositional heterogeneity
- Segregation error that results from distributional heterogeneity.

The fundamental error is fixed at approximately 4% (Ramsey and Suggs 2001) due to the nature of the TCLP procedure (i.e., 100-g sample and passing through a 1-cm sieve). However, the segregation error can vary widely if no subsampling control at the laboratory is incorporated. The segregation error can be minimized by ensuring that subsampling is performed to ensure that an equi-distribution of particles is sampled for the TCLP on untreated and stabilized soil samples. This can be done by spreading the sample out as far as practicable to minimize vertical segregation and measure equal-volume scoops from the distribution as best as can be done semi-quantitatively. The lack of this essential subsampling can lead to significant segregation error.

4.2 Stabilization

The stabilization reagents will be dry-mixed and stored before being added to the waste form so that it will be ready when the samples are ready. There will be one 2-kg sample obtained for each site. One portion of the sample will be stabilized in accordance with the formulation specified. The soil will be placed in the mixing vessel (see equipment list below). The top of the mixing vessel will be covered to prevent spills during mixing. Efforts will be made to minimize contact between the mixture and its surroundings. Drip pans and other precautions will be used to minimize contact. Where practical, equipment will be rinsed; however, if this results in the generation of too much waste or it is impractical, the vessels will be disposed of without rinsing.

Several years of mixed waste treatability studies performed for INEEL's Waste Reduction Operations Complex have yielded preferred procedures for mixing cement mixtures (Gering and Schwendiman 1997). The general recommended procedure is to place the preweighed amount of soil in the mixing vessel, followed by the total amount of added water. Next, this (waste + water) combination is mixed until the waste becomes an even consistency. At this point, any pretreatment reagents (e.g., Na_2S) can be introduced to the wetted waste as mixing continues. Mixing during pretreatment should be of sufficient duration to allow the pretreatment processes (e.g., redox reactions) to near completion. Next, the hydraulic binders are slowly added as mixing continues.

While the above is the preferred procedure based on INEEL experience, the currently planned operational mode adds reagent and waste to the mixer together. Therefore, the mixing mimics the operational mode. This mode is to place the preblended reagents and the soil into a Hobart mixer and dry mix for 30 seconds. The water is then added and mixed for an additional 30 seconds. This needs to be performed in a ventilated hood as there is a potential for hydrogen sulfide evolution.

Following mixing, the stabilized samples will be tested by the paint filter test to determine free liquid (EPA 1986b). The TCLP analysis will evaluate the leaching properties of the samples in accordance with Method 1311 (EPA 1986a).

4.3 Sulfide Demand

The TCLP of the untreated soil sample may be augmented by a sulfide demand test. The sulfide demand test provides an accurate estimate of the amount of sulfide a soil requires. This is a simple test requiring a small amount of soil (25 g) placed in a beaker with distilled water adjusted to pH >7 to prevent hydrogen sulfide (H_2S) emissions. This water is stirred while adding small amounts of Na_2S until the ion-specific electrode (ISE) indicates a steady signal. The time to perform this process should be 5–10 minutes. The results provide the amount of sulfide per unit mass of soil.

4.4 Water Determination

Water is not a controlled parameter during these tests but the amount will be measured. This will be determined by taking part of the 2-kg sample and using an oven or other thermal device to drive off the water. The sample weight change provides the water content of the soil. Also, all added water will be measured and recorded during sample stabilization.

4.5 Equipment

- Hobart Mixer or equivalent
- Oven
- Beaker
- Magnetic stirring device
- Spoons or spatulas for transfer of treated mixtures
- Stainless steel beakers (mixing vessels)
- Thermometer
- Scale
- Dry mix storage container
- Dry mix preparation container
- Tamping rods

- Rubber mallet
- Handling tongs
- Waste containers
- Glovebox or hood
- Other standard laboratory equipment as required
- Ion-specific electrode for sulfide demand (Thermo Orion Solid State Half Cell or equivalent) with display monitor and ancillary equipment
- Paint filter test equipment per Method 9095A (EPA 1986b)
- TCLP equipment per Method 1311 (EPA 1986a).

4.6 Materials

- Type I Portland cement
- Class F flyash
- BFS (ground, granulated - rapid water quenching)
- Na_2S powder
- Water (standard, plant potable water)
- TCLP reagents per Method 1311.

5. SAMPLING AND ANALYSIS

There could be up to four stabilized samples per waste generated from this treatability study. As requested by 40 CFR 268.40(e), characteristic waste will be analyzed for the TCLP metals and all underlying hazardous constituents to demonstrate compliance with the UTS. All samples will also be analyzed for free liquids (PFT). The generators are required to provide representative samples to the testing laboratory. Sample identification and tracking will conform to company policies and procedures. Samples will be presumed to be hazardous and radioactive and will be shipped in accordance with company policies.

6. DATA MANAGEMENT

Data generated during treatability studies will be managed in accordance with guidelines provided in Section 10 of the O&M Plan (DOE-ID 2003a). This plan provides or references procedures and requirements necessary to develop a database of relevant information that can be readily accessible and accurately maintained. The plan describes the data-flow process, data custodianship, and organizational and individual responsibilities associated with data management. The plan also provides the project file and reporting requirements and identifies extensive database capability requirements to allow selective data sorting, analysis, formatting, and reporting.

Section 10 (DOE-ID 2003a) provides the necessary requirements for this treatability study. There may be some deviations from the Section 10. Deviations are due to information that is not directly recorded in logbooks or from laboratory data that may not be tracked. For this treatability study, the following tests may result in information considered variances:

- Mixing and grouting performance - laboratory data not tracked
- Paint filter test - laboratory data not tracked.

In each of the above-mentioned cases, the data and information may be placed in Information Repository and Document Control, if it is not tracked. Specific DQOs and data validation requirements will be specified in the test plans. Data will be handled in accordance with Section 10 of DOE-ID (2003a).

7. DATA ANALYSIS AND INTERPRETATION

Upon completing the treatability study, the data will be summarized and evaluated to determine the validity of the data and to assess the performance of the stabilization process. To accomplish this goal, results will be reduced to a useful form in accordance with applicable data uses, including specifically

- Characteristic metals immobilization
- Free liquid determination by the paint filter test.

The data will be both qualitative and quantitative. The qualitative data will include visual observations, logbook entries, descriptions, etc. The quantitative data will include timing of events, measurements of the amount of materials used, chemical concentration measurements, physical measurements, mixing parameters, and laboratory analyses.

Data produced from testing will be reported as described in Section 6. In addition to the analytical data collected during the study, data packages will also contain relevant observations of key parameters and unknowns encountered during the testing.

Test results are to be interpreted in the context of the formulation's effectiveness, i.e., does the best stabilized waste sample, as determined from the testing results, pass the TCLP and PFT.

8. HEALTH AND SAFETY

All health and safety issues associated with required treatability studies will be consistent with the ICDF Complex Health and Safety Plan (INEEL 2003).

9. RESIDUALS MANAGEMENT

The treatability studies discussed herein will be performed on wastes containing heavy metals, organic compounds, and radioactive materials. Wastes generated as a consequence of this study may include the following:

- Unused, untreated, waste samples
- Pretreated wastes, if any
- Stabilized waste forms
- Treatment residues
- Extraction fluids (TCLP)
- Contaminated equipment wash/rinse water
- Contaminated protective clothing and other PPE
- Contaminated sampling materials and debris.

9.1 Waste from Tests

The analyses of waste samples may reveal heavy metals, radionuclides, and organic compounds; therefore, a potential exists for dealing with mixed waste. Through careful planning, the amount of mixed waste will be minimal. All material will be consumed during the stabilization tests (i.e., all material except the untreated soil will undergo stabilization). Incidental lab waste that is not hazardous or radioactive will be disposed of as cold waste.

9.2 Hazardous Waste Determination

Hazardous waste determinations will be based on TCLP analysis or other appropriate testing methods and/or process knowledge. The samples for this determination are considered ICDF Complex-generated wastes and will be addressed in the ICDF Operations Waste Management Plan (DOE-ID 2003b).

10. REPORTS

During the course of the treatability study, open lines of communication are essential to ensure a smooth and accurate flow of information to all parties directly or indirectly involved. The pertinent information will be disseminated in a timely manner to interested parties, using informal project meetings or conference calls and notes, as well as more formal written reports.

The organization performing the treatability study will document activities by preparing the following reports:

- Reports documenting the performance of CFS formulations, including any trending information, recommended formulations, along with affected methodologies used and experimental data.

11. REFERENCES

40 CFR 261, 2000, "Identification and Listing of Hazardous Waste," *Code of Federal Regulations,* Office of the Federal Register, July 2000.

40 CFR 261.24, 2000, "Toxicity Characteristic," *Code of Federal Regulations,* Office of the Federal Register, July 2000.

40 CFR 268.40, "Applicability of Treatment Standards," *Code of Federal Regulations*, Office of the Federal Register, July 2000.

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42 USC 6921 et seq., 1976, Subtitle C, "Hazardous Waste Management," *Resource Conservation and Recovery Act of 1976*, as amended, United States Code.

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Appendix A

Data Quality Objectives

Appendix A

Data Quality Objectives

The data quality objective (DQO) process is used to specify, qualitatively and quantitatively, the objectives for the data collected. The DQO process is described in the Environmental Protection Agency (EPA) documents "Guidance for the Data Quality Objectives Process" (EPA 1994) and in "Data Quality Objectives for Hazardous Waste Site Investigations" (EPA 2000). The DQO process includes seven steps, each of which has specific outputs. The first seven subsections below correspond to a step in the DQO process, and the output for each step is provided as appropriate.

A-1. PROBLEM STATEMENT

The problem statement is to determine whether the stabilized waste sample, following stabilization treatment, meets the requirements of the INEEL CERLCA Disposal Facility (ICDF) landfill Waste Acceptance Criteria.

A-2. DECISION STATEMENT

This step in the DQO process is used to identify the decisions and the potential actions that will be taken based on the data collected. This is done by specifying principal study questions (PSQs), alternative actions (AAs) that could result from resolution of the PSQs, and combining the PSQs and AAs into decision statements (DSs).

The objective of this waste characterization activity is to answer the following PSQ1:

PSQ1: Does the stabilized waste sample meet the land disposal restriction (LDR)?

The AAs to be taken based on resolutions of the PSQ are

AA1: If some of the regulated metals are not immobilized, then the waste loading (WL) requires reduction.

Combining the PSQ and AA results in the following DS:

DS1: Reduce the WL to the next lowest step in the plan

AA2: If all of the Universal Treatment Standard (UTS) metals are not immobilized at any of the WLs, the chemical fixation and stabilization (CFS) formula requires modification

DS2: Using the toxicity characterization leaching procedure (TCLP) results, examine trending and adjust formula accordingly and/or determine cause.

A-3. DECISION INPUTS

Determination on whether or not the stabilized samples contain hazardous constituents or materials will be based on UTS data for the metals (see Table A-1). Data collected during this activity will be used to determine constituents of concern that may be present at levels above the maximum concentration of contaminants for UTS defined in 40 CFR 268.49. Therefore, for this treatability testing, there are constituent-specific numerical values for the action level. That is, for each constituent of concern, an action level is specified, i.e., the UTS for metals. If it is found that all of the stabilized sample batches possess a hazardous characteristic, it will be concluded that the formula is incorrect.

Table A-1. UST data for metals.

Metal	10 × UTS, mg/L (40 CFR 268.49)
Antimony (Sb)	11.5
Arsenic (As)	50
Barium (Ba)	210
Beryllium (Be)	12.2
Cadmium (Cd)	1.1
Chromium (Cr)	6.0
Lead (Pb)	7.5
Mercury (Hg)	0.25
Nickel (Ni)	110
Selenium (Se)	57
Silver (Ag)	1.4
Thallium (Tl)	2
Vanadium (V)	16
Zinc (Zn)	43

To resolve the DSs, the formula will require modification or additional additive be used.

A-4. STUDY BOUNDARIES

The spatial boundaries of concern for this study are confined to the samples of the waste soils from the sites. The data collected from the analysis of the waste samples will be used to make independent decisions concerning each site's waste. The data obtained from the treatability testing are used to determine the formula in terms of WL and proportions of the four components making up the formula. The characteristics that define the population of interest are the concentrations of UTS metals found in the toxicity (TCLP) leachates produced from the stabilized samples. Additional tests will be run if the TCLP results are close to the appropriate limit, or several tests have failed.

A-5. DECISION RULE

The decision rules relevant to this activity are

- *If* the concentration in all batches of a test, for any constituent of concern, indicates that the materials have constituents that are greater than the constituent-specific maximum concentration of a contaminant for the UTS, *then* the WL requires reduction.

- *If* the concentration in at least one of the test batches, for all constituents of concern, indicates that the materials have no constituents that are greater than the UTS, *then* the test is deemed successful.

A-6. DECISION ERROR LIMITS

The two types of decision errors for the TCLP results of the stabilized samples are determining that the contents do not exceed regulatory values, when in fact they do, or determining that the contents exceed regulatory values, when in fact they do not. The consequences of each decision error must be considered.

Because the regulatory Agencies will state that the more severe decision error occurs when it is determined that the stabilized samples do not contain hazardous waste, when in fact they do. The null hypothesis for the TCLP is that "metals greater than the UTS indicates that the TCLP failed." The alternative hypothesis then becomes "the metals less than the UTS indicates that the TCLP passed."

TCLP Fails	TCLP Passes
M >10 × UTS	M <10 × UTS

A-7. DESIGN OPTIMIZATION

No optimization is required for this testing. Based on the small amount of material requiring treatment, the expense of optimization is not justified.

A-8. MEASUREMENT QUALITY OBJECTIVES

Measurement quality objectives (MQOs) are specifications that measurements must meet to produce acceptable data. The technical and statistical quality of these measurements is required to be properly documented. Precision, accuracy, method detection limits, and completeness must be specified for physical/chemical measurements. Additional analytical requirements are described qualitatively in terms of representativeness and comparability. Table A-2 lists the MQOs for this testing.

Table A-2. MQOs for untreated and stabilized samples.

Measurement	Method	Validation	Data Uses	P/A[a]	RQL[b]
UTS metal (solids)	1311, 3000/7000	Level B	Treatability Test	QAPjP (DOE-ID 2002)	QAPjP (DOE-ID 2002)

a. Precision/accuracy.

b. Required quantification limits.

A-9. REFERENCES

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