5 TECHNOLOGY SHORTCOMINGS AND LIMITATIONS

This chapter discusses some of the shortcomings and limitations of S/S technology pertaining to S/S processes/binders, waste form and treatability/performance testing, and other issues. The topics discussed should be viewed as examples of issues rather than an exhaustive list of technology limitations.

5.1 PROCESS/BINDER CONSIDERATIONS

5.1.1 Hierarchy of Waste Management

As discussed in Chapter 1, technologies that lead to the recycling, recovery, or reuse (3R) of some portion of the contaminant or waste material are preferred over treatment technologies in the waste management hierarchy. Technologies such as incineration that destroy the contaminant also are typically preferred over S/S processes. However, S/S is still an important treatment option because of its versatility and effectiveness (Section 1.1).

5.1.2 Scale-Up Uncertainties

Process scale-up from bench-scale to full-scale operation involves numerous complex issues that should not be taken for granted. These issues are no less important for S/S technology than for any other remediation technology. Variables such as ingredient flow rate control, materials mass balance, mixing, and materials handling and storage, as well as the unpredictability of the outdoor elements compared with the more controlled environment in the laboratory, all may affect the success of a field operation. These potential difficulties underline the need for a field demonstration prior to full-scale implementation (Section 2.8).

5.1.3 Proprietary Binders

The nature of the S/S business at present is such that most vendors protect their exact binder formulations as proprietary or trade secret. Relatively few formulations are covered by patent. The proprietary designation protects the formulations from being readily recognized by competitor vendors (Section 4.1). The reality is that there are several different generic binder systems that are used by the majority of S/S vendors, and each vendor has its own variations in the form of special additives. Binder ingredients are frequently designated in the literature as, for example, "fly ash A" or "proprietary additive." As a result, the report on a treatability study lacking information on binders and additives has no technology transfer value, and the ability to evaluate the data in terms of chemical mechanisms is absent, because binder chemistry is unknown or unreported.

5.1.4 Binder "Overkill"

Too much of a particular binder ingredient can lead to unnecessary expense and even to an improperly stabilized waste form. For example, many metals are amphoteric, meaning that they are soluble under both acidic and basic conditions (Section 4.2). The metal will be at minimum solubility when a sufficient base (in the form of an S/S ingredient) is added to make the waste moderately alkaline. Too much base will cause the metal to resolubilize and/or make the waste hazardous by virtue of the RCRA corrosivity characteristic (i.e., pH >12.5).

5.2 WASTE FORM/CONTAMINANT ISSUES

5.2.1 <u>Complications of Physicochemical Form of the Target Contaminants</u>

In a recent S/S field demonstration (Means et al., 1991b), the unsatisfactory degree of stabilization of the copper and lead was a direct result of their encapsulation in organic coatings of various types (antifouling compounds, pigments, etc.). People conducting S/S treatability tests frequently measure the type and amount of contaminant present, but, in complex waste forms such as sandblasting grit, the type and amount of contaminant do not provide sufficient information. It is important to understand the physicochemical form of the contaminant as well. However, the chemical analyses necessary to characterize the physicochemical form of the contaminant can be expensive and nonroutine (Section 3.5).

5.2.2 Interferences and Incompatibilities

As discussed in Section 4.3, numerous chemical constituents may interfere with various S/S processes. Thus, specific chemical incompatibilities should be recognized and avoided.

5.2.3 <u>Volatile Organic Contaminants</u>

Several studies have been performed that strongly indicate the inadvisability of using S/S as the principal remediation technology for organic wastes, particularly wastes containing hazardous volatile organics (Wiles and Barth, 1992). The following guidance is provided based on the current state of knowledge about using S/S for treating organics (Section 4.4):

- According to the hierarchy of waste management, treatment by a destructive technology (e.g., incineration) is preferable to contaminant immobilization (e.g., by S/S) because the former processes eliminate the contaminant and the concern over the long-term stability of the S/S process. The same is true for removal processes, such as thermal desorption, that concentrate the contaminant into a much smaller volume of material which can then be either reused as a raw material or incinerated and destroyed.
- Generally, S/S should not be used to treat a site containing only organic waste. Alternative technologies (e.g., incineration, steam stripping, vacuum extraction) should be used to remove and/or destroy the organics. If residues remain after this primary treatment, S/S treatment may be effectively used to stabilize the residue. However, a welldesigned and controlled treatability study should be conducted to assess S/S effectiveness and to select and design a proper S/S process.
- There are exceptions to avoiding S/S treatment of organic wastes. For example, if the organic is generally not mobile through air, soil, and water (e.g., low levels of oil and grease), then S/S may be an acceptable, cost-effective treatment alternative for a given site. Careful attention must be paid to any existing state and federal environmental regulations concerning the particular organic contaminant (e.g., dioxins, etc.). Treatability studies must be performed incorporating appropriate test methods to evaluate the organic waste's potential for escape.
- Based on existing data, volatile organic compounds (VOCs) usually cannot be treated by current S/S technology. Whether a site containing VOCs as a minor constituent can be treated by S/S will depend on specific conditions existing at the site.
- Available data also indicate that semivolatile organic compounds generally cannot be effectively treated by

current S/S techniques. Whether a site containing low to moderate concentrations of semivolatile organics should be treated using S/S also depends upon sitespecific factors.

• Notwithstanding the above factors, there are situations in which S/S can be a satisfactory treatment method for wastes containing organics. When S/S treatability tests are performed on such matrices, it is important to understand that (a) aqueous leaching tests will be a meaningless indicator of the degree of immobilization for organic compounds having low solubility in water and (b) in the aggressive chemical environments associated with certain binders, certain organic contaminants may be degraded or transformed into by-products that, in some cases, may be as toxic as or more toxic than the parent compounds.

5.2.4 <u>Multicontaminant Wastes</u>

Wastes containing a large number of contaminants are generally more difficult to stabilize than wastes containing one or a few contaminants, particularly when the multiple contaminants have widely varying chemistries (Section 4.2). The problem is that a given type of binder might be more compatible with an organic waste than with a primarily metallic waste. Therefore, when both organics and metals occur in the same waste form, the binder selected will not be optimal for both types of contaminants. On a more specific level, because metal chemistry varies widely, metals will respond differently to the same binder. As a general rule, a physical encapsulation process (solidification) may be the best compromise for a multicontaminant waste, whereas a chemical stabilization process may be the best approach when there is only one contaminant or when the contaminants present have similar chemical properties.

5.2.5 Limitations of Cement-Based Waste Forms

The weaknesses of cement-based waste forms are as follows:

- The fate of the waste species within the waste form is unknown.
- They are porous solid bodies.
- The total volume of material to be disposed of usually increases.

- Small changes in the waste composition or mix proportions can alter the properties, sometimes without the knowledge of those utilizing the waste form.
- Managers and operators charged with the task of waste disposal frequently do not understand the complexity of the heterogeneous material they are attempting to create.

It is of utmost importance that users of these waste forms be aware of these weaknesses and their ramifications. In most instances, problems originating from the weaknesses can be avoided or circumvented. Future research is expected to help explain and overcome these weaknesses (McDaniel et al., 1990).

5.2.6 <u>Sample Heterogeneity</u>

Solid wastes can be highly heterogeneous in composition, both macroscopically and microscopically. A person can analyze two different portions of the sample and obtain two very different analytical results. Therefore, sample heterogeneity should be recognized as a possible causative factor when explaining treatability data that are discrepant or difficult to interpret.

5.3 TREATABILITY AND PERFORMANCE TESTING ISSUES

5.3.1 Testing Limitations

Several unresolved issues pertain to S/S processes. In particular, tests that have been developed to assess technology performance are not applicable to every disposal scenario. Testing methodologies must be tailored to the specific nature of the S/S-treated waste. Personnel involved in treatability testing should be aware of the various tests' limitations when interpreting the data (Chapter 3).

Examples of the limitations of treatability studies and S/S-treated waste testing based on actual field experience are as follows:

• Although the principal objective of the site sampling is to obtain a sample that is representative of the waste as a whole, variation from sample to sample is common and must be considered when interpreting the



analytical data. Many factors affect site sampling. If the goal is a single composite sample, site debris, such as large boulders or rocks, timbers, shingles, etc., usually should be segregated by physical screening before samples are collected from a wide range of locations, in order to produce a representative sample.

- No single leaching methodology is suitable for all waste forms or target contaminants, and none of the leaching methodologies is calibrated in terms of contaminant migration in actual groundwater. The TCLP does not provide data on long-term stability; in fact, different results are frequently obtained when the TCLP test is conducted on the same stabilized waste at different cure times. Leach tests in general are probably most useful for assessing the relative stabilization efficiencies of different binders.
- Some leaching test methods are more appropriate for metals, some are not applicable to nonvolatile organics, and others are applicable only to monolithic wastes that do not change in surface area appreciably during testing. Batch methods usually do not use sufficient acid to exhaust the acid-neutralizing capacity of most stabilized waste forms. Sequential methods accelerate leaching to assess long-term performance. The interpretation of results is difficult, however. Accelerated leaching in the laboratory may occur by different mechanisms than the longer term leaching that occurs in the field.
- At times it is appropriate to modify a standard leaching protocol to address a specific issue. Examples include the following:
 - Eliminate the leachate filtration step to address colloidal contaminant transport.
 - Use site-specific groundwater as the leachant instead of the generic leachant specified in the procedure.
 - Consider use an organic solvent (e.g., acetone) as the leachant instead of an aqueous leachant for addressing the S/S of organic contaminants (see Section 4.4.3 for discussion of pros and cons).
 - Determine when it is appropriate to create an artificial surface area prior to leaching (e.g., by crushing).
 - Deionized water can be more aggressive than acid in some cases.



- Microbes may eventually affect the long-term performance of certain waste forms, particularly organic binders (Section 3.4). However, these microbial reactions can be very slow, and accelerated tests that are generally recognized and approved and that closely simulate real-world biochemical reactions are not available.
- Bioassay data may conflict with chemical data.
- There are limitations to interpreting and applying the results of physical tests. For example:
 - The unconfined compressive strength test is not appropriate for noncohesive substrates and is not a direct indicator of constructability.
 - No correlation has been identified between the physical strength of a waste form and its leaching behavior.
 - Permeability measurements are difficult to conduct and are subject to wide variation. Also, large differences have been observed between values measured in the laboratory and in the field for the same substrates.
- With the exception of a small group of "regulatory" tests, no performance standards or acceptance criteria exist for many tests. In fact, acceptance criteria should vary, depending on waste composition, disposal or reuse site characteristics, and other factors. This leaves much to the interpretation of individual S/S project personnel.
- In general, the bench-scale treatability study should exceed the performance criteria established for the project. That is, a margin of safety should be established that allows for the greater variability of the process when implemented in the field, especially in the area of mixing. The necessary magnitude of the safety margin, however, is unknown and probably varies from project to project (Sections 2.6 and 2.7).

5.3.2 Long-Term Performance

The long-term performance of treated waste is not clearly understood, and no definitive test procedures exist to measure or assess this property. The TCLP is not an adequate measure of long-term leaching. Monitoring data from field disposal sites are needed to detect the premature deterioration of solidification or stabilization of previously processed wastes. Because of the uncertainties surrounding long-term performance, wastes previously treated using S/S and disposed of may have to be retrieved and retreated in the future (Section 4.7).

5.3.3 <u>Reproducibility</u>

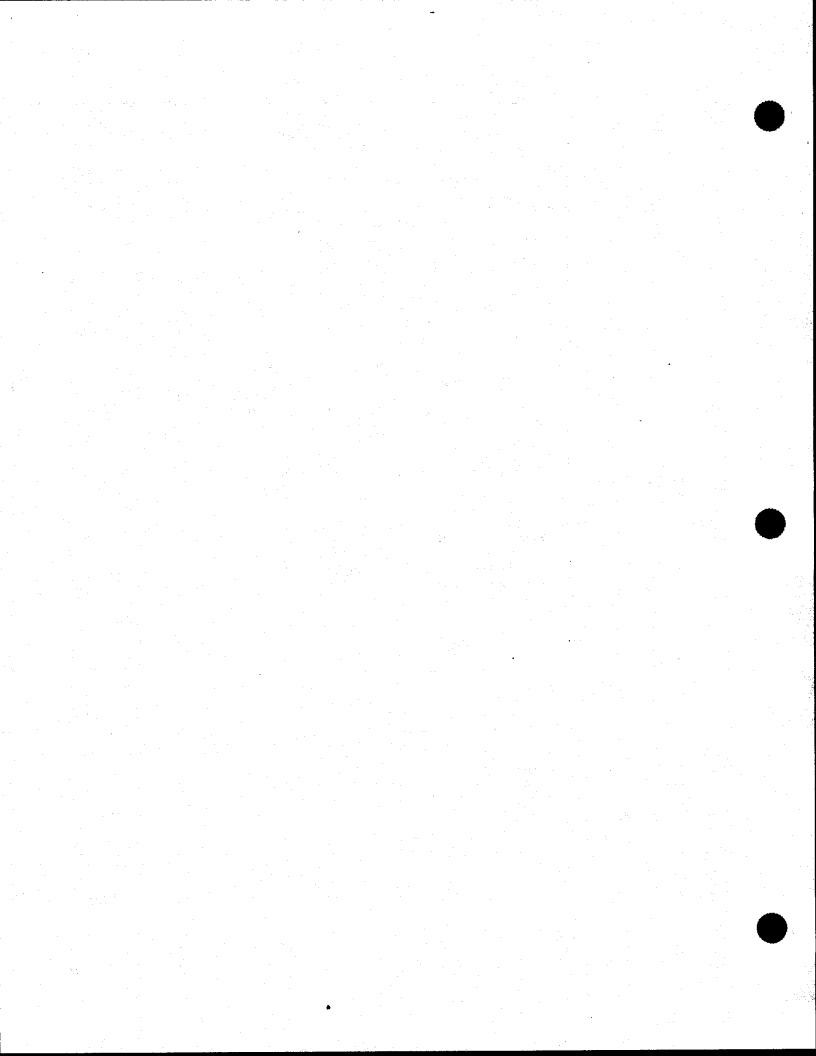
The reproducibility of treatability data can be poor because of sample heterogeneity, uneven mixing, the complexity of S/S chemical reactions, and other reasons. Timing is also a critical variable. It is not unusual to see different analytical results when samples from the same treatability study are cured for different periods of time prior to leaching.

5.3.4 Limitations in S/S Treatability Reference Data

S/S processes would be used more successfully if experiences were shared more effectively. However, well-documented S/S treatability data are scarce. Many of the common reporting deficiencies are as follows:

- 1. <u>Proprietary binders</u> (Section 5.1.3). Without specific information on binder characteristics the process is not reproducible, and the treatability data have no technology transfer value.
- Incomplete treatability data and data gaps. Certain types of data that are needed to evaluate the stabilization efficiency and help understand the chemical mechanism(s) of stabilization are frequently missing, for example:
 - <u>Baseline soluble metal concentrations in the</u> <u>untreated waste</u>. This is needed as a point of comparison for the soluble metal concentrations in the treated waste so that the percent reduction attributable to treatment can be assessed.
 - <u>Total metal concentration in the untreated</u> waste and the treated waste. The latter is necessary to demonstrate that a low posttreatment soluble metal concentration is not attributable simply to sample heterogeneity.
 - <u>Binder-to-waste ratio</u>. This is needed to estimate the volume expansion of the waste during treatment and the effect of dilution on posttreatment soluble metal concentrations.

- <u>pH of the leachate from the untreated and</u> <u>treated wastes</u>. This is an important parameter for interpreting the performance data. Frequently, high soluble-metal concentrations are due to pH. The pH parameter should be routinely measured at the conclusion of leach testing.
- Extent of dilution from binder ingredients. This can be estimated from the binder/waste ratio, where given, but should be carefully characterized in each treatability study so that the performance data can be corrected for dilution. Frequently, a significant proportion of the reduction in soluble metal concentration in the treated waste can be attributed to dilution from the binder ingredients.
- 3. <u>Data reliability</u>. Many treatability reports do not indicate whether data were collected under an appropriate quality assurance/quality control (QA/QC) program. Therefore, many existing S/S performance data have unknown validity.
- 4. <u>Treatability procedures</u>. Similarly to data reliability, the frequent absence of detailed treatability procedural information greatly limits the technology transfer value of a treatability study. The success or failure of a treatability study may depend on small variations in the amounts of the ingredients and in the order and timing of ingredient addition.
- 5. <u>Bias of existing S/S performance data toward</u> <u>successful treatability studies</u>. Treatability projects that achieved a high degree of metal stabilization are reported more frequently in the literature than projects in which the treatment systems worked poorly. Therefore, the existing S/S database is probably biased toward the most successful treatability studies.



6 CURRENT RESEARCH AND FUTURE DEVELOPMENT NEEDS

6.1 CURRENT RESEARCH

Solidification/stabilization is the subject of active research aimed at improving the range and efficiency of S/S process application. Some of that research is described in sections 6.1.1 through 6.1.8.

6.1.1 Binders

Experimental Study of S/S Treatment of Hazardous Substances.

Statistically designed treatability studies are being applied to identify environmentally acceptable and economically feasible methods for S/S processing of organic and inorganic wastes. The work focuses on inexpensive pozzolanic binders such as fly ash, silica fume, lime kiln dust, cement kiln dust, and ground blast furnace slag. Waste types tested include electric arc furnace dust (K061) and arsenic-contaminated soil (Fan, L.T., 1991, personal communication).

Improvement in S/S Treatment of Hazardous Inorganic Wastes by Silica Fume (Microsilica) Concrete. A preliminary experimental program is being conducted to assess the potential of silica fume concrete for solidification/stabilization of KO61 metal arc dust from steel manufacturing. TCLP leaching tests are being used to investigate the effectiveness of the various methods of S/S processing. The study is testing S/S process performance for condensed silica fume and cement binder or fly ash, cement kiln dust, and cement binder. It was concluded that silica fume concrete can significantly enhance the stabilization of furnace arc dust as compared with the other S/S processes. The results were based on studying the concentration of metals in the leachant as specified by U.S. EPA (Fuessle and Bayasi, 1991).

<u>Physical and Chemical Aspects of Immobilization</u>. Recent studies are using sodium as an internal marker for physical retardation. Almost any product will contain some Na, K, or Cl, which can be used independently as indicators for tortuosity. The difference between the mass transfer coefficients for Na and other elements derived from leaching tests, such as the modified ANSI/ANS/16.1, reflects the contribution of chemical retention in the product matrix to the overall mass transfer coefficient for the product. The types of release mechanisms that can be distinguished are:

- dissolution
- surface wash-off
- diffusion (de Groot and van der Sloot, 1990)

Evaluation of Solidification/Stabilization of RCRA/CERCLA Wastes.

U.S. EPA Risk Reduction Engineering Laboratory is sponsoring a project to do bench-, pilot-, and field-scale evaluation of the performance of cementitious binders in S/S treatment of metal-contaminated wastes over time (Trish Erickson, 1992, personal communication). Performance will be measured in terms of lab leachability tests, solids composition and actual water quality of infiltration/runoff. Field measurements will extend over at least 5 years, while smaller tests are intended to simulate field results at a much-accelerated pace. The University of Cincinnati protocol for accelerated weathering testing described below can be tested in this project.

6.1.2 <u>Mechanisms</u>

Review and Analysis of Treatability Data Involving S/S Treatment of

<u>Soils</u>. This project is using geochemical equilibria models to determine minimally soluble forms of the eight Toxicity Characteristic Leaching Procedure (TCLP) metals. Emphasis is on identifying physicochemical forms of these metals that are relevant to the stabilization or solidification of typical hazardous wastes and the chemical conditions needed to produce the physicochemical forms of these metals.

These data are being analyzed to identify empirical or theoretical geochemical relationships that appear to govern the success of S/S applied to metal-contaminated soils. Relationships for multiple metal systems are being quantified, where possible (Means et al., 1991c).

<u>Morphology and Microchemistry of S/S-Treated Waste</u>. Scanning electron microscopy and X-ray diffraction techniques along with solvent extractions are being used to investigate waste/binder interactions. The objectives of these investigations are to better understand S/S processes by characterizing the binder phase composition and structure and the distribution of the contaminants in the solid phases, and to determine if microstructure can be correlated to macroscale physical properties (U.S. EPA, 1990f; and

several other papers in preparation). Contaminant distribution data include analysis of the contaminant concentration, chemical forms and crystal structure, and binding mechanisms in each phase.

Fate of PCBs in Soil Following Stabilization with Quicklime.

Several researchers have reported destroying polychlorinated biphenyls (PCBs) in contaminated soil by applying quicklime. These reports are based on retrospective data from site remediation programs, anecdotal information and results of one bench-scale project. Accordingly, an investigation was conducted to verify claims that use of quicklime alone can promote decomposition of PCBs. Synthetic soil samples were spiked with three PCBs and treated with quicklime and water. Significant PCB losses (60% to 85%) were evidenced after five hours of treatment. However, evaporation and steam stripping at elevated temperature conditions, rather than PCB decomposition, accounted for most of the losses observed. Low levels of partially dechlorinated PCBs were detected in lime-treated samples, but the quantities were stoichiometrically trivial. The amounts of observed dechlorination products were not dependent on the duration of lime treatment, and no evidence of phenyl-phenyl bond cleavage was found. The use of quicklime alone as an in-situ treatment for removal of PCBs is not supported by these results (U.S. EPA, 1991c).

<u>S/S Treatment of Salts of As, Cd, Cr, and Pb</u>. The behaviors of various metal salts in cement-based S/S processes are being studied through leaching tests, conduction calorimetry, and solid-state NMR. The research is aimed at identifying the chemistry involved during cement hydration reactions in S/S processes treating metal salts (U.S. EPA, 1990f).

The Nature of Lead, Cadmium, and Other Elements in Incineration on Residues and Their Stabilized Products. A detailed laboratory study of metal species in raw and S/S-treated wastes is being conducted to test how the chemical nature and binding state affect leachability. Focus will be on the application of sophisticated surface analysis techniques to characterize poorly crystalline inhomogeneous metal forms. Existing geochemical models will be applied to test if they can predict the formation of solubilitycontrolling solid phases as determined analytically (Eighmy et al., 1992).

6.1.3 Interferences

Factors Affecting the S/S Treatment of Toxic Waste. Research on interfering agents is being done to quantify the physical and performance characteristics of S/S-treated waste containing interfering chemicals. The data are being analyzed to determine whether physical properties can be correlated with durability and leach resistance. Interferences from inorganics such as Pb, Cd, and Zn and from sulfates and organics such as oil, grease, hexachlorobenzene, trichloroethylene, and phenol are being studied (Jones et al., 1992).

Effects of Selected Waste Constituents on S/S-Treated Waste Leachability. The effects of 10 common waste constituents on the strength and contaminant immobilization of S/S-treated waste were studied. The contaminants were cadmium, chromium, mercury, and nickel. The potential interferences were nitrate salts, sodium hydroxide, sodium sulfate, and five organic substances. The S/S binders tested were Portland cement, cement plus fly ash, and lime/fly ash (Jones et al., 1992).

6.1.4 Organics and Air Emissions

Roles of Organic Compounds in Solidification/Stabilization of <u>Contaminated Soils</u>. Organic compounds pose problems for solidification/ stabilization processes in three ways:

- 1. Nontarget organics can interfere with the immobilization of target metals.
- 2. Target organics are more difficult to stabilize than metals.
- 3. Some organics can volatilize during mixing with treatment agents, leading to unacceptable air emissions.

The University of Cincinnati, on behalf of the U.S. EPA, is evaluating the effectiveness of S/S processing for organic/metal wastes, in terms of organic immobilization and organic-induced effects on metal immobilization. Organic emissions during S/S processing are being measured. Polyaromatic hydrocarbons (PAHs) will be used in this project to represent a common class of organic compounds of concern in waste remediation.

<u>Measurement of Volatile Emissions from S/S-Treated Waste</u>. Although the mechanical strength and leaching characteristics of S/S-treated wastes have been investigated, few data are available on the emissions of organics from the S/S process and from the treated waste. Acurex Corporation at Research Triangle Park, North Carolina, is developing organic measurement methods and using them to test S/S-treated waste to address this data gap. A "Wind Tunnel" system, a "Modified Headspace" sampling system, and a "Sample Venting" system have been developed and are being used to measure organic releases from S/S-treated waste (Weitzman et al., 1990).

<u>Field Assessment of Air Emissions From Hazardous Waste S/S Process-</u> <u>ing</u>. The U.S. EPA is collecting information to develop standards necessary to control air emissions from hazardous waste treatment, storage, and disposal facilities. Field tests have been conducted to quantify emissions of volatile, semivolatile, and particulate emissions from S/S treatment processes (Ponder and Schmitt, 1991).

<u>S/S Treatment of Metal Wastes Contaminated with Volatile Organics</u>. S/S-treatment of sludge contaminated with about 1% metal ions and about 0.04 % VOCs was tested. Waste sludge containing 11 metal contaminants was spiked with 8 VOCs. Four different cement based S/S processes were applied to treat sludge samples (Spence et al., 1990).

Immobilization of Organics in S/S Waste Forms. U.S. EPA RREL is sponsoring a laboratory study to investigate (1) the immobilization of target organics by selected S/S formulations and (2) the effects of nontarget organics on the immobilization of target metals. Initial studies will be performed on spiked soils to systematically vary relative contaminant concentrations (Trish Erickson, U.S. EPA, personal communication, 1992).

6.1.5 Test Methods

<u>Method Development</u>. Laboratory and field test methods are needed to support optimum binder selection, assess short-term and long-term performance of S/S-treated waste, and allow better correlation of laboratory and field tests. A project is being conducted to study these three areas (U.S. EPA, 1991a):

- Evaluate the effect of sample size and configuration on results from leaching tests.
- Assess durability tests such as ANSI/ANS/16.1 and the accelerated aging/weathering protocol being developed through cooperative agreement between the U.S. EPA and the University of Cincinnati.
- Evaluate methods to monitor S/S-treated waste in situ.

Investigation of Test Methods for Solidified Waste. An effort was conducted with Environment Canada to evaluate several leaching and physical property measurement methods. This research is leading toward development of a protocol for evaluating S/S-treated waste. The protocol is based on the measurement of several physical, engineering, and chemical properties of S/Streated wastes to allow different use and disposal scenarios to be evaluated. Several of the testing methods in the protocol have been evaluated in a cooperative project with industry initiated by Environment Canada. Others are methods recommended by standards organizations in the fields of hazardous and radioactive wastes. Finally, some properties of S/S wastes were measured using methods in the developmental stage (Stegemann and Côté, 1991).

<u>Critical Characteristics of Hazardous S/S-Treated Waste</u>. The physical and chemical characteristics of the waste affect performance, as do the climatic (temperature and humidity) conditions during curing and after placement in the final disposal or reuse environment. This research is being conducted to determine the critical characteristics affecting waste performance and how to measure them. The work is leading to quality control procedures for use in the field to better assure performance of S/S-treated waste (Wiles and Howard, 1988).

<u>Advanced Test Methods</u>. A program evaluating test methods for construction materials and stabilized waste is ongoing at Enegieonderzoek Centrum Nederland (ECN). Aspects being dealt with are changes within the product with time, problems in determining the proper geometrical surface area, boundary conditions for modeling the release from products, development of a three-dimensional leaching model, and chemical speciation within a waste form. Testing involves radionuclide tracers in specific chemical forms in the S/S-treated waste (van der Sloot, ECN, personal communication, 1991).

Assessment of Long-Term Durability of Solidified/Stabilized Hazard-

<u>ous Waste Forms — Lab Component and Field Component</u>. U.S. EPA RREL is sponsoring a laboratory study of synthetic and real hazardous wastes to develop a protocol for accelerated weathering testing of cementitious waste forms. Durability testing is focused on the use of elevated temperature or acid to speed degradation reactions.

A field project is also being conducted to develop and utilize sampling and analysis methods that allow assessment of waste form durability after various periods of exposure to field conditions. Early efforts are concentrating on detection of the interface between buried waste forms and adjacent fill material. Subsequent work will focus on sampling to obtain surficial (<1 cm) weathered material for analysis as well as bulk sampling. The observed weathering patterns will be compared with those induced under laboratory or lysimeter conditions. (Trish Erickson, U.S. EPA, personal communication, 1992).

6.1.6 Leaching and Transport Models

<u>Contaminant Profile Analysis</u>. Chemical and X-ray diffraction analysis methods are being used to determine the composition profiles in blocks of S/S-treated waste that have experienced long-term leaching. These analyses evaluate the actual release from S/S-treated waste and provide insight into the processes occurring within the waste during leaching (Hockley and van der Sloot, 1991).

<u>The Binding Chemistry and Chemical Leaching Mechanism of Hazardous</u> <u>Substances in Cementitious S/S Binders</u>. Type I Portland cement samples containing the soluble nitrates of the priority pollutant metals chromium, lead, barium, mercury, cadmium, and zinc have been investigated using thermogravimetric and Fourier-transform infrared techniques, including diffuse reflectance. The major vibrational bands and thermal stability of the carbonate, sulfate, silicate, water, and nitrate species have been tabulated in comparison to uncontaminated Portland cement. Immobilization mechanisms and their effect on contaminant leaching are being studied (Ortego et al., 1989; Ortego, 1990; and Ortego et al., 1991).

Development of a Numerical Three-Dimensional Leaching Model. The overall goal of this research effort is to improve the fundamental understanding of binding chemistry and leaching mechanisms in S/S-treated waste and to apply this understanding to development of improved S/S technology and of improved methods for predicting the environmental impacts of disposing of S/Streated waste. This work is taking the approach of developing mechanistic leach models and developing characterization methods that can be used with the leach models. An underlying theme throughout this research is the need to separately describe the physical and chemical immobilization mechanisms. A set of simple leach models has been developed based on various simple reaction systems and rectangular geometry. Irreversible immobilization, reversible linear sorption, reversible precipitation, and reaction between a precipitate and inwardly diffusing reactant are the mechanisms considered in the simple leach model. A general numerical three-dimensional leaching model is being developed based on the Crank-Nicholson finite difference algorithm (Batchelor, 1991, personal communication).

<u>Acid Leaching Rate and Advancement of Acid Front in S/S-Treated</u> <u>Waste</u>. This program is studying the behavior of leaching of a cement-based waste form. The investigations indicate that acids in the bulk solution diffuse through the pores of the waste form leading to a reduction in pH and dissolution of metals.

The dissolved metals leach out of the solid matrix into the bulk solution, leaving a leached layer on the surface of the waste form. A sharp leaching boundary was identified in every leached sample, using pH indicators. The movement of the leaching boundary was found to be a single diffusioncontrolled process. Studies were conducted using both static and semidynamic leaching procedures (Cheng and Bishop, 1992).

Leaching Test Methods and Models. Several leaching mechanisms, including dissolution of the matrix, washoff of surface contaminants, and diffusion-controlled release, were studied. A variety of leach testing methods were described and the capabilities compared. A diffusion model for leaching was developed (de Groot and van der Sloot, 1992). Review and Analysis of Treatability Data Involving Solidification/Stabilization of Soils. A paper study of existing treatability data for S/S of 18 metals and application of geochemical models is being conducted to identify factors controlling metal solubility. The data base contains approximately 2600 records representing approximately 80 studies. Despite the volume of data, inconsistent data collection and procedural uncertainties limit interpretation. No statistically significant correlations could be found when post-treatment parameters were tested against measured waste characteristics. However, subsets of the data base will continue to be tested to identify chemical controls as the modeling work proceeds (Means et al., 1991a).

6.1.7 Compatibility with Disposal or Reuse

Assessment of Long-Term Durability of S/S-Treated Waste. The mechanisms governing the durability of S/S-treated waste are not well understood. Studies are needed to examine how the disposal environment interacts to modify the physical and chemical performance of the waste. In one study, S/S-treated waste is being tested to quantify waste form performance and examine degradation mechanisms. Testing involves accelerated freeze/thaw and wet/dry cycles and various environments, such as high or low pH, high pressure, high- or low-redox potential. Conventional and advanced large-scale leaching tests are being performed. The S/S-treated waste is being characterized by sophisticated techniques such as laser holography, acoustic stress wave testing, and dye injection (Bishop et al., 1990a).

Effect of Curing Time on Leaching. The effect of curing time on metal leaching, as measured by the TCLP test, is being studied in synthetic wastes for a variety of metal contaminants. Initial results indicate a significant effect of curing time, both on TCLP results and on the chemical structure of the stabilized waste as evidenced by spectroscopic analyses (Akhter and Cartledge, 1991; Cartledge, 1992). Both increased and decreased leaching is being observed, depending on the metal contaminant, binder, and other factors. These observations underline the limitations of the TCLP test as an indicator of the long-term leaching of stabilized waste and emphasize the need for other types of leaching data. Field Performance of S/S-Treated Waste. Solidification/stabilization is used at CERCLA sites and in other waste treatment applications. However, durability of S/S-treated waste remains unclear due, in part, to the relative newness of the technology and the lack of information from sites currently applying S/S processes. A three-phase project is under way (U.S. EPA, 1991b):

- Identify sites using S/S processes.
- Core sample and test S/S-treated waste from several sites.
- Design and implement a program to solidify representative wastes by various S/S processes and monitor the wastes over an extended period.

<u>Utilization and Disposal</u>. The performance of S/S-treated waste depends on the environment the material is exposed to as well as the treated waste and contaminant properties. The Waste Technology Centre in Canada is developing an evaluation protocol as a decision-making tool for management of S/S-treated waste. One factor in the protocol is identification and definition of use and disposal scenarios. Scenarios include unrestricted use, approved use, sanitary landfill, segregated landfill, and secure landfill (WTC, 1990b).

6.1.8 Treatability Tests and S/S Process Application

<u>Superfund Innovative Technology Evaluation (SITE) Program</u>. The SITE Program was established to accelerate the development and use of innovative cleanup technologies at hazardous waste sites across the country. The Demonstration Program of SITE focuses on field demonstration of emerging site remediation technologies. The Demonstration Program has 37 active tests, including the eight low-temperature S/S technologies summarized in Table 6-1.

<u>Municipal Waste Combustion Residue S/S Program</u>. Vendors of S/S processes are cooperating with the U.S. EPA Office of Research and Development Risk Reduction Engineering Laboratory to demonstrate and evaluate the performance of S/S processes for treating residues from the combustion of municipal solid waste (MSW). The program includes four S/S processes: cement, silicate, cement kiln dust, and a phosphate process. The aim of the project is to

TABLE 6-1. SUPERFUND INNOVATIVE TECHNOLOGY EVALUATION PROGRAM: SOLIDIFICATION/STABILIZATION TECHNOLOGIES

	Solidification/ Stabilization	Applicable	App1	Applicable Waste
	Technology	Waste Media	Inorganic	Organic
Chemfix Technologies, Inc. Metairie, LA	Soluble silicates and silicate setting agents	Soil, sludge, other solids	Metals	High-molecular- weight organics
HAZCON, Inc. Brookshire, TX	Cement and proprietary additive	Soil, sludge	Metals	Not an inhibitor
International Waste Technologies/Geo-Con, Inc. Wichita, KS	In situ — silicate and proprietary additives	Soil, sediment	Nonspecific	PCBs, other nonspecific organic compounds
S.M.W. Seiko, Inc. Redwood City, CA	In situ — proprietary binder	Soil	Metals	Semivolatile organic compounds
Separation and Recovery Systems, Inc. (SRS) Irvine, CA	Lime and proprietary additives	Liquid/solid	Low-level metals	Specific for acidic sludges with at least 5% hydrocarbons
Silicate Technology Corp. Scottsdale, AZ	Silicate, cementitious material, and proprietary additives	Groundwater, sludge, soil	Metals, cyanide, ammonia	High-molecular- weight organics
Soliditech, Inc. Houston, TX	Pozzolan or cement and proprietary liquid additives	Soil, sludge	Metals	Nonspecific
Wastech, Inc. Oak Ridge, TN	Proprietary	Soil, sludge, liquid waste	Nonspecific, radioactive	Nonspecific

Nonspecific = Technology is generally applicable to that waste type.

Sources: U.S. EPA, 1988d and Barth, 1991)

enhance the environmental performance of S/S-treated MSW combustion residue in a range of final environments. The final environment may be disposal in the land or use as roadbed aggregate, building blocks, or artificial reefs for shore erosion control (Wiles et al., 1991a and b).

Leaching Mechanisms and Performance of S/S-Treated Hazardous Waste Substances in Modified Cementitious and Polymeric Matrices. In this study, a latex polymer additive is being used with Portland cement to treat inorganicand organic-contaminated waste. The latex polymer is used to reduce the porosity of the S/S-treated waste in order to improve immobilization (Daniali, 1990).

Stabilization Potential of Lime Injection Multistage Burner (LIMB) Product Ash Used With Hazardous Distillation Residues. A study is under way to investigate the trace metal binding mechanisms in S/S high-sulfur coal fly ash and flue gas desulfurization (FGD) sludges. Fly ash and sludge from a typical wet FGD process and dry flue gas desulfurization by-product from a demonstration LIMB process are being evaluated. The latter material contains substantial portions of available lime and may prove amenable as a solidifying agent with the fly ash. This work is being done to characterize the waste, determine the solidified/stabilized waste formulation, and measure the influence of liquid/solid ratio on metal leaching from the waste forms (Bishop et al., 1992; Dusing et al., 1991).

<u>Stabilized Incinerator Residue in a Shore Protection Device</u>. The goals of this research are to stabilize potentially toxic incineration residues and to use the stabilized material to construct energy-deflecting or absorbing structures to reduce shore erosion. The initial phases of the project will deal with developing the proper mix design for stabilized materials in high-wave energy environments and with determining their engineering properties, leachate characteristics, and potential toxicity to organisms. Permits will be secured to construct a model wave deflector/absorber in a marine system. The actual construction will occur in the next phase (Swanson, 1990).

6.2 FUTURE DEVELOPMENT

For more than 20 years, S/S processes have been used to treat industrial and radioactive waste. More recently, the technology has been used to treat contaminated soils at CERCLA sites, fly ash, incinerator ash, and metal-contaminated sludges.

Despite extensive application and considerable research, there still are areas that could profit from additional effort. An increased understanding of S/S mechanisms, interferences, leaching behavior, and long-term performance would all help to improve process efficiency and increase confidence in the technology. Some areas to consider for future research are summarized in sections 6.2.1 through 6.2.8.

6.2.1 Binders

- Increase immobilization performance by modifying existing binders.
- Develop advanced binders to minimize volume increase inherent in most existing S/S processes.
- Develop advanced binders with better tolerance to organic contaminants and interferences.
- Determine factors affecting optimum binder addition rate. Too much of a particular binder ingredient can lead to an improperly stabilized waste form. For example, many metals are amphoteric, meaning that they are soluble under both acidic and alkaline conditions. The metal will be at minimum solubility when a sufficient base (S/S ingredient) is added to make the waste moderately alkaline. Too much base will cause the metal to resolubilize and/or make the waste hazardous by virtue of the RCRA corrosivity characteristic (i.e., pH >12.5).

6.2.2 Mechanisms

- Develop an understanding of chemical speciation and how it affects immobilization.
- Gain understanding of S/S process bonding mechanisms with presently used binders and additives.
- Gain understanding of microstructure and chemistry of the complex interactions among binder phases and contaminants (McDaniel et al., 1990).

6.2.3 <u>Interferences</u>

- Organic matter in the waste can prevent setting of the S/S-treated waste or reduce the strength or immobilization performance of the final product. Research is needed to determine threshold levels for interfering organic compounds with inorganic and organic S/S binders.
- Interfering agents should be classified into groups based on similarity of interference mechanisms. Once the mechanisms are defined and interferences grouped, control parameters could be set for interfering chemicals such as volatile organics, insoluble organics, soluble organics, soluble salts, sulfates, and ammonia.

6.2.4 Organics and Air Emissions

- Develop methods to efficiently remove organic contaminants from sludge, soil, and soil-like wastes (Barth, 1990).
- Develop methods to determine whether bonding occurs between binder and organic waste. Increased understanding of the mechanisms for organic immobilization will speed development of better binders for organic contaminants.

6.2.5 Test Methods

- Characterize the chemical interaction within the S/S-treated waste and at the waste/soil interface by diffusion tube measurements with radiotracers.
- Develop methods to more accurately predict and measure the performance of S/S processes and products in the laboratory and to improve the correlation of laboratory results with performance in the field (McDaniel et al., 1990).
- Develop and evaluate simple methods for determination of metal speciation for use in binder evaluation and selection.
- Develop and evaluate methods for inexpensive determination of metal speciation.
- Develop better test methods for detailed research of S/S-treated waste performance (e.g., X-ray fluorescence, computer imaging, laser holography).

- Identify factors affecting scale-up of treatability test results to determine the safety margin needed in performance measures. Scale-up from bench-scale to field-scale involves a number of variables that cannot be exactly replicated in the bench-scale experiments, e.g., field-curing conditions, degree of mixing, and ingredient control, among others. Therefore, the results of the bench-scale tests should exceed the performance measures for the field project by a wide enough margin to allow for unknown contingencies. As a general rule, if a bench-scale test meets the field performance measures by only a slim margin, then one may expect problems with fullscale implementation.
- Quantify the effect of the small-scale treatability test environment on S/S-treated waste performance. The jar environment promotes good contact between the binder and waste form and can enhance the degree of stabilization.

6.2.6 Leaching and Transport Models

- Develop approaches to better predict field performance from laboratory results.
- Quantify containment release rates by diffusion and advection over long-term exposure to environmental conditions. Use the transport data to evaluate the acceptability of the release rates.
- The TCLP does not fully address the main leaching mechanisms for many organics. In many cases, the organics in leachates are associated with particulate matter. Methods need to be developed to assess the fraction of organics mobilized by mechanisms not directly related to diffusion or dissolution such as sorption on particulates.
- Develop better, more economical, and more rapid leaching tests that allow reliable prediction of long-term performance of S/S-treated waste.

6.2.7 Compatibility with Disposal or Reuse

- Identify and validate methods to produce S/S-treated waste that can be reused or recycled (Barth, 1990).
- Determine the long-term physical durability and contaminant retention properties of S/S products by the following means:

- Define the physical and chemical environments for various end uses.
- Develop accelerated weathering tests.
- Define biodegradation potential.
- Determine the relative merits of granular versus monolithic materials.
- Analyze the conditions needed for long-term environmental protection for S/S-treated waste placed in a disposal or use environment. Analysis will include determination and evaluation of the ultimate release pathways.
- Evaluate and develop criteria for reuse of S/Streated waste (e.g., bricks or subgrade fill).

6.2.8 <u>Treatability Tests and S/S Application</u>

- Determine the effectiveness of S/S processes and equipment for treating contaminated soil and impounded liquid.
- Determine effectiveness of mixing methods (including in situ methods).
- Evaluate effectiveness of slag addition or other pretreatment options to alter the valence states of metal contaminants prior to S/S processing.
- Establish a database recording important characteristics of S/S processing, such as binders, waste characteristics, interferences, and performance.
- Develop expert systems to aid in planning and evaluating treatability studies, S/S processes, and pretreatment options. The expert systems can be used to screen potential S/S processes for specific waste types and contaminated site conditions.
- Develop real-time QA/QC methods for S/S process control.
- Evaluate uses, based on experience with S/S treatment of industrial sludge, for similar wastes such as dredged materials from harbors and waterways or ashes and residues from combustion of coal and municipal solid waste.
- Develop strategies to optimize sample collection and analysis to increase efficiency and reduce cost.

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APPENDIX A

SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS

INTRODUCTION

This section summarizes the steps in the technology screening process for S/S technology. It provides a checklist of the material described in detail in Chapter 2. The organization of the checklist parallels the organization of Chapter 2, integrating the issues covered in that section into a user-friendly format. The checklist worksheets help the uninitiated user to follow orderly and comprehensive screening procedures. The screening could be repeated at several stages throughout a project, as appropriate. For the first use, the checklist would serve as a tool to guide preparation of test plans. The checklist would then be applied at major milestones, such as selection of an S/S process or completion of bench-scale screening, to review progress, identify weaknesses in the project, and develop methods to improve the testing. Later in the testing the checklist would be applied to review and evaluate the project.

INSTRUCTIONS

Each major subheading in the checklist is followed by 1) a brief statement or question that clarifies the scope and aspect of S/S technology covered in that section and 2) a series of questions to guide evaluation of the S/S project with respect to that aspect. The question can be evaluated as "favorable," "neutral," "unfavorable," "not known," or "not applicable." "Favorable" means lower complexity or a higher probability of success for the S/S project. "Neutral" means that the issue has a known effect but the effect is not significant to the outcome of the project. "Unfavorable" means greater challenges to S/S technology. "Not known" means there is high probability of an effect but the magnitude and/or direction are not known. "Not applicable" means a low probability of any effect. The questions are typically clarified or elaborated with notes in the "Issues" column. In most cases the evaluations are qualitative, but in a few cases quantitative performance criteria are given as guidance. Typically, an answer of "yes" to the question equals a favorable condition. Cases where the reverse is true are noted.

A summary sheet for tallying the responses for each subheading is provided at the conclusion of this chapter. The purpose of the summary sheet

is to assist in identifying trends or possible weaknesses in the treatability study.

Not every issue listed in the checklist is applicable to every treatability study. Irrelevant issues should be ignored. It is hoped that, through consideration of the issues contained herein, future S/S treatment projects can be improved in terms of both planning and conduct.

					Indica	tor		
		Information Requirements	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	Issues
		ECIFIC BASELINE INFORMATION EMENTS						· · ·
1.1	ac ch	aste Sampling - Do the waste samples curately reflect the chemical and physical aracteristics of the entire volume of the aste?						
	1.	Are preliminary field surveys available?						Planning for sampling
	2.	Are waste sampling procedures documented and consistent with guidance in SW-846 (U.S. EPA, 1986a) and/or other agency guidance?						Representativeness, holding times, chain-of-custody, etc.
	3.	Are sampling locations statistically randomized?						Representativeness
	4.	Is sample variability addressed by statistical analysis?						Representativeness
	5.	Were samples composited prior to analysis?						Composites preferred for comparative treatability testing but do not define extremes in waste composition. Variation is particularly important for testing of continuous processes, e.g., pug mill mixing.
	6,	Were debris, large rock fragments, vegetative material, etc. removed prior to analysis?						Representativeness
	7.	Is material available sufficient for pilot- scale testing?						Need to support waste characterization and bench- and pilot-scale tests.
	8.	Is some material being archived for possible later tests?						QA/QC

			I	ndica	tor		
	Information Requirements"	Favorable	Neutral	Unfavor able	Not Known	Not Applicable	Issues
1.2	Waste Acceptance - Is the waste material toxicity low enough to allow contact handling needed for S/S testing and application?						
	1. Was a representative sample analyzed prior to shipping?						Identification of chemical hazards
	2. Is waste composition in compliance with shipping regulations?						Toxicity and U.S. DOT shipping regulations
	3. Is the hazard to S/S workers acceptably low?						Worker safety
1.3	Waste Characterization - Is there an adequate, statistically valid database to support selection of binding agents?	1					
	1. Is historical information available?						Optimize data collection
	 Does characterization include a "total waste analysis"? 						Identify target contaminants
	3. Were TCLP data generated on the untreated waste?						Baseline leaching data; RCRA toxicity characteristic
	4. Have other hazard characteristic tests been performed or are they known to be unnecessary?						Ignitability, corrosivity, reactivity, toxicity, infectivity
	5. Have other chemical analyses been performed to establish baselines and possible S/S interferences?						pH, redox potential, acid neutralization capacity, etc.; Interferants screen, e.g., oil and grease, salt content, nitrate, sulfate, etc.
	6. Have baseline physical characteristics of the untreated waste been measured?						UCS, specific gravity, Paint Filter Test, permeability, etc.

* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

				Indica	ator	······	• · · · · · · · · · · · · · · · · · · ·
		favorable	lar.	Jnfavorable	Not Known	Not Applicable	
	Information Requirements	Favo	Neutral	Unfa	Not	Not	Issues
	7. Are any other data available on physico-chemical form of the ta contaminants?						X-ray diffraction, SEM-EDXA, microscopy, spectroscopy, etc.
1.4	Site Characterization - Are fundame characteristics established to give ba data for the design of the treatment	Iseline					
	1. Does the site support the setup operation of S/S equipment?	and					Available space, topography, excavation difficulty, climate
	2. Are necessary resources close to site?	o the					Design flexibility
	• Water, gas, electricity						
	• Supplies and chemicals						
ų	• Equipment						
Ň	Access routes						
	• Disposal facilities						
	3. What proportion of the waste or above the groundwater table (or uppermost aquifer)?						Excess water can make excavation difficult and require dewatering of waste material.
	100% = favorable						
	4. Has the total waste volume been estimated, measured, or calculate						Smaller volumes, more limited treatability study; larger volumes, more extensive treatability study
	5. Does the waste contain debris th interfere with field treatment?	at may					Pretreatment and handling requirements; interferences may be process-specific.
	no = favorable				ļ		e process-specific.

* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

		<u></u>	I	ndica	tor		
	Information Requirements*	Favorable	Neutral	Unfavorab le	Not Known	Not Applicable	Issues
		1	Γ				133469
	6. What are the textural characteristics of the waste?						Pretreatment and handling requirements
	dry, granular = favorable clayey, sludge, or liquid = neutral hard, blocky = unfavorable						
	7. How heterogeneous is the distribution of the target contaminant(s) within the waste?						More analytical data needed to compensate for higher variability.
•	fairly homogeneous = favorable						
1.5	Quality Assurance/Quality Control - Is QA/QC sufficient to determine and document data quality?						
*	1. Does the analytical laboratory performing the analyses on the untreated waste possess appropriate qualifications/certifications?						CLP, other qualifications/certifications
	2. Are the characterization data collected under an appropriate QA/QC program, or is there some other indication of the quality of the analytical measurements?						Blind replicates, duplicates, bracketed calibration, standard additions, blanks, etc.
	3. Are there a sufficient number of replicates analyzed to permit a statistical analysis of the results?						Mean, standard deviation, confidence intervals, etc.
	4. Is a second analytical laboratory available for interlaboratory verification on a portion of the more critical measurements?						Data accuracy, interlaboratory verification

Indicator Not Applicable Unfavorable Not Known Favorable Neutral Information Requirements* Issues PERFORMANCE OBJECTIVES 2 2.1 Regulatory Requirements - Have CERCLA and RCRA regulatory-driven requirements been considered in developing performance requirements? 1. Is the site close to possible receptors of Possible source of locationnoise, fugitive dust, volatiles, or odors? specific ARAR 2. Is the site close to sensitive Possible source of locationenvironmental areas such as floodplains, specific ARAR wetlands, or the breeding grounds of protected species? 3. Are the primary contaminants metals or S/S BDAT for many metals; some organics, or both? types of organics may require pretreatment unless present in low metals = favorableconcentrations. metals and organics = neutral organics only = unfavorable 4. If mostly metals, how many metals are Potential for incompatible present in regulated concentrations? chemistries; complex wastes are more difficult to satisfactorily 1 = favorablestabilize 2-3 = neutral4 or more = unfavorable 5. If arsenic and chromium are among the Toxicity issues; may affect binder target contaminants, have their valence selection; data may also be states been determined? inferred from waste origin in some cases.

SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

" An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

			<u> </u>	I	ndicat	tor		
			_ ·		ble	Ę	icable	
			Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
		Information Requirements	E.	ž	5	ž	ž	Issues
	6.	If mercury, nickel, tin, arsenic or lead is among the target contaminants, are analyses planned for organic (e.g., tetraethyl lead, tributyl tin, organoarsenic) or other unusual and						Toxicity issues; may affect binder selection; data may also be inferred from waste origin in some cases.
	7.	toxic forms (e.g., nickel carbonyl)? Does the waste contain volatile organic contaminants and, if so, in what concentrations?			:			Levels of concern will vary with the contaminant; S/S not demonstrated for volatiles; probable release during mixing
•		no or < 50 ppb = favorable						and curing; pretreatment probably necessary.
	8.	Does the waste contain other high hazard or special contaminants, such as PCBs, dioxins, pesticides, chlorophenols, radionuclides, or cyanide?						Levels of concern vary with the contaminant; pretreatment will likely be necessary; S/S may not be preferred approach, unless a strong rationale is provided.
		no = favorable						
	Hav con	chnical and Institutional Requirements - ve technical and institutional factors been usidered in developing performance uirements?						
	1.	Will testing determine the leaching (e.g., TCLP) or physical properties (e.g., compressive strength) of treated waste?						Demonstrate basic feasibility.
	2.	Are reagent costs consistent with project economics?						Calculate binder cost per volume of stabilized waste.
	3.	Does the waste contain compounds that may decompose or volatilize to produce off-gas?						Off-gas treatment increases processing costs.
		no = favorable						

			<u> </u>		ndica			
		Information Requirements*	Favor able	Neutral	Unfavorable	Not Known	Not Applicable	Issues
	4.	Will the waste mix well with the binder	?					Good mixing and wetting is needed to ensure a strong, uniform product.
	5.	Does the waste interfere with setting or cause unfavorable reactions with the binder?						Interferences should be identifie
		no = favorable						an a
	6.	Is the waste/binder mixture fluid and amenable to material handling and mixing?						Pumpable waste/binder mix mak handling easier.
	7.	Does S/S increase waste volume significantly?						Large volume increase raises cos and increases disposal problems.
		no = favorable						
	8.	Is the S/S-treated waste amenable to placement?						Need long-term structural integri and ability to support heavy equipment soon after placement.
	9.	Is the binder material subject to possible biodegradation?						Long-term stability
		no = favorable						
	10.	Are longer-term leaching tests on the treated waste planned?						TCLP is not a good indicator of long-term stability.
IN	ITIA	L TECHNOLOGY SCREENING						
3.1	Has alter	hnology Screening/Feasibility Study - S/S been compared to other treatment matives and been found to be the most ropriate technology?						

			I	ndicat	tor		
- - -	Information Requirements	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	Issues
3.1.1	CERCLA Technology Screening						
	1. Do the selected methods protect human health and the environment?						Methods should attain threshold criteria.
	2. Do the selected methods meet ARARs?						Methods should attain threshold criteria.
	3. Do the selected methods reduce toxicity, mobility, or volume?						Methods should provide good trade-off of primary balancing criteria.
	4. Do the selected methods minimize impact to human health and the environment?						Methods should provide good trade-off of primary balancing criteria.
	5. Do the selected methods reliably maintain low residual risk to human health and the environment?						Methods should provide good trade-off of primary balancing criteria.
	6. Do the selected methods allow efficient, cost-effective application at the site?						Methods should provide a good trade-off of primary balancing criteria.
	7. Are the selected methods likely to receive state acceptance?						Modifying criteria are evaluated after the public comment period.
	8. Are the selected methods likely to receive community acceptance?						Modifying criteria are evaluated after the public comment period.
3.1.2	Technology Screening at RCRA TSD Facilities						
	1. Is the waste banned under another regulatory system such as TSCA?						Review waste for suitability of S/S treatment.
	yes = not suitable for S/S						

* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.



			ŀ	ndicat	or		
		able .	al I	Unfavorable	Not Known	Not Applicable	
	Information Requirements*	Favorable	Neutral	Unfav	Not K	Not A	Issues
· ·	2. Is the waste classified as "not suitable" for S/S or land disposal under the landbans, or is a technology other than S/S recommeded as BDAT?						Adherence to RCRA landban and BDAT recommendations
	yes = not suitable for S/S						
	3. Is the waste not yet covered or extended under landbans?						Landban requirements
	yes = S/S not required4. Does the generator certify that the waste						Landban requirements
	yes = S/S not required						
	 5. Is the waste restricted or banned under site permit conditions or otherwise unacceptable to a TSD facility? 						Permit compliance
	yes = not suitable for S/S						
	6. Is treatment required to prepare waste for a TSD facility's S/S system?						Treatment process complexity
	yes $=$ less favorable						
3.2	General Criteria for Not Using S/S - Is the waste compatible with S/S technology?						na an an Araba an Araba. An Araba an Araba an Araba an Araba an Arab
	1. Is the waste amenable to recycling, reuse, or recovery technology, all other factors being equal?						Recycling, reuse, and recovery are preferred over treatment or disposal.
	no = favorable for S/S						
÷							

* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

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	-		olo	g	Not Applicable		
	Favorable	7	Unfavorabl	Not Known	ppli		
	ů0/	Neutral	favi	Ľ.	Ę ,		
Information Requirements	Far	Ň	n -	Ŷ	Ň	Issues	
· · · · · · · · · · · · · · · · · · ·		T	Τ				
2. Is the waste treatable by a destruction						Contaminant destruction is	
technology, all other factors being						preferred over disposal.	
equal?						presented over disposal.	
no = favorable for S/S							
3. Are there ARARs that cannot be						Can S/S meet regulatory	
satisfied by existing S/S technology?					1	requirements?	÷.,
no = favorable for S/S							
4. Is S/S waste treatment inefficient or						Cost effectiveness	
expensive when compared to another					1		
remedy?							
no = favorable for S/S							
5. Does the waste exhibit poor mixing,						Amenability to S/S	
incompatibility, or other unacceptable						-	
characteristics?							
							14
no = favorable for S/S							
6. Does the waste contain volatile organics						Organics can be difficult to	· · ·
or a large fraction of total organics?						stabilize.	
no = favorable for S/S							
				1			
ASTE/BINDER COMPATIBILITY							
ITERATURE SCREENING - Has a omprehensive review and selection process						•	
und a group of test S/S binder formulations							
at have a high probability of providing good							
abilization?							
	1						



]	Indica	tor		
				Information Requirements*	Favorabie	Neutral	Unfavorable	Not Known	Not Applicable	Issues
			2.	Are interferences and chemical incompatibilities considered as part of the binder selection? Has metal chemistry been considered in the binder formulation? Is S/S-treated waste compatible with the planned end use? Are the S/S costs known, and are they competitive with other treatment and disposal methods? Does the S/S process have a proven track record on similar wastes?						 Pozzolanic binders are incompatible with high concentrations of oil, grease, organics, chlorides, and other soluble salts. Sodium sulfite binder is incompatible with acids. Formation of metal hydroxides is an important stabilization mechanism with alkaline binders; however, high pH can increase the solubility of some metals (e.g., As and Cr). Possible end use includes disposal such as landfill, monofill, or burial or reuse as fill, road base, or construction material. Cost is a consideration but should be secondary to performance. While proven performance is desirable, innovative methods should not be discouraged.
5	S N t a s s t t c	SCR MIX be co in in teps implester	EE TU ond tern le t l ag nia.	ATORY BENCH-SCALE NING OF THE WASTE/BINDER RES - Although laboratory screening can ucted in a variety of ways, it is typically active process involving two sequential A wide range of formulations are given ests. Then a more refined group are gainst more complex or demanding Test criteria and issues are discussed						

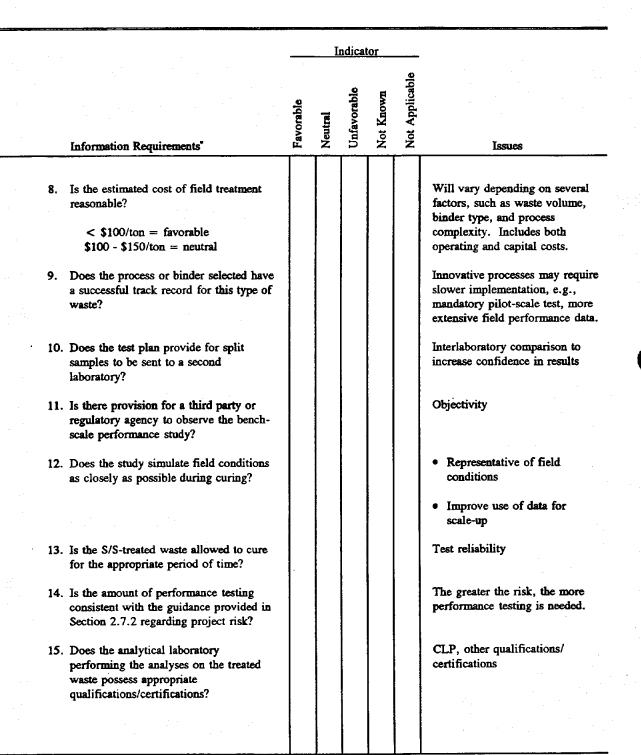
			[ndicat	LOT		
						ble	
		favorable	la l	Unfavorable	Not Known	Not Applicable	
	Information Requirements	Favo	Neutral	Unfa	Not]	Not ,	Issues
	·····	1					
1.	Has an appropriate pretreatment step been devised, if necessary?						Highly toxic constituents; contaminants that do not respond well to S/S; interferants; debris
2.	Have at least 3 to 4 different binders been selected for bench-scale testing?						Maximize potential for successful treatability study.
3.	Are several different binder-to-waste ratios used in the testing?						Cost/benefit; excess binder may hinder S/S process.
4.	Have waste/binder compatibility issues been considered in selecting a binder?						Target contaminants; interferants; compatibility with disposal environment
5.	Is laboratory testing being based on composite or "worst-case" samples, or both?						Composite best for process comparison; may be necessary to design for worst case.
	issue was considered = favorable						
6.	Are any chemical additives to the binder carefully monitored and controlled?						Reproducibility, interpretability, sensitivity analysis
7.	Are several rounds of bench-scale testing performed, i.e., have the most successful processes been adapted to the site-specific waste form?						Process optimization is an iterative process; ability to "engineer" solutions to treatability problems
8.	Are the chemical compositions of the binder and of any other chemicals added during S/S (e.g., fairy dust) known?						Hazardous properties
9.	Are all of the additives mentioned in item 8 above nontoxic and nonhazardous?						Corrosivity (pH), reactivity (free sulfide or lime), etc.

			<u> </u>	ndicat	lor		
	Information Requirements*	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	Issues
10	 Are there any new ARARs that result from the binder additives? no = favorable 						Toxicity and hazard characteristics, e.g., pH, reactive sulfide, metal leach criteria, volatile emissions, dust, etc.
11	I. Is there provision for a third party or regulatory agency to observe the treatability study?						Objectivity
12	2. Were anticipated field conditions simulated during waste curing?						"Jar effect" enhances performance
13	B. Were the samples allowed to cure for an appropriate time period prior to analysis?						28 days recommended before UCS testing for most pozzolans
. 14	I. Does the test program cover critical ARARs?						Leaching and critical chemical/physical properties
15	5. Does the test plan provide for split samples to be sent to a second laboratory?						Interlaboratory comparison to increase confidence in results
16	 Does the test include good statistical design, replication, blind controls, laboratory QA/QC, etc? 						Data accuracy and reliability
17	7. Is the waste volume increase resulting from binder additions determinable from the test?						End use compatibility, economical feasibility
							and a second second Second second second Second second

		Indicator						
		Favorable	뎥	Unfavorab io	Vot Known	Not Applicable		
	Information Requirements"	Favo	Neutral	Unfa	Not 1	Not /	Issues	
	BENCH-SCALE PERFORMANCE TESTING/PROCESS OPTIMIZATION - Does the bench-scale performance test demonstrate that the S/S-treated waste meets predetermined performance standards?							
	1. Are the guidelines applied in the bench- scale screening also considered in the bench-scale performance testing?						Completeness and consistency	
	2a. If subsurface disposal is anticipated, are appropriate physical tests being conducted?						e.g., UCS, permeability etc.	
	2b. If surface or near-surface disposal is anticipated, are the appropriate physical tests being conducted?						e.g., wet/dry, freeze/thaw, etc.	
	2c. Is the longer-term stability of the waste toward leaching being evaluated?						e.g., multiple extraction procedure, ANSI/ANS/16.1, etc.	
	2d. For wastes containing organic contaminants with low aqueous solubilities, are leaching tests in an organic solvent being conducted?						Aqueous leachate is a meaningless indicator of process effectiveness because of low solubility of contaminant.	
	2e. Are there any technical reasons to suspect that colloidal contaminant transport may be important at this site?						Assess in leach test by modifying or eliminating filtration step.	
	no = favorable							
	2f. Is there any technical reason for conducting leach tests with site-specific groundwater as leachant?no = favorable						e.g., humic-rich groundwater, or groundwater with other complexing ligands (e.g., carbonate, fluoride, high chloride, etc.)	

]	indica	tor		
Information Requirements*	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	Issues
2g. If the binder is biodegradable, is a biodegradation performance test being conducted?						Waste form stability
2h. If the disposal site could potentially leak into an aquatic system, are leachate bioassays being performed?						Leachate toxicity to aquatic ecosystem
2i. Are specific binding agent properties considered in the test plan?						Examples include:
						• pH and reactive sulfide analyses for sulfide-containing treatment chemicals
						 biodegradation tests for thermoplastic or other organic binders
3. Is a total metal analysis being performed on the same subsample as the leach test?						Eliminate false negatives.
4. Have the leaching performance data been corrected for dilution by binder additives?						Subtract out effect of dilution.
5. Is there a safety margin in the performance data compared to the performance criteria?						Mixing, ingredient control, and curing environments are not as well controlled in the field.
6. Is the process implementable in the field?						Materials handling issues; process complexity; mixing, throughput, and storage requirements
7. Is the bulking factor (volumetric expansion of the waste due to binder additives and water) compatible with disposal constraints?						Criteria will vary depending on the site.
< 25% expansion = favorable						

* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.



				I	ndica	tor		
	Inform	nation Requirements"	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	Issues
	under or is ti quality 17. Have a been a	the performance data collected an appropriate QA/QC program, here some other indication of the v of the analytical measurements? a sufficient number of replicates nalyzed to permit a statistical is of the results?						Binder replicates, duplicates, bracketed calibration, standard additions, blanks, interlaboratory verification, etc. Mean, standard deviation, confidence intervals, etc.
)T-SCALE / IONSTRATI							
7.1	technical, 1 confidence binder/was	for Process Scale-Up - Is regulatory, and institutional in the S/S binder and te ratio high enough to obviate the ench-scale testing?						
		e binder been used successfully in pplications?	L					Field application increases confidence.
	physic: similar	he waste to be treated have al and chemical characteristics to waste successfully treated in a ield application?						 Similar wastes' characteristics imply similar binder performance. Particular attention should be
								given to complex mixtures and possible interferences.
	3. Are sit	e surroundings similar?						Review site-specific performance and institutional issues.
	4. Are re	gulatory requirements similar?						Site-specific regulatory issues and ARARs
	5. Are pr unders	ocess scale-up issues well bood?				-		 Material handling Mixing Vapor evolution

				I	ndica	tor		
			Favorable	Neutral	Unfavorab le	Not Known	Not Applicable	
<u>.</u>		Information Requirements"	Fav	Zer Zer	C III	ž	Not	Issues
ı	б.	Are process costs known?						• Pilot plant test will improve accuracy of cost estimate.
	7.	Is waste reasonably homogeneous and well characterized?						Waste composition variations can affect S/S binder performance.
7.2		ale-Up Issues - Do your pilot-scale tests dress the major remediation steps?						
•	1.	Is the performance of earth-moving or other waste removal equipment known?						 Throughput Free liquid handling Operator safety
	2.	Is the performance of material-handling equipment known?						 Throughput Caking/Plugging Spillage
	3.	Is the storage and handling system for the S/S binder known?						Inventory needsThroughputSpace
	4.	Is waste pretreatment needed to improve material handling?						 Size adjustment by crushing and/or screening Moisture adjustment
	5.	Is waste pretreatment needed to improve binder compatibility or efficiency?						Blending, homogenization, pH adjustment, volatile organic removal
	6.	Are the mixing system for the S/S binder and the waste disposal approach known?						In situ, batch, continuous
	7.	Is the S/S-treated waste disposal approach known?						Handling, placement, compaction, moisture content, final closure and capping
		n an						

			<u> </u>	ndica	tor		
• • •		e.		able	IIMO	Not Applicable	
		Favorable	Neutral	Unfavorable	Not Known	ot App	
	Information Requirements*	<u><u></u><u></u><u></u><u></u><u></u></u>	Ž	5	ž	Ž	Issues
7.3	Analytical Testing of the Treated Waste - Is sampling and analysis of pilot plant S/S- treated waste sufficient to determine performance?						
	1. Is basic testing included?						Leaching and physical strength
	2. Are additional tests required?						Permeability, moisture content, chemistry
							an an an guideach a' stàiteach ann an t- ann an t-airteachtachtachtachtachtachtachtachtachtacht
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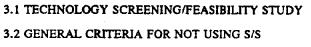
SUMMARY SHEET

- I. <u>Site</u>:
- II. <u>Reviewer</u>:
- III. Date:
- IV. <u>Review Summary</u>:

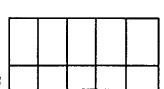
1.1 WASTE SAMPLING		
1.2 WASTE ACCEPTANCE		
1.3 WASTE CHARACTERIZATIO	N	
1.4 SITE CHARACTERIZATION		
1.5 QUALITY ASSURANCE/QUA	LITY CONTROL	
	Subtotal, Waste and Site Char	acterization
· ,		

2.1 REGULATORY REQUIREMENTS2.2 TECHNICAL AND INSTITUTIONAL REQUIREMENTS2.3 APPROACH TO SETTING PERFORMANCE CRITERIA

Subtotal, Performance Objectives



Subtotal, Initial Technology Screening





Not Applicable

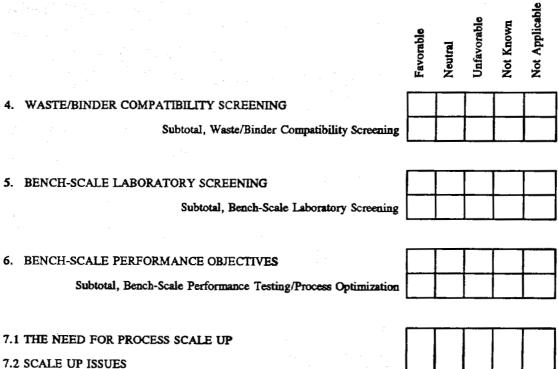
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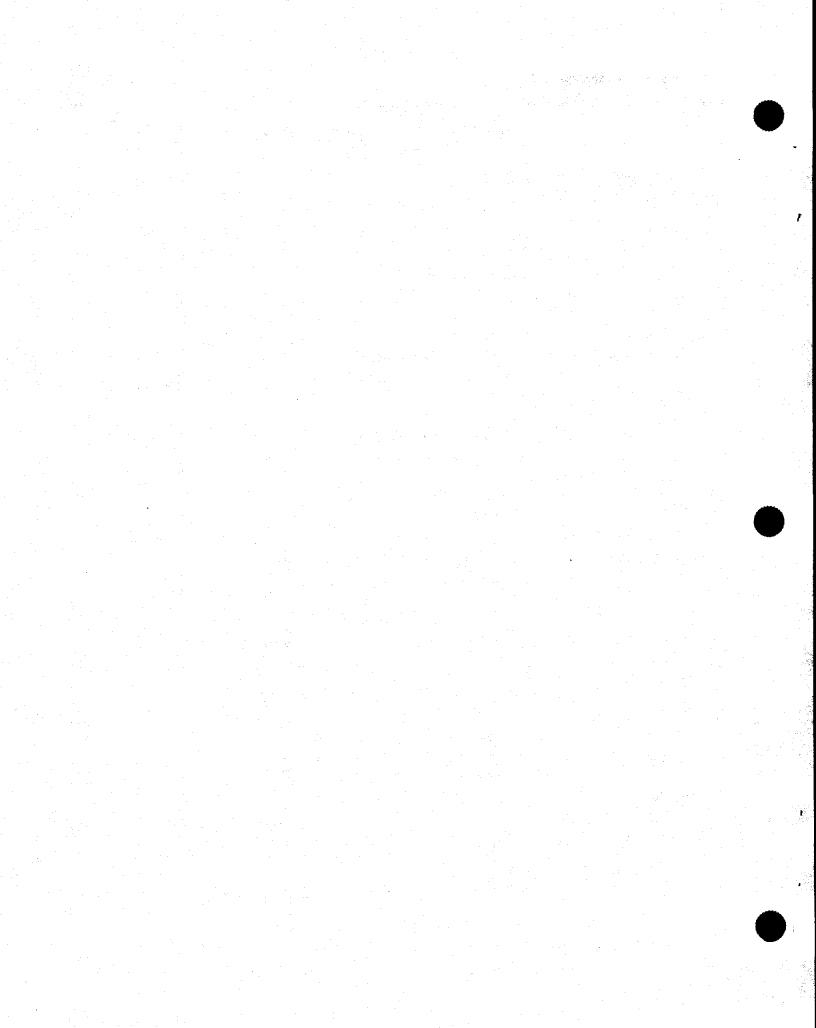
Not Known

SUMMARY SHEET (Continued)



7.3 ANALYTICAL TESTING OF THE TREATED WASTE

Subtotal, Pilot-Scale and Field Demonstration



APPENDIX B

DRAFT REPORT: SAMPLING AND ANALYTICAL PROCEDURES

Note: The sampling and analytical procedures document presented in this appendix was developed for sampling piles of waste material contaminated with copper and lead. The document is included here only as an example and has been modified to protect client confidentiality.

DRAFT REPORT

SAMPLING AND ANALYTICAL PROCEDURES

February 25, 1992

by

Andrea Leeson Jeffrey Means Gregory Headington Bruce Buxton

BATTELLE Columbus Division 505 King Avenue Columbus, Ohio 43201-2693



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DRAFT REPORT

FOR

SAMPLING AND ANALYTICAL PROCEDURES

1.0 INTRODUCTION

[The introduction is specific to each project and should briefly describe the project background and objectives.]

2.0 PROJECT SCOPE

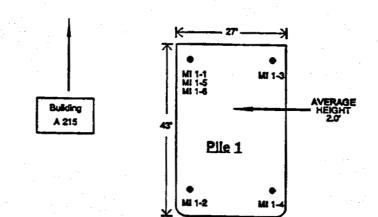
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The existing waste consists of three accumulated piles of material situated on pavement in an approximately rectangular shape (Figure 2-1). Approximate estimates of the dimensions of the piles are: Pile 1: 43 ft by 27 ft and 2 ft deep; Pile 2: 53 ft by 38 ft and 2 to 2.5 ft deep; and Pile 3: 53 ft by 20 ft and 3 ft deep.

A preliminary sampling of the waste was conducted by Battelle to obtain an estimate of the number of contaminants of concern as well as the concentrations. In addition, previous sampling of other similar which had been collected in rolloff boxes and stored in the parking area was analyzed in order to obtain a better estimate of the contaminants likely to be found in the piles. Copper and lead were the primary contaminants from both sampling surveys. Average concentrations of copper and lead from the rolloff boxes and piles are shown in Tables 2-1, 2-2, and 2-3. These preliminary measurements of the metal concentrations were used to design the sampling program.

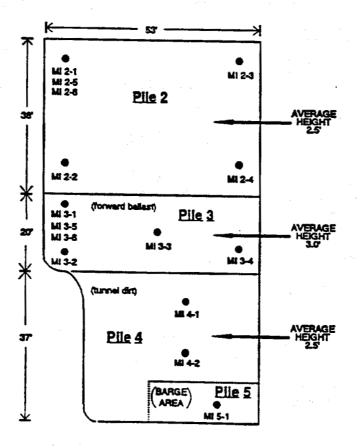
The waste tends to be fairly uniform in consistency throughout, but possible variations in metal concentrations require that samples be collected at varying locations, both spatially and as a function of depth. Specific details of the sampling design are discussed in the following section.

(B-6)



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(B-7)

Results by Analytical Methods	Copper	Lead
STLC		
Regulatory Threshold (mg/L)	25	5.0
Mean (mg/L) ¹	35	2.2
Coefficient of Variation	0.97	0.43
TILC		
Regulatory Threshold (mg/kg)	2500	1000
Mean (mg/kg) ¹	3240	28
Coefficient of Variation	0.33	0.40

TABLE 2-1. SUMMARY OF COPPER AND LEAD LEVELS IN WASTE BOXES

1

Samples which contained nondetectable concentrations were used in calculations as the mean between 0 and the detection limit.

Results by Analytical Methods		Pile #	
	1	2	3
STLC			
Regulatory Threshold (mg/L)	25		
Mean (mg/L) ¹	45	79	31
Coefficient of Variation	0.33	0.91	0.85
TTLC			
Regulatory Threshold (mg/kg)	2500		
Mean (mg/kg) ¹	2550	3080	2600
Coefficient of Variation	0.14	0.27	0.11
Average of four samples.			

5

TABLE 2-2. SUMMARY OF COPPER LEVELS IN WASTE PILES

- -											
Results	by Analytical Methods		Pile #								
	<u> </u>		1	2	- 3						
STLC											
	Regulatory Threshold (mg/L)		5.0								
	Mean (mg/L) ¹		3.0	2.0	2.4						
	Coefficient of Variation	1	0.23	0.26	0.33						
TTLC											
	Regulatory Threshold (mg/kg)	2.1	1000								
	Mean (mg/kg) ¹		66	58	64						
	Coefficient of Variation		0.21	0.11	0.05						
1	Average of four samples.										

TABLE 2-3. SUMMARY OF LEAD LEVELS IN WASTE PILES

6

(B-10)

3.0 SAMPLING PROGRAM

The sampling design will be of a random grid layout. Piles 1 and 3 will be gridded into equal surface areas by marking a coordinate every 3 ft, both horizontally and laterally. Due to the variation in size between piles, this will result in approximately 130 grids for Pile 1 and approximately 120 grids for Pile 3. Each grid will have a surface area of 9 square ft. Pile 2 will be gridded into equal surface areas by marking a coordinate every 4 ft, both horizontally and laterally. This will result in approximately 125 grids. Each grid will have a surface area of 16 square ft. The grid areas will be numbered consecutively so that sample locations may be referenced.

Six different samples will be collected along with two blind replicates for each pile. Location of the sampling points will be selected for each of the sampling locations from a random number table (see Section 5.2.3).

Trained technicians will be required to collect samples of waste from the piles using the techniques described in Sections 5.0 through 10.0. Composite samples will be collected from each randomly selected grid. Composite sampling will consist of collecting five subsamples from each of two different depths in the randomly selected grid for a total of ten subsamples. The depths will be 0.5 ft from the surface of the pile and approximately 0.5 ft from the pavement. Subsamples will be collected from four corners of the grid in addition to one subsample from the center. The subsamples will then be composited in a tray and mixed using a stainless steel or Teflon spoon. The composited sample will be split and placed into two or three precleaned polyethylene bottles for analysis as follows:

- 500 cc from all sampling points. These samples will be sent to the primary analytical laboratory.
- 1000 cc from all sampling points. These samples will be archived in the event that additional analyses are required.
- 500 cc from 1 out of 10 sampling points. These samples will be sent to a separate analytical laboratory to verify results from the primary laboratory.

This type of sampling versus a single grab sample will provide a better estimate of the mean concentration of the contaminants within the sampling grid and, correspondingly, a better estimate of the mean concentration of the contaminants in the waste pile.

(B-11)

4.0 ANALYSIS PROGRAM

One sample from each pile will be analyzed for the seventeen California Assessment Manual (CAM) metals plus Cr (VI). Total metal concentration is to be compared to California Total Threshold Limit Concentrations (TTLCs) for the seventeen metals plus Cr (VI) using appropriate methods as found in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, 3rd Edition. The remaining samples need be analyzed for only total copper and lead since previous testing has shown these to be the major metals. The waste will be analyzed for soluble metals using the following two methods:

> The Toxicity Characteristic Leaching Procedure (TCLP) will be carried out on 1 out of 5 samples in future sampling programs to ensure the waste is not a RCRA waste. The waste piles which are now undergoing analyses have already been tested by this method.

> Soluble metal concentrations using the California Title 22 Waste Extraction Test (WET), to be compared with the California Soluble Threshold Limit Concentrations (STLCs) standards for these metals.

The total metal analyses (all 17 metals plus Cr(VI)) are conducted first and are conducted to determine major metals for subsequent analysis. A major metal is one whose total concentration is ten times above the STLC for that metal. Then all the remaining samples are analyzed for total metals content for just the major metals. Finally, any sample whose total metal concentration is \geq ten times its STLC should be analyzed by the WET to determine any STLC exceedances. The approach to analysis described in this paragraph is relatively simple, quick, and cost-effective.

It is important to inform the analytical laboratory to use as large a sample volume as possible for analyses in order to obtain an accurate representation of the metal concentrations in each sample. A minimum of 100 g of sample should be used for the WET and a minimum of 5 g of sample should be used for acid digestion.

The analytical laboratory must meet the following quality control and quality assurance standards:

•

The minimum acceptable detection limit is 100 times lower than TTLCs and 10 times lower than STLCs for WET analysis.

Results from spike analyses must be provided to demonstrate the accuracy and reproducibility of laboratory methods. An error of $\pm 20\%$ is acceptable.

Also, in future sampling programs we recommend that approximately one out of ten samples be analyzed for total metal concentrations of all 17 CAM metals plus CR(VI). It is not necessary or cost-effective to analyze every sample for all 17 metals. However, a representative fraction of the samples used needs to be completely characterized in order to determine the major metals present.

Additional details on the statistical design of the sampling program are provided in Section 5.0. Sampling equipment and operation, sample collection and preservation, personal protective equipment and decontamination, and quality assurance and quality control are discussed in Sections 6.0 through 10.0.

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5.0 STATISTICAL DESIGN

An overview of the sampling effort will be discussed first followed by details on each aspect of the sampling design. The overview is intended to provide a general understanding of how the waste will be sampled. The details which follow the overview will include information on how the number of samples and grid sizes were selected, as well as detailing the method for selection of the grids and the sampling method within a grid.

5.1 Overview

Each waste pile will first be subdivided into either 3 ft by 3 ft grids (Piles 1 and 3) or 4 ft by 4 ft grids (Pile 2). Random sampling will then be used to select six grids for sampling. Within each of these grids, ten samples will be taken and composited, five samples from each of two levels.

The key elements which must be defined for this type of sampling design include: 1) the number of samples; 2) the grids (spatial area) to be sampled; 3) the selection of the grids; 4) the sampling method within a grid; and 5) the estimators used to characterize the population.

5.2 Approach

5.2.1 NUMBER OF SAMPLES PER WASTE PILE

Factors affecting the number of samples which should be collected are the homogeneity of the contaminant in the waste, the desired confidence interval, and the cost per sample. Based on previous sampling at the site (Tables 2-1 - 2-3), an estimate of the number of samples which would provide statistical confidence in the results may be determined.

In order to provide a basis for the determination of the number of samples to acquire per pile, a table was generated which compares the coefficient of variation of a sample set (standard deviation/mean) versus K, which is a ratio of the mean of the sample set to the regulatory threshold (Table 5-1). In order to generate this table, the sample mean, standard deviation, and sample size are related to determine an upper bound, T_U , which represents the highest value for the

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		K = 100X/RT 10 30 50 70 90										
CV	10	30	50	70	90							
		80% CON	FIDENCE LEVE	L								
0.1	1	1	1	1	1							
0.5	1	1	1	2	15							
0.9	1	1	1	4	38							
1.3	1	1	2	6	63							
1.7	1	1	2	8	87							
2.0	1	1	3	9	103							
		90% COM	FIDENCE LEVE	L								
0.1	1	1	ĩ	1	2							
0.5	1	1	1	3	34							
0.9	1	1	3	8	108							
1.3	1	2	4	13	147							
1.7	1	2	- 5	18	202							
2.0	1	2	6	21	239							
		95% CON	NFIDENCE LEVE	IL.								
0.1	· 1	1	1	1	3							
0.5	1	- 1	2	5	55							
0.9	1	2	4	13	145							
1.3	1	3	6	22	242							
1.7	1	3	8	29	332							
2.0	1	4	10	35	393							

TABLE 5-1. SAMPLE SIZE¹ REQUIRED TO DEMONSTRATE COMPLIANCE WITH REGULATORY THRESHOLD (RT) AS A FUNCTION OF ANTICIPATED AVERAGE CONTAMINATION LEVEL (X) AND COEFFICIENT OF VARIATION

These sample sizes correspond to a statistical power of 50% at a contamination level x, and were calculated assuming a lognormal probability distribution for the metal concentrations, along with assumptions that the standard deviation of the measurements is known, and that spatial correlation effects are not important.

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concentration that is plausible based on the samples taken. If T_U is found to be below the regulatory threshold, then it is decided that the true average concentration is also below that threshold. From an environmental point of view, the use of T_U is probably most defensible because it requires that an area be demonstrated free of contaminants at the regulated levels.

T_U is calculated from the statistical formula shown below:

$$\ln(T_0) = m + g_{1-e} \frac{\sigma}{\sqrt{n}} + \frac{\sigma^2}{2}$$
 (5.1)

where m is the mean of the log-transformed metal concentrations:

$$n = \frac{\sum_{i=1}^{n} t(x_i)}{n}$$
 (5.2)

where:

 $t(x_i)$ = the log-transformed metal concentrations

1

n = sample number

 $g_{1-\alpha}$ = the (1- α) percentile point of the standard normal distribution

 σ = the standard deviation of the log-transformed metal concentrations

The sample sizes shown in Table 5-1 have been generated by assuming an average metal concentration (x), a standard deviation (σ), and a desired T_U to give a range of CVs (σ /x) and Ks (100x/RT). In order to use Table 5-1, it is necessary to either assume an expected x and CV or a small preliminary sample should be taken to provide an estimate of x and the CV. These values can then be used to select an appropriate sample size. The mean and standard deviation of the sample set may be calculated in the standard method as shown. The mean of a sample set may be calculated as follows:

$$x = \frac{\sum_{i=1}^{n} x_i}{n}$$

(5.3)

)

The standard deviation of the sample set may be calculated as follows:

(B-15)

 $S = \sqrt{\frac{\sum_{i=1}^{n} (y_i - y)^2}{n - 1}}$ (5.4)

The coefficient of variation (CV) is simply the ratio of the sample standard deviation to the sample mean:

$$CV = \frac{s}{x} \tag{5.5}$$

From Table 5-1, one can see that as the K value increases or the coefficient of variation increases, a greater number of samples are required to demonstrate compliance. In other words, as the expected sample mean approaches the regulatory threshold, it will require many more samples to demonstrate that the actual metal concentration in the waste is below the threshold.

As shown by the preliminary sampling (Tables 2-1 - 2-3), the results demonstrated that most of the waste in the piles contain copper concentrations above the regulatory thresholds for both soluble and total metals content, although a high coefficient of variance was often found with these results. Theoretically, additional sampling of any pile of waste might result in finding the metal concentrations to be below the regulatory limits (although this is not recommended for these particular piles because the soluble copper content is too high); however, one must balance the cost of sampling with the likelihood of being able to dispose of the waste as nonhazardous.

Although the calculations in Table 5-1 show that in some cases one sample would be sufficient to demonstrate compliance, this would be difficult to justify from a regulatory perspective. From a statistical standpoint, a minimum of six samples per waste pile (where a waste pile is equal to 300 yd³ or less) would provide relatively good confidence in the calculated average metal concentration. The number of samples required if, for example, the average metal concentration is expected to be close to the regulatory threshold and the coefficient of variation is high, can be as high as 390 samples, which would clearly be economically unfeasible. Therefore, it is recommended that six samples per pile be taken to determine the average metal concentration. If waste piles generated in the future are significantly larger than those now in question, sample size should increase proportionally.

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5.2.2 GRID SIZE

The grid size selected was based upon the area required to collect the samples and a "rule of thumb" that for a sample of size n, there should be $20 \times n$ grids. There are six samples to be taken from each waste pile, therefore, 120 grids would be adequate. This number of grids indicates a grid size of 3 ft by 3 ft would be appropriate for Piles 1 and 3 (generating approximately 130 and 120 grids, respectively), while a grid size of 4 ft by 4 ft would be appropriate for Pile 2 (generating approximately 125 grids).

For sampling of other piles, the following steps may be followed:

- 1) Determine the number of samples to be taken as discussed in the previous section.
- 2) Multiply the number of samples, n, by 20 to determine the number of grids required per strata.
- 3) Based upon the dimensions of the pile, determine the size of the grids required. For example, to take 5 samples from a waste pile with dimensions of 40 ft by 50 ft would require 100 grids. Selecting a grid size of 4.5 ft by 4.5 ft would yield approximately 100 grids.

5.2.3 SELECTION OF GRIDS

Grid areas should be numbered consecutively. Selection of the grids for sampling will be done randomly. In order to select the grids, use the set of random numbers shown in Table 5-2. Select the first, middle, or last three digits from each five-digit number, but decide which digits will be selected prior to beginning. Choose any number randomly in the table as a starting point. From this number, go down the column, then to the top of the next column on the right, until six numbers have been selected with no repetitions. If a number is selected for which there is no grid, select the next consecutive random number. For example, if we choose to select the middle three digits from the five-digit number and we begin in the seventh column, proceeding down column 7 would give us the numbers 46, 119, 75, 22, 95, and 130. The grids corresponding to these numbers would then be selected for sampling.

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TABLE 5-2. RANDOM NUMBERS TABLE¹

15

ine/Col.	(1)	(2)	(3)	(4)	(5)	(8)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)
t	10480	15011	01536	02011	81547	91646	69179	14194	62590	36207	20969	99570	91 291	90700
2	22368	48573	25595	85393	30995	89198	27982	53402	93965	34095	52668	19174	39615	9950
3	24130	48360	22527	97255	76393	64809	15179	24630	49340	32081	30680	19655	63348	5862
4	42187	93093	06243	81680	07856	16376	39440	53537	71341	57004	00849	74917	97758	1537
5	37570	39975	81637	16656	06121	91782	60468	\$1305	49684	50672	14110	06927	01263	5461
6	77521	06907	11008	42751	27756	53498	18602	70659	90655	15053	21916	81825	44394	4288
7	99562	72905	56420	69994	98872	31016	71194	18738	44013	48840	53213	21069	10634	1295
8	96301	91977	05463	07972	18876	20922	94595	56869	69014	50045	18425	84903	42508	3230
9	89579	14342	63661	10281	17453	18103	57740	84378	25331	12566	58678	44947	05585	5694
10	85475	36857	53342	53988	\$3060	59533	38867	62300	08158	17983	16439	11458	18593	649
11	28918	69578	88231	33276	70997	79936	56865	05859	90106	31595	01547	85590	\$1610	781
12	63553	40961	46235	03427	49626	69445	18663	72695	52180	20847	12234	90511	33703	903
13	09429	\$3969	52636	92737	88974	33488	36320	17617	30015	08172	84115	27156	30613	749
14	10365	61129	87529	85689	48237	52267	67689	\$3394	01511	26358	85104	20285	29975	898
15	07119	97336	71048	08178	77233	13916	47554	81056	97735	85977	29372	74461	28551	907
16	51085	12765	51 62 1	51259	77452	16308	60756	52144	49442	53900	70960	63990	75601	407
17	02365	21382	52404	60268	89368	19885	55322	44819	01188	65255	64835	44919	05944	551
18	01011	54092	33362	94904	31273	04146	18594	29852	71585	85030	51132	01915	92747	549
19	52162	53916	46369	58586	23216	14513	83149	96738	23495	64350	94738	17752	35158	357
20	07056	97828	33787	09998	42698	06691	75988	13602	51851	45104	88916	19509	25625	581
21	48663	91245	85828	14345	09172	30168	90229	04734	59153	22178	30421	61666	99904	328
22	54164	58492	22421	74103	47070	25306	75468	26384	58151	06646	21524	15227	96909	445
23	32639	32363	05597	24200	13363	38005	94342	28728	35806	05912	17012	64151	18296	228
24	29334	27001	87637	87308	56731	00258	45834	15398	46557	41135	10367	07684	36188	185
25	02488	33052	28834	07351	19731	92420	60952	81280	50001	67658	32586	86679	50720	549

¹ Ott, L. 1984 <u>An Introduction to Statistical Methods and Data Analysis</u>, Second Edition, Duxbury Press, Boston

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5.2.4 SAMPLING METHOD WITHIN A GRID

Spatial composite sampling will be used to characterize the waste within a grid. Five subsamples will be taken within each grid from the corners of the grid and the center at a depth of 0.5 ft from the surface. An additional five subsamples will be taken in the same manner from a depth of 0.5 ft from the pavement. These ten subsamples will then be composited via mixing in a lined container into a homogenous sample for the various analyses.

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6.0 SAMPLING EQUIPMENT AND OPERATION

The following pieces of equipment will be used to perform sampling of the waste placed in roll-off bins, grit piles, and the rinsate water. The two main requirements for the sampling equipment are:

• The tool must not contribute any chemical contamination to the sample, and

• The tool must be capable of collecting a representative sample.

Stainless steel equipment is generally the most durable and is often used for sampling sludge, sediments, and soils. The following paragraphs below discuss the pieces of sampling equipment which are recommended for use in sampling the waste and the rinsate water resulting from decontamination.

6.1 Dipper

A dipper consists of stainless steel, glass, or Teflon beaker constructed with or clamped to the end of a handle (Figure 6-1). Dippers are used for sampling tanks, bins, outfalls, and discharge. The following precautions should be observed:

- A stainless steel dipper should have a riveted handle not a soldered handle, because metals from the solder could leach into and contaminate the sample.
 - Use only Teflon, stainless steel, or glass to sample wastes containing organic materials.

When using a beaker clamped to a pole, the handle and clamp should be painted with a 2-part epoxy or other chemically-inert paint when sampling either alkaline or acidic materials.

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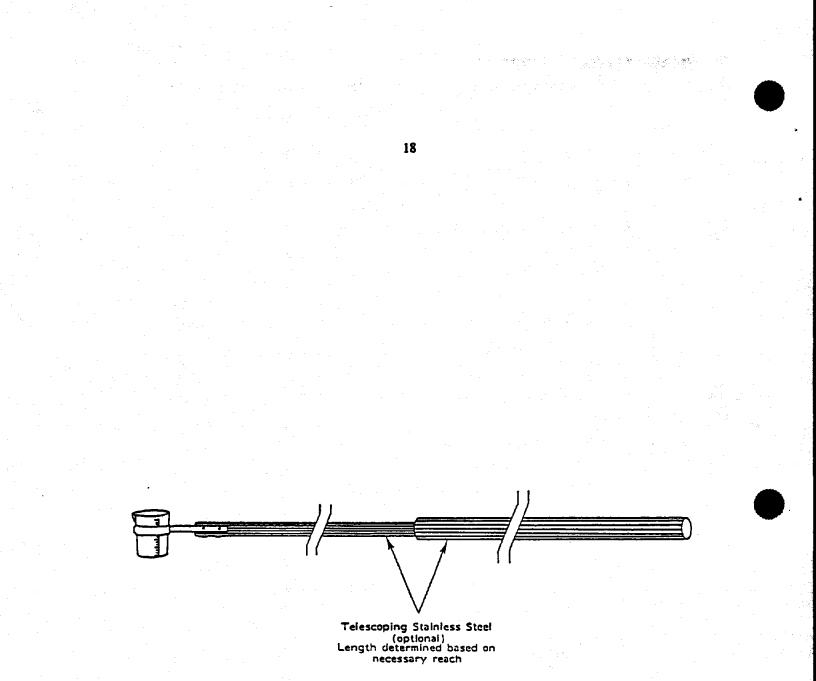


FIGURE 6-1. SCHEMATIC DIAGRAM OF DIPPER

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Procedures for Use:

1. Decontaminate the dipper, clamp, and handle (see Section 6.2).

2. In tanks, turn the dipper so the mouth of the dipper faces down and insert it into the waste material. Turn dipper right side up when dipper is at desired depth. Allow dipper to dill completely as shown by the cessation of air bubbles. When sampling drums, submerge the dipper to the desired depth, allow the beaker to fill.

3. Raise dipper and pour the sample material into an appropriate container.

4. Decontaminate the dipper.

6.2 Stainless Steel Spoon or Scoop

A stainless steel spoon or scoop is the simplest, most direct method for collecting soil samples. In general, the procedure is used to sample the first three inches of surface soil. However, samples from greater depths and samples of sludges, sediments and bulk samples may also employ this technique in some situations.

Procedures for use:

1. Collect and composite samples from the top three inches of soil.

2. Mix the samples in a lined container, then deposit in the appropriate container.

3. Wipe sample containers clean of surface contamination.

4. Place in individual plastic bags in an insulated ice chest with freezer packs if refrigeration is necessary.

6.3 Glass Tube Thief

A hollow glass tube is a simple tool which is used to sample liquids from drums (Figure 6-2). The advantages of using a glass tube thief include inexpensive cost, ease of disposal, its availability in variable lengths, and capability to sample a vertical column of waste. The tool

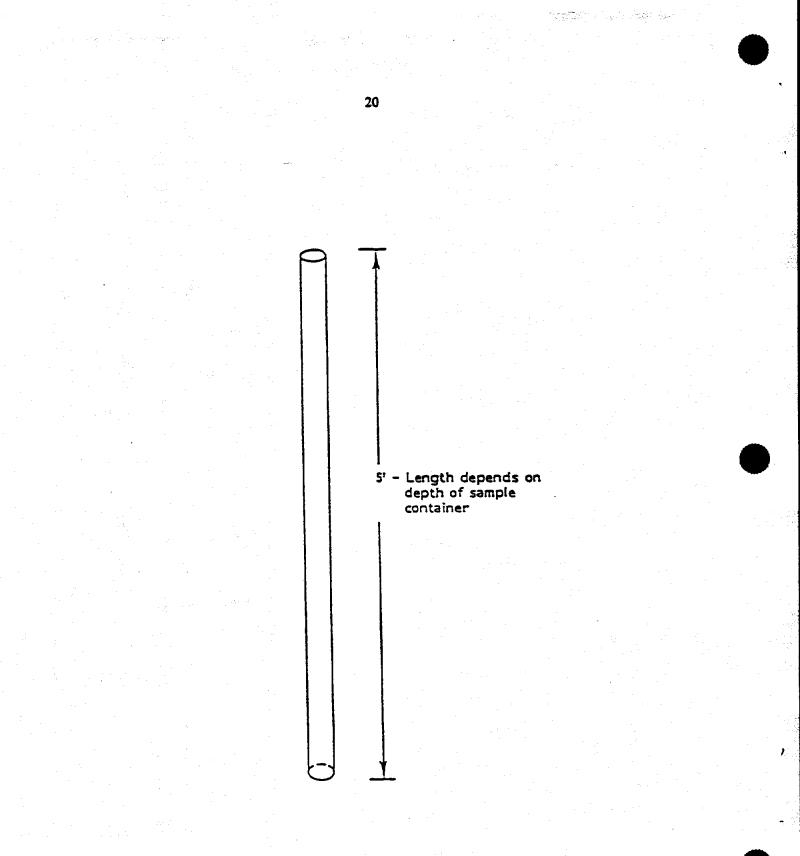


FIGURE 6-2. SCHEMATIC DIAGRAM OF GLASS TUBE THIEF

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consists of a glass tube, typically between 8 and 16 mm in diameter. This device will be used to sample the drums containing rinsate from the decontamination of the dipper.

Procedures for use:

3.

- 1. Decontaminate the glass tube (see Section 6.2)
- 2. Slowly insert the tube into the waste container. This should be done at a rate which permits the level of the liquid inside and outside the sampler to remain the same. If the level of waste in the sampler tube is lower inside than outside, the sampling rate is too fast and may yield a non-representative sample.
 - When the tube contacts the bottom of the waste container, place a rubber stopper or attach a squeeze bulb over the exposed end of the sampling tube. The use of a squeeze bulb improves the ability of a glass tube to retain very viscous fluids during sampling. It is important that none of the fluid comes in contact with the rubber squeeze bulb. If using your thumb, ensure your hands are protected by gloves which are resistant to the chemicals sampled. With the end of the tube plugged, slowly draw the tube from the waste container. In order to enable the sampler to retain the fluid in the glass tube, the glass tube may be withdrawn at an angle such that the thumb may be kept over the end of the glass tube.
- 4. Place the end of the glass tube in the sample container and remove plug from the end of the tube.
- 5. Repeat steps 2 through 5 until the required amount of sample has been collected.
- 6. Place the contaminated glass tube in a plastic storage tube for subsequent cleaning, as described in Section 6.2. If used to sample a drum of waste, the glass tube may be disposed in the drum prior to resealing the bung. Notch the glass with a steel file to avoid shattering the glass when breaking long pieces.

6.4 Auger and Thin-Wall Tube Sampler

The system consists of an auger bit, a series of drill rods, a "T" handle, and a thin-wall corer (Figure 6-3). The auger bit is used to bore a hole to the desired sampling depth and is then withdrawn. The auger tip is replaced with the tube corer, lowered down the borehole, and forced into the soil at the completion depth. The corer is then withdrawn and the sample collected.

Alternatively, the sample may be recovered directly from the auger. This technique however, does not provide an "undisturbed" sample as would be collected with a thin-tube

(B-25)

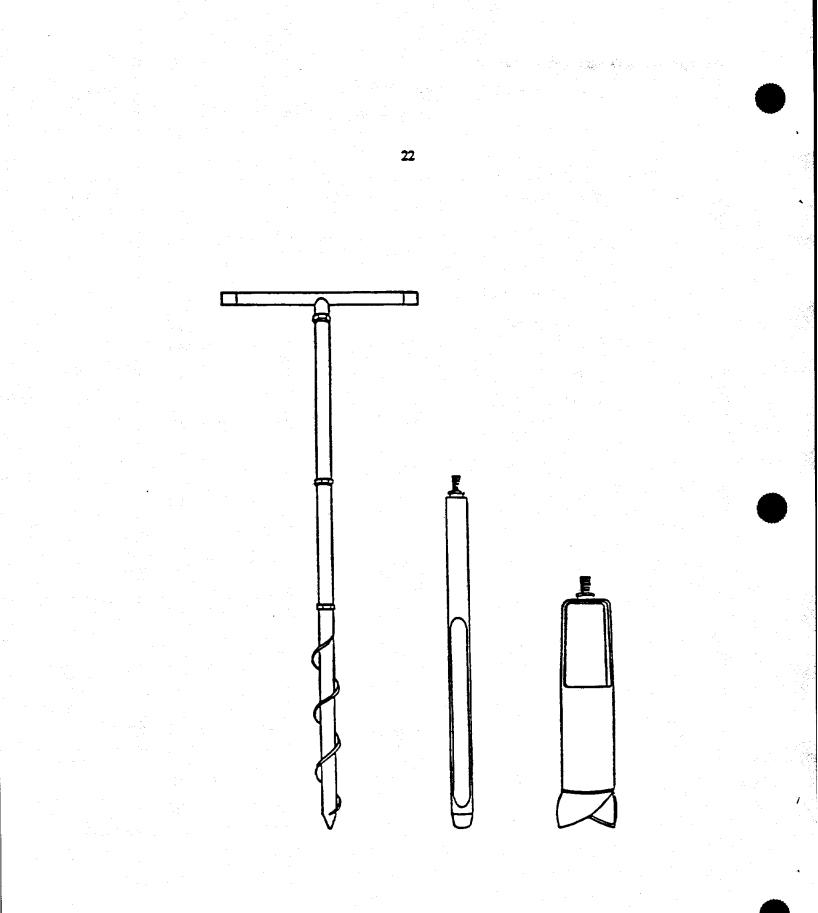


FIGURE 6-3. SCHEMATIC DIAGRAM OF AUGERS AND THIN-WALL TUBE SAMPLER

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sampler. When the soil is rocky, it may not be possible to force a thin-tube sampler through the soil or sample recovery may be poor. Sampling directly from the auger may be the only viable method. Several auger types are available: bucket type, continuous-flight (screw), and posthole augers. Bucket types are good for direct sample recovery, are fast, and provide a large volume of sample. When continuous flight (screw) augers are used, the sample may be collected directly off the flights, however, this technique will provide a somewhat unrepresentative sample as the exact sample depth will not be known. The continuous-flight augers are satisfactory for use when a composite of the entire soil column is desired. Posthole augers have limited utility for sample acquisition as they are designed more for their ability to cut through fibrous, heavily rooted, swampy areas. In soils where the borehole will not remain open when the tool is removed, a temporary casing may be used until the desired sampling depth is reached.

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Procedures for use:

- 1. Attach the auger bit to a drill rod extension and attach the "T" handle to the drill rod.
- 2. Clear the area to be sampled of any surface debris (twigs, rocks, litter). It may be advisable to remove the first 8 to 15 cm of surface soil from a 30-cm diameter area around the drilling location.
- 3. Begin drilling, periodically removing accumulated soils. This prevents accidentally brushing loose material back down the borehole when removing the auger or adding drill rods.
- 4. After reaching desired depth, slowly and carefully remove anger from boring. (Note: When sampling directly from auger, collect sample after auger is removed from boring and proceed to Step 10).
- 5. Remove auger top from drill rods and replace with a precleaned thin-wall tube sampler. Install proper cutting tip.
- 6. Carefully lower corer down borehole. Gradually force corer into soil. Take care to avoid scraping the borehole sides. Do not hammer the drill rods to facilitate coring as the vibrations may cause the boring walls to collapse.
- 7. Remove corer and unscrew drill rods.
- 8. Remove cutting tip and remove core from device.

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9. Discard top of core (approximately 2.5 cm) which represents material collected by the core before penetrating the layer in question. Place remaining core into sample container.

10. Verify that a Teflon liner is in the cap if required. Secure the cap tightly.

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11. Label the sample bottle with the appropriate sample tag. Label the tag carefully and clearly, addressing all the categories or parameters. Complete all chain-of-custody documents and record in the field logbook.

7.0 SAMPLE COLLECTION AND PRESERVATION

7.1 Sample Collection

The following procedures will be followed for sampling waste from waste piles:

1. Identify the specific pile which will be sampled.

б.

- 2. Construct the sampling grid as described in Section 5.2.3.
- 3. Go to the random numbers table (Table 5-2) and select six numbers. Each number represents the grid unit which will be sampled.
- 4. Use the appropriate instrument to obtain the sample. Follow the recommended procedures for use as stated in Section 6.0.
- 5. Collect a composite sample from each randomly selected grid. Composite sampling will consist of collecting five subsamples at two different depths (0.5 ft from the surface and 0.5 ft from the pavement) from each corner of the randomly selected grid in addition to one sample from the center for a total of 10 subsamples. The samples will then be composited in a tray and mixed using a stainless steel or Teflon spoon. The composited sample will be placed in precleaned polyethylene bottles for analysis.
 - From each sampling point, split the composite sample into a 500 cc subsample for the analytical laboratory and a 1000 cc subsample to archive. From 1 out of 10 sampling points, reserve 500 cc of the composite sample to send to a separate analytical laboratory. No preservation is required for samples. Rinsate blanks must be preserved with a solution of nitric acid. This can be provided in the sample jar by the analytical laboratory. Holding time for the samples is 6 months, unless sampling for mercury which has a holding time of 28 days.
- 7. The collection of the sample does not require filling the sample jar in any special manner.
- 8. Discard the outer latex gloves after each sample into an appropriate container and then replace them for the next sampling event.

9. For the rinsate blank (which will be required once for every twenty samples), simply run deionized water over the sampling instrument after it has been decontaminated.

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10. The sampler must pay attention while filling the sample bottle for the rinsate blank due to the fact that the sample bottle will have a preservative already in it. If the bottle were to be overfilled during collection, some of the preservative would be lost resulting in insufficient preservative remaining in the bottle and an inaccurate analysis.

7.2 Sample Preservation

No preservatives will be required for the sampling of the waste itself. Only the rinsate blank (equipment washing) will require a preservative of nitric acid in order to lower the pH of the sample below 2. The analytical laboratory can provide the sample containers containing the appropriate quantities of preservative for this. Caution should be exercised when these samples are collected to prevent accidental exposure by splashing.





8.0 PERSONAL PROTECTIVE EQUIPMENT AND DECONTAMINATION

8.1 Personal Protective Equipment

8.1.1 SAMPLING

waste:

The following personal protective equipment shall be worn during the sampling of the

- Tyvek suit
- Latex gloves (two pairs)
- Dust protector
- Safety glasses with splash shields (only necessary for when rinsate blanks (equipment washes) are collected).

8.1.2 CLEANING OPERATIONS (DECONTAMINATION)

The following personal protective equipment shall be worn during all cleaning operations for sampling equipment:

- Safety glasses with splash shields
- Latex gloves (water decontamination)
- Neoprene or nitrile gloves (when using solvents)
- Tyvek or cloth coveralls

8.2 Decontamination

Decontamination (cleaning) of sampling devices prior to and after use is required. Decontamination is important so that material from a previous sampling event does not contaminate subsequent samples. Decontamination should be performed as follows:

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- Scrub the sampling tool with a brush in a laboratory-grade detergent (Alconox, Liquinox, or the equivalent) and tap water solution
- Rinse with water
- Rinse again with deionized water or the equivalent
- If sampling for organic contamination, rinse a final time with pesticide-grade isopropanol or pesticide-grade acetone or methanol (a minimal amount is necessary for rinsing and this should be conducted under a fume hood or in the open, but never in a closed room without adequate ventilation)
- Allow sampling tool to air dry
- Wrap in aluminum foil or other similar protective covering to avoid contamination before the next use
- No eating, smoking, drinking, chewing, or any hand to mouth contact will be permitted during cleaning operations.

The following are cleaning procedures for the glass tube thief:

- Wash thoroughly with laboratory detergent and hot water using a brush to remove any particulate matter or surface film
- Rinse thoroughly with hot tap water
- Rinse with at least a 10 percent nitric acid solution
- Rinse thoroughly with tap water
- Rinse thoroughly with deionized water
- Rinse twice with solvent and allow to air dry for at least 24 hours
- Wrap completely with aluminum foil to prevent contamination during storage

The following are cleaning procedures for stainless steel sampling equipment:

- Wash thoroughly with laboratory detergent and water with a brush
- Rinse thoroughly with tap water
- Rinse thoroughly with deionized water
- If sampling for organic contamination, rinse twice with solvent and allow to air dry

Wrap completely with aluminum foil

Rinse with tap water after use in the field

Decontamination wash waters should be collected and containerized separately from solvent rinses in a 55-gallon drum. Since potentially hazardous wastes are being rinsed from sampling equipment, the collected rinse waters should be handled and sampled for hazardous constituents using a glass tube thief prior to disposal. The storage area should have a drum staged for the disposal of rinse waters and one for disposal of solvents. Upon filling the rinse water drum, it should be sampled for metals to determine if it must be disposed of as a hazardous waste or down the industrial drain. The contents of the solvent drum may be recycled.

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9.0 SAMPLE CUSTODY, LABELING, PACKAGING, AND TRANSPORTATION

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9.1 Sample Custody

The purpose of a sample chain-of-custody is to document the possession of a sample from the time of collection, through all transfers of custody, until it is delivered to the analytical laboratory. This requires that a form (Figure 9-1) be filled out in permanent ink and sent along with the samples to the storage area. This form will contain the following minimum information:

- Sample number
- Date and time of collection
- Shipyard location
- Waste type
- Signature of collector
- Preservation
- Container type
- Analysis request
- Appropriate notations relative to sample integrity and handling practices
- Signature of all persons involved in the chain of possession
- Inclusive dates and times of possession

9.2 Sample Labeling

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A sample label is applied to a sample container before a sample of waste is collected (Figure 9-2). The label will be completely filled out with permanent ink. It will contain the following information:

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FIGURE 9-1. CHAIN-OF-CUSTODY SHEET

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SAMPLE	PRESERVATIVE	<u> </u>	· · · · · · · · · · · · · · · · · · ·	-
SAMPLI	G LOCATION			
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FIGURE 9-2. SAMPLE LABEL

(B-36)



Sample number

- Date and hour the sample was taken
- Sampler's initials
- Sampling site
- Tests required, if known
- Preservative used, if any

9.3 Sample Packaging

The laboratory will typically provide all sample containers, preservatives, and packaging for transportation of samples. Proper preparation of sample containers for transport to the laboratory is essential to prevent breakage of containers and spillage of potentially hazardous material. The following steps will be taken during sample packaging:

- Ensure sample container is labeled correctly
 - After sampling activities are complete, clean the outer surface of all sample containers
 - Wrap each glass container with plastic insulating material (bubble wrap) and enclose in a plastic bag to prevent contact with other sample containers. Plastic containers also should be placed into bags, however, bubble wrap is not needed.
- Place sample containers in thermally-insulated, rigid ice chests which contain ice or reusable ice packs if the temperature must be held at 4°C. If the sample does not need to be held at 4°C, an ice chest is not required. However, an ice chest is a lightweight, rigid, and easily secured container in addition to being thermally efficient.
 - Ensure the chain-of-custody forms are filled out and secure the inside the sample chests. Packers should retain one copy.

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9.4 Sample Transportation

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Transport samples to the laboratory as soon as possible after collection.

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10.0 SAMPLE QUALITY ASSURANCE AND QUALITY CONTROL

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10.1 Rinsate Blanks (Equipment Washes)

Equipment washes serve as checks of field decontamination procedures. They are obtained after final wash and decontamination of equipment by pouring reagent-grade water into/through/over a decontaminated piece of sampling equipment. The water is collected in appropriate sample containers and transported to the laboratory with other samples. The equipment blanks are analyzed in the same manner as the field samples. Equipment blanks should be collected prior to each sampling event at each sampling site. However, once good equipment decontamination technique (equipment blanks are "clean") has been demonstrated, the frequency of equipment wash samples may be reduced to an occasional basis. Initially, one rinsate blank (equipment wash) will be collected for every twenty samples taken.

10.2 Laboratory Quality Control and Certification

Laboratory quality control procedures are instituted to ensure the reliability of analytical data obtained throughout the sampling effort. Procedures include the analysis of laboratory samples to measure the accuracy and precision of laboratory procedures. A laboratory duplicate should typically be analyzed one time in twenty samples. Any analytical laboratory used should have current certification from the state of California for performing all the necessary chemical analyses.

10.2.1 MATRIX SPIKE ANALYSIS

Matrix spike analyses are performed to assist the accuracy of laboratory methods. Spiked samples are used to determine if chemical interferences are occurring. One spike analysis per sample set is generally adequate.

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10.2.2 MATRIX SPIKE DUPLICATES

Matrix spike duplicates are used to evaluate the reproducibility of the analytical procedures. A field sample is analyzed and the results are compared to the original matrix spike sample test results. In general, this is only necessary for large numbers of samples (>30).

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10.2.3 METHOD BLANK TESTS

Method blank tests are performed in the laboratory by analyzing distilled, deionized water for all analytical methods employed by the laboratory. Method blanks are analyzed for each matrix to verify that laboratory-induced contaminants are identified and distinguished from environmental contaminants of concern.

APPENDIX C

GLOSSARY OF SOLIDIFICATION/STABILIZATION TERMS AND ABBREVIATIONS*

AA - atomic absorption spectroscopy, a microcharacterization method.

ANC - Acid Neutralization Capacity, a chemical test.

ANS - American Nuclear Society.

ANSI - American National Standards Institute.

ANSI/ANS/16.1 - American Nuclear Society test 16.1, a leaching test.

AOC - area of contamination.

APC - air pollution control.

API - American Petroleum Institute.

ARAR — applicable or relevant and appropriate requirement. These are cleanup standards, standards of control, and other substantive requirements, criteria, or limitations promulgated under federal, state, or local environmental laws or facility siting laws that: (l. applicable) specifically address a hazardous substance, pollutant, contaminant, remedial action, location, or other circumstance found at a CERCLA site or (2. relevant and appropriate) address problems or situations similar to those encountered at a CERCLA site (40 CFR 300.5, pp. 7 and 12).

ASTM - American Society for Testing and Materials.

absorption - assimilation of fluids into interstices (ASTM D 653, p. 129).

acidity - the quantitative capacity of materials to react with hydroxyl ions.

additives — materials included in the binder to improve the S/S process. Examples of some types of additives are: (1) silicates or other materials that alter the rate of hardening, (2) clays or other sorbents to improve retention of water or contaminants, or (3) emulsifiers and surfactants that improve the incorporation of organic compounds.

adsorption - attraction of solid, liquid, or gas molecules, ions, or atoms to particle surfaces by physiochemical forces. The adsorbed material may have different properties from those of the material in the pore space at the same temperature and pressure due to altered molecular arrangement (after ASTM D 653 and Parker, 1989, p. 37).

advection - unidirectional, progressive bulk movement, such as water under the influence of a hydraulic gradient.

alkalinity — the quantitative capacity of aqueous media to react with hydrogen ions.

*Acronyms and abbreviations are listed at the beginning of each letter of the alphabet.

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anion — an ion that is negatively charged.

asphalt — a brown, black, hard, brittle, or plastic bituminous material composed principally of hydrocarbons. It is found in nature or can be prepared by pyrolysis of coal tar, certain petroleums, and lignite tar. It melts on heating and is insoluble in water but soluble in gasoline.

BDAT — Best Demonstrated Available Technology.

BNA — base, neutral, and acid (organic) compounds, a chemical analysis identification.

bentonite — a clay formed from volcanic ash decomposition and largely composed of montmorillonite and beidellite. Usually characterized by high swelling on wetting.

binder — a cement, cementlike material, or resin (possibly in conjunction with water, extender, or other additives) used to hold particles together.

bitumen — naturally occurring or pyrolytically obtained dark or black colored, tarry hydrocarbons consisting almost entirely of carbon and hydrogen, with very little oxygen, nitrogen, or sulfur.

buffer — a solution selected or prepared to minimize changes in pH (hydrogen ion concentration). Also known as buffer solution.

CAA — Clean Air Act.

Cal WET — California Waste Extraction Test, a leaching test.

CERCLA — Comprehensive Environmental Response, Compensation, and Liability Act.

CERCLA hazardous substance — any substance, pollutant, or contaminant as defined in CERCLA sections 101(14) and 101(33), except where otherwise noted in the Hazard Ranking System (see 40 CFR 302.4).

CERCLA hazardous wastestream — any material containing CERCLA hazardous substances that was deposited, stored, disposed, or placed in or that migrated to a site being evaluated by the HRS; any material listed in the NPL.

CERCLA waste — a term with no regulatory meaning that is often used as a shortened form of CERCLA hazardous wastestream.

CFR — Code of Federal Regulations.

CLP — Contract Laboratory Procedures.

COE — U.S. Army Corps of Engineers.

CRN — Core Research Needs for Containment Systems.

CSH — Calcium Silicate Hydrate.

CWA — Clean Water Act.

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CWARP — Coal Waste Artificial Reef Program.

cation — a positively charged atom or group of atoms.

cement — a mixture of calcium aluminates and silicates made by combining lime and clay while heating.

characteristic waste — see RCRA characteristic waste

clay — fine-grained soil or the fine-grained portion of soil that can be made to exhibit plasticity (putty-like properties) within a range of water contents and that exhibits considerable strength when air-dry.

colloid — the phase of a colloidal system made up of particles having dimensions of 1 to 1000 nanometers and which is dispersed in a different phase.

colloidal system — an intimate mixture of two substances, one of which, called the dispersed phase (or colloid), is uniformly distributed in a finely divided state through the second substance, called the dispersion medium.

compressive strength (unconfined or uniaxial compressive strength) — the load per unit area at which an unconfined cylindrical specimen of soil or rock will fail in a simple compression test. Commonly the failure load is the maximum that the specimen can withstand in the test.

contaminant — typically undesirable minor constituent that renders another substance impure.

corrosiveness characteristic — exhibiting the hazardous characteristic of corrosivity due to extreme pH or failing under the test conditions defined in 40 CFR 261.22.

DLT — Dynamic Leach Test, a leaching test where the specimen is exposed to an actual or simulated flow of the leachant.

DQO — Data Quality Objective, a planned quantitative measure of precision, accuracy, and completeness of data.

DRE — destruction-removal efficiency. The combined efficiencies of one or more processes intended to reduce the target contaminant(s). The DRE may be expressed as a ratio or percentage.

density, apparent (of solids and liquids) — the mass of a unit volume of a material at a specified temperature. Only the volume that is impermeable is considered.

density, bulk (of solids) — the mass of a unit volume of the material at a specified temperature.

diffusion — movement of molecules towards an equilibrium driven by heat or concentration gradients (mass transfer without bulk fluid flow).

diffusivity — diffusion coefficient, the weight of material, in grams, diffusing across an area of 1 square centimeter in 1 second due to a unit concentration gradient.

dimensional stability — the ability of the solidified/stabilized waste to retain its shape.

disposal facility — a facility or part of a facility at which waste is intentionally placed into or on any land or water, and at which waste will remain after closure.

durability — the ability of solidified/stabilized wastes to resist physical wear and chemical attack over time.

ECN — Energieonderzoek Centrum Nederland (Netherlands Energy Research Foundation).

EDXA — energy dispersive X-ray analysis, a microcharacterization method.

EE/CA — Economic Evaluation/Cost Analysis, CERCLA technology screening process for a removal action 40 CFR 300.415.

ELT — Equilibrium Leach Test, a leaching test where, under the conditions of the test, an equilibrium between the specimen and the leachant is attained.

EP Tox — Extraction Procedure Toxicity Test, a regulatory leaching test used since 1980 to determine if a waste is toxic (40 CFR 261, Appendix II).

embedment — the incorporation of waste masses into a solid matrix before
disposal.

emulsifier — a substance used to produce an emulsion of two liquids which do not naturally mix.

emulsion — a colloidal mixture of two immiscible fluids, one being dispersed in the other in the form of fine droplets.

ettringite — a mineral composed of hydrous basic calcium and aluminum sulfate. The formula for ettringite is $Ca_{k}Al_{2}(SO_{k})_{3}(OH)12.26 H_{2}O_{k}$

extender — an additive whose primary function is to increase the total bulk of the S/S-treated waste.

FGD — flue gas desulfurization, a pollution abatement process.

FR -- Federal Register.

FS — Feasibility Study, a study undertaken to develop and evaluate options for a treatment process.

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FTIR — Fourier transform infrared spectroscopy, a microcharacterization method.

FY — fiscal year.

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fly ash — the finely divided residue from the combustion of ground or powdered coal and which is transported from the firebox through the boiler by flue gas.

free water — water that is free to move through a soil or rock mass under the influence of gravity.

freeze/thaw cycle — alternation of a sample temperature to allow determination of weight loss and visual observation of sample disintegration resulting from phase change from water to ice.

GC/MS — gas chromatography/mass spectrometry.

grout — as used in soil and rock grouting, a material injected into a soil or rock formation to change the physical characteristics of the formation. The term "grout" is not used in this document but is frequently encountered in the S/S industry as a synonym for the term "binder."

HCB — hexachlorobenzene.

HRS — Hazard Ranking System, the primary mechanism for considering sites for inclusion on the NPL.

HSL — Hazardous Substance List, a list of designated CERCLA hazardous substances as presented in 40 CFR 302.4.

HSWA — Hazardous and Solid Waste Amendments of 1984.

hazardous characteristics — ignitable, corrosive, reactive, and toxic as defined in 40 CFR Part 261.10.

hazardous waste — see RCRA hazardous waste, CERCLA hazardous substance, and CERCLA hazardous wastestream.

heat of hydration (in S/S reactions) — the heat generated due to the reaction of cementitious or pozzolanic materials with water.

hydrate — a compound containing structural water.

ICP — inductively coupled plasma atomic emission spectroscopy.

ignitability characteristic — exhibiting the hazardous characteristic of ignitability as defined in 40 CFR 261.21.

immobilization — the reduction in the ability of contaminants to move through or escape from S/S-treated waste.

incineration — a treatment technology involving destruction of waste by controlled burning at high temperatures.

inhibitor — a material that stops or slows a chemical reaction from occurring. Used in this document to apply to stopping or slowing of the setting of S/Streated material. interference (S/S) — an undesirable change in the setting of the S/S material resulting in lower strength, poorer leach resistance, or evolution of noxious or hazardous gases, or other degradation of the S/S-treated material.

ion — an atom or molecule which by loss or gain of one or more electrons has acquired a net electric charge.

interstitial — see pore water.

kaolin — a variety of clay containing a high percentage of kaolinite.

kaolinite — a common clay mineral having the general formula $Al_2(Si_2O_1)(OH_4)$.

kiln — a heated and usually rotating enclosure used for drying, burning, or firing materials such as ore or ceramics. In this document "kiln" typically refers to a kiln used for production of lime or cement.

kiln dust — fine particulate by-product of cement production or lime calcination.

LDR - Land Disposal Restriction.

LIMB — Lime Injection Multistage Burner.

LRT — Liquid Release Test.

leachability — a measure of release of constituents from a waste or solidified/stabilized waste. Leachability is one measure of the mobility of a constituent. High leachability means high constituent mobility.

leachant — liquid that comes in contact with a material either from natural exposure (e.g., water in a disposal site) or in a planned test of leachability. The typically used leachants are pure distilled water or water containing salts, acids, or both.

leachate — any liquid, including any suspended components in the liquid, that has soaked, percolated through, or drained from material during leaching.

leaching — the release of constituents from a solid through contact with the leachant. The leaching may occur by either natural mechanisms at waste sites or as part of a laboratory leaching test.

leaching agent — leachant.

leaching rate — the amount of a constituent of a specimen or solid waste form which is leached during a unit of time (usually normalized by sample volume, area, or weight).

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leaching resistance — the inverse of leachability. High leach resistance means low contaminant mobility.

leaching test — exposure of a representative sample of contaminated waste, S/S-treated waste, or other material to a leachant under controlled conditions to measure the release of constituents. lime — specifically, calcium oxide (CaO); also loosely, a general term for the various chemical and physical forms of quicklime, hydrated lime, and hydraulic hydrated lime.

listed waste — see RCRA listed waste.

long-term stability — the ability of solidified/stabilized wastes to maintain their properties over time while exposed to the environment.

MCL - maximum concentration limit.

MEP — Multiple Extraction Procedure, a leaching test in which the sample is repeatedly leached with fresh batches of leachant.

MSDS - Material Safety Data Sheet.

MSW -- municipal solid waste.

MWEP - Monofilled Waste Extraction Procedure, a leaching test.

macroencapsulation — a process of encasing a mass of solid or S/S-treated waste in a protective layer, such as bitumen (thermoplastic).

meq — milliequivalent.

microencapsulation — containment of the contaminants on a microscopic or molecular scale.

microstructure — the structure of an object or material as revealed by a microscope at a magnification over 10 times.

mixer — machine employed for blending the constituents of grout, mortar, or other mixtures.

modified clays — clays (such as bentonite) that have been modified by ion exchange with selected organic compounds that have a positive charged site (often a quarternary amine), hence rendering the clay/organo complex hydrophobic.

monolith — a free standing solid consisting of one piece.

monomer — a simple molecule which is capable of combining with a number of like or unlike molecules to form a polymer.

montmorillonite — a group of clay minerals characterized by a weakly bonded sheet-like internal molecular structure; consisting of extremely finely divided hydrous aluminum or magnesium silicates that swell on wetting, shrink on drying, and have ion exchange capacity.

multimedia — air, land, and water.

NAAQS - National Ambient Air Quality Standards.

NCP -- National Oil and Hazardous Substances Contingency Plan, provides the organizational structure and procedures for preparing and responding to

discharges of oil and releases of hazardous substances, pollutants, and contaminants (40 CFR 300.1).

NESHAP — National Emission Standards for Hazardous Air Pollutants.

NMR — nuclear magnetic resonance spectroscopy, a microcharacterization method.

NPL — National Priorities List, list of CERCLA sites (40 CFR Part 300 Appendix B).

NRC — U.S. Nuclear Regulatory Commission.

NYSC-HWM — New York State Center for Hazardous Waste Management.

OAQPS — Office of Air Quality Planning and Standards (of the U.S. EPA).

OSHA — Occupational Safety and Health Act; Occupational Safety and Health Administration.

PAH — polynuclear aromatic hydrocarbon.

PCB — polychlorinated biphenyl.

PFT - Paint Filter Test, a physical characterization test.

ppb — part per billion.

ppm — part per million.

PRP — potentially responsible party, potentially liable for the contamination and cleanup of CERCLA sites.

percolation — movement of water under hydrostatic pressure or gravity through the smaller interstices of rock, soil, wastes, or S/S-treated wastes.

performance criterion — a measurable performance standard set for an individual property or parameter.

performance indicator — an easy-to-measure property or parameter selected to characterize the S/S process or S/S-treated waste.

permeability — a measure of flow of a fluid through the tortuous pore structure of the waste or S/S-treated waste. It is expressed as the proportionality constant between flow velocity and the hydraulic gradient. It is a function of both media. If the permeating fluid is water, the permeability is termed as hydraulic conductivity.

phase (of a material) — a region of a material that is physically distinct and is homogeneous in composition and morphology.

polymer — a chemical with repetitive structure formed by the chemical linking of single molecules (monomers).

pore — a small cavity or void in a solid.

pore size distribution — variations in pore sizes in solids; each material has its own typical pore size distribution and related permeability.

pore water — water contained in voids in the solid material.

porosity — the ratio of the aggregate volume of voids or interstices to the total volume of the medium.

Portland cement — a hydraulic cement produced by pulverizing clinker consisting essentially of hydraulic calcium silicates, usually containing one or more of the forms of calcium sulfate.

pozzolan — a siliceous or siliceous and aluminous material, which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds with cementitious properties. The term is derived from an early source of natural pozzolanic material, Pozzuoli, Italy.

QA/QC — Quality Assurance/Quality Control.

QAPjP — Quality Assurance Project Plan.

QAPP — Quality Assurance Program Plan.

3Rs - recovery, reuse, and recycle.

RCRA — Resource Conservation and Recovery Act.

RCRA characteristic waste — any solid waste exhibiting a characteristic of ignitability, corrosivity, reactivity or toxicity, as defined in 40 CFR 261, Subpart C.

RCRA hazardous waste — any RCRA solid waste, as defined by 40 CFR 261.3, that is not excluded from regulation under 40 CFR 261.4 and that meets any one of the characteristic or listing criteria (including mixtures) described in 40 CFR 261.3(a)(2). For more detail, see 40 CFR 260, Appendix I.

RCRA listed waste — any solid waste listed in 40 CFR 261, Subpart D; or a mixture that contains a solid waste listed in 40 CFR 261, Subpart D that has not been excluded under the provisions of 40 CFR 261.3 in accordance with 40 CFR 260.20 or 40 CFR 260.22.

RCRA solid waste — any garbage, refuse, or sludge; or any solid, liquid, semisolid or contained gaseous material that is: (1) discarded, (2) no longer to be used for its original purpose, or (3) a manufacturing or mining by-product and is not excluded by the provisions of 40 CFR 261.4(a). For more detail, see 40 CFR 260, Appendix I. Also note that the definition of solid waste includes materials that are not "solids" in the normal sense of the word.

RI — Remedial Investigation, a process undertaken by the lead agency to determine the nature and extent of the problem presented by a CERCLA site (40 CFR 300.430(d)).

RI/FS - Remedial Investigation/Feasibility Study, see RI or FS.

ROD — Record of Decision, a document prepared to explain and define the final remedy selected for a CERCLA site (40 CFR 300.430 (f)(4)(i)).

RP — Responsible Party, persons or corporate entities found to be responsible for contamination and cleanup at a CERCLA site.

RPM — Remedial Project Manager, the official designated by the lead agency to coordinate, monitor, or direct remedial or other response actions under subpart E of the NCP (40 CFR 300.5).

RREL - Risk Reduction Engineering Laboratory (of the U.S. EPA).

reactivity characteristic — exhibiting the hazardous characteristic of reactivity as defined in 40 CFR 261.23.

redox — abbreviation for oxidation-reduction, now accepted as a word.

residual liquid — free liquid remaining in the S/S-treated waste after treatment.

SARA — Superfund Amendments and Reauthorization Act.

SDWA — Safe Drinking Water Act.

SCE — sequential chemical extraction, a leaching test with a variety of aqueous chemicals used sequentially to characterize the contaminant bonding.

SEM — scanning electron microscopy, a microcharacterization method.

SET — Sequential Extraction Test, a leaching test with a series of sequential acid extractions used to determine the sample buffering capacity.

SITE — Superfund Innovative Technology Evaluation.

SRS — Separation and Recovery Systems, Inc.

S/S — solidification/stabilization, used in this document to encompass the variety of processes that may contribute to increased physical strength and/or contaminant immobilization.

S/S-treated waste — a waste liquid, solution, slurry, sludge, or powder that has been converted to a stable solid (granular or monolithic) by an S/S treatment process.

STLC — Soluble Threshold Limit Concentration, limit applied to Cal WET leaching results (Ca 22 California Code of Regulations 66699).

silica fume — very fine silica dust produced by condensation of silica fumes.

sludge — in this document, sludge means a viscous semi-solid or fluid containing contaminants requiring treatment. The regulatory definition is any solid, semi-solid, or liquid waste generated from a municipal, commercial, or industrial wastewater treatment plant, water supply treatment plant, or air pollution control facility with the exception of specific exclusions such as the treated effluent from a wastewater treatment plant (40 CFR 260.10).

solid waste - see RCRA solid waste.

solidification — a process in which materials are added to the waste to convert it to a solid or to simply improve its handling and physical properties. The process may or may not involve a chemical bonding between the waste, its contaminants, and the binder. In solidification, the mechanical binding of contaminants can be on the microscale (microencapsulation, absorption, or adsorption) or the macroscale (macroencapsulation).

solubility — the maximum concentration of a substance dissolved in a solvent at a given temperature.

solubility product — a type of simplified equilibrium constant defined for and useful for equilibria between solids and their respective ions in solution.

solution — a single, homogeneous phase of liquid, solid, or gas in which a solute is uniformly distributed.

sorption — a general term used to encompass the processes of adsorption, absorption, desorption, ion exchange, ion exclusion, ion retardation, chemisorption, and dialysis.

stability — the stabilization and solidification provided by an S/S process.

stabilization — a process by which a waste is converted to a more chemically stable form. The term may include solidification, but also includes chemical changes to reduce contaminant mobility.

storage — the holding of hazardous waste for a temporary period, at the end of which the hazardous waste is treated, disposed of, or stored elsewhere (40 CFR 260.10).

surfactant — surface-active agent, a soluble compound that reduces the surface tension of liquids, or reduces interfacial tension between two liquids or a liquid and a solid.

TCE — trichloroethylene.

TCLP — Toxicity Characteristic Leaching Procedure, the primary leach testing procedure required by 40 CFR 261.24 and the most commonly used test for degree of immobilization offered by an S/S process.

TDS — total dissolved solids.

TOC — total organic carbon, a chemical analysis.

TRD - Technical Resources Document.

TSCA — Toxic Substances Control Act.

TSD — treatment, storage, and disposal facility (RCRA).

TTLC — Total Threshold Limit Concentration, limit applied to Cal WET leaching results (Ca 22 California Code of Regulations 66699).

TWA — Total Waste Analysis, total concentration of priority pollutants, organics, and metals in the waste

technology screening — the logistic of technology selection, evaluation, and optimization. A treatment technology properly screened prior to full-scale implementation has the highest probability of success in the field.

thermoplastic resin — an organic polymer with a linear macromolecular structure that will repeatedly soften when heated and harden when cooled; for example styrenes, acrylics, cellulosics, polyethylenes, vinyls, nylons, and fluorocarbons.

thermosetting resin — an organic polymer that solidifies when first heated under pressure, and which cannot be remelted or remolded without destroying its original characteristics; for example epoxies, melamines, phenolics, and ureas.

tortuosity — the ratio of the length of a sinuous pathway between two points and the length of a straight line between the points.

toxicity characteristic — exhibiting the hazardous characteristic of toxicity as defined in 40 CFR 261.24.

transportation — the movement of hazardous waste by air, rail, highway, or water (40 CFR 260.10).

treatability study — a study in which hazardous waste is subjected to a treatment process to determine: (1) whether the waste is amenable to the treatment process, (2) what pretreatment (if any) is required, (3) the optimal process conditions needed to achieve the desired treatment, (4) the efficiency of a treatment process for a specific waste or wastes, or (5) the characteristics and volumes of residuals from a particular treatment process (40 CFR 260.10).

treatment — any method, technique, or process, including neutralization, designed to change the physical, chemical, or biological character or composition of any hazardous waste so as to neutralize such waste, or so as to recover energy or material resources from the waste, or so as to render such waste non-hazardous, or less hazardous; safer to transport, store, or dispose of; or amenable for recovery, amenable for storage, or reduced in volume (40 CFR 260.10).

triaxial compression — compression caused by the application of normal stress in lateral directions (ASTM D 653, p. 152).

triaxial shear test (triaxial compression test) — a test in which a cylindrical specimen encased in an impervious membrane is subjected to a confining pressure and then loaded axially to failure.

UCS — unconfined compressive strength, the load per unit area at which an unconfined cube or cylindrical specimen of material will fail in a simple compression test without lateral support.

U.S. DOE — United States Department of Energy.

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J.S. DOT - United States Department of Transportation.

U.S. EPA — United States Environmental Protection Agency.

VOC - volatile organic compound, an organic compound with a low boiling point.

WDW - wet/dry weathering.

WET - see Cal WET, a leaching test.

WTC - Wastewater Technology Centre, formerly of Environment Canada.

wet/dry cycle — alternation of soaking and drying a sample to allow determination of material loss and visual observation of sample disintegration resulting from repeated soaking and drying cycles.

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