

WASHINGTON STATE DEPARTMENT OF **ECOLOGY**



An Assessment of Laboratory Leaching Tests for Predicting the Impacts of Fill Material on Ground Water and Surface Water Quality

--A Report to the Legislature--

December 2003

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STATE OF WASHINGTON

DEPARTMENT OF ECOLOGY

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December 31, 2003

The Honorable Bob Morton, Chair Senate Committee on Natural Resources, Energy and Water Washington State Senate 210 John A. Cherburg Building P.O, Box 40466 Olympia, WA 98504-0466

The Honorable Kelli Linville, Chair House Committee on Agriculture and Natural Resources Washington House of Representatives John L O'Brien Building 2nd Floor, Room 207-A P.O. Box 40600 Olympia, Washington 98504-0600

Dear Senator Morton and Representative Linville:

In accordance with Substitute Senate Bill 5787, we are transmitting our report on leaching tests utilized for evaluating the potential impacts of fill material to water quality. The report was prepared under the direction of Ecology staff by Science Applications International Corporation (SAIC), a consultant retained by the Department of Ecology's Toxics Cleanup Program for this work.

SAIC was selected based on their qualifications and best value for the estimated cost through a process of soliciting and evaluating proposals using General Administration's list of prequalified consultants. Consultants providing services related to the 3rd runway expansion of the Sea-Tac Airport to any party involved in the litigation pertaining to this project or previously provided such services were disqualified to avoid any actual or potential appearance of conflict of interest.

This report indicates that the leaching of contaminants from fill materials is a complex process and that the use of leaching tests to predict these processes is an evolving area of science. As such, no one single laboratory leaching test can evaluate the leaching behavior of a wide variety of materials in a broad range of management scenarios. However, when used within the proper framework, leaching tests can provide useful information for environmental decision-making. Senator Morton and Representative Linville December 31, 2003 Page two

Based on this report, we do not recommend any specific legislative or rule changes at this time. Ecology staff are available to brief your committees about this work if you so desire. Questions regarding the technical aspects of this report can be directed to Pete Kmet of my staff. Mr. Kmet can be reached at <u>pkme461@ecy.wa.gov</u> or (360) 407-7199.

Thank you for the opportunity to work on this very interesting project. We believe this report provides valuable information that will help guide future Department decisions regarding the use of leaching tests, not only in 401 certifications and site cleanup decisions, but in other Department programs that use leaching tests in decision-making.

We look forward to further discussions on this topic with you and your staff.

Sincerely,

James J. Pendowski Program Manager Toxics Cleanup Program

JJP:cp

cc:

Senator Hewitt Senator Fraser Senator Doumit Senator Hale Senator Hale Senator Hargrove Senator Honeyford Senator Oke Senator Regala Genevieve Pisarski Representative Rockerfeller Representative Schoesler Representative Chandler Representative Eickmeyer Representative Grant Representative Holmquist Representative Hunt Representative Kristiansen Representative McDermott Representative Orcutt Representative Quall Representative Sump Caroleen Dineen



An Assessment of Laboratory Leaching Tests for Predicting the Impacts of Fill Material on Ground Water and Surface Water Quality

A Report to the Legislature

Prepared by Science Applications International Corporation Washington State Department of General Administration Contract No. 30700-100968

> Toxics Cleanup Program Olympia, Washington 98504-7600

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LIST OF ACRONYMS

AGP-ANP – Acid generation potential/acid neutralization potential ALT-MAT – Alternative materials APC – Air pollution control ASTM – American Society for Testing and Materials BUD – Beneficial use determination CDF – Confined disposal facility CEN - Committee European de Normalization CFR - Code of Federal Regulations CWA – Clean Water Act DAF – Dilution Attenuation Factor DRET - Dredge Elutriate Test **EP** – Extraction Procedure EPA – U.S. Environmental Protection Agency GC/MS – Gas chromatograph & mass spectroscopy HSWA - Hazardous and Solid Waste Amendments LDR – Land Disposal Restrictions LEP - Canadian Leachate Extraction Procedure L/S – Liquid to solid ratio MCL – Maximum contaminant level mg/kg – milligrams per kilogram mg/L – milligrams per liter MSW - Municipal solid waste MSWI - Municipal solid waste incinerator MTCA - Model Toxics Control Act m/v - mass to volume MWC - Municipal waste combustion MWEP - Monofilled Waste Extraction Procedure MWMP – Meteoric Water Mobility Procedure NEN – Netherlands Normalization Institute PAH – Polycyclic aromatic hydrocarbon PCBs - Polychlorinated Biphenyls PCLT - Pancake Column Leach Test RCRA - Resource Conservation and Recovery Act RCL - Residual contaminant level RCW - Revised Code of Washington RSD - Relative standard deviation SBLT – Sequential Batch Leachate Test SET – Standard Elutriate Test SPLP – Synthetic Precipitation Leaching Procedure SSB - Substitute Senate Bill SWEP - British Columbia Special Waste Extraction Procedure TARP - Technology Acceptance and Reciprocity Partnership TCLP - Toxicity Characteristic Leaching Procedure TKN – Total Kjeldahl nitrogen

TSS – Total suspended solids

 $\mu g/l - Micrograms per liter$

USEPA – U.S. Environmental Protection Agency

UST – Underground storage tank

VOC – Volatile Organic Compound

v/v - volume to volume

WAC - Washington Administrative Code

ZHE – Zero headspace extractor

ABSTRACT

Washington State Legislature Substitute Senate Bill (SSB) 5787 added new part 90.48.531 to the Revised Code of Washington (RCW) directing the Washing Department of Ecology to submit a report to the legislature by December 31, 2003 to identify leaching tests for evaluating the potential impacts to water quality in situations where fill material is imported. The legislature directed Ecology to assess whether the tests provide appropriate methods for analyzing water quality impacts for all types of projects and in all circumstances where fill material is imported and identify any gaps in leaching test methodology. Ecology retained SAIC to prepare this report in response to the requirements of RCW 90.48.531.

A literature search was conducted, and sixteen leaching tests were identified based on their capabilities to evaluate impacts to water quality from fill materials. Detailed descriptions of each test are given including those leaching tests identified in the soil clean-up rules at Chapter 173–340 Washington Administrative Code (WAC) Model Toxics Control Act (MTCA) adopted under Chapter 70.105D RCW (i.e., the SPLP and the TCLP).

To evaluate the extent to which leaching tests accurately predict leaching, a literature search was conducted to identify studies in which leaching test results were compared to actual field leachates. Relatively few studies were found, most focusing on inorganic constituents rather than organic constituents. The results of these studies were mixed in that some leaching tests over-predicted the field leaching, some under-predicted field leaching, and others provided ambiguous results.

An assessment was conducted of techniques used to evaluate leaching test results. Techniques used depend on the testing objectives and end use of the data. In general, leaching test results are compared to regulatory or risk-based levels (e.g., to classify a waste or soil) using direct comparison or statistical methods, or they are used to predict the constituent release under an anticipated field scenario (e.g., by using models that take into account site-specific release mechanisms, pH, and liquid-to-solid ratio over a specified timeframe).

An assessment was conducted to identify gaps in leaching test methodologies. The assessment evaluated each of the sixteen leaching tests against criteria such as implementability, accuracy, reproducibility, and ability of the test to address scenario-specific factors. The findings were consistent with the growing consensus among researchers: Evaluating the leaching behavior of a wide variety of materials in a broad range of management scenarios cannot be addressed adequately by one single laboratory leaching test. Existing leaching tests may be adequate where there is a reasonable match between laboratory test and field conditions, or where the test provides conservative results. An alternative to the use of single scenario batch tests is to use a framework to define the question to be answered, specify the disposal or use scenario, identify relevant parameters influencing leaching, perform tests from a suite of leaching tests for those parameters, and model leaching behavior to simulate and forecast release under the specified time and use scenario. This step-wise approach is used in Europe and a similar framework has been proposed in the United States. The study recommends that Ecology consider such an approach using a small number of leaching tests and a hierarchy in which the type and number of tests employed is scaled to the amount of leaching information required by the user.

ACKNOWLEDGEMENTS

Development of this report was funded by the Washington Department of Ecology (Ecology), Toxics Cleanup Program, under the Washington State Department of General Administration Contract No. 30700-100968. It has been reviewed by Ecology and approved for publication.

This report was developed under the direction of Mr. Pete Kmet (Ecology). Authors included Ray Anderson, Adria Diaz, Sara Hartwell, Doug Pearman, and Robert B. Stewart of Science Applications International Corporation (SAIC). Ecology gratefully acknowledges the contributions of the technical reviewers involved in this effort, including the following Ecology staff: Pete Kmet, John Keeling, Jim Knudson, and Brett Betts.

EXECUTIVE SUMMARY

Washington State Legislature Substitute Senate Bill (SSB) 5787 was approved by the Governor of the State of Washington on May 9, 2003. This bill added two new parts to the Revised Code of Washington (RCW). New part RCW 90.48.530 authorizes the Washington State Department of Ecology (Ecology) to require the use of leaching tests adopted by rule under Chapter 70.105D RCW (Model Toxics Control Act or MTCA) to evaluate the suitability of potential fill material in connection with any water quality certification issued under Section 401 of the federal Clean Water Act (CWA) and in connection with any administrative order issued under Chapter 90.48 RCW, Water Pollution Control. The leaching tests allowed under the MTCA rules (WA State's Cleanup Regulations or WAC 173-340) include the EPA Method 1312, Synthetic Precipitation Leaching Procedure (SPLP), EPA Method 1311, Toxicity Characteristic Leaching Procedure (TCLP), and other unspecified alternative leaching tests. Under the cleanup regulations, these tests can be used for deriving soil concentrations protective of ground water at contaminated sites.

New part RCW 90.48.531 directed Ecology to prepare and submit a report to the legislature by December 31, 2003 to identify leaching tests used for evaluating the potential impacts to water quality in situations where fill material is imported. Furthermore, the legislature directed Ecology to assess whether the tests provide appropriate methods for analyzing water quality impacts for all types of projects and in all circumstances where fill material is imported and identify any gaps in leaching test methodology. Ecology prepared this report in response to the requirements of RCW 90.48.531.

The assessment of leaching tests was conducted in a series of steps that included scoping of the project, identifying and describing available leaching tests, assessing the performance of leaching tests for specific applications, and identifying gaps in leaching test methodologies that could be used to determine the suitability of fill material.

First, a broad search was conducted of regulatory and non-regulatory programs that employ leaching tests (e.g., to evaluate wastes or soil in connection with disposal, remediation, materials reuse, or risk assessment). The search included programs of federal agencies, states, and other countries. Following this analysis, a list of sixteen leaching tests was identified based on their capabilities to evaluate impacts to water quality from fill materials. As suggested by SSB 5787, this list of leaching tests included those identified in the soil clean-up rules at Chapter 173–340 Washington Administrative Code (WAC) Model Toxics Control Act (MTCA) adopted under Chapter 70.105D RCW (i.e., the SPLP and the TCLP).

Third, to evaluate the extent to which leaching tests results accurately predict leaching, a literature search was conducted to identify studies in which leaching test results were compared to actual field leachates. The literature search revealed relatively few studies involving comparisons of laboratory leaching test results to actual field data, and most of the work to date has focused on leaching of inorganic constituents rather than organic constituents. Of those studies reviewed, the results were mixed in that some leaching tests over-predicted the field leaching, some under-predicted field leaching, and others provided ambiguous results. For example, in one study (Lackovic, et al., 1997) the SPLP was found to be more realistic than the

TCLP for assessing the mobility of metals in soils, however, the SPLP over-estimated the mobility of most metals (e.g., arsenic, lead, and zinc) but under-estimated the mobility of chromium. Importantly, batch tests such as the TCLP and SPLP are designed to screen or categorize wastes or materials based on a single disposal scenario -- not to predict constituent concentrations in leachate on a site-specific basis. Thus, the results of batch leaching tests are not expected to match field leachates except where there is a reasonable match between field and laboratory test conditions (such as pH and liquid-to-solid ratio).

Fourth, an assessment was conducted of the various techniques available for evaluating leaching test results. There are various types of leaching tests, each designed to address some specific aspect of leaching. Some tests are designed to model a specific leaching scenario (e.g., the TCLP models co-disposal of industrial waste with municipal solid waste) and others measure some intrinsic property of leaching (such as constituent solubility as a function of pH). Thus, the methods used to evaluate and interpret the test results must consider the test objectives, the manner in which the result is expressed, and the scientific, quality, and regulatory objectives.

Finally, an assessment was conducted to identify any gaps in leaching test methodology. The assessment evaluated each of the sixteen leaching tests against criteria such as implementability, accuracy, reproducibility, and ability of the test to address scenario-specific factors (e.g., aerobic vs. anaerobic conditions, saturated vs. unsaturated environments). The findings of the assessment are consistent with the growing consensus among researchers: Evaluating the leaching behavior of a wide variety of materials in a broad range of management scenarios cannot be addressed adequately by one single laboratory leaching test. For example, batch tests such as the SPLP or TCLP may be adequate for screening or categorizing materials where the test is known to produce sufficiently conservative (environmental protective) results. Batch tests also can be used to predict constituent concentrations in leachate for selected constituents where the anticipated field conditions (e.g., pH, liquid-to-solid ratio) match the laboratory test conditions. Tests such as the Sequential Batch Leachate Test (SBLT) may be more appropriate for evaluating fill material emplaced in a saturated freshwater or marine environment under anaerobic conditions.

An alternative to the use of single scenario batch tests is to use a framework to define the question to be answered, specify the disposal or use scenario, identify relevant parameters influencing leaching, perform tests from a suite of leaching tests for those parameters, and model leaching behavior to simulate and forecast release under the specified time and use scenario. This step-wise approach is used in Europe (CEN, 1997) to evaluate materials for disposal and beneficial reuse, and a similar framework has been proposed in the United States (Kosson, et al., 2002) in response to criticisms of the TCLP. A common theme of these leaching frameworks is the use of a hierarchy of leaching tests in which the type and number of tests employed is scaled to the type and amount of leaching information required by the user. One option is for Ecology to explore the use of such a framework that would include a small number of existing (or modified) leaching tests capable of addressing a broad range of management scenarios for fill material.

1.0 INTRODUCTION

1.1 Background

Washington State Legislature Substitute Senate Bill (SSB) 5787 was approved by the Governor of the State of Washington on May 9, 2003. This bill added two new parts to the Revised Code of Washington (RCW). Part 1 added 90.48.530, entitled "Construction projects involving fill material -- Leaching test" and part 2 added 90.48.531, entitled "Leaching tests -- Identification -- Report to the legislature." The text of SSB 5787 is given in **Appendix A**.

New part RCW 90.48.530 establishes laboratory leaching tests that the Washington Department of Ecology (Ecology) may use to evaluate the suitability of potential fill material in connection with any water quality certification issued under Section 401 of the federal Clean Water Act (CWA) and in connection with any administrative order issued under Chapter 90.48 RCW, Water Pollution Control. The leaching tests include those specified in Ecology's soil clean-up rules at Chapter 173–340 Washington Administrative Code (WAC) Model Toxics Control Act (MTCA) adopted under Chapter 70.105D RCW. The leaching tests at WAC 173-340-747(7) include (1) EPA Method 1312, Synthetic Precipitation Leaching Procedure (SPLP), (2) EPA Method 1311, Toxicity Characteristic Leaching Procedure (TCLP), and (3) "alternative leaching tests" [per WAC 173-340-747(7)(d)] for deriving soil concentrations for ground water protection.

New part RCW 90.48.531 directs Ecology to prepare and submit a report to the legislature by December 31, 2003 to:

- Identify leaching tests (including those identified in the MTCA soil clean-up rules)
- Assess whether the leaching tests provide appropriate methods for analyzing water quality impacts for all types of projects and in all circumstances where fill material is imported, and
- Identify any gaps in leaching test methodology.

This report responds to the requirements of RCW 90.48.531.

1.2 Approach

This assessment of leaching tests was conducted in a series of steps that included scoping of the project, identifying and describing available leaching tests, assessing the performance of leaching tests for specific applications, and identifying gaps in leaching test methodologies that could be used to determine the suitability of fill material. The steps are outlined below.

• Scoping – Upon passage of SSB 5787, Ecology conducted a scoping analysis that included a review of the legislative direction given in Section 2 of SSB 5787, available resources, and the required schedule. Based on this scoping analysis, Ecology identified

specific tasks to identify leaching tests for evaluating water quality impacts where fill materials is used, assess whether the available tests are appropriate for all project and circumstances where fill material is used, identify gaps in the leaching test methods, and report the findings to the legislature. Based on time and resource constraints, the assessment was conducted using only information obtained from existing literature (e.g., from scientific journals), standards setting organizations, and government sources. No new primary data were generated (e.g., via field sampling and laboratory analysis) as part of this study.

- Identify and Describe Available Leaching Tests The language of SSB 5787 required Ecology to "identify the leaching tests utilized for evaluating the potential impacts to water quality in situations where fill material is imported." This requirement was addressed in a two-step process. First, a broad search was conducted of regulatory and non-regulatory programs that employ leaching tests (e.g., to evaluate wastes, soil, or other materials in connection with disposal, remediation, materials reuse, or risk assessment). The search included programs of federal agencies, states, and other countries. Following this analysis, a subset of the leaching tests was identified based on their capabilities to evaluated impacts to water quality from fill materials. As suggested by SSB 5787, this list of leaching tests (sixteen in all) included those identified in the soil clean-up rules at Chapter 173–340 Washington Administrative Code (WAC) Model Toxics Control Act (MTCA) adopted under Chapter 70.105D RCW (i.e., the SPLP and the TCLP).
- Assess Performance and Application of Leaching Tests SSB 5787 required Ecology to "assess whether [the] list of leaching tests provides appropriate methods for analyzing water quality impacts for all types of projects and in all circumstances" where fill material is used. For the purpose of this assessment, Ecology interpreted the term "fill material" to include any material that is not a dangerous waste or extremely hazardous wastes and that is exempt from the State's solid waste permitting requirements. Ecology interpreted the term "projects" to include construction projects and the phrase "all circumstances" to include the use of fill materials in an upland setting above the saturated zone, in a wetland, in a freshwater environment, and in a marine environment. Two analyses were conducted to assess the performance of leaching tests applied to fill materials placed in saturated or unsaturated environments in construction projects. (1) A literature search was conducted to identify studies in which leaching test results were compared to actual field leachates. The objective of this task was to evaluate the extent to which leaching tests results accurately predict leaching. (2) An assessment was conducted of the various techniques available for evaluating leaching test results.
- Identify Gaps in Leaching Test Methodologies Each leaching test was evaluated to determine to what extent the test is suitable for analyzing water quality impacts for fill projects. The coverage and gaps in the capabilities of the leaching test methods were evaluated by identifying the major factors that influence leaching behavior, identifying the aspect of leaching each test was designed to model or simulate, and evaluating the potential suitability of each test for assessing the leaching characteristics of fill materials.

2.0 FUNDAMENTALS OF LEACHING AND LEACH TESTING

This section provides an overview of leaching processes that occur under field conditions and provides an introduction to how laboratory tests are used to evaluate leaching phenomena.

2.1 Leaching of Fill Material: Basic Concepts

Leaching is the process by which soluble constituents are dissolved from a solid material (such as rock, soil, or waste) into a fluid by percolation or diffusion. Thus, when fill materials come into contact with liquid (including percolating rainwater, surface water, groundwater, and liquids present in the fill material), constituents in the solid phase will dissolve into the liquid forming a leachate. The extent to which the constituents dissolve into the contact liquid will depend upon site- and material-specific conditions (chemical, physical, and biological factors) and the length of time involved. The composition of the leachate generated from the material and its potential to impact water quality are key factors in evaluating the suitability of the material for use as fill.

Figure 2-1 presents a conceptualized view of the water balance at a hypothetical site at which fill material has been emplaced. Water enters the site via net precipitation (that is, precipitation minus evapotranspiration), run-on, and groundwater or surface water intrusion. In the figure, fill material can be subject to leaching due to exposure to moisture present within the fill material, infiltrating precipitation, run-on, and due to placement in a saturated environment.

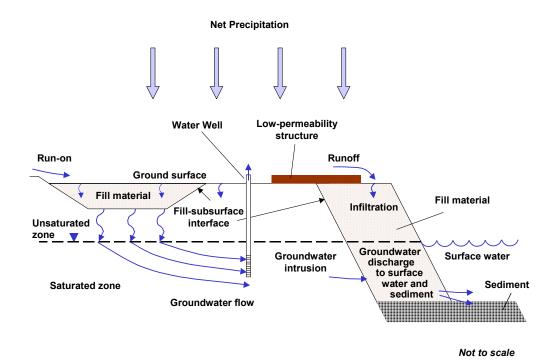


Figure 2-1. Conceptual model of water balance at a fill site

2.2 Factors Controlling Leaching Under Field Conditions

Various physical, chemical, and biological factors influence leaching from waste and soil. These factors are related to the management scenario and material-specific properties.

Physical factors related to the management scenario include the following:

- *Climate and meteorological conditions.* The amount of net precipitation at a fill site will influence the amount of water available for infiltration through the site.
- Design/configuration of the fill site. The depth of the fill will affect the quality of the leachate. Water entering the top surface of the fill material (as infiltration) will travel down through it from interconnected void spaces until it eventually reaches the bottom of the unit as leachate. Dissolution from the solid phase to the water will occur until solubility limits are reached. The deeper the unit, the greater the contact time between the percolate and the fill material and thus there will be a greater opportunity for the leachate to reach saturation limits (Lu, et al., 1985). Also, the deeper the unit, the longer it will take for contaminants to be depleted. In addition, the presence of a cap, pavement, or other low permeability structure will reduce the opportunity for infiltration and leachate generation. Topography will affect the site's runoff pattern and the amount of water entering (via run-on) and leaving (via run-off) the site.
- Vegetation. Vegetation limits infiltration by intercepting precipitation directly (thereby improving evaporation from the surface) and by taking up soil moisture and transpiring it back to the atmosphere (i.e., via evapotranspiration). A fill site lacking vegetative cover may experience erosion that cuts gullies through the cover material and allows precipitation to flow directly into it.
- Hydrogeological conditions. Subsurface geologic conditions and depth to groundwater at a fill site can impact the generation of leachate. For materials that are emplaced at or below the water table, ground water flowing through the material can provide a source of water that is in addition to precipitation and run-on.

Characteristics of the fill material that influence leaching include the following (modified from van der Sloot, et al. 1997):

- Particle size, shape, and surface area exposed to leaching
- Permeability of the matrix (during testing or under field conditions) and flow rate of the leaching fluid
- Physical properties of the leaching fluid (e.g., viscosity)
- Heterogeneity of the fill material
- Temperature during leaching

Chemical factors that influence leaching include equilibrium- (relatively quick chemical dissolution reactions) and kinetic- (e.g., relatively slow desorption reactions) based reactions, solubility/desorption characteristics of constituents, the presence of non aqueous phase liquid (NAPL), pH of the fill material, pH of the leaching fluid, and complexation with inorganic or organic compounds (van der Sloot, et al., 1997).

Biological factors that influence leaching include the presence of microorganisms that, through the process of biodegradation, can change redox and pH conditions within the fill material, affecting the solubility of contaminants. These microorganisms can also biotransform or change contaminants into compounds that are more or less toxic than the parent contaminant. On a larger scale, roots and burrowing animals may create pathways that increase infiltration.

2.3 Leaching Tests: Basic Concepts

Leaching behavior in the environment is studied by means of field or laboratory experiments.

Field experiments generally involve the use of controlled pilot landfill cells or field lysimeters. A field lysimeter is container usually installed in the unsaturated zone used to measure percolation, leaching and evapotranspiration losses from the contained material. It may be less than a cubic yard to several hundred cubic yards in size and can be constructed outside to permit exposure to the natural environment or inside in closely controlled laboratory conditions (see **Figure 2-2**). Field lysimeter tests address different aspects of leaching, such as the physical mechanisms involved, chemical interactions between the waste and the leaching fluid, the kinetics of leaching, and leaching as a function of pH, time, and liquid-to-solid (L/S) ratio (Kim, 2002).

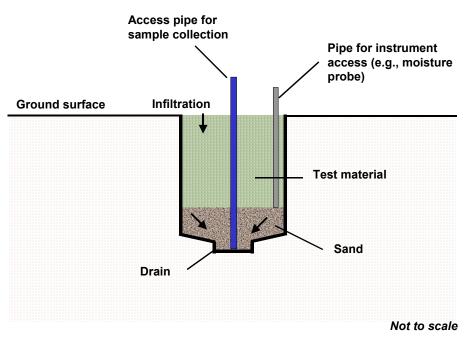


Figure 2-2. Schematic diagram of a field lysimeter

Laboratory leaching tests involve contacting one or more samples of the study material with a liquid to determine which constituents will be leached by (or dissolved into) the liquid and potentially released to the environment in a liquid phase (e.g., to ground water or surface water). See **Figure 2-1**.

Laboratory experiments may include single extraction/batch tests or multiple extraction/flowaround/flow-through ("dynamic") leaching tests. One feature common to all leaching tests is the output of a leachate that is then used to assess some specific property of the material or to simulate a field leaching scenario.

Batch extraction tests typically involve mixing a sample of waste or other fill material with a specific amount of leaching solution without renewal of the leaching solution. The mixing is performed over a relatively short time period (hours to days) with the aim of reaching equilibrium conditions. The mixing is followed by filtration and analysis of the filtered liquid phase (the laboratory leachate). The U.S. EPA Method 1312, Synthetic Precipitation Leaching Procedure (SPLP) is an example of such a test. **Figure 2-3** provides a simplified schematic of a batch extraction test.

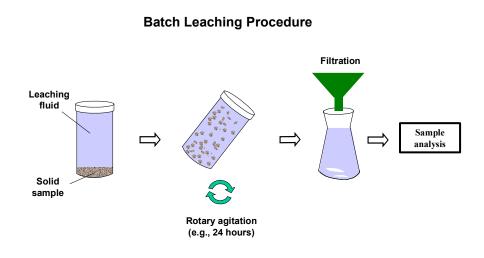
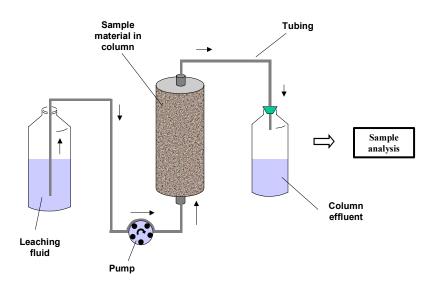


Figure 2-3. Simplified schematic of a batch equilibrium leaching procedure

Dynamic tests typically address some aspect of leaching in which time is an important variable. In dynamic tests, a specific amount of leaching solution and test material are mixed and the leaching solution is periodically or continuously renewed. The mixing is performed over a relatively long time period (days to months) compared to extraction tests. An example of a dynamic leaching test is the ASTM column test, D 4874 Standard Test Method for Leaching Solid Waste in a Column Apparatus. **Figure 2-4** provides a simplified schematic of a column leaching test.



Column Leaching Procedure

Figure 2-4. Simplified schematic of a column leaching test

The sample analysis results from leaching tests are used either to simulate a field-leaching scenario or to assess some specific intrinsic property of the material. For example, leaching tests are designed with specific objectives in mind such as the following:

- Classify a waste as hazardous or nonhazardous (e.g., via the TCLP)
- Determine the effectiveness of a waste treatment process (e.g., via the TCLP)
- Estimate a source term or end point for risk assessment purposes; or
- Assess the release potential under specified reuse or disposal conditions.

The specific test employed must be selected based on regulatory requirements, technical objectives, and consideration of the end use of the data.

As mentioned previously, various physical, chemical, and biological factors influence leaching from a waste or soil under field conditions. To evaluate leaching phenomena in the laboratory setting, however, a small number of the most easily modeled factors typically are incorporated into a laboratory test. The factors are related to the leaching fluid, the particle size, and the management scenario being evaluated. A brief description of these factors is provided in the following sections.

2.3.1 Leaching Fluid

Leaching is the extraction of constituents from a solid matrix by a liquid (the leaching fluid). Solubility of the constituents in the leaching fluid is a major mode of leaching behavior. The

solubility of inorganic constituents is strongly influenced by pH and redox potential, while the solubility of organic constituents is driven by polarity and partitioning effects.

The solubility of inorganics can be influenced by the presence of other constituents. In particular, adsorption phenomena, competition for ligands (i.e., groups or ions attached to a central metal ion), and the "common ion effect" influence the solubility of inorganic constituents, and consequently, their availability for leaching.

Adsorption phenomena are known to play a significant role in the leachability of inorganic constituents. For example, the ability of iron oxides to adsorb metals, removing them from solution, has a powerful impact on leachability. Similarly, the release of these metals adsorbed to ferric oxides impacts concentrations of ions in solution (leachability). For example, arsenic adsorbed to ferric oxides is released from oxidized sediments during the transformation between oxidizing and reducing conditions (Loeppert, et al., 1995).

Similarly, the solubility of metals can be influenced by competition with other metals in solution for organic ligands. For examples, the competition between lead (Pb) and iron (Fe) for ligands in soils may be important in the aqueous solubility of Pb, and is believed to explain field observations of Pb solubility that are greater than predicted values (Dong, et. al, 2000).

The common ion effect is the disturbance of an ionic equilibrium by the addition of one or more of the ions involved. Disturbance of this ionic equilibrium can influence solubility of one or more species in solution, because the solubility of a slightly or moderately soluble compound is smaller in a solution that contains an excess of a soluble compound that has an ion in common, than it is in reagent water. For example, PbCl₂ is moderately soluble in water:

 $PbCl_2 \leftrightarrow Pb^{2+} + 2Cl^{-}$

The equilibrium of PbCl₂, Pb²⁺, and Cl⁻ is disrupted by the addition of an excess of soluble chloride (Cl⁻) by adding NaCl. This disruption will shift the equilibrium expression to the left, precipitating additional PbCl₂ - in other words, limiting its solubility (Segal, 1989).

The volume of leaching fluid available for constituents to leach into, relative to the mass of constituents available to leach, can influence the ultimate concentration in the leachate. This relationship is controlled by the ratio of liquid (L) to solid (S), or L/S ratio. A smaller L/S ratio may result in lower concentrations of less soluble species, and higher concentrations of more soluble species. A smaller L/S ratio may limit the amount of constituent leached because of the common ion effect (Lowenbach, 1978). A higher L/S ratio may result not only in higher concentrations of some constituents, but in a larger total number of constituents leaching (Lowenbach, 1978; van der Sloot, et al., 1997). In general, the cumulative amount of constituents leached generally increases as the L/S ratio increases (Kmet, 1984). The L/S ratio of the disposal scenario can be estimated by dividing the total amount of liquid that will contact the solid material over a period of time by the total quantity of disposed material. This estimation may be more useful in a relative sense. That is, the L/S ratio of a marine or freshwater disposal scenario will be very high compared to that of the L/S ratio of an upland disposal scenario. However, the L/S ratio of the upland disposal scenario will depend on

precipitation and infiltration rates, the fill density, and the geometry of the fill (e.g., thickness and exposed surface area).

The ionic strength of the leaching fluid also influences solubility and leaching behavior. Ionic strength is the relationship of the concentration of ions in solution and the charges of those ions. Ionic strength impacts reaction rates as well as the solubility of ionic species, with solubility generally increasing as ionic strength increases (Lowenbach, 1978). This relationship is particularly important when considering leaching behavior in marine and estuarine environments, where the water has relatively high concentrations of ions in solution.

2.3.2 Particle Size

Leaching is a function of the surface exposed to the leaching fluid. The ratio of the particle surface area to the volume occupied by the particles, the average particle size, and internal pore structures in the material all control the surface area where dissolution from the solid to the liquid can occur (van der Sloot, et al. 1997). Smaller particle sizes produce larger surface area, allowing for increased contact between the solid material and the leaching fluid, resulting in increased contact between the leaching fluid and leachable constituents.

Multiple phenomena can affect particle size after placement of the fill material. Crushing, grinding, and compaction, which usually occur as the fill material is placed, can reduce the size and increase the surface area of larger particles. Subsequent vehicular traffic can subject the solid material to compressive and flexural forces that can lead to cracking and crumbling. If the fill material is exposed to the weather following placement, physical (and chemical) degradation can be caused and/or accelerated by the sun, wind and rain erosion, freeze/thaw cycles, and by wetting/drying cycles. For example, nightly freezing and thawing cycles during the winter can lead to cracking and crumbling of the fill material, and hence an increase in leachable surface area.

Homogeneity of particle sizes can have a strong influence on how well laboratory-based testing predicts leaching behavior. The less homogeneous a material is, the more difficult it becomes to collect a representative sample. This may result in a test sample with a surface area that is significantly larger or smaller than the average surface area in the fill material, with a consequent distortion in leaching performance. Particle size reduction and/or control techniques employed in leaching tests may exacerbate this distortion, particularly if the fill material tends to comprise large chunks of material that will not reduce in size over time due to external forces.

2.3.3 Management Scenario/Conditions

The chemical conditions at the disposal/management site will strongly influence leaching, and should, therefore, be considered when selecting a leaching test to predict leaching performance. Most leaching tests are conducted under aerobic conditions (the leaching fluid contains oxygen), however, most marine and freshwater sediments are anaerobic just a few centimeters below the surface (Hartwell/NOAA, 2003; Myers, Brannon, and Price, 1992). Fill material deposited below the water surface in marine and freshwater environments will most likely also become anaerobic. Under anaerobic conditions (no oxygen present), there are multiple species of

bacteria capable of growth by respiratory reduction of metal species. For example, sulfurospirillum barnesii is capable of anaerobic growth using either ferric iron or arsenate as electron acceptors. In this case, arsenate is reduced by the bacteria to arsenite, and Fe+3 (as ferrihydrite) is reduced to Fe+2, a soluble form of iron (Zobrist, et al., 2000). Further, the mobility of cadmium has been reported to be strongly dependent on microbial activity, and is greater under anaerobic than aerobic conditions (Lodenius and Autio, 1989).

2.3.4 Leaching Test Types

Laboratory tests fall into two general categories: (1) single extraction/batch tests (sometimes referred to as "static" extraction tests); and (2) multiple extraction/flow-around and flow-through leaching tests (sometimes referred to as "dynamic" tests).

Single extraction tests include all tests in which a specific amount of leaching fluid is put into contact with a specific amount of waste for a specified length of time, without renewal of the leaching fluid. The resulting leachate is removed from the test, either at various times to derive kinetic information (changing concentrations over time) or, more commonly, at the end of the test, and then analyzed (see **Figure 2-3**). The assumption made when conducting a single extraction test is that a steady-state condition (equilibrium) is achieved by the end of the testing period, though this may not necessarily be the case in practice. Reaching equilibrium in single extraction leach testing is critical to predicting leaching behavior over long periods of time. If tests are conducted at non-equilibrium conditions, leaching behavior does not reach capacity, and predictions of long-term leaching behavior will be based on leachate concentrations that are too low or too high (EQM, 1998). Single extraction leaching tests discussed in this report include the following:

- ASTM D 3987, Standard Test Method for Shake Extraction of Solid Waste with Water
- ASTM D 6234 (ASTM, 2002), Standard Method for Shake Extraction of Mining Waste by the Synthetic Precipitation Leaching Procedure
- SPLP, Synthetic Precipitation Leaching Procedure
- TCLP, Toxicity Characteristic Leaching Procedure
- DRET, Dredge Elutriate Test
- SET, Standard Elutriate Test
- NEN 7341, Availability Test
- EN 12457/1-4, Compliance Test for Granular Waste Materials and Sludges

Multiple extraction tests continuously or intermittently renew the leaching fluid to maintain a driving force for leaching. Multiple extraction tests provide information about the kinetics of contaminant mobilization. There are three primary types of multiple extraction tests: serial batch

tests, flow-around tests, and flow-through tests. In a serial batch test, a portion of a granular sample is mixed with the leaching fluid and agitated at a set L/S ratio for a specified period of time. The leachate is then separated from the solids and replaced with a fresh leaching fluid until the desired number of leaching periods has been completed. Data from these tests can be used to infer temporal release of leachable constituents. Serial batch tests discussed in this report are:

- ASTM D 4793, Standard Test Method for Sequential Batch Extraction of Waste with Water
- ASTM D 5744, Standard Test Method for Accelerated Weathering of Solid Materials Using a Modified Humidity Cell
- SBLT, Sequential Batch Leachate Test

Flow-around tests, as their name implies, are usually applied to monolithic samples. The sample is placed in the test vessel and leaching fluid is added. The flow of leaching fluid around the waste provides the driving force to maintain leaching. The flow-around test discussed in this report is:

• NEN 7345, Tank Leach Test

In a flow-through test, the leaching fluid is passed, either intermittently or continuously, through an open container packed with a porous solid sample. The leachate is periodically sampled and analyzed for the parameters of interest. The results are used to examine contaminant release over time and as a function of L/S ratio. There are two typical types of flow-through tests: lysimeter tests (see **Figure 2-2**) and column tests (see **Figure 2-4**), which differ primarily in size and duration. The flow-through tests discussed in this report are:

- ASTM D 4874, Standard Test Method for Leaching Solid Waste in a Column Apparatus
- PCLT, Pancake Column Leachate Testing
- NEN 7343, Column Test
- prEN 14405, Upflow percolation test

3.0 LEACHING TESTS USED FOR EVALUATING POTENTIAL IMPACTS TO WATER QUALITY IN SITUATIONS WHERE FILL MATERIAL IS IMPORTED

SSB 5787 required Ecology to "identify the leaching tests utilized for evaluating the potential impacts to water quality in situations where fill material is imported." This requirement was addressed the in two steps that are described in this section. Section 3.1 describes a broad literature search conducted of regulatory and non-regulatory programs that employ leaching tests (e.g., to evaluate wastes or soil in connection with disposal, remediation, materials reuse, or risk assessment). The search included programs of federal agencies, states, and other countries. A subset of sixteen leaching tests was identified based on their capabilities to evaluate impacts to water quality from fill materials. Section 3.2 describes the sixteen leaching tests including information about the technical basis for the test and information about the reproducibility of the test.

3.1 Leaching Tests Used by the Federal Agencies, States, and Other Countries

Initial information sources for this section included previous literature research conducted by the Washington Department of Ecology in support of previous regulatory development efforts and existing information on leaching tests obtained from the U.S. Environmental Protection Agency (USEPA) and other government sources.

Supplemental searches were conducted to fill any information gaps. These searches were conducted using resources such as the Bureau of National Affairs Inc. (BNA) Environment and Safety Library on the Web (ESLW), individual state web sites, and direct contact with state regulators.

3.1.1 Federal Hazardous Wastes Regulations – Characteristic of Toxicity

In the Hazardous and Solid Waste Amendments of 1984 (HSWA), Congress directed EPA to revise and expand the existing toxicity characteristics, which are used to identify solid wastes that are hazardous due to their potential to leach toxic constituents. Specifically, HSWA required EPA to reevaluate its use of the leaching test known as the Extraction Procedure (EP) to determine whether wastes exhibited a toxic characteristic and to identify additional hazardous waste characteristics. These mandates were prompted by the concern that the EP inadequately represented the mobility of toxicants under a wide variety of conditions and failed to evaluate the mobility of organic toxicants.

To fulfill these statutory mandates, EPA promulgated the revised Toxicity Characteristics (TC) on March 29, 1990 (55 FR 11798). The TC revisions included a second generation leaching test known as the Toxicity Characteristic Leaching Procedure (TCLP) to replace the EP leach test and added 25 organic chemicals to the list of toxic constituents of concern. The rule also established regulatory levels for contaminants of concern based on health-based concentration limits and dilution-attenuation factors (DAFs) developed using a subsurface fate and transport

model (USEPA, 1990a). The Federal regulations for use of the TCLP are given at 40 CFR 261.24.

Description of Test

The TCLP test involves the extraction of contaminants from a 100-g size-reduced sample of waste material with an appropriate extraction fluid (see **Figure 3-1**). A 20:1 liquid to solid (L/S) ratio (mass/mass, m/m) is employed, and the mixture is rotated for 18 ± 2 hr at 30 rpm using a rotary agitation apparatus (see **Figure 3-2**). Where volatile constituents must be evaluated, a smaller sample mass is used (25 g) and a specialized zero-headspace extraction (ZHE) vessel is employed (see **Figure 3-3**). The extraction fluid used for the extraction depends on the alkalinity of the waste material. Very alkaline waste materials are leached with a fixed amount of acetic acid without buffering the system (pH 2.88 ± 0.05), while other waste materials are leached with acetic acid buffered at pH 4.93 ± 0.05 with 1-N sodium hydroxide. After rotation, the final pH is measured, and the mixture is filtered using a glass fiber filter (see **Figure 3-4**). The filtrate is collected in an appropriate container, and preservative may be added if needed. The filtrate is analyzed for a number of constituents. If these constituent concentrations equal or exceed the concentrations described in 40 CFR 261, then waste is characteristically hazardous for toxicity (unless otherwise excluded).

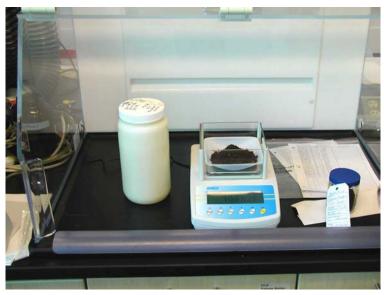


Figure 3-1. Weighing of 100-grams of sample (right) prior to the sample being added to the TCLP bottle extractor (left) containing 2-liters of extraction fluid



Figure 3-2. Rotary agitation device used for sample extraction in the TCLP and SPLP



Figure 3-3. Zero-Headspace Extractor (ZHE) used for liquid/solid separation, extraction, and filtration in the TCLP and the SPLP when the sample is being tested for volatile analytes



Figure 3-4. Filtration unit used for liquid/solid separation in the TCLP and SPLP when the sample is being tested for nonvolatile constituents

Technical Basis of Test

In developing the original toxicity characteristic, EPA designed the EP based upon a "mismanagement scenario" in which potentially hazardous wastes would be co-disposed with municipal solid waste (MSW) in a landfill with actively decomposing material overlying an aquifer. Consistent with this mismanagement scenario, the EP required that a liquid extract be obtained from solid waste (following particle size reduction, if necessary) by exposing the waste to organic acids (the acids likely to be found in a landfill containing decomposing municipal wastes). In conjunction with the co-disposal scenario, EPA assumed that the most likely pathway for human exposure to toxic constituents would be through drinking water contaminated by leachate from the landfill. Analyses of the EP extract were to be compared to the National Interim Primary Drinking Water Standards (NIPDWS). To account for the likely dilution and attenuation of the toxic constituents that would occur as they traveled from the landfill to a drinking water source, regulatory limits were established by multiplying the NIPDWS by a "dilution and attenuation factor" (DAF) of 100. The DAF of 100 was not derived from any model or empirical data, but rather was an estimated factor that EPA believed would indicate substantial hazard.

The TCLP was developed using the same mismanagement assumptions that formed the basis for the development of the EP -- that wastes would be co-disposed with actively decomposing MSW in a landfill. Under this co-disposal scenario, infiltrating precipitation combined with water-soluble products of MSW biodegration act as the leaching fluid. In the TCLP, this is represented

by a sodium acetate buffer solution with a pH of 4.93 (or pH 2.9 for highly alkaline wastes). In selecting the TCLP leaching fluid, it was assumed that the concentration of acetic acid and acetate in the TCLP extract would approximate concentrations of volatile fatty acids likely to occur in actual landfill leachates during the acid generation phase of landfill decomposition. Acetic acid was selected because it was considered to be the most prevalent acid found in MSW leachates (45 FR 33084, May 1, 1980).

The TCLP uses a 20:1 L/S ratio for waste extraction. EPA adopted the 20:1 L/S ratio for its apparent suitability for simulating a mismanagement scenario. The 20:1 TCLP methodology was determined using a combination of factors related to climate, waste characteristics, and disposal practices. The TCLP methodology assumes a 3-meter-landfill depth, 100 cm annual rainfall, 5 percent co-disposal with municipal waste, 100 percent rain percolation through the landfill, 1 gm/cm³ waste density, and three years of leaching (Frampton, 1998). However, the 20:1 ratio of the TCLP methodology represents a significantly longer leaching period of perhaps 3 to 10 years (Kimmell and Friedman, 1986). The TCLP is intended to provide a representation of the average leachate concentration over this 3 to 10 year leaching period, and not the peak concentration.

At the same time that it promulgated the TCLP, EPA also expanded the list of contaminants for the Toxicity Characteristics by adding 25 organic compounds. The new constituents were selected based on the availability of chronic toxicity reference levels. Regulatory levels for these and existing Toxicity Characteristic constituents were established based on chronic toxicity reference levels: Maximum Contaminant Level (MCLs) promulgated under the Safe Drinking Water Act were used when available, while reference doses (RfDs), and risk-specific doses (RSDs) were used for those constituents for which no MCLs had been promulgated at the time. (The RfD is an estimate of a daily oral exposure of a contaminant to the human population that is likely to be without an appreciable risk of deleterious effects during a lifetime. The RSD is the daily dose of a carcinogen over a lifetime that will result in an incidence of cancer equal to a specific risk level.). A subsurface fate and transport model (EPA's Composite Model for Landfills or "EPACML") was employed to determine compound-specific DAFs. EPA stated that a DAF of 100 is sufficient to capture only those wastes that are clearly hazardous.

3.1.2 Arkansas Pollution Control and Ecology Commission/Department of Environmental Quality – Regulations for Site Characterization of Landfills

Regulation No. 22, Section 22.1102 of the Arkansas Pollution Control and Ecology Commission/Department of Environmental Quality establishes site characterization requirements for Class 3 landfills. (In Arkansas, Class 1 landfills accept non-hazardous, household, commercial, and industrial solid waste. Class 3 landfills accept non-hazardous industrial waste. Class 4 landfills accept non-hazardous, bulky, inert, non-putrescible solid waste. Arkansas does not define a Class 2 landfill.) The regulation specifies that requirements for site characterization for Class 3 landfills are dependent on the potential for the waste to impact surface- or groundwater quality as determined by the leaching test ASTM D-3987 (ASTM, 1985). The leaching test results are to be compared to groundwater standards set forth in Appendix 3 and Section 22.523 of the regulations to determine appropriate site characterization requirements. If groundwater standards are exceeded, then Class 1 landfill characterization requirements are applicable. Otherwise, the minimum site characterization requirements for Class 4 landfills apply.

Description of Test

ASTM D-3987 is an agitated extraction procedure that employs reagent water as the extraction fluid. (Reagent water is purified water in which an analyte or interferant is not observed at or above the method's detection limit of the analyte(s) of interest.) In this procedure, a water/solid waste mixture (L/S = 20:1, volume to mass {v/m}) is mechanically agitated for 18 hours using a device that rotates sample containers on a central axis at a rate of 29 rotations/minute. After rotation, the aqueous phase is separated from the solid material (decantation followed by filtration) for analysis. Particle size reduction is not required. The test is applicable to the extraction of inorganic constituents.

Technical Basis of Test

ASTM D-3987 does not modify the pH of the leaching process, instead providing for the determination of the final pH of the leachate at the completion of the test. This allows the user to evaluate the acidity and buffering capacity of the waste matrix. The test is intended to simulate conditions where the solid waste is the dominant factor in determining the pH of the leachate. The test is not intended to simulate site-specific leaching conditions.

The authors have contacted Arkansas Department of Environmental Quality by e-mail to obtain information on why the state selected ASTM D-3987. The information was not available as of the date of this report.

3.1.3 California Regulations for Identification and Listing of Hazardous Wastes

Title 22, Chapter 11, Article 3, Identification and Listing of Hazardous Wastes (§66261.24, Characteristic of Toxicity) of the California Code of Regulations specifies that a waste exhibits the characteristic of toxicity, and thus is a hazardous waste, if a representative sample of the waste has any of eight properties. Two of these properties involve the use of leaching tests: the TCLP and the Waste Extraction Test. According to the regulation, a waste exhibits the characteristic of toxicity if concentrations of contaminants in the extract obtained using the TCLP are equal to or greater than the maximum contaminant concentrations listed in the regulation (which are based on those concentration limits given in 40 CFR Section 261.4). When using the Waste Extraction Test, a waste is characteristically hazardous if the concentrations in the extract equal or exceed the contaminants' soluble threshold limit concentrations (STLC) or total threshold limit concentrations (TTLC).

Description of Test

TCLP - See description given in Section 3.1.1.

Waste Extraction Test - The Waste Extraction Test procedure calls for the extraction of a waste with citrate buffer solution (pH 5) in a closed extraction vessel for a period of 48 hours. A 10:1 L/S ratio (m/m) is used in this method.

Technical Basis for Test

The Waste Extraction Test was incorporated into California regulations for identification of hazardous wastes in 1984 because it was considered to be more representative of leaching conditions found in landfills and soils compared to the federally required method at the time, the Extraction Procedure (EP).

As with the TCLP, the Waste Extraction Test was intended to model the mobility of organic and inorganic constituents from an industrial waste co-disposed with municipal waste in a landfill. However, there are significant differences between these two procedures in terms of the approach utilized to evaluate the leaching potential of a waste. These differences are reflected in the choice of extraction fluid, liquid-to-solid ratio, and extraction time.

California adopted the Waste Extraction Test on the belief, at the time, that the use of the citrate buffer solution in the method simulated more closely conditions found in landfills and soils than the acetate buffer used in the EP. However, while the citrate buffer used in the Waste Extraction Test has been demonstrated to be a more aggressive leaching fluid than the one used in the TCLP, current knowledge of MSW leachate composition does not support the assumption that the citrate buffer best represents the chemical composition of MSW leachates (Frampton, 1998).

The 10:1 L/S ratio (m/m) used in the Waste Extraction Test was chosen to facilitate mixing and handling. An important assumption that was made in adopting a 10:1 ratio was that the total amount of contaminant extracted would be independent of the L/S ratio, however, current understanding of leaching phenomena indicates that the L/S ratio directly influences the quantity of constituents extracted from a waste sample based on principles of chemical thermodynamics (Frampton, 1998).

3.1.4 California Regulations for Recyclable Hazardous Wastes - Requirements for Recyclable Materials that Are Placed on the Land

California Code of Regulations, Title 22, Division 4.5, Chapter 16, Article 3 applies to a recyclable hazardous waste material that is placed on land, either by itself or mixed with other materials. It specifies that a product that uses recyclable hazardous waste materials should not have hazardous constituents in excess of Soluble Threshold Limit Concentrations (STLCs) as specified in California Code of Regulations, Title 22, Division 4.5, Chapter 11, Article 3. Analysis of the recyclable material and the product is to be conducted according to the Waste Extraction Test. The Waste Extraction Test results for each hazardous constituent is multiplied by a dilution factor (calculated by dividing the weight of the final product made with the recyclable material by the weight of the recyclable material used in the product) to obtain the final concentration of hazardous constituents in the product, which should not exceed the STLCs.

Description of Test

Waste Extraction Test - See description in Section 3.1.3

Technical Basis for Test

See discussion in Section 3.1.3.

3.1.5 Illinois Requirements for New Steel and Foundry Industry Wastes Landfills

Illinois Administrative Code Title 35, Part 817 specifies requirements for landfill units that accept waste from the steel and foundry industries. Section 817.103 requires that all individual waste streams to be disposed of or utilized be tested annually using ASTM Method D3987-85 to characterize the expected constituents and concentrations of the leachate.

Description of the Test

ASTM Method D3987-85 - See Section 3.1.2 for a description of this method.

Technical Basis for Test

ASTM D-3987 does not modify the pH of the leaching process, instead providing for the determination of the final pH of the leachate at the completion of the test. This allows the user to evaluate the acidity and buffering capacity of the waste matrix. The test is intended to simulate conditions where the solid waste is the dominant factor in determining the pH of the leachate. The test is not intended to simulate site-specific leaching conditions.

The authors have contacted Illinois Environmental Protection Agency by e-mail to obtain information on why the state selected ASTM D-3987. The information was not available as of the date of this report.

3.1.6 Indiana Department of Environmental Management - Guidance on Lead Issues at Small Arms Firing Ranges

This guidance document (IDEM, 2000) was prepared by the Indiana Department of Environmental Management (IDEM) to address environmental and legal issues regarding lead deposited at outdoor shooting ranges. The document indicates that lead bullet and lead shot impact areas at small arms firing ranges are not regulated under the hazardous waste regulations since these regulations do not extend to products whose normal use involves application to the land. Only when debris from these ranges is generated (i.e., excavated) and destined for disposal would solid or hazardous waste rules apply. IDEM notes that small arms firing ranges do not present extreme environmental hazards and recommends that remedial actions take place at closing ranges prior to re-use of range land. To assess lead impacts on areas surrounding shooting ranges, IDEM recommends that leaching methods such as the Indiana Neutral Leaching Method, ASTM Water Leach Method (ASTM D 3987-85) or SPLP be used to determine lead mobility. IDEM states that the premise behind the TCLP makes it a poor candidate for assessing the level of leachable lead at a firing range, because the amount and type of acids the [TCLP] model uses typically would significantly exceed those types and amounts found naturally.

Description of Tests

Indiana Neutral Leaching Method – In addition to its use in connection with firing range waste, the Neutral Leaching Method is a testing requirement for coal ash, flue gas desulfurization byproducts, and foundry waste that will be disposed in a restricted waste site in accordance with 329 Indiana Administrative Code (IAC) 10-9. The test is the same as SW-846 Method 1311 (TCLP) but uses deionized water instead of the Method 1311 extraction fluids. pH must be analyzed at the end of the 18 hour extraction period.

ASTM Water Leach Method (ASTM Method D3987-85) - See description in Section 3.1.2.

SPLP - The Synthetic Precipitation Leaching Procedure (SPLP) test (EPA Method 1312) is a single batch extraction method used to assess the leaching potential of a waste exposed to rainfall. The procedure is performed using the same equipment that is used to perform the TCLP and calls for the use of one of two leaching fluids depending on the location of the waste disposal site. A 20:1 L/S (m/m) ratio and an extraction time of 18 ± 2 hours are used in this test.

Technical Basis for Test

IDEM notes in the guidance document that TCLP is not a good test for determining leachable lead at a firing range because the amount and type of acids used in the test do not match those found naturally. On the other hand, the Indiana Neutral Leaching Method and the ASTM Water Leach Method, both of which employ a neutral leaching fluid (deionized water), would be the most appropriate tests to assess the leachability of lead at shooting ranges located over standing bodies of water. The size and buffering capacity of the standing water body and its matrix would cause the pH of rainfall to approach neutral.

Noting the acidic nature of rainfall in Indiana, IDEM indicates that the SPLP would be the appropriate test for most shooting ranges. Rainfall, with a pH range of 4.5 to 5, is the major source of water at most shooting ranges; therefore, the leaching fluids used in the SPLP would adequately simulate the acidity and types of acid found in rainfall. Use of a leaching fluid that has a pH of 4.2 ± 0.05 would effectively model a worst-case scenario of lead mobilized by acid rain, according to the guidance document.

3.1.7 North Dakota Department of Health Division of Waste Management - Guideline 11, Ash Utilization for Soil Stabilization, Filler Materials, and Other Engineering Uses

"Guideline 11" (NDDH, 2002), issued by the North Dakota Department of Health, addresses the beneficial reuse of coal-fired fly ash and bottom ash for engineering purposes including their use for soil stabilization and as filler material. Approval of beneficial reuse of ash is contingent upon demonstration by interested persons that the material will not adversely affect the environment. The guideline specifies information that should be included in a proposal for beneficial reuse of

ash. Along with information regarding source, quality, quantity and proposed use of ash, the guideline specifies the need for leach analysis of the ash and a laboratory simulation of the environmental properties of the proposed use. Leach analysis is to be performed using either: 1) a modified EPA SPLP, with a L/S ratio of 4:1, or 2) a modified ASTM D-3987 test, with a L/S ratio of 4:1.

According to the North Dakota Department of Health (NDDH), Division of Waste Management the 4:1 ratio was selected because it was believed to be more reflective of the state's semi-arid climate and probably mimics more accurately the interaction of rainfall with ash, which is probably the worst case scenario. In addition, experts in ash analysis have recommended the use of a 4:1 ratio. However, they have received and approved at least one proposal to use coal combustion material in feedlots that included data using a 20:1 SPLP and analyzed the leachate at time intervals of 18 hours, 30 days and 60 days.

It was noted that the TCLP would be inappropriate since most of the ash disposal and utilization (beneficial use) applications in North Dakota involve the interplay of ash with predominantly alkaline soils and groundwater. Rainfall, surface water and groundwater interaction with the material is the most significant factor in contaminant leachability.

No published comparisons are available, but data reviewed by the State so far seem to be similar to what their experience with landfill runoff and surface impacts suggest.

According to NDDH, the beneficial use applications they have approved include the use of ash as an admixture for a controlled flowable fill in engineered settings and for stabilization of sugar beet piling sites, road stabilization, and other stabilization projects. Stabilization of mine haul roads and other roads also has been studied. Use of bottom-ash, a sand-like product, for winter traction has been accepted in North Dakota and other states. Bottom ash also is being marketed as abrasives and sand blast medium (Tillotson, 2003).

Description of Test

Modified EPA SPLP – The Modified SPLP is similar to SW-846 Method 1312 (SPLP) described in Section 3.6 with the exception that a L/S ratio of 4:1 is used rather than 20:1 (m/m).

Modified ASTM D-3987 – The Modified ASTM D-3987 is similar to the ASTM D-3987 procedure described in Section 3.2 with the exception that a L/S ratio of 4:1 is used rather than 20:1 (v/m).

Technical Basis for Test

The authors have contacted the North Dakota Department of Health by phone and e-mail to obtain information on why the state selected the modified SPLP and ASTM D-3987. The information was not available as of the date of this report.

3.1.8 Texas Commission on Environmental Quality - Class 3 Waste Determination

Texas regulations under Section 335.507 of Title 30, Environmental Quality, specify conditions for classification of an industrial solid waste as a Class 3 waste. Class 3 wastes, which may include materials such as rock, brick, glass, dirt, plastics and rubber, are inert and essentially insoluble. A material is deemed "essentially insoluble" if the following conditions exist:

- The analysis of leachate obtained by the Seven-Day Distilled Water test indicate constituents are below the Maximum Contaminant Levels listed in Sec. 335.521 (adapted from 40 CFR Part 141, Subparts B and G, Maximum Contaminant Levels)
- Leachate obtained from TCLP analysis has no detectable levels of constituents listed in Sec. 335.509; and
- Analysis of a representative sample indicates no detectable levels of petroleum hydrocarbons (TPH).

Description of Test

Texas Seven-Day Distilled Water Leachate Test – This test is intended only for dry, solid wastes, i.e., waste materials without any free liquids. In this test, a 250-gram (dry weight) representative sample of a waste material is mixed with one liter of deionized or distilled water and mechanically stirred at a low speed for five minutes. The sample-water mixture is then allowed to stand for seven days. At the end of seven days, the solution is filtered and the filtered leachate is analyzed.

TCLP - See description given in Section 3.1.1.

Technical Basis for Test

Texas Seven-Day Distilled Water Leachate Test – According to the Texas Commission on Environmental Quality, the Seven-Day Distilled Water Leachate Test was selected to test those materials that are destined for disposal at sites other than landfills and, therefore, will not be exposed to acidic media. The Seven-Day Distilled Water Leachate Test is believed to simulate more accurately conditions in which the material is contacted with surface water and precipitation (Green, 2003).

3.1.9 New Jersey Administrative Code - Sanitary Landfill Environmental Performance Standards

The New Jersey Administrative Code (NJAC) 7:26-2A.6 establishes performance standards for the design and construction of sanitary landfills. For Class II sanitary landfills (i.e., accepts nonhazardous solid waste), performance is based on the waste type to be disposed of at the sanitary landfill. Historical data of the waste type to be disposed of at the sanitary landfill can be used to demonstrate the degradation and immobilization of the waste within the soil matrix under similar disposal conditions. In the absence of historical data, waste analysis must be performed

including extraction procedures for metals such as the TCLP and USEPA "Solid Waste Leaching Procedure SW 924".

Description of Tests

TCLP - See description given in Section 3.1.1.

Solid Waste Leaching Procedure SW 924 - Monofilled Waste Extraction Procedure (MWEP) -This procedure is a sequential batch extraction test developed to predict the composition of leachate produced from solid waste under field conditions. In this procedure, solid materials are crushed to pass a 9.5 mm sieve and are combined with reagent grade water as extraction fluid in a 10:1 L/S (m/m) ratio. The waste/water mixture is tumbled at room temperature for 24 hours. In addition to reagent water, the procedure can be conducted using process waters, ground water, or other fluids that occur at a site. Following extraction, the leachate is filtered and analyzed. The solid residue is returned to the extraction vessel and the leach process is conducted using fresh extraction fluid. Four leachings per sample are recommended, however, the test has been modified with the use of only two sequential batch extractions (USEPA, 1987).

Technical Basis for Test

The technical basis for the TCLP is given in Section 3.1.1.

The Monofilled Waste Extraction Procedure (MWEP) uses distilled or deionized water to evaluate leaching of a waste disposed in a monofill. In this situation, acidity of the leachate is determined by the characteristics of the waste itself, rather than the environment in which the waste might be buried. The justification for use of the method is that it may be a better predictor of leaching in a monofill scenario.

3.1.10 TARP Program Tier II Guidance Document for Beneficial Use Determination of Non-hazardous Materials

Under the Technology Acceptance and Reciprocity Partnership (TARP) program (http://www.dep.state.pa.us/dep/deputate/pollprev/techservices/tarp/), the states of Illinois, Massachusetts, New Jersey, New York, Pennsylvania, and Virginia have developed a Beneficial Use Determination (BUD) protocol that details the requirements to be met regarding the use of non-hazardous RCRA solid wastes as a valuable material. The TARP Tier II Guidance Document For Beneficial Use Determination of Non-Hazardous Materials (TARP, 2002) provides vendors and States with general guidance on testing and administrative procedures for obtaining comprehensive testing required to receive beneficial use determinations for a nonhazardous RCRA solid waste as a valuable material.

Information required for a beneficial use determination includes various technical, scientific, and engineering information and data including testing data to characterize the materials. "Standard testing" methods given in the guidance include various laboratory leaching tests, as described below.

Description of Tests

TCLP - See description given in Section 3.1.1.

SPLP - See description given in Section 3.1.6.

Dutch Total Availability Leaching Test (NEN 7341) - The availability test (NEN 7341) is an agitated extraction test performed at two controlled pH values, pH = 4 and pH = 7. In this test, a finely ground sample (< 125 µm) is leached at a 50:1 L/S (v/m) ratio for 3 hours.

Monolithic Inorganic Leach Test ASTM C 1308 - This test method provides a method for accelerating the leach rate of solidified waste to determine if the release is diffusion-controlled. This test method is applicable to any material that does not degrade, deform, or change leaching mechanism during the test. If diffusion is the dominant leaching mechanism, then results of this test can be used to model long-term releases from waste forms. Diffusion can be confirmed as the leaching mechanism through the use of a computerized mathematical model for diffusion from the finite cylinder. The leaching mechanism should be verified as diffusion-controlled by a means other than analysis of the leach test data. For this purpose, analysis of post-leaching concentration profiles within the solid waste form is recommended (ASTM, 2001a).

Dutch Tank Leach Test (NEN 7345) - In this test a monolithic specimen is subjected to leaching in a closed tank to evaluate surface area related release. The leaching fluid (demineralized water) is renewed after 8 hours and 1, 2, 4, 9, 16, 36, 64 days using a leaching fluid with a volume of five times the volume of the material to be tested (i.e., a L/S of 5:1, v/v). The results are expressed in mg/m² (ECN, 2003).

Column Leach Test Using ASTM D 4874 - ASTM D 4874 describes a procedure for generating aqueous leachate from a solid material using a column apparatus. The maximum particle size for the column procedure is 10 mm; particle size reduction is not recommended. This procedure uses reagent water in a continuous up-flow mode to leach a sample of a solid material.

Dutch Column Test (NEN 7343) - In the NEN 7343 column test, demineralized water, adjusted to pH 4, is passed upward through a column of ground waste (< 4 mm). Seven consecutive leachate fractions are collected, each representing an L/S ratio within the range of 0.1 to 10 L/Kg (v/m). Total test duration is approximately 21 days. The test is designed to simulate the leaching behavior of a waste in the short and medium term. However, the test does not account for aging effects and slow changes in mineral composition. In addition, there is no direct correlation between the test and field conditions since variables such as temperature, channeling, aging and contact time are not considered (NEN, 1995 and Van der Sloot, et al., 1997).

Neutral Water (ASTM 3987) - See description given in Section 3.1.2.

Technical Basis for Test

According to the Tier II Guidance (TARP, 2002), actual testing and other informational requirements are dependent upon the hazards associated with the material or processing of it.

The guidance further states that the analytical plan should describe the methods that will be used to test the samples collected to ensure the consistency and integrity of the testing to meet the data quality objectives.

According to staff in the New Jersey Department of Environmental Protection, Division of Solid and Hazardous Waste – Bureau of Resource Recovery and Technical Programs, Beneficial Use Projects, the suite of tests specified in the Tier II Guidance were selected by reputation, and in the case of the Dutch leaching tests, as part of an agreement with the government of the Netherlands (Patrijin, 2003).

3.1.11 Iowa - Beneficial Use Determinations: Solid By-Products as Resources and Alternative Cover Material.

Chapter 108 of Title VIII Solid Waste Management and Disposal Regulations for the state of Iowa establishes rules for approval of beneficial uses of solid by-products, including, but not limited to, coal combustion byproducts, foundry sand, and paper mill sludge. Section 108.6 (455B, 455D) specifies requirements to be met when solid by-products are being used beneficially as fill material. One of the requirements involves the use of the SPLP to measure the leaching characteristics of the solid by-product. The regulations require the SPLP results To be less than or equal to ten times the maximum contaminant levels (MCL) for drinking water for the material to be acceptable as fill material.

Iowa's beneficial use rules were finalized in late April of 2003 and so far only a limited number of BUD applications have been received. Most of the BUD applications received involve the use of coal combustion residues. The Iowa Department of Natural Resources indicated that the SPLP results they have reviewed have all been below the MCL standards. No field studies have been performed to validate the SPLP results (Myrom, 2003).

Description of Test

SPLP - See description in Section 3.1.6.

Technical Basis for Test

The State of Iowa selected the SPLP based on a recommendation from the U.S. Department of Energy related to the beneficial use of coal byproducts (Myrom, 2003). The SPLP models leaching in a monofill environment exposed to precipitation (acid rain). Iowa selected the SPLP the state regulators felt that the SPLP more accurately models the field conditions under which coal combustion byproducts would be beneficially used. Iowa also referenced the EPA Guide for Industrial Waste Management (EPA530-R-03-001) (USEPA, 2003b) for additional information on leaching tests.

3.1.12 Rhode Island and Providence Plantations Department of Environmental Management – Management of Dredge Materials

Rhode Island regulation # DEM-OWR-DR-02-03 establishes requirements for dredging operations in marine waters of the State of Rhode Island and for the management of dredged materials resulting from these activities. The regulation specifies that the material to be dredged be characterized and that all applications for dredging activities include a sediment sampling plan that outlines the area to be dredged, proposed depth of dredging, location of sampling points, sampling procedures and testing protocols. For upland disposal or beneficial use of dredged material, the regulation requires that the material be tested for grain size, PCBs, total petroleum hydrocarbons (TPH), total metals, and semivolatiles at a minimum. Additional testing may include leachability testing using either the TCLP or SPLP to determine compliance with GA Leachability Criteria (as defined in Table 2 in Section 8 of the Rhode Island Rules and Regulations for the Investigation and Remediation of Hazardous Material Releases) and chloride sampling in case of material dewatering. ("GA" refers to one of four classes of groundwater designated under Rhode Island regulations. GA Leachability Criteria refer to concentrations in leachate that are protective of groundwater designated to be suitable for public or private drinking water use without treatment.)

Description of Tests

TCLP - See description given in Section 3.1.1.

SPLP - See description given in Section 3.1.6.

Technical Basis for Tests

The authors have contacted the Rhode Island Department of Environmental Management by email to obtain information on why the state selected the TCLP and SPLP. The information was not available as of the date of this report.

3.1.13 Wisconsin Department of Natural Resources - Interim Guidance on the Use of Leaching Tests for Unsaturated Contaminated Soils to Determine Groundwater Contamination Potential

In 1997, the Wisconsin Department of Natural Resources issued an interim guidance document (WDNR, 1997) that addresses the use of leaching tests to establish site-specific residual contaminant levels (RCLs) according to NR 720.19 (4)(b)2, Wisconsin Administrative Code (i.e., procedure for determining soil cleanup standards specific to a site or facility). The document provides guidance on the appropriate application of leaching tests (i.e., to determine RCLs protective of groundwater for soil contaminated with heavy metals), sample collection and handling activities, and on the use of leaching test data to develop RCLs.

The interim guidance identifies the SPLP as the recommended procedure for determining the leaching potential of soil contaminants. The document also notes some limitations of the SPLP including loss of volatile compounds or residual non-aqueous phase liquids (NAPLs) during the

particle size reduction and filtration steps and underestimation of metal leachability due to the inability to reach equilibrium within the time allotted for the test.

Description of Test

SPLP - See description given in Section 3.1.6.

Technical Basis for Test

The guidance recommends the use of a leaching test to determine the potential for the remaining contaminants in soil at a waste site to desorb from the soil and contaminate groundwater to a level that may exceed the groundwater standards. The guidance specifically recommends use of EPA Method 1312, SPLP but notes limitations such as losses from volatilization, potential overestimation of leachability due to particle size reduction, potential underestimation of leachability due to a short test duration, and other analytical difficulties. The guidance also notes the limitation of using the TCLP. The guidance notes that the aggressive leaching fluid used in the TCLP was designed to simulate the conditions found in the core of a landfill. Thus, the TCLP test could underestimate the amount of contaminant leached by immobilizing constituents that would be mobile under normal soil conditions. The TCLP could overestimate the amount of contaminant mass by leaching constituents that would not be mobile under normal soil conditions. The TCLP is not recommended for use in determining site specific RCLs.

3.1.14 British Columbia Special Waste Regulation - Waste Management Act

The Province of British Columbia (Canada) Special Waste Regulation (BC Reg. 63/88) provides detailed provisions for the application, siting, operation, storage, and management for special waste facilities in British Columbia. This regulation specifies the use of the Leachate Extraction Procedure, also known as the British Columbia Special Waste Extraction Procedure (SWEP), to classify wastes as special (i.e., hazardous) waste (British Columbia Ministry of Water, Land and Air Protection, 1988).

The Modified Leachate Extraction Procedure under this same regulation is used to determine if a waste is prohibited from disposal in a secure landfill or a long-term storage facility.

Description of Test

Leachate Extraction Procedure - The British Columbia Leachate Extraction Procedure (also known as the Special Waste Extraction Procedure (SWEP)), is a single batch extraction that uses acetic acid as the leaching fluid, a 16:1 L/S mass ratio, and an extraction time of 24 hours. Variations of the method for extraction of mine wastes in British Columbia include the use of distilled water or 0.1 N hydrochloric acid as the extract fluid, a L/S mass ratio of 3:1, and an extraction time of 24 hours.

Modified Leachate Extraction Procedure - The Modified SWEP is identical to the Leachate Extraction Procedure except that reagent water (Type IV, ASTM D1193) is used as the leaching fluid instead of the acetic acid.

Technical Basis for Test

The SWEP is designed to provide information on the solubility and release potential of constituents from a waste. The test is operationally similar to the TCLP and serves a similar purpose in the British Columbia regulations. The Waste Management Act and regulations that require the use of the SWEP are currently under comprehensive review by the Ministry of Water, Land and Air Protection with the goal to provide more effective and efficient protection of human health and the environment.

3.1.15 Ontario Regulation 347 - General Waste Management Regulation

Ontario Regulation 347, Ontario's hazardous waste management regulation under the Ontario Environmental Protection Act, was amended in 2000 through Ontario Regulation 558/00 (Ontario Ministry of the Environment, 2000). The amendments included: 1) adopting the Toxicity Characteristic Leaching Procedure (TCLP); 2) adopting the "derived from" rule; 3) amending Schedules 1, 1.1, 2A, 2B, and 4 which list hazardous wastes and 4) administrative amendments. The TCLP replaced the Canadian Leachate Extraction Procedure (LEP) as the required test for identification of toxic wastes.

Description of Test

TCLP - See description given in Section 3.1.

Technical Basis for Test

The decision to replace the Canadian LEP with the TCLP was based in part on the results of a review of the appropriateness of the Canadian LEP procedure, as prescribed in Canada's Transportation of Dangerous Goods, by the Hazardous Waste Task Group (HWTG) of the Canadian Council of Ministries of the Environment. The HWTG review concluded that the TCLP was the most cost-efficient, cost-effective and realistic approach for the determination of the potential of toxic constituents to leach from a waste matrix into the environment. The HWTG recommended the adoption of the TCLP to measure the leachability of hazardous constituents in hazardous wastes and hazardous recyclable materials.

On the other hand, by replacing the Canadian LEP with the TCLP, Ontario sought to make its regulations more consistent with U.S. regulations, Alberta regulations (which adopted the TCLP in 1996) and future amendments to the Canadian federal regulations (Revised Regulations of Ontario, Regulation 347, 2000).

3.1.16 European Union - Criteria and Procedures for the Acceptance of Waste at Landfills

In 2002, the Council of the European Union issued a regulation that establishes criteria and procedures for acceptability of wastes at landfills (Council of the European Union, 2002). Waste acceptance criteria are specified for various types of landfills including inert waste landfills, non-

hazardous waste landfills, hazardous waste landfills, and for underground storage (e.g., isolation of waste within geologic barriers, cavities, or engineered structures). The regulation outlines a procedure for acceptance of waste at landfills that consists of three steps with associated testing requirements: basic characterization, compliance testing and on-site verification. Basic characterization constitutes a full characterization. Characterization testing focuses on understanding the long term behavior and parameters influencing leaching behavior; compliance testing is used for regulatory control once the basic leaching characteristics of a material have been established; and on-site verification testing is used as a quick control to verify that the material meets the specifications.

For basic characterization, leachability testing, if needed, is to be performed using a batch leaching test and/or percolation test and/or a pH dependence test. The draft CEN ("Committee European de Normalization") standard listed in the regulation for this type of testing is prEN 14405. For compliance testing, a batch leaching test is required and the recommended method is EN 12457/1-4.

Description of Tests

prEN 14405 Upflow Percolation Test - This test provides for sequential flushing of a column packed with granular (<4mm) material with acidified water at increasing liquid to solid ratios (L/S = 0.1-10, v/m). The column is eluted by pumping the leaching fluid from the bottom of the column to the top, minimizing the creation of channels and column plugging.

EN 12457/1-4, Compliance Test for Granular Waste Materials and Sludges - This test consists of four procedures:

- Part 1 One-stage batch test at a L/S ratio of 2 L/kg (v/m) for materials with high solid content and with particle size < 4 mm (with or without size reduction)
- Part 2 One-stage batch test at a L/S ratio of 10 L/kg (v/m) for materials with particle size < 4 mm (with or without size reduction)
- Part 3 Two-stage batch test at a L/S ratio of 2 L/kg (v/m) and 8 L/kg (v/m) for materials with high solid content and with particle size < 4 mm (with or without size reduction)
- Part 4 One-stage batch test at a L/S ratio of 10 L/kg (v/m) for materials with particle size < 10 mm (with or without size reduction)

Technical Basis for Tests

prEN 14405 – The upflow percolation test is intended to determine the rate of contaminant leaching as a function of L/S ratio, particularly at the low L/S ratios prevailing in disposal scenarios.

EN 12457/1-4, Compliance Test for Granular Waste Materials and Sludges is designed to assess leachability under mild extraction conditions for waste disposal or material reuse options. The

test provides a relative timeframe for contaminant release when compared with availability for leaching. Waste acceptance criteria for landfilling include limit values for leachability of metals, Cl and SO₄ published by the EA and EC.

3.1.17 Michigan Department of Environmental Quality - Alternate Soil Leaching Procedures

Subrule (a) of the Michigan Environmental Response Act (MERA) Administrative Rule 299.5711(2) requires the use of the TCLP to determine the potential impacts of soil contaminants on groundwater. However, the rule, in Subrule (b), allows the Department of Environmental Quality to approve other methods to be used in lieu of the TCLP if they are demonstrated to more accurately simulate site conditions. In 1995, DEQ issued Memorandum #12 (MDEQ, 1995) that identifies alternative leaching tests approved by DEQ for use in lieu of the TCLP. The tests listed in this memorandum were reviewed by DEQ and deemed acceptable for specific applications as listed below. The memorandum also indicates that proposals for the use of other methods and/other applications of the approved test may be considered by DEQ. **Table 3-1** summarizes the methods and appropriate uses.

TEST METHOD	EXTRACTION FLUIDS(S)	APPROPRIATE FOR:	INAPPROPRIATE FOR:
"Totals" methods	As per each analytical method	All, see MERA Operational Memo #6 for correct methods	
Toxicity Characteristic Leaching Procedure (TCLP) EPA Method 1311	Buffered Acetic Acid, pH 2.88 or 4.93	Metals, semi-volatiles, pesticides, PCBs, volatiles	Cyanide, sulfides, hexavalent chromium
Synthetic Precipitation Leaching Procedure (SPLP) EPA Method	Fluid #1: H2SO4 & HNO3 @ pH 4.20	Extraction Fluid #1: metals, semi-volatiles, pesticides, PCBs	
1312	Fluid #3: Reagent Water	Extraction Fluid #3: cyanide, sulfides, volatiles, hexavalent chromium	
ASTM D3987-85 (ASTM Neutral Leach)	Reagent Water	Semi-volatiles, pesticides, PCBs cyanide, sulfides, hex. chromium	Metals, volatiles
ASTM D5233-92 (ASTM Single Batch)	Buffered Acetic Acid, pH 2.88 or 4.93	Metals, semi-volatiles pesticides, PCBs	Volatiles, cyanide, sulfides, hexavalent chromium

Table 3-1: MDER Acceptable Leaching Methods

Description of Tests

TCLP - See description given in Section 3.1.1.

SPLP - See description given in Section 3.1.6.

ASTM D3987-85 - See description given in Section 3.1.2.

ASTM D5233-92, Standard Test Method for Single Batch Extraction Method for Wastes – This test (ASTM, 1992) is an agitated extraction procedure that employs one of two extraction fluids (buffered acetic acid, pH 2.88 or pH 4.93, depending on the acidity and buffering capacity of the solid waste). In this procedure, a water/solid waste mixture (20:1) is mechanically agitated for 18 hours using a device that rotates sample containers on a central axis at a rate of 29 rotations per minute. After rotation, the aqueous phase is separated from the solid material for analysis. Particle size reduction is not required. The test is applicable to the extraction of both organic and inorganic constituents, including mercury.

Technical Basis for Test

Michigan employs a risk-based clean-up standard in most beneficial use programs, whereby a material meets the inert (uncontaminated) standard if contaminants of interest are not detected at the method detection level, if they are detected below the state-wide default background concentration, or if the concentration meets the risk-based criteria.

Memorandum #12 (MDEQ, 1995) indicates that TCLP may be too aggressive to accurately simulate site conditions. MDEQ staff have also said that the TCLP is too aggressive to predict what actually happens to beneficial reuse of waste in the environment, and that the SPLP (pH=4.2 east of the Mississippi and 5.0 west of the Mississippi), seems to be a better predictor of mobility of contaminants. MDEQ indicated that there is limited information regarding actual field studies or comparisons. However, in their experience with unlined landfills for industrial wastes, the SPLP seemed to predict what was found in the groundwater. Michigan DEQ further indicated that as is the case with most states, they do not generally perform compliance at land application or reuse sites, so there has not been any validation of SPLP results in the field (Roskoskey, 2003).

According to Michigan DEQ, the tests identified in Memorandum #12 were selected because they were routinely used by national organizations (EPA and ASTM) to investigate leaching of compounds from wastes and soils. There were no comparisons made with other tests for the purpose of selecting one test(s) over others (Curtis, 2003).

See Section 3.1.1 for a description of the technical basis of the TCLP.

See Section 3.1.6 for a description of the technical basis of the SPLP.

See Section 3.1.2 for a description of the technical basis of ASTM D-3987.

ASTM D-5233 differs from ASTM D-3987 in one important aspect. ASTM D-5233 does modify the pH of the leaching process, in a manner intended to reflect the acid available from the leachate in a landfill where municipal and industrial wastes are co-disposed. Two leaching fluids

are provided, and selection is made based on acid neutralization capacity of the solid waste. The test is intended to generate leachate concentrations representative of the expected release under a co-disposal scenario.

3.1.18 New Jersey Department of Environmental Protection - Pollutant Discharge Elimination System, Requirements for Discharges to Ground Water, Permits for Disposal of Dredge Spoils

New Jersey Environmental Codified Regulations (Title 7, Department of Environmental Protection, Chapter 14A, Pollutant Discharge Elimination System, Subchapter 7, Requirements for Discharges to Ground Water (DGW)) establish requirements for obtaining a New Jersey Discharge Elimination System (NJPDES) discharge to groundwater permit. Permits are required for specific activities, pollution sources, or regulated units to include surface impoundments, spray irrigation, overland flow, infiltration/percolation lagoons, residual surface impoundments, injection wells and land disposal of dredge spoils. The regulation provides information on the general requirements for applications for NJPDES-DGW permit as well as specific additional requirements for each of the activities, pollution sources, or regulated units identified above.

For disposal of dredge spoils, additional requirements, as specified in Section 7:14A-7:15, include the submittal of a proposed dredge spoils disposal plan that outlines the engineering design and construction plan, operation/maintenance plan, a groundwater protection program and a closure/post closure care plan. As part of the groundwater protection program, a monitoring system is to be used in which leaching tests are employed to determine the maximum leachate concentration of the dredge spoils. The regulation at 7:14A-7.15(b)(3)(i) specifies that the maximum leachate concentration of the dredge spoils shall be determined by subjecting an adequate number of samples to leaching tests ... and "leaching tests shall be performed according to the methods described by the U.S. Army Corps of Engineers, Waterways Experiment Station (WES), or other test approved by the Department."

Description of Test

The Waterways Experiment Station Sequential Batch Leach Test (WES-SBLT) challenges sediment solids with successive aliquots of distilled-deionized water (L/S = 4:1, m/m) in an agitated system. After the aqueous and solid phases have reached steady-state, the phases are separated by centrifugation and filtration, and the leachate is analyzed for contaminants of concern. The solid phase is then reequilibrated with fresh distilled-deionized water, and the process of phase separation and leachate analysis is repeated. Each cycle in the WES-SBLT involves an equilibration step, a phase separation step, and a leachate analysis step. A table of solid phase and aqueous phase concentrations is developed from chemical analysis of the leachates, and these data are plotted to produce sorption isotherms. From the resorption isotherms, contaminant-specific equilibrium distribution coefficients are obtained. WES-SBLTs are conducted under nitrogen (anaerobically), and in the presence of air (aerobically).

Technical Basis for Test

The WES-SBLT was developed specifically to address the conditions found when dredge spoils (sediments) are disposed of in a confined facility. Tests are conducted anaerobically to simulate the conditions that prevail throughout most of a confined disposal facility (CDF) for dredged material, and aerobically to simulate the conditions that exist as a CDF dewaters. Testing has shown that the mobility of contaminants under anaerobic and aerobic conditions is substantially different.

Because there are no artificial adjustments of pH, the WES-SBLT allows the sediment biogeochemistry to adjust pH to values that are representative of field pH in anaerobic (saturated) and aerobic (unsaturated) zones.

3.1.19 North Carolina Department Of Environment and Natural Resources -Requirements for Beneficial Use of Coal Combustion By-Products

Sections .1700 - .1710, Chapter 13 of Title 15A of the North Carolina Code of Regulations establishes provisions for the siting, design, construction, operation and closure of projects that employ coal combustion by-products as structural fill material. The regulations specify that prior to the use of coal combustion by-products in structural fill projects, a TCLP analysis is to be performed on a representative sample of the material, to determine the concentrations of arsenic, barium, cadmium, lead, chromium, mercury, selenium and silver, at a minimum.

Description of Test

TCLP - See description given in Section 3.1.1.

Technical Basis for Test

The primary motivation for use of the TCLP to test coal combustion by-products prior to use as structural fill is to demonstrate that the material is not a RCRA hazardous waste (Barber, 2003). In North Carolina, as long as the ash tests non-hazardous, then it can be used for structural fill material. The coal combustion by-products regulations are the only specific rules that North Carolina has for a specific structural fill activity. The state has a statute for recovered materials, for handling other proposals that deal with beneficial reuse of a specific waste material, such as foundry sand, off-specification tile waste, or concrete product waste. When requesting a approval for the use of a specific product as a recovered material, the applicant can choose which testing method they wish to use; either TCLP or SPLP to demonstrate that any constituent leaching from the material will not exceed the Division of Water Quality groundwater standards set forth in part 2L of the regulation (Barber, 2003).

3.1.20 Nevada Division of Environmental Protection, Bureau of Mining Regulation and Reclamation - Guidance Document on Alternate Use of Mine Waste Solids

The Nevada Department of Conservation and Natural Resources (NDCNR), Division of Environmental Protection (NDEP), Bureau of Mining Regulation and Reclamation have

published a guidance document (NDCNR, 1996a) for the reuse or disposal of mine waste solids outside of containment. The guidance document specifies that a proposal be developed and approved prior to relocation of mine waste material. The proposal must include a detailed description of the proposed place and manner of use of the material. It also requires that the waste material be characterized for its potential to release contaminants to the environment. For spent heap leach ore or tailings the required testing includes the TCLP, the Meteoric Water Mobility Procedure (MWMP) (NDCNR, 1996b) and Acid Generation Potential - Acid Neutralization Potential testing (AGP-ANP). For waste rock, MWMP and AGP-ANP are required by the state for characterization.

Description of Tests

TCLP - See description given in Section 3.1.1.

Meteoric Water Mobility Procedure (MWMP) - The MWMP is a single-pass column leach test developed by the State of Nevada for use in determining the potential for waste rock, spent ore, and tailings to release contaminants to the environment. In this test a 5-kg waste material is crushed to particle sizes smaller than 5 cm, and loaded in an extraction column. Using a ratio of 1:1 sample to extraction fluid, deionized water (used as the extraction fluid) is passed through the column in a 24-hour period.

Acid Generation Potential / Acid Neutralization Potential testing (AGP-ANP) - Acid-Base Accounting (ABA) - The purpose of acid-base accounting (ABA) is to identify the potential for acid generation based on the balance between acid forming species and acid neutralizing species within the rock. Net neutralizing potential (NNP) is calculated by subtracting the acid generation potential (AGP) from the acid neutralization potential (ANP) of the rock (ANP – AGP = NNP). ABA is the most common basis for predicting post-mining water quality largely because of its simplicity

Technical Basis for Tests

The state guidance (NDCNR, 1996a) indicates that analytical results will be considered along with site-specific conditions to determine the potential of the proposed activity to degrade waters of the state.

The authors have contacted the Nevada Department of Conservation and Natural Resources NDEP to obtain information on why the state selected the various leaching tests. The information was not available as of the date of this report.

3.1.21 Summary

Of the States and Countries interviewed, the overwhelming majority have specified either the TCLP or the SPLP in their regulations and/or guidance on leaching determinations. As shown in **Table 3-2**, the only other test specified by more than one State or Country is ASTM D 3987. Interestingly, the primary difference between the TCLP, SPLP and ASTM D 3987 is the pH of the leaching fluid.

Leaching Test Method	State or Country Where Method Is Used in a Waste or
	Materials Management Program
TCLP	CA, MI, NC, NJ, NV, RI, TX, TARP, Ontario
SPLP	IN, IA, MI, ND (mod), RI, WI, TARP
ASTM D 3987	AR, IL, IN, MI, ND (mod)
ASTM D 5233	MI
ASTM C 1308	TARP
ASTM D 4874	TARP
SBLT	NJ
California Waste Extraction Test	CA
Indiana Neutral Leaching Method	IN
NEN 7341	TARP
NEN 7343	TARP
NEN 7345	TARP
Canadian Leachate Extraction Procedure	British Columbia
(LEP)	
MWMP	NC
AGP-ANP	NC
Texas 7-day Water Leachate Test	TX
EN 12457/1-4	EU
prEN 14405	EU

 Table 3-2.
 Leaching Test Methods Specified by States or Countries

Very little information was found justifying the selection of these test methods. **Table 3-3** provides contact information for the individuals that were interviewed.

State	Contact Name	Address	Phone	e-mail
Florida	Richard Tedder	Florida Department of Environmental Protection Solid Waste Management 2600 Blair Stone Road MS# 4565 Tallahassee, FL 32399	850-488- 0300	richard.tedder@dep.state.fl.us
Iowa	Jeff Myron	Energy & Waste Management Bureau Iowa Dept. of Natural Resources		
Massachusetts	Sean Griffin	Massachusetts Department of Environmental Protection, Bureau of Waste Prevention 1 Winter Street	617-338- 2255	sean.griffin@state.ma.us

 Table 3-3.
 State Contacts

State	Contact Name	Address	Phone	e-mail
		Boston, MA 02108		
Michigan	Duane Roskoskey, P.E.	Department of Environmental Quality Waste and Hazardous Materials Division PO Box 30241 Lansing, Michigan 48909	517-335- 4712	Roskoskd@Michigan.gov
Missouri	Scott Waltrip	Missouri Department of Natural Resources Solid Waste Management Program P.O. Box 176 Jefferson City, MO 65102	573-751- 5401	
New Jersey	Ravi Patrijin	New Jersey Department of Environmental Protection Division of Solid and Hazardous Waste	609-292- 0125	
North Dakota	Steven J. Tillotson	Environmental Health Section North Dakota Department of Health 1200 Missouri Avenue P. O. Box 5520 Bismark, ND 58506-5520	701-328- 5166	stillots@state.nd.us
Wyoming	Bob Doctor	Department of Environmental Quality 122 West 25th St, Herschler Building Cheyenne 82002	307-777- 7937	BDOCTO@state.wy.us

3.2 Description of Leaching Tests Available for Evaluating Potential Impacts to Water Quality in Situations Where Fill Material Is Imported

Based on the survey of commonly used leaching tests (Section 3.1), a set of leaching tests was identified for further research. As indicated in Section 2.3.4, commonly used laboratory leaching tests can be divided into two broad categories: (1) single extraction/batch tests carried out in the form of leaching tests on a single portion of material using a single portion of leaching fluid (with no renewal of leaching fluid), or (2) multiple extraction/flow-around and flow-through leaching tests. **Table 3-4** presents commonly used leaching tests by test category.

Single Extraction/Batch Leaching Tests	 ASTM D 3987 - Standard Test Method for Shake Extraction of Solid Waste with Water
	 ASTM D 6234 - Standard Method for Shake Extraction of Mining Waste by the SPLP
	 SPLP - Synthetic Precipitation Leaching Procedure
	 TCLP - Toxicity Characteristic Leaching Procedure
	 DRET - Dredge Elutriate Test
	 SET - Standard Elutriate Test
	 NEN 7341 - Availability Test
	 EN 12457/1-4 - Compliance Test for Granular Waste Materials and Sludges
Multiple	Sequential Batch Tests
Extraction/Flow-around and Flow-through	 ASTM D 4793 - Standard Test Method for Sequential Batch Extraction of Waste with Water
Leaching Tests	 ASTM D 5744 - Standard Test Method for Accelerated Weathering of Solid Materials Using a Modified Humidity Cell
	 SBLT - Sequential Batch Leachate Test
	Flow-around Test
	 NEN 7345 - Tank Leach Test
	Flow-through Tests
	 ASTM D 4874 - Standard Test Method for Leaching Solid Waste in a Column Apparatus
	 PCLT - Pancake Column Leach Test
	 NEN 7343 - Column Test
	 prEN 14405 - Upflow percolation test

Table 3-4. Commonly Used Leaching Tests

Sections 3.2.1 through 3.2.16 provide a description of each test, as well as a discussion of the conceptual basis of the test, major test variables, sample preparation methods, and information on the expected method precision (reproducibility) of the test. The format of this precision information varies from test to test, depending on the design of the data-gathering study, and available documentation. The following terms are used in the discussions of method precision:

- *Precision* the degree of mutual agreement among a series of individual measurements, values, or results.
- *Reproducibility* the variation in average measurements obtained when two or more people measure the same parts or items using the same measuring technique. Reproducibility is often expressed as the variability between two or more laboratories.

- *Repeatability* the variation in measurements obtained when one person or one laboratory measures a group of samples or repeated measurement on the same sample. Repeatability is often expressed as the variability within a single laboratory.
- *Standard deviation* a statistic used to measure the variation in a distribution. The relative standard deviation (RSD) is often times used, expressed as percent. %RSD is a dimensionless quantity used to measure the spread of the data relative to the size of the numbers. For a normal distribution, the %RSD is expressed as the standard deviation (s) divided by the sample mean (\bar{x}), multiplied by 100.

Section 3.2.17 identifies laboratories that can perform the tests and the estimated cost for performing the tests. Initial information sources for this section of the report included literature identified by Ecology in support of previous regulatory development efforts and existing information on leaching tests obtained from the U.S. Environmental Protection Agency (USEPA) and other government sources. Additional literature and information was obtained from library services, Internet searches, and contacts with standards-setting organizations.

A table comparing the major variables of the leaching tests (e.g., liquid to solid ratio, test duration, leaching fluid, etc.) is given in **Appendix B**.

3.2.1 ASTM D 3987 Standard Test Method for Shake Extraction of Solid Waste with Water

Method Description and Sample Preparation

ASTM D 3987 describes an agitated extraction method in which contaminants are extracted from a sample of waste material with a volume of Type IV reagent water (e.g., prepared by either distillation, ion exchange, reverse osmosis, or electrodialysis) equal to 20 times the weight of the solid phase. Thus, the test employs a 20:1 L/S ratio on a volume to mass (v/m) basis. ASTM D 3987 requires the analyst to determine the solids content of the sample (as percent solids), however, the entire sample is used to generate the leachate, regardless of solids content. A minimum of 70 grams of sample is recommended for the extraction. Starting with a sample size of at least 5,000 grams, the sample size is reduced to that amount needed for the test by quartering the sample received from the field on an impermeable sheet of glazed paper or oil cloth. The size of the sample particles is not reduced prior to testing. After an 18-hour (± 0.25 hours) agitation period, the extraction fluid/sample mixture is filtered. The filtrate is collected, preserved if needed, and analyzed for the constituents of interest.

ASTM D 3987 was first approved by the American Society for Testing and Materials (ASTM) in 1985, and re-approved in 1999. The standard is intended to provide a rapid mechanism for obtaining an extract of a solid waste for the purpose of estimating the release of non-volatile, inorganic constituents.

Conceptual Basis

The final pH of the leachate generated with ASTM D 3987 is intended to reflect the interaction of the leaching fluid with the buffering capacity of the waste. The test is not intended to provide a leachate that is representative of the actual leachate produced from a solid waste in the field, nor is it intended to produce extracts that will serve as the sole basis of engineering design (ASTM, 1999a). Information available from ASTM does not elucidate the basis of the 20:1 L/S ratio, however, the standard acknowledges that this L/S ratio may not be appropriate or adequate for all waste types.

No reproducibility data are available for ASTM D 3987. Neither the paper referenced in the standard ("Statistical Analysis and Description of Factors Affecting the ASTM Leaching Test") nor the contact person cited in the standard (Dr. Robert Paule, National Institute of Standards and Technology) could be located.

3.2.2 ASTM D 4793 Standard Test Method for Sequential Batch Extraction of Waste with Water

Method Description and Sample Preparation

ASTM D 4793 is a serial batch test that employs reagent water as the extraction fluid. The procedure calls for ten sequential extractions of the same sample of waste with water followed each time by separation of the aqueous phase for analysis. The final pH of the leachate is to reflect the interaction of the leaching fluid with the buffering capacity of the waste.

In this procedure, a 100-g sample (on a dry weight basis) is extracted with Type IV reagent water at a L/S ratio of 20 to 1 (v/m). The volume of water added to the sample is determined by correcting for the mass of moisture present in the sample. The sample/water mixture is constantly agitated for approximately 18 hours and then filtered using a pressure filtration device equipped with a 0.45 or 0.8-µm filter. The damp solid material is transferred back to the extraction vessel and re-extracted with a fresh volume of water. The procedure is performed ten times in sequence on the same sample to generate ten aqueous solutions. At least the first four extraction sequences must be conducted without interruption.

As with other ASTM methods, this procedure is not intended to provide a leachate that is representative of the actual leachate under field conditions, as it does not simulate site-specific conditions. The test may be used to estimate the release of inorganic constituents only under the specified test conditions.

Reproducibility

A collaborative study of ASTM D 4793 was conducted using a raw oil shale sample (ASTM, 1999b). Eight laboratories conducted extractions of the single sample in duplicate and analyzed the extracts generated in the first, third, fifth, seventh, and tenth extraction steps. Data generated by these analyses were used to calculate the total standard deviation, which includes both the extraction procedure and analytical errors. Standards containing high, medium, and low

concentrations of the elements of interest were also analyzed to determine analytical precision. The estimated standard deviation of the procedure (Ste) for each analyte was calculated using the equation $S_{te} = (S_{tt}^2 - S_{ta}^2)^{1/2}$ and is presented in **Table 3-5**.

				Element			
	Aluminum	Calcium	Copper	Iron	Magnesium	Nickel	Zinc
Extract 1							
\overline{x}_t	75.4	982.0	12.3	68.2	189.0	63.3	237.0
Ste	8.75	*	2.65	8.49	10.7	4.66	15.4
Extract 3							
\overline{x}_t	10.5	72.1	0.990	1.87	7.78	2.74	11.3
S _{te}	9.17	30.6	0.950	*	2.30	0.996	5.24
Extract 5							
\overline{x}_t	6.23	52.8	0.322	1.85	4.73	2.01	8.46
S _{te}	2.03	12.3	*	0.252	1.04	0.333	2.37
Extract 7							
\overline{x}_t	5.13	52.7	0.416	1.53	3.95	1.59	6.65
Ste	1.74	4.56	*	0.455	1.43	*	0.626
Extract 1)						
\overline{x}_t	1.46	62.3	0.444	1.56	2.72	1.36	6.71
S _{te}	0.406	21.6	*	0.125	0.556	*	1.88

Table 3-5. Total Mean (\bar{x}_t) and Estimated Standard Deviation of the Extraction Procedure (S_{te}) for Elements of Interest - ASTM D 4793

* The standard deviation of the extraction procedure (S_{te}) could not be determined in some cases since the analytical standard deviation (S_{ta}) was higher than the total standard deviation value (S_{tt}) . Also, for some elements in some extracts, it was not possible to use the analytical standard deviation to calculate Ste since the element concentration in the extract varied significantly from the element's closest concentration among the analytical standards. (S_{te} for a particular element is calculated using the S_{ta} for analysis of the analytical standard containing the concentration of the element closest to its concentration in the extract.)

The statistical analysis of the collaborative test data indicated that the estimated precision of the sequential batch extraction procedure varied with the concentration of each constituent of interest, but no specific trend could be identified due to the limited data available (ASTM, 1999b).

3.2.3 ASTM D 4874 Standard Test Method for Leaching Solid Waste in a Column Apparatus

Method Description and Sample Preparation

ASTM D 4874 describes a procedure for generating aqueous leachate from a solid material using a column apparatus, using dynamic partitioning. The procedure allows for selection of specific operating conditions for the column in order to meet the objectives of individual studies. The column procedure is intended for evaluation of the leachability of low concentrations of semi-volatile and nonvolatile organic compounds as well as inorganic constituents.

The sample packed into the column is collected representatively from 5000-g or three column volumes, whichever is larger. The method directs the analyst to adjust waste variables prior to column packing in order to simulate the state of the waste in the field. These variables include moisture content, density, curing and particle size distribution. The maximum particle size for the column procedure is 10-mm; particle size reduction is not recommended.

The column is eluted with reagent water in a continuous up-flow mode. Reagent water is delivered to the column using a pressurized reservoir vessel. After saturation of the column with reagent water according to the method outlined in ASTM D 2434 (Test Method for Permeability of Granular Soils), the effluent flow is set so that one complete void volume exchange rate is accomplished in approximately 24 hours. The method recommends that void volumes 1, 2, 4 and 8 be collected and analyzed. Effluent samples are stored under refrigeration and analyzed for parameters of interest.

ASTM D 4874 was first approved by ASTM in 1995, and re-approved in 2001.

Conceptual Basis

ASTM D 4874 can be used to evaluate the leachability of both semivolatile and nonvolatile organic compounds, as well inorganic constituents. However, as indicated in the procedure, it is not meant to be used to determine leachability of volatile compounds. In addition, the method is not intended to be used as the sole basis for engineering design of a disposal site or waste characterization based on its leaching characteristics (ASTM, 2001b). In addition, the procedure notes that it is inappropriate for the evaluation of materials that have sufficient aqueous solubility to impact the void volume of the packed column.

No information is available that elaborates on the testing duration specified in the test, although the test does acknowledge that other testing periods (measured either in elapsed time or number of void volumes collected) may be desirable. While no documentation could be found discussing the rationale for eluting the column from the bottom to the top ("up-flow"), this is generally believed to minimize plugging of the column due to movement of small particles ("fines"), as well as channeling of the packed column.

Reproducibility

A three-laboratory study using fly ash, and involving triplicate columns at each laboratory, was conducted to determine the precision of this method for inorganic materials. In this study, the precision identified was a measure of the ability of the method to generate effluents of similar quality from a single waste material analyzed at different laboratories, or obtained from multiple columns at a single laboratory. The pooled within-interlaboratory relative standard deviation for metals data averaged 33%. The overall interlaboratory single test reproducibility in terms of standard deviation was 54% (ASTM, 2001b).

3.2.4 ASTM D 5744 Standard Test Method for Accelerated Weathering of Solid Materials Using a Modified Humidity Cell

Method Description and Sample Preparation

ASTM D 5744 describes a procedure that accelerates the natural weathering rate of a solid material by controlling exposure of the solid material to temperature, volume and application rate of both water and oxygen, and generates a leachate for the quantification of diagnostic weathering products. This method is only applicable to non-volatile inorganic constituents. It may not be suitable for testing materials containing plastics, polymers, or refined materials.

In this procedure, bulk samples are air- or oven-dried, and screened through a 6.3-mm screen prior to subsampling. Oversized material is crushed so that 100% of the material passes the screen. The procedure calls for bulk samples to be mixed and divided to obtain a representative "test unit". The test unit is divided into nominal 1-kg test specimens, one of which is selected at random for use in the accelerated weathering test method. This 1000-g solid sample is then leached once a week, for a minimum of 20 weeks, using a fixed volume of de-ionized water. In each of the 20 weeks of testing, the sample is subjected to a three-day dry-air period and a three-day wet-air period, followed by the water leach on the seventh day. The weekly water leach can be applied drop-wise (drip trickle) or as a flooded leach (by pouring water down the sides of the cell wall until the sample is flooded). A 500-mL or 1-L volume (yielding a L/S ratio of 0.5:1 or 1:1, v/m) of deionized water may be used, depending on the weekly pore volume desired or the quantity of solution needed for analysis. The test is conducted using a cylindrical humidity cell, and multiple cells can be arranged in parallel to allow the testing of different solid materials simultaneously.

ASTM D 5744 was first approved by ASTM in 1996, and re-approved in 2001.

Conceptual Basis

ASTM D 5744 is intended for use to meet kinetic testing regulatory requirements for mining wastes and ores, and is a modification of an accelerated weathering test developed originally for mining wastes. Accelerated material weathering in this test method is accomplished by controlling exposure of the solid material to temperature, volume and application rate of both water and oxygen. The procedure has been demonstrated to accelerate the weathering rates of metal-mine waste rock by at least one order of magnitude greater than observed field rates.

The test is not intended to simulate site-specific leaching conditions, nor is it intended to produce leachates that will serve as the sole basis of engineering design (ASTM, 2001c).

The weekly leachates can be used to determine the sample's tendency to produce acidic, alkaline, or neutral effluent. They may also be analyzed to determine pH, Eh, conductivity and dissolved gaseous oxygen and carbon dioxide, as well as the concentrations of cations, anions and metals.

Reproducibility

No precision data are currently available for this method, as studies are undergoing to determine the precision for measuring the rate of accelerated weathering using waste-rock samples (ASTM, 2001c).

3.2.5 ASTM D 6234 Standard Method for Shake Extraction of Mining Waste by the Synthetic Precipitation Leaching Procedure

Method Description and Sample Preparation

ASTM D 6234 describes a procedure obtaining an extract of a mining waste that is at least 80 percent dry solids, for the purpose of estimating the release of non-volatile, inorganic constituents. The testing conditions specified in the standard conform to the specifications of the Synthetic Precipitation Leaching Procedure (SPLP, SW-846 Method 1312).

The test is a single-batch, agitated extraction method in which contaminants are extracted from a sample of mining waste of known weight (at least 100-g) with a volume of acidic extraction fluid of specified composition, for approximately 18 hours at a 20:1 (mass to mass (m/m)) liquid to solid ratio. The pH of the extraction fluid is dictated by the expected pH of the precipitation in the geographic region in which the waste is to be disposed. Prior to extraction, the sample is sieved using a 9.5-mm sieve, and oversized material is reduced in size by crushing. After the extraction, the waste/leaching solution is filtered using a pressure filtration device equipped with a 0.45 or 0.8-µm filter. The filtrate is collected, preserved if needed, and analyzed for the constituents of interest.

ASTM D 6234 was first approved by ASTM in 1998, and re-approved in 2002.

Conceptual Basis

The pH of the leaching fluid used in the test is intended to reflect the pH of acidic precipitation in the geographic region in which the waste is to be disposed. The procedure is not intended to produce a leachate that is representative of the actual leachate that would occur in the field. In addition, the applicability of this procedure to extraction of organic compounds, volatile matter and biological active samples has not been determined. The standard is based specifically on the Synthetic Precipitation Leaching Procedure (SPLP, SW-846 Method 1312) (ASTM, 2002).

Reproducibility

An interlaboratory study was conducted to determine the precision of the method on two wastes from six data sets. The repeatability (within-laboratory) was measured using six analytical standards, in triplicate. The reproducibility (between-laboratory) was measured on the mining waste using a pH 5.0 + 0.05 synthetic precipitation leaching fluid. The results are summarized in **Table 3-6**.

Analyte	Mean concentration (µm/mL)	Repeatabilty, r (µg/mL)	Reproducibility, R (µg/mL)
Barium	1.9	0.6	0.8
Calcium	1815	389	666
Lead	636	8	22
Magnesium	107	20	34
Manganese	36.7	5.6	15.4
Silicon	55	6	58
Zinc	297	87	98

 Table 3-6. ASTM D 6234 – Reproducibility (ASTM 2002)

3.2.6 Toxicity Characteristic Leaching Procedure

Method Description and Sample Preparation

The Toxicity Characteristic Leaching Procedure (TCLP) was adopted by the USEPA in 1990 to replace the Extraction Procedure (EP) as the USEPA regulatory method for classifying wastes as hazardous based on toxicity. To make a hazardous waste determination, concentrations of specific contaminants in the TCLP extract are compared to thresholds levels established for the Toxicity Characteristic (TC) constituents in 40 CFR §261.24 including eight metals, 20 volatile organics, 16 semi-volatile organics, and two pesticides. If the TCLP extract contains any one of the TC constituents in an amount equal to or exceeding the concentrations specified, then the waste possesses the characteristic of toxicity and is a hazardous waste (USEPA, 1990c).

The TCLP, described in SW-846 (USEPA, 1996a) Method 1311, is an agitated extraction method in which contaminants are extracted from a size-reduced sample of waste material with an appropriate extraction fluid equal to 20 times the weight of the solid phase (i.e., a 20:1 L/S (m/m)). Prior to extraction, particle size reduction is required, unless the solid has a surface area per gram of material equal to or greater than 3.1 cm, or is smaller than 1 cm in its 2 narrowest dimensions (i.e., is capable of passing through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, the sample must be prepared for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. If the solids are prepared for organic volatiles extraction, special precautions must be taken as described in the method.

The extraction fluid used for the extraction depends on the alkalinity of the waste material. Very alkaline waste materials are leached with a fixed amount of acetic acid without buffering the system (pH 2.88 ± 0.05), while other waste materials are leached with acetic acid buffered at pH 4.93 ± 0.05 with 1-N sodium hydroxide. After an 18-hour (± 2 hours) agitation period, the extraction fluid/sample mixture is filtered using a glass fiber filter. The filtrate is collected, preserved if needed, and analyzed for the constituents of interest.

The TCLP calls for a percent solids determination prior to extraction of the sample. If the percent solids is found to be less than 0.5%, the liquid phase after filtration is defined as the TCLP extract, which can be analyzed directly. For wastes containing greater than or equal to 0.5% solids, the liquid and solid portions are separated, with the solid phase undergoing particle size reduction, if necessary. An amount of extraction fluid equal to 20 times the weight of the solid material is used to extract the waste. Following extraction, the sample is filtered, combined with the initial liquid portion (if miscible), preserved and analyzed. The TCLP procedure prescribes the use of a zero-head extractor (ZHE) for sample extraction when volatile compounds are of interest.

Conceptual Basis

The TCLP was designed to simulate the leaching a waste will undergo if co-disposed with municipal solid waste (MSW) in a sanitary landfill. The co-disposal scenario was selected as the most reasonable worst-case mismanagement scenario for industrial wastes, and thus the TCLP was developed to incorporate key factors that affect waste leachability in an MSW landfill. Under a co-disposal scenario, infiltrating precipitation combined with water-soluble products of MSW biodegration act as the leaching fluid. In the TCLP, this phenomenon is represented by a sodium acetate buffer solution with a pH of 4.93 (or pH 2.9 for highly alkaline wastes). In selecting the TCLP leaching fluid, it was assumed that the concentration of acetic acid and acetate in the TCLP extract would approximate concentrations of volatile fatty acids likely to occur in actual landfill leachates during the acid generation phase of landfill decomposition. Acetic acid was selected because it was considered to be the most prevalent acid found in MSW leachates (see 45 FR 33112, May 19, 1980).

As noted previously, the TCLP uses a 20:1 liquid-to-solid ratio for waste extraction. EPA adopted the 20:1 liquid to solid ratio for its apparent suitability for simulating a mismanagement scenario. The 20:1 TCLP methodology was determined using a combination of factors related to climate, waste characteristics, and disposal-practice characteristics. The TCLP methodology assumes a 3-meter landfill depth, 100 cm annual rainfall, 5 percent co-disposal with municipal waste, 100 percent rain percolation through the landfill, 1 gm/cm3 waste density, and three years of leaching (USEPA, 1989a). Operational considerations also played a role in selection of the 20:1 liquid to solid ratio employed in the TCLP since the use of low L/S presents practical difficulties in separating the leachate from the solid fraction following a leaching test.

While the TCLP was developed to predict leaching in a MSW landfill, in practice regulators and industry have used the TCLP in applications that depart from the original basic assumptions. The use of TCLP in this manner may be inappropriate as the TCLP is not designed to address all

scenarios and it may over- or under-predict leaching potential in scenarios other than the one for which it was originally intended (USEPA, 1999a).

Reproducibility

In SW-846 Method 1311, EPA notes that "many TCLP precision (reproducibility) studies have been performed, and have shown that, in general, the precision of the TCLP is comparable to or exceeds that of the EP toxicity test and that method precision is adequate." EPA notes that variability introduced during sampling contributes significantly to overall variability of the procedure.

The method provides precision data (including the mean, standard deviation, and percent relative standard deviation (%RSD) by constituent) for TCLP analysis of metals, semi-volatile organics, and volatile organic compounds (VOCs). These data are summarized in **Table 3-7** below.

Waste Types Evaluated	Constituent Group	%RSD Range	Mean % RSD
Ammonia Lime Still Bottoms, API Sludge/Electroplating Waste (EW) Mixture, and Fossil Fuel Fly Ash	Metals	17 - 118	74**
Ammonia Lime Still Bottoms and API sludge/EW Mixture	Semi-Volatiles	1 - 33	12*
Ammonia Lime Still Bottoms, API sludge/EW Mixture, and Fossil Fuel Fly Ash	Semi-Volatiles	0 – 164	54**
Mine Tailings, Ammonia Lime Still Bottoms	VOCs	17 - 144	75

Table 3-7. Summary of Precision Data Given in SW-846 Method 1311 TCLP

*Results are from a single laboratory study.

**Results are from a multi-laboratory study.

As stated in the method, the results of a multi-laboratory study indicate that a single analysis of a waste may not be adequate for waste characterization and identification requirements. For semi-volatile organic compounds, the single laboratory precision was excellent with greater than 90 percent of the results exhibiting an RSD less than 25 percent. Over 85 percent of all individual compounds in the multi-laboratory study fell in the RSD range of 20 - 120 percent. Both studies concluded that the TCLP provides adequate precision. It was also determined that the high acetate content of the extraction fluid did not present problems (i.e., column degradation of the gas chromatograph) for the analytical conditions used.

A collaborative study of the use of the ZHE for evaluation of VOCs found that precision results for VOCs tend to occur over a considerable range, similar to that found for metals.

3.2.7 Synthetic Precipitation Leaching Procedure (SPLP)

Method Description and Sample Preparation

The Synthetic Precipitation Leaching Procedure (SPLP) is an agitated extraction that is used to provide information on the mobility (leachability) of organic and inorganic constituents from liquids, soils and wastes. This procedure is similar to the TCLP but instead of the acetic acid mixture used with the TCLP to simulate landfill leachate, nitric and sulfuric acids are utilized to simulate the acid rain resulting from airborne nitric and sulfuric oxides. The SPLP, which is described in SW-846 Method 1312, calls for the use of one of two leaching fluids depending on the location of the sample site in the case of soil samples. For sites east of the Mississippi River, the leaching fluid is a solution of sulfuric acid and nitric acid in water with a pH of 4.2. For sites west of the Mississippi River, a solution of sulfuric acid and nitric acid in reagent water, pH 5.0, is used as the leaching fluid. If the sample is a waste or wastewater, the extraction fluid employed is the pH 4.2 solution.

As in the TCLP, a percent solids determination is performed prior to sample extraction. For samples with less than 0.5% solids, the liquid obtained after filtration through a 0.6 to 0.8 μ m glass fiber filter, is defined as the SPLP extract and analyzed for constituents of interests without extraction. If the waste contains greater than or equal to 0.5% solids, the solid portion is separated from the liquid, subjected to particle size reduction, if necessary, as described in the TCLP, and extracted with the appropriate extraction fluid.

For sample extraction, a 20:1 liquid to solid ratio (m/m basis) and an 18-hr agitation period are employed. When testing for volatile compounds, the ZHE is used for the extraction. Following extraction, the sample is filtered through a glass fiber filter. The filtrate is collected, combined with the initial liquid portion (if miscible), preserved if needed, and analyzed (USEPA, 1996a).

Conceptual Basis

The SPLP was designed to assess the leaching potential of contaminated soils and wastes disposed of in a monofill when exposed to rainfall. As stated above, the SPLP is similar to the TCLP with the exception that the acetic acid buffer extraction fluid has been replaced by a dilute nitric acid/sulfuric acid solution. This solution was selected to represent the acidity of rainfall in the area where the monofill is located.

The nitric acid/sulfuric acid solution was deemed more appropriate than the TCLP extraction fluid for extraction of contaminated soils and wastes destined for disposal in a monofill environment since the sanitary landfill co-disposal scenario is not applicable in these situations. It was believed that use of the acetic acid leaching fluid could selectively solubilize contaminants (i.e., lead) that would, otherwise, not leach under actual field conditions, leading to the soil or waste being incorrectly classified as hazardous when no such leaching would be expected to occur in the environment (USEPA, 1989b).

Reproducibility

SW-846 Method 1312 provides precision data for the SPLP extraction of metals, semivolatile organics, and volatile organic compounds. These data were obtained from a single laboratory precision evaluation of Method 1312 for soils only and are summarized in **Table 3-8**. In this study, conducted in two phases, several soil types were fortified with semi-volatiles, metal salts and volatile organic compounds, and then leached in replicates of 3 or 6 analyses to determine method precision.

Soil Samples Evaluated	Constituent Group	% RSD Range*	Mean % RSD*
Eastern soil, Western soil	Semi-volatiles	5 - 173	29
Eastern soil, Western soil	Metals	2 - 71	32
Western soil (Superfund site)	Volatiles	3 - 68	15
Eastern soil (Superfund site)	Volatiles	2 - 73	24
Western soil/sludge-1	Volatiles	11 - 120	41
Western soil/sludge-2	Volatiles	5 - 115	33

Table 3-8. Summary of Precision Data Given in SW-846 Method 1312, SPLP	Table 3-8. Summary	of Precision	Data Given	in SW-846 Metho	d 1312, SPLP
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* Results from a single laboratory study.

In the first phase of the study, method precision was evaluated by measuring the repeatability of recovery of 14 semi-volatile compounds and two metals: lead and cadmium. As stated in the method, for semi-volatiles and metals the concentrations of contaminants leached from both types of soils were reproducible as indicated by the moderate relative standard deviations (RSDs), which averaged 29% for all the compounds and elements analyzed. The RSDs of the recoveries of most compounds were less than 15 percent; only four of the 16 compounds had large RSDs (above 15 %), which was attributed to analytical difficulties due to constituent volatility or reactivity (USEPA, 1989b).

For volatiles, method precision was determined by measuring the repeatability of recovery of 27 volatile organic compounds using four different types of soil. For the western and eastern soils, 85 percent and 65 percent of analytes, respectively, had RSDs less than 20 percent. Only four analytes had RSDs greater than 50 percent, due to significant analytical difficulties during the purge-and-trap GC/MS analysis. In general, replicate leachings of the western soil/sludge mixtures showed lower precision than the leachates from the Superfund soils.

3.2.8 Standard Elutriate Test (SET)

Method Description and Sample Preparation

The Standard Elutriate Test (SET) is a simple batch test in which an undisturbed sediment sample from a dredging site is added to dredging-site water at a 4:1 liquid to solid ratio (v/v). The water-sample solution is mechanically agitated for 30 minutes and then allowed to settle for one hour and filtered to remove particulates prior to analysis of an aliquot of the supernatant (Havis, 1988).

Although SET has also been used to predict contaminant concentrations at the point of dredging, experiments have shown that SET overestimates the expected release for some contaminants in this application (DiGiano, et al., 1995). The SET was developed in the 1970s, and has no numerical designation.

Conceptual Basis

SET is used to predict contaminant release from dredged material at the point of disposal (openwater disposal). The SET procedure, in a modified version, has also been used to predict the release of contaminants during disposal in a confined disposal facility (CDF). This modified elutriate test (MET) employs a liquid to solid ratio of 17:1 (v/v), which is believed to be more representative of conditions at CDFs.

Reproducibility

In the past, the SET was required by the Army Corps of Engineers as a standard test for dredged material bioassays. The elutriate preparations were intended primarily for toxicity tests and not for chemical analyses, so that little, if any, chemical data are available (Carr, 2003).

3.2.9 Dredge Elutriate Test (DRET)

Method Description and Sample Preparation

The Dredge Elutriate Test (DRET) is a modification of the SET procedure, used to predict the release of contaminants at the point of dredging. The major difference in conditions at the point of disposal and the point of dredging, which the procedures are designed to reflect, is related to the total suspended solids (TSS) concentrations. In the DRET, the maximum initial TSS is 10 g/L, which translates to a ratio of water to sediment of 226:1 (v/v). The DRET has no numerical designation.

Conceptual Basis

In the development of the DRET, an evaluation was conducted to determine how well the DRET simulates field conditions. In this study, comparison of DRET results and field data showed that DRET produce a TSS similar to those encountered in the field when using two types of dredgeheads. Also, DRET was found to be a reasonable predictor of soluble PCB concentration

for these two types of dredgeheads giving a conservative estimate of soluble concentrations. During evaluation of the DRET protocol it was determined that an initial TSS of 10 g/L was adequate, even though this TSS was well above field observations, and should give a conservative prediction of soluble contaminants. An aeration time of one hour and settling time of one hour were chosen since it was determined that increasing aeration time did not increase release of soluble PCB. A settling time of one hour was demonstrated to be sufficient to remove from suspension all but the particles less than 10 mm in diameter (DiGiano, et al., 1995).

Reproducibility

In the past, the DRET was required by the Army Corps of Engineers as a standard test for dredged material bioassays. The elutriate preparations were intended primarily for toxicity tests and not for chemical analyses, so that little, if any, chemical data are available (Personal communication, Scott Carr, USGS/TAMU).

3.2.10 Pancake Column Leach Test (PCLT)

Method Description and Sample Preparation

In the Pancake Column Leaching Test (PCLT), formerly known as the thin-layer column leach test, water is passed at a constant rate through a sample of the dredged material loaded in a column leach apparatus. After passing through the dredged material, the water is analyzed for contaminant concentrations.

Conceptual Basis

The pancake or thin-layer column leach test is designed to simulate contaminant leaching in a CDF. It may be used to confirm the results of batch testing (SBLT) and can also be used with mass transport modeling to estimate the long-term water quality impact and contaminant flux in a confined disposal site (Brannon, et al., 1994).

Reproducibility

No reproducibility data have been found.

3.2.11 Sequential Batch Leachate Test (SBLT)

Method Description and Sample Preparation

The Sequential Batch Leach Test (SBLT) is an agitated extraction method where sediment samples are extracted with deionized water under anaerobic conditions. Samples and deionized water are loaded into centrifuge tubes at a liquid to solid ratio of 4:1 using a glove box with a nitrogen atmosphere to maintain anaerobic conditions. The tubes are placed in a rotary tumbler and turned at 40 rpm for 24 hours. After filtration, the sediment is returned to the centrifuge tube and fresh deionized water is added to the centrifuge tube at a L/S ratio of 4:1 (m/m). The

sediment-water mixture is again tumbled and filtered. The tumble/filter/replenish procedure is repeated a minimum of four times.

The SBLT has been recommended for leachate testing of freshwater sediments (Brannon, et al., 1994). It is generally used to provide an estimate of the maximum contaminant leachate concentration, but can also be used to calculate contaminant distribution coefficients for sediment types that can be employed to predict leachate concentrations within different areas of a CDF.

Conceptual Basis

A liquid to solid ratio of 4:1 was selected for the SBLT based on experiments that indicated this is the highest ratio that can be used without solids concentration effects becoming important. Experiments also demonstrated that a contact time of 24 hours is sufficient to achieve steady-state conditions for organics in leachate. In the SBLT, leaching takes place under anaerobic conditions to simulate leaching in the saturated-anaerobic zone of a CDF.

Reproducibility

No reproducibility data have been found.

3.2.12 NEN 7341 Availability Test (The Netherlands)

Method Description and Sample Preparation

The availability test (NEN 7341), developed in the Netherlands, is an agitated extraction test performed at two controlled pH values, pH = 4 and pH = 7. In this test, a finely ground sample (< 125 mm) is extracted twice in succession at a L/S ratio of 50:1 (L/kg, v/m). In the first phase, leaching is continued for 3 hours at constant pH 7. In the second stage, the material is leached for 3 hours at constant pH 4, or a lower pH if the sample drives the pH lower. The leachates are filtered, combined, and analyzed. The results of this test give the maximum quantity of each inorganic component that can leach. The test data can also be used to calculate the acid-neutralizing capacity of the material (<u>http://www.leaching.net/DaviWB/Pagina3.html</u>).

Conceptual Basis

By grinding the material finely (maximizing surface area), and using a large L/S ratio, NEN 7341 is intended to determine the maximum leachable quantity of a given component over the very long term. Solubility constraints are minimized in the test by using a high L/S ratio and very small particle size (van der Sloot, et. al., 1994).

Reproducibility

No information on method reproducibility was identified for inclusion in this report.

3.2.13 NEN 7343 Column Leach Test (The Netherlands)

Method Description and Sample Preparation

The Column Leach Test (NEN 7343) describes a procedure for determining leachability of inorganic components from solid earthy and stony materials and wastes as a function of the value of L/S. The method involves passing demineralized and acidified water (HNO₃, pH = 4) upward through a vertical column of particulate material (4 mm or smaller). Seven consecutive leachate fractions are collected, corresponding to a liquid-to-solid ratio range of 0.1 to 10 L/kg (v/m). The total test duration is approximately 21 days. Very slow changes in mineral composition are not addressed by this test; and NEN 7343 test conditions do not correlate on a one-to-one basis with field conditions because several variables, e.g., temperature, channeling, aging, and degree and length of contact, must be considered (<u>http://www.leaching.net/DaviWB/Pagina3.html</u>).

Conceptual Basis

The test is intended to simulate the leaching behavior of inorganic components from powdered and granular materials in an aerobic environment. By running the test at a range of L/S ratios, the leachability of a waste material can be estimated in the short, medium, and long term by relating contaminant release, expressed as mg/kg leached, to the L/S ratio (Sorini, 1996). The column test is designed to reflect the dynamic aspects of leaching that may occur due to slow transformation processes, sequential release and depletion of species. (van der Sloot, et al., 1994)

Reproducibility

A round-robin study, comprising ten laboratories and three waste types, was conducted to evaluate the precision of NEN 7343. The wastes studied were incinerator fly ash, E-bottom ash (bottom ash suitable for use in an environmentally-friendly application), and incinerator bottom ash, covering all grain size classes to which the test applies. Care was taken to exclude inhomogeneous components of the wastes, and to minimize imprecision due to sample preparation. Two estimates of precision were calculated: repeatability (r), variation within a single laboratory, and reproducibility (R), variation between different laboratories. These data are summarized in **Table 3-9**.

Table 3-9. Reproducibility and Repeatability - NEN 7343

	Median Value (relative standard deviation)	Range (relative standard deviation)
r- repeatability limit (within-laboratory)	11%	5% - 25%
<i>R</i> - reproducibility limit (between laboratories)	21%	6% - 29%

3.2.14 NEN 7345 Tank Leach Test (The Netherlands)

Method Description and Sample Preparation

The Dutch Tank Leach Test (NEN 7345) is an immersion test used to determine the leachability of inorganic constituents from building materials, monolithic waste and stabilized waste materials. Samples (minimum size =40 mm) are supported or suspended in a tank to allow contact with de-mineralized water (pH = 4) leaching fluid along all sides. The tank is filled with water at a volume of five times the volume of the material to be tested (L/S ratio = 5:1, v/v). The liquid is changed and analyzed at pre-set intervals (8 hours and 1, 2, 4, 9, 16, 36, 64 days). The analysis of the extracts indicates the controlling mechanism: dissolution, erosion or diffusion. Multiple, simultaneous mechanisms are possible (http://www.leaching.net/DaviWB/Pagina3.html).

Conceptual Basis

The Dutch Tank Leach Test is a flow-around test that provides a mechanism for evaluation of surface area-related (diffusion) release.

Reproducibility

Information on reproducibility was not available at the time this report was prepared.

3.2.15 prEN 14405 (2002) Upflow Percolation Test

Method Description and Sample Preparation

The Upflow Percolation Test describes a column packed with granular (<4mm) material, eluted sequentially with acidified water at increasing L/S ratios (L/S = 0.1 to 10). Similar to the procedure in ASTM D 4874 and NEN 7343, the column is eluted by pumping leaching fluid from the bottom of the column to the top, in order to minimize the creation of channels and column plugging and to provide better control of flow rate.

Conceptual Basis

The Upflow Percolation Test is intended to determine the rate of contaminant leaching as a function of liquid to solid ratio, particularly at the low L/S ratios prevailing in disposal scenarios.

Reproducibility

No reproducibility data have been found.

3.2.16 EN 12457 Compliance Test for Granular Waste Materials.

Method Description and Sample Preparation

EN 12457 describes an agitated leaching procedure, using deionized water and granular materials less than 4-mm in diameter. Because the pH of the leaching fluid is not controlled, the final pH of the leachate is controlled by the test sample. The test consists of four procedures, depending on the characteristics of the waste to be tested. A waste matrix may be leached by more than one procedure:

Part 1 - One-stage batch test at a liquid to solid ratio of 2 L/kg (v/m) for materials with high solid content and with particle size < 4 mm (with or without size reduction)

Part 2 - One-stage batch test at a liquid to solid ratio of 10 L/kg (v/m) for materials with particle size below 4 mm (with or without size reduction)

Part 3 - Two-stage batch test at a liquid to solid ratio of 2 L/kg and 8 L/kg (v/m) for materials with high solid content and with particle size below 4 mm (with or without size reduction)

Part 4 - One-stage batch test at a liquid to solid ratio of 10 L/kg (v/m) for materials with particle size below 10 mm (with or without size reduction)

The standard is based on the assumption that equilibrium or near equilibrium is achieved between the liquid and solid phases during the test period. EN 12457 was developed primarily to support the requirements for compliance testing with the European Union

Conceptual Basis

EN 12457 is intended to assess waste leachability under mild extraction conditions for waste disposal or material reuse options. When more than one of the test procedures is used, the analyst may estimate a relative timeframe for contaminant release when compared with availability for leaching. The test focuses on release by diffusion from granular materials, a phenomenon found when fine-grained materials are surrounded by coarser particles, or when materials are compacted to a low permeability (van der Sloot, et al., 1994).

Reproducibility

A study was conducted with 12 to 14 European laboratories, utilizing seven types of waste materials, selected to represent as broad a range of waste types as possible and covering all grains sizes addressed by the test. The leachates were tested for inorganic constituents. Typical values and observed ranges of the repeatability (measurements made by a single lab) and reproducibility (between multiple labs) found are summarized in **Table 3-10**.

Table 3-10. EN 12457 Repeatability and Reproducibility

	Typical value (Relative Standard Deviation)	Observed range (Relative Standard Deviation)
r- repeatability limit (within-laboratory)	24 %	7 % - 100 %
<i>R</i> - reproducibility limit (between laboratories)	72 %	20 % - 160 %

The heterogeneity of some materials and low concentrations of some target analytes clearly contributed to high instances of repeatability and reproducibility. However, both the repeatability and reproducibility were found to be good for a range of elements in four different materials, leading to the conclusion that the leaching test is suitable and provides adequate results, provided that the samples are sufficiently homogeneous, and contain sufficient concentrations of the target analytes (Van der Sloot, et al., 1994).

3.2.17 Survey of Laboratories

While a large number of leaching tests exist, practical applications of the tests in the U.S. are limited by the capabilities, experience, and capacity of laboratories to conduct the tests. To characterize the capabilities of laboratories to conduct the leaching tests identified in Section 3.2, the project team contacted laboratories in the U.S. (with initial emphasis on commercial laboratories in the Pacific Northwest) to determine their capabilities and associated costs to perform any of the tests. A summary of the results of the survey is given in **Table 3-11**. A complete list of laboratories contacted is given in **Appendix C**.

To obtain information on laboratory capabilities for performing leaching tests, SAIC contacted more than 70 laboratories in the U.S. (primarily in the Pacific Northwest). Unfortunately, the number of replies was limited. We believe the response rate was low due to the fact that the laboratories were requested to respond to a survey of capabilities rather than a request for quote.

TEST	LABORATORY	EXPERIENCE PERFORMING THE TEST	COST PER SAMPLE*	COMMENT
ASTM D 3987	Laucks Testing Laboratories, Inc. 940 South Harney St. Seattle, WA 98108 206-767-5060 http://www.lauckslabs.com/	Equipment available, but no experience	\$100-400	Test is similar to EP Toxicity and TCLP.
	Severn Trent Laboratories 5701 Executive Drive Baltimore, MD 21228 410-869-0085 http://www.stl-inc.com/	Supported primarily for wet chemistry tests	\$85	
	Harbor Branch Environmental 5600 US 1 North Fort Pierce, FL 34946 772-465-2400 http://www.hbel.com/	Extensive	\$100	
	Inttp://www.nbei.com/ North Creek Analytical, Inc. 11720 North Creek Pkwy. North Suite 400 Bothell, WA 98011 425-420-9200 http://www.ncalabs.com/			
	Anatek Labs, Inc. 504 E. Sprague Suite D Spokane, WA 99202 509-838-3999 http://www.anateklabs.com/	Equipment available, but no experience	\$100	

 Table 3-11. Capabilities of U.S. Laboratories to Perform Leaching Tests

TEST	LABORATORY	EXPERIENCE PERFORMING THE TEST	COST PER SAMPLE*	COMMENT
ASTM D 4793	Laucks Testing Laboratories, Inc	Equipment available, but no experience	\$100-400	Test is similar to EP Toxicity and TCLP.
	Analytical Resources, Inc. 4611 S. 134th Place Tukwila, WA 98168 206-695-6200 http://www.arilabs.com/	Equipment available, but no experience	\$950/10 cycles	Test very similar to the Corps SBLT.
	Soil Technology, Inc. 7865 N.E. Day Road West Bainbridge Island, WA 206-842-8977 <u>http://www.soils-sti.com/</u>	None		
	North Creek Analytical, Inc.	None		
	Anatek Labs, Inc.	Equipment available, but no experience	\$500	
ASTM D 4874	Laucks Testing Laboratories, Inc	None		
	Analytical Resources, Inc.	10-15 tests	\$1,000/30 pore volumes	
	Soil Technology, Inc.	None		
	North Creek Analytical, Inc.	None		
	Anatek Labs, Inc.	Equipment available, but no experience	\$300 setup and \$25 per sample	
ASTM D 5744	Laucks Testing Laboratories, Inc	None		
	Analytical Resources, Inc.	None		
	Soil Technology, Inc.	None		
	North Creek Analytical, Inc.	None		
	Anatek Labs, Inc.	None		
ASTM D 6234	Laucks Testing Laboratories, Inc	None		
	Analytical Resources, Inc.	Limited	\$100	Similar to SPLP.
	Soil Technology, Inc.	None		
	North Creek Analytical, Inc.	None		
	Anatek Labs, Inc.	None		

TEST	LABORATORY	EXPERIENCE PERFORMING THE TEST	COST PER SAMPLE*	COMMENT
Dredge Elutriate Test	Laucks Testing Laboratories, Inc	None		
(DRET) - Army Corps of Engineers	Analytical Resources, Inc.	Dozens of samples	\$125	
5	Soil Technology, Inc.	Yes	\$1,200 per 20 liter batch	
	Severn Trent Laboratories	Approximately 5 projects	\$195	
	Anatek Labs, Inc.	Equipment available, but no experience	\$250	
Standard Elutriate Test	Laucks Testing Laboratories, Inc	None		
(SET) - Army Corps of Engineers	Analytical Resources, Inc.	Dozens of samples	\$125	
	Soil Technology, Inc.	Yes	\$1,200 per 20 liter batch	
	North Creek Analytical, Inc.	None		
	Anatek Labs, Inc.	One year	\$250	
Pancake Column	Laucks Testing Laboratories, Inc	None		
Leachate Testing (PCLT) - Army Corps of Engineers	Analytical Resources, Inc.	5 tests in last 2 years	\$10,000 per 30 pore volumes	Anaerobic conditions specified. Complex test requiring a minimum of 30 weeks.
	Soil Technology, Inc.	Yes	\$36,000 per 30 pore volumes	Anaerobic conditions specified. Complex test requiring a minimum of 30 weeks.
	North Creek Analytical, Inc.	None		
	Anatek Labs, Inc.	Equipment available, but no experience	\$500 setup and \$25 per sample	

TEST	LABORATORY	EXPERIENCE PERFORMING THE TEST	COST PER SAMPLE*	COMMENT
Sequential Batch Leachate	Laucks Testing Laboratories, Inc	None		
Test (SBLT)- Army Corps of	Analytical Resources, Inc.	4-5 tests per year	\$850/7cycles	
Engineers	Soil Technology, Inc.	Yes	\$250/cycle	
	North Creek Analytical, Inc.	None		
	Anatek Labs, Inc.	Equipment available, but no experience	\$500	
TCLP - SW- 846 Method	Laucks Testing Laboratories, Inc	Extensive	\$85 tumbler \$85 ZHE	
1311	Analytical Resources, Inc.	15 years	\$100 tumbler \$100 ZHE	
	Severn Trent Laboratories	> 5,000 samples	\$45 tumbler \$45 ZHE	
	North Creek Analytical, Inc.	Extensive	\$72 tumbler \$85.50 ZHE	
	Cascade Analytical, Inc. 3019 G.S. Center Road Wenatchee, WA 98801 (800) 545-4206 <u>http://www.cascadeanalytical.com</u>	Extensive	\$80 tumbler \$85 ZHE	
	Anatek Labs, Inc.	10 years	\$100 \$150 ZHE	
SPLP - SW-846 Method 1312	Laucks Testing Laboratories, Inc	Extensive	\$85 tumbler \$85 ZHE	
	Analytical Resources, Inc.	15 years	\$100 tumbler \$100 ZHE	
	Severn Trent Laboratories	> 1,000 samples	\$45 tumbler \$45 ZHE	
	North Creek Analytical, Inc.	Extensive	\$72 tumbler \$85.50 ZHE	
	Cascade Analytical, Inc.	Extensive	\$80 tumbler \$85 ZHE	
	Anatek Labs, Inc.	2 years	\$100 \$150 ZHE	

TEST	LABORATORY	EXPERIENCE PERFORMING THE TEST	COST PER SAMPLE*	COMMENT
NEN 7341 - Dutch Total	Laucks Testing Laboratories, Inc.	None		
Availability Test	Analytical Resources, Inc.	None		
	Soil Technology, Inc.	None		
	North Creek Analytical, Inc.	None		
	Anatek Labs, Inc.	Equipment available, but no experience	\$500	
NEN 7343 Column Test (The Netherlands)	None identified in the U.S.			
NEN 7345 Tank Leach Test (The Netherlands)	None identified in the U.S.			
prEN 14405 Upflow percolation test (European Union)	None identified in the U.S.			
EN 12457/1-4, Compliance Test for Granular Waste Materials and Sludges (European Union)	None identified in the U.S.			

Notes to table:

* Unit costs assume a total of 10 samples of granular fill material with a standard 30-day turnaround time. Estimated costs are for sample preparation and extraction only. Additional costs would be incurred for extract analyses depending on the desired constituents.

Blank cells in the table indicate no information was obtained.

Other leaching tests with a capability as listed by Analytical Resources:

- Modified Elutriate Test (MET) Army Corps of Engineers \$125
- ASTM D 4646 24 hr batch sorption test (organics) \$125
- MWEP SW-924 (10 mL/g waste, 4 cycles, 18hrs/cycle) \$375
- ANSI/ANS 16.1 short term (90 day leaching test) \$325
- ASTM D 5285 24 hr batch sorption test (volatile organics) \$125

Laboratories contacted with an emphasis on regional eastern Washington locations:

- AAA Lab, Inc., Cheney, WA Have the ability to perform TCLP metals only.
- Ana Laboratories, Spokane, WA No environmental testing performed at this facility.

- Anatek Labs, Inc., Spokane, WA Experienced with three leaching tests and also submitted costs for several tests for which they have no previous experience. These costs should be viewed as hypothetical.
- Cascade Analytical, Inc., Wenatchee, WA Only experienced with TCLP and SPLP testing.
- Energy Northwest Environmental Lab, Richland, WA No response was received to several requests for information.
- North Creek Analytical, Inc., Montgomery St., Spokane, WA This facility is part of the North Creek Analytical lab network consisting of six domestic locations. The response received from the Bothell location, noted in the table, applies to the entire network regardless of location.
- Valley Environmental Laboratory, Yakima This lab has the ability to perform TCLP metals and may have the capability to perform other leaching tests, but those listed in the survey have never been requested.

4.0 ASSESSMENT OF LEACHING TEST METHODS FOR ANALYZING WATER QUALITY IMPACTS FOR ALL TYPES OF PROJECT AND IN ALL CIRCUMSTANCES WHERE FILL MATERIAL IS IMPORTED

This section presents an assessment of leaching test methods that can be used to evaluate water quality impacts for projects where fill material is a candidate for use. The first part of the assessment (Section 4.1) compares leaching test results to actual field leachates to evaluate the ability of leaching test to accurately predict constituent concentrations in field leachates. The second part of the assessment (Section 4.2) describes other methods used to interpret leaching test results.

4.1 Leaching Test Results and Actual Field Leachate Concentrations

A fundamental question in the assessment of leaching test methods is: How well do the leaching test data represent the scenario in the field (Inyang, 2003)? To address this question, a literature search was conducted to obtain data or other information comparing the results of laboratory leaching tests to analytical results of field leachates for a given waste or material. The initial literature review focused on research previously performed by Ecology on leaching tests. Additional information was obtained in the form of journal articles, government publications, and other published reports.

Field leachates described in the literature were collected from leachate collection systems, controlled landfill cells, field lysimeters, or large-scale column tests. Groundwater and surface water monitoring data were not evaluated because dilution, attenuation, transformation, and other processes that occur in groundwater and surface water greatly hinder the ability to correlate laboratory leach tests results with field leachate characterization results.

4.1.1 Summary of Findings

Table 4-1 presents a summary of the literature reviewed to address the question of how well leaching test data represent field leachates. The literature search revealed relatively few studies involving comparisons of laboratory leaching test results to actual field data, and most of the work to date has focused on leaching of inorganic constituents rather than organic constituents. Of those studies reviewed, the results were mixed in that some leaching tests over-predicted the field leaching, some under-predicted field leaching, and others provided ambiguous results.

A number of factors contribute to the difficulty in comparing laboratory leaching test results to field leachate analysis results. These factors include heterogeneity of the waste or material under field conditions, difficulties in obtaining representative samples, differences in the liquid-to-solid ratio under field versus laboratory test conditions. As discussed in the following paragraphs, comparisons of a leaching test result to actual field leachate data are more meaningful when the purpose of the test and its underlying assumptions are understood.

Single scenario/batch leaching tests, such as the TCLP and the SPLP, typically are designed as compliance tests and not necessarily to predict the character of leachate generated at a specific

site. For example, the TCLP was designed to model average leaching of industrial waste codisposed with municipal solid waste over a moderate to long-term (3 to 10 years) exposure period (Kimmel and Friedman, 1986), and not the peak concentration. Moreover, the test was designed to differentiate between clearly hazardous waste and nonhazardous waste per the Toxicity Characteristic (40 CFR 261.24) – not to predict the actual leachate concentration on a site-specific basis. Some of the batch tests used in the studies evaluated (see Table 4-1) were shown to be inaccurate predictors of concentrations in field leachates due to failure of the tests to simulate certain factors that influence field leaching such as biological activity, oxidation, or pH changes due to carbonation. In fact, one should not expect batch test (e.g., TCLP or SPLP) results to match actual field leachates except where there is a reasonable match between field and laboratory test conditions. Even where the leaching test does not accurately predict field conditions, the test may still serve its purpose (e.g., for waste classification) where the leaching test provides conservative (over-predictive) results.

An alternative to the use of a single batch test to assess leaching on site-specific basis is the use of multiple tests designed to measure intrinsic leaching properties, better understand release mechanisms, and provide more accurate translation (e.g., using release modeling) between the laboratory test data and the release under field conditions (see also Section 4.2.5). Intrinsic leaching parameters that can be measured using laboratory testing are:

- Total long-term availability (e.g., using NEN 7341)
- Solubility and release as a function of pH (e.g., prEN 14429 or Kosson, et al.'s "SR002.1" (Kosson, et al., 2002))
- Solubility and release as a function of L/S ratio (prEN 14405) to relate leach test results to a time scale of interest based on infiltration rate, and
- Mass transfer from a monolithic waste (NEN 7345) or from fully saturated compacted granular materials (Kosson, et al., 2002).

While tests for intrinsic leaching parameters provide significantly more information than single batch tests, they too can yield ambiguous or inaccurate results because they do not take into account all factors that influence leaching in the field.

In summary, in spite of the large number of leaching tests available (see Section 3), no single leaching test or combination of tests can provide a complete match between the laboratory test(s) and field conditions. Leaching tests designed to model a specific disposal scenario tend to be inaccurate predictors of field leachates where the conditions in the field do not match the assumptions of the test. Tests designed to measure intrinsic properties of material provide a means for evaluating trends and predicting release under site-specific conditions but also do not take into account all factors that influence leaching in the field.

Field Scenario	Tested Material	Compared Leaching Test	Parameters	Observations/ Conclusions	Reference	Section
Field vaults	Ash and MSW + Ash	 4-ft. leaching columns (laboratory-scale lysimeters) TCLP SW-846 Method 1311 SPLP SW-846 Method 1312 California Waste Extraction Test Multiple Extraction Procedure (MEP) 	Temperature, pH, oxidation- reduction potential, conductivity, total dissolved solids, chemical oxygen demand, non- purgeable organics, anions, cations, alkalinity, ammonia.	Sodium, potassium, calcium, and magnesium showed good correlation of cumulative amounts leached between the vault, lysimeters, SPLP, and other batch test results.	Townsend, Jang, and Tolaymat, 2003a	4.1.2.1
Continuous Column Leaching Experiments	Soil from four sites representing typical glacial till and stratified drift deposits found in New England. Low organic carbon and low clay content.	 Toxicity Characteristic Leaching Procedure (TCLP) Synthetic Precipitation Leaching Procedure (SPLP) Deionized Water Leaching Procedure (DWLP) California Waste Extraction Test 	Site 1: Cd, Cr, Cu, Pb, Ni, Zn, final pH Site 2: Pb, Zn, final pH Site 3: Cd, Cr, Cu, Pb, Ni, Zn, final pH Site 4: As, Cr, Ni, final pH	The SPLP was determined to be more realistic than the TCLP for assessing the mobility of metals in glaciated soils, however, the SPLP overestimated the mobility of most metals (As, Pb, and Zn) in the soils studied and under- predicted leachate concentrations of Cr.	Lackovic, et al., 1997	4.1.2.2

Table 4-1. Summary of Findings of Literature Search for Data Correlating Leaching Test Results with Actual Leachate Concentrations

Field Scenario	Tested Material	Compared Leaching Test	Parameters	Observations/ Conclusions	Reference	Section
Landfill simulator	Ash and MSW + Ash	 One step leaching test with L/S of 4 ml/g One-step DIN 38414 with L/S 10 Sequential test modified "S4" DIN 38414-S4 Sequential Swedish Envi. Protection Agency "ENA" test 	Conductivity, chloride, volatile solids, COD, TOC, TKN, Cr, Ni, Zn, S	For MSW, leaching tests such as shake leaching tests (SLTs) that do not promote biological activity cannot be considered good tools for prediction of leachate quality from MSW landfills over time.	Kylefors, et al., 2003	4.1.2.3
Lysimeter	Air-cooled blast furnace (ACBF) slag	 Two-stage serial batch leaching test at L/S = 0-2 l/kg and 2-10 l/kg CEN prEN 12457-3 or Nordtest NT ENVIR 005 	pH, Ni	pH in lysimeter was lower than batch tests due to oxidation of sulfides, a phenomena not reproduced in the lab test. Batch test under-predicted leaching of metals.	European Commission, 2001	4.1.2.4
Lysimeter	Air-cooled blast furnace (ACBF) slag	Column leaching test to L/S = 2 l/kg, analysis of 5 eluate fractions, Nordtest NT ENVIR 002	pH, Co, Cu, Ni, Zn	Column test under- predicted leaching of metals.	European Commission, 2001	4.1.2.4
Lysimeter	Crushed Concrete	Two-stage serial batch leaching test at L/S = 0-2 l/kg and 2-10 l/kg, CEN prEN 12457-3 or Nordtest NT ENVIR 005	рН	Batch test did not reproduce pH changes that occurred in lysimeter due to carbonation of the crushed concrete	European Commission, 2001	4.1.2.4

Field Scenario	Tested Material	Compared Leaching Test	Parameters	Observations/ Conclusions	Reference	Section
Lysimeter	Crushed Concrete	Column leaching test to L/S = 2 l/kg, analysis of 5 eluate fractions, Nordtest NT ENVIR 002	pH, Cr, Cu, Ni, Pb, sulfate	The column leaching test is appropriate for this waste because it does not underestimate the leaching in a field situation for Cr, Cu, Ni or Pb	European Commission, 2001	4.1.2.4
Field lysimeter	Cement-stabilized air pollution control (APC) residues, from MSW incineration, in the form of blocks, 0.5m on each side.	Batch leaching test with L/S ratios of 5, 10, and 20 equilibrated for 27 days. Final pH of 12.45, 12.23, and 12.02 respectively.	pH, Cd, Co, Cu, Mn, Ni, Mo, Pb, W, Zn, plus major species of Al, Ca, K, Na, and Si. Chloride and SO ₄ .	Batch leaching test results and thermodynamic modeling could not explain leaching behavior of most heavy metals. Concentrations of Cd, Co, Cu, Mn, Ni, Mo, Pb, W, and Zn were often lower in the field leachate than expected from batch experiments.	Baur, et al., 2001	4.2.1.5
Field lysimeter	Cement-stabilized APC residues, from MSW incineration, in the form of blocks, 0.5m on each side.	Tank dynamic leaching test similar to ANSI/ANS-16.1-1986	pH, Cd, Co, Cu, Mn, Ni, Mo, Pb, W, Zn, plus major species of Al, Ca, K, Na, and Si. Chloride and SO ₄ .	Results of tank leaching test were ambiguous for some species, but seem to be correct for alkalis (Na and K) which leach primarily by diffusion.	Baur, et al., 2001	4.1.2.5

Field Scenario	Tested Material	Compared Leaching Test	Parameters	Observations/ Conclusions	Reference	Section
Pilot landfill cell (12,000 m ³)	Inorganic waste and mixtures of inorganic waste and organic (shredder waste and sewage sludge)	Percolation test PrEN 14405 and pH- dependence test PrEN 14429	pH, DOC, and Zn	Mutual consistency of the data at the field, lysimeter, and laboratory scales is promising and may provide a means for assessing long-term leachate quality for predominantly inorganic wastes.	Van der Sloot, et al., 2002	4.1.2.6
Lysimeters (three at 1.5 m ³ each)	Inorganic waste and mixtures of inorganic waste and organic (shredder waste and sewage sludge)	Percolation test PrEN 14405 and pH dependence test PrEN 14429	pH, DOC, and Zn	Mutual consistency of the data at the field, lysimeter, and laboratory scales is promising and may provide a means for assessing long-term leachate quality for predominantly inorganic wastes	Van der Sloot, et al., 2002	4.1.2.6
Field lysimeter	Alkaline fly ash	NEN 7343 (column test)	pH, Al, As, Ca, Fe, K, Mg, Mo, Na, P, Si, S	There was a "great discrepancy" between the column test results and field lysimeter results.	Janssen- Jurkovieova, et al., 1994	4.1.2.7
Field lysimeter	Alkaline fly ash	NEN 7341 (availability test)	pH, Al, As, Ca, Fe, K, Mg, Mo, Na, P, Si, S	Availability test is not adequate to determine the maximum amount of elements available from the ash for leaching.	Janssen- Jurkovieova, et al., 1994	4.1.2.7

Field Scenario	Tested Material	Compared Leaching Test	Parameters	Observations/ Conclusions	Reference	Section
Field lysimeters	Blast furnace slag, steel slag, MSWI bottom ash, wood ash	Availability test similar to NEN 7341 and column leach test	pH, redox, conductivity, total dissolved solids, metals, and salts	Estimated accumulated leached amounts in the lysimeter were 10 to more than 100 times the column test results, except for MSWI bottom ash where field and column test pH and redox were similar.	Fällman and Hartlén, 1994	4.1.2.8
Large-scale field drainage lysimeters	Coal combustion residue (fly ash)	Laboratory batch and column experiments	pH, conductivity, alkalinity. Sulfate, fluoride, chloride, nitrate, nitrite, ammonium, phosphate, B, Na, K, Ca, Mg, As, Ba, Cd, Co, Cr, Cu, Hg, Mo, Ni, Pb, Se, V, and Zn.	The results of the accelerated leaching test correlated well with the results of the large- scale lysimeter experiments.	Hjelmar, 1990	4.1.2.9
Pressure vacuum lysimeters	Ferrous foundry wastes	EP and EP (with deionized water)	pH, conductivity, TOC, phenols, CN, fluoride, As, Ba, B, Cd, Cr, Cu, Fe, Pb, Mg, Hg, Se, Ag, and Zn	The EP was basically equal to the EP-water test in predicting the presence of constituents in foundry waste leachate. However, substantial variability was observed between leach test and field results.	Ham, Boyle, and Blaha, 1986 and Ham et al., 1986 (see Section 4.1)	4.1.2.10

Field Scenario	Tested Material	Compared Leaching Test	Parameters	Observations/ Conclusions	Reference	Section
Saturation extract (equivalent to field pore water)	Coal combustion byproducts (e.g., fly ash)	TCLP	As, Ba, Cr, Pb, Se	The ratios of TCLP to saturation extract values indicate that the TCLP consistently overestimated the concentration of Ba and consistently underestimated the concentration of Se. Results for other metals (As, Cr, and Pb) differed by no more than one order of magnitude.	EPRI, 1995	4.1.2.11
Large columns	Electroplating sludge, Electric arc furnace dust, Paint incinerator ash, Municipal refuse incinerator ash, Mine tailings (MT)	 Monofilled Waste Extraction Procedure USEPA Extraction Procedure Acetate Buffer Extraction Procedure Ham Procedure C Saturated Paste Procedure 	pH, Electrical conductivity, Cr, Ni	For selected metals, the authors concluded that "batch extraction procedures that employ acetic acid or acetate buffer are less effective for assessing the leachability of monofilled wastes than extraction methods that use de-ionized water as the extraction fluid."	Jackson and Bisson, 1990	4.1.2.12

Field Scenario	Tested Material	Compared Leaching Test	Parameters	Observations/ Conclusions	Reference	Section
Concentration profiles of road-based materials	Coal fly ash and MSWI bottom ash used as stabilization materials in roadbase applications.	Study did not specify the actual leaching tests employed. Leaching data were obtained from previous leaching studies performed on similar materials to those studied.	Cr, Mo, V, Zn, SO ₄	For most of the constituents evaluated, the results indicated a good to very good agreement (defined as a difference of less than a factor of 2) between predictions based on laboratory tests and field data.	Schreurs, et al., 1997	4.1.2.13
Leachate collection sumps, leachate collection lines, and lysimeters at ash monofills	Municipal waste combustion residues	 EP-TOX (Extraction Procedure) TCLP SW-924 (MWEP) CO2 saturated deionized water Simulated acid rain (SAR) 	Semivolatile compounds, PCBs, dioxins and furans, metals, and several miscellaneous conventional compounds	 Extracts from EP- TOX and TCLP contained higher concentrations of metals than the other leaching test methods Extracts from the SW-924, CO2, and SAR extraction procedures simulated field concentrations of Pb and Cd better than the other three procedures Few organics were detected in the field leachates and leaching test extracts. 	USEPA, 1990c	4.1.2.14

Field Scenario	Tested Material	Compared Leaching Test	Parameters	Observations/ Conclusions	Reference	Section
Leachate collection system at K088 hazardous waste monofill	Treated K088: listed hazardous waste generated from the primary reduction of aluminum ("pot liner")	TCLP	As, fluoride, pH, CN, toxic metals and PAHs	Field leachate had higher levels of arsenic and fluoride than predicted by TCLP. Weakly acidic extraction fluid in TCLP does not reflect actual disposal conditions for K088, which are highly alkaline (pH 12.5). Arsenic is more soluble under highly alkaline than weakly acidic conditions.	USEPA, 1997	4.1.2.15
Dredging site	Dredging material	Standard Elutriate Test (SET)	Cd, Zn, Pb, Cu, Hg, As, Cr, Ni, PCBs, Mn, Fe, Total phosphorus, Total Kjeldahl nitrogen, NH ₃	For most constituents, the SET was shown to be a conservative predictor of dissolved concentrations in the water column at the point of dredging	Havis, 1988	4.1.2.16

Field Scenario	Tested Material	Compared Leaching Test	Parameters	Observations/ Conclusions	Reference	Section
Dredge material rehandling facility	Dredging material	 One-day Modified Elutriate Test (MET) Seven-day Modified Elutriate Test (MET) 	Metals, Tributyltin, chlorinated pesticides, PCBs, and semivolatile organics (including PAHs, phenols, phthalates, chlorobenzenes, and other extractables)	In general, there was good agreement between the effluent concentrations predicted by the one- day and seven-day MET and actual concentrations in the field samples.	Thornburg et al., 2002	4.1.2.17

4.1.2 Literature Review

The literature search was conducted to obtain data and other information correlating leaching test results with actual field leachate concentrations. The following sections present summaries of literature reviewed.

4.1.2.1 Leaching Tests for Evaluating Risk in Solid Waste Management Decision Making (Townsend, et al., 2003a)

The Florida Center for Solid and Hazardous Waste Management conducted a two-year study (Townsend, et al., 2003a) on the use of leaching tests for risk-based decision-making in solid waste management. The study evaluated three waste streams using laboratory batch and column leaching tests and field vault experiments. Sample analysis focused on the leachability of selected heavy metals, and the leaching experiments evaluated the role of key leaching factors including leaching time (contact time), pH, and liquid-to-solid (L/S) ratio.

Wastes evaluated in the study included the following:

- Spent abrasive blasting media (ABM) from shipyards and private contractors
- CCA-contaminated soils obtained from a CCA wood treating facility, and
- Wood and tire ash obtained from a waste to energy generation plant.

Leaching of the waste was evaluated by means of laboratory batch leaching tests (including the Toxicity Characteristic Leaching Procedure (TCLP), Synthetic Precipitation Leaching Procedure (SPLP), California Waste Extraction Test, and the Multiple Extraction Procedure (MEP)), laboratory leaching lysimeters (4-foot columns), and field leaching vaults. Field leaching vaults (four total) were described as 2.5-foot by 2.0-foot by 7.0-foot ash-filled concrete vaults lined with high-density polyethylene with drainage holes at the bottom of each. The vaults were used to simulate field leaching under natural weathering conditions. Leachate samples were collected once a week or after each rain event and actual precipitation was monitored via a weather station. The use of the various leaching test methods offered the investigators the ability to examine leaching under a wide range of L/S ratios, with the vault tests providing the lowest L/S ratios and the batch tests the highest L/S ratios.

One aspect of the study involved a detailed examination of the leaching of the wood-tire ash including an examination of how to relate batch leaching tests to field leaching. Analysis included total metals aluminum, arsenic, barium, calcium, cadmium, Co, chromium, copper, vanadium, iron, potassium, lead, magnesium, manganese, sodium, nickel, and zinc. Metal mobility was assessed using TCLP, Waste Extraction Test (WET), SPLP, Deionized (DI) water extraction, and the Multiple Extraction Test (MEP). L/S ratio, contact time, and pH effects also were examined.

For the leaching study of the wood and tire ash, California Waste Extraction Test extracted the highest levels, followed by the TCLP, and the SPLP and deionized (DI) water extractions. The pH and complexation of organic acid with metals were the major controlling factors in metal leachability. In the pH static tests, metal concentrations in the extracts were the highest at low

pH values and lowest around pH 9. Less than three hours of leaching time (or contact time) was needed to reach equilibrium conditions, indicating metal leaching from an alkaline waste occurred rapidly. This indicates that the 18-hour leaching required by most batch extraction tests was enough to reach chemical equilibrium for leaching experiments of the wood and tire ash. The trends of metal leaching with different L/S ratios noticeably varied, depending on the type of metal.

Lysimeter results showed that many inorganic constituents (especially soluble ions) typically leached at high levels at the beginning of the experiments and rapidly decreased over time, with the exception of calcium, lead, and zinc. Townsend, et al. attribute the high concentrations observed at the initial stages to a surface wash-off mechanism. Of the metals analyzed for in the leachate from the vault tests only four metals (aluminum, barium, copper, and iron) were consistently detected above the detectable limits.

The field vault test results were compared to the lysimeters (column) and batch leaching test results. Data comparisons were presented for sodium, potassium, calcium, and magnesium. For each constituent, plots of constituent concentration (mg/L) as a function of L/S ratio and cumulative constituent released (mg/kg) as a function of L/S ratio were presented. Each plot combined vault, lysimeters, SPLP and other batch test results. All four constituents showed a good continuity of cumulative amounts leached between the vault, lysimeters, SPLP, and other batch test results, though, at the L/S ratio of 20, the SPLP slightly over-predicted concentrations of sodium and potassium compared to the lysimeters results.

4.1.2.2 Evaluation of Batch Leaching Procedures for Estimating Metal Mobility in Glaciated Soils (Lackovic, et al. 1997)

Lackovic, et al. (1997) conducted a study of four batch leaching procedures and compared the batch test results to the results of continuous column leaching experiments. The column leaching tests served to represent field conditions. The objective was to evaluate the batch tests for their ability to realistically quantify the mobility of metals from previously contaminated glaciated soils as compared to continuous column leaching experiments. Leaching tests were performed on samples of contaminated soils obtained from four sites (three in Connecticut and one in Maine). The soils selected tended to have relatively low organic carbon and low clay content, and represent typical glacial till and stratified drift deposits found in New England.

Batch leaching procedures included the Toxicity Characteristic Leaching Procedure (TCLP); Synthetic Precipitation Leaching Procedure (SPLP); Deionized Water Leaching Procedure (DWLP); and the California Waste Extraction Test. In addition, continuous column leaching experiments were conducted to quantify the amount of metals leached from soil under different pH and flow conditions. The authors state that continuous column leaching tests are usually more representative of the actual environmental [field] conditions than are batch leaching tests. The continuous column leaching test was carried out by filling the glass column (3.5 cm inside diameter by 45 cm height) with 500 grams of contaminated soil (<2 mm fraction) and passing an eluant through the soil under an oxygen-free environment for eight weeks. An oxygen-free solution of 0.1 M NaNO3 containing 0.02 percent sodium azide (to inhibit biological growth) was used as the eluant. To assess the impact of pH and hydraulic loading on leachability of metals, the column leaching was carried out with eluant at two different pH values (4 and 7) and two different nominal flow rates (0.1 mL/min and 1.0 mL/min).

Comparing the metal concentrations from the four batch leaching test revealed that the California Waste Extraction Test is the most chemically aggressive test due to the relatively low buffered pH and citrate chelation used as the leaching solution. The TCLP has a similar buffered pH as the California Waste Extraction Test but leached a lower concentration of metals. The SPLP and DWLP provided similar results to each other in the concentration of metals leached, and at concentrations less than the TCLP or the California Waste Extraction Test.

The results of the TCLP and SPLP leaching tests were compared with a set of continuous column leaching experiments. The basis for comparison of the TCLP and SPLP results to the column leaching test results was the "average cumulative concentration at 10 liters" or ACC10. The ACC10 is the average of the cumulative mass of each metal in the first 10 liters of column effluent passing through the 500-gram column sample, thus corresponding to the same liquid-to-solid (L/S) ratio used in the TCLP and SPLP (that is, 2 L/100-g). The data used in the comparison are reproduced in **Table 4-2**.

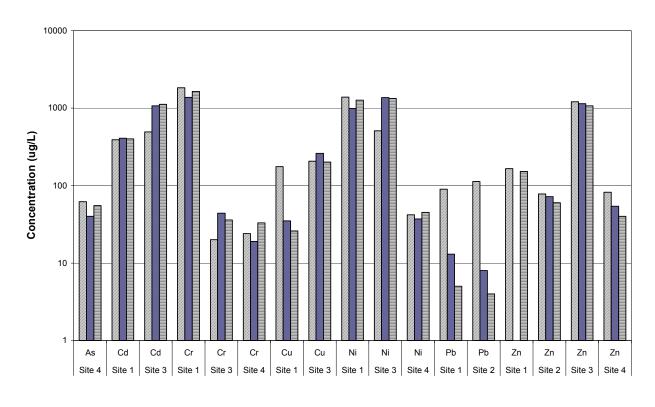
		Concentration (µg/L)						
Site	Metal	TCLP	SPLP	ACC10 Low flowrate pH = 4.0	ACC10 Low flowrate pH = 7.0	ACC10 Hi flowrate pH = 4.0	ACC10 Hi flowrate pH = 7.0	
Site 1	Cd	531±24	391±23	774±92	409±31	526±60	400±76	
Site 1	Cr	2443±272	1838±272	1313±123	1380±121	1608±365	1638±185	
Site 1	Cu	222±12	176±31	366±3	35±1	442±28	26±4	
Site 1	Ni	2010±37	1383 ± 120	2400±139	988±90	1947±255	1268±104	
Site 1	Pb	ND	90±8	22±4	13±2	30±2	5±2	
Site 1	Zn	283±22	165±35	397±12	ND	368±148	152±17	
Site 2	Pb	188±40	113±0	29±9	8±2	47±33	4±1	
Site 2	Zn	260±26	78±9	613±23	72±6	481±34	60±7	
Site 3	Cd	7599±207	494±176	3457±420	1070±38	7160±1580	1122±21	
Site 3	Cr	38±3	20±2	36±5	44±5	55±5	36±1	
Site 3	Cu	2772±138	207±36	401±29	260±7	455±98	201±5	
Site 3	Ni	3156±124	508±122	5465±482	1365±49	5315±611	1335±32	
Site 3	Zn	ND	1209±451	8558±3184	1141 ± 20	12835±1602	1069±31	
Site 4	As	116±11	62±6	49±2	40±2	52±3	55±2	
Site 4	Cr	24±6	24±4	26±3	19±3	14±2	33±1	
Site 4	Ni	52±12	42±3	44±3	37±2	79±4	45±2	
Site 4	Zn	172±21	82±13	104±41	54±8	407±132	40±9	

Table 4-2. Comparison of TCLP and SPLP Results to Column Leaching Test Resu	lts (data
from Lackovic, et al., 1997)	-

Values after " \pm " are the standard deviation.

Lackovic, et al. (1997) concluded that overall, out of the 17 sets of metals results examined across the four sites, SPLP and TCLP concentrations were statistically equal to or greater than the high flowrate ACC10 concentrations for both pH-4 and pH-7 test conditions. **Figure 4-1** presents a comparison of the SPLP results to the column test results run at pH 7. The SPLP tests had final pH values between 5.5 and 7.2, consistent with the pH values of the column test and consistent with the soil pH values reported for the four sites (5.6 to 7.4 SU).

The SPLP results were consistently conservative (greater than the column test results) for arsenic, lead, and zinc. Compared to the column test results, the SPLP showed mixed results for cadmium, nickel, and copper and under-predicted leachate concentrations of chromium. Lackovic, et al. (1997) concluded that the SPLP is a conservative method to determine the mobility of metals from sandy, low organic content, glaciate soils.



■ SPLP ■ ACC10 Lowflow pH-7 ■ ACC10 Hiflow pH-7

Figure 4-1. SPLP results compared to continuous column leaching test results (data from Lackovic, et al., 1997)

4.1.2.3 A Comparison of Small-Scale, Pilot-Scale and Large-Scale Tests for Predicting Leaching Behavior of Landfilled Wastes (Kylefors, et al., 2002)

Kylefors, et al. (2002) conducted a study to compare the leaching behavior in small-scale, pilotscale and large-scale tests. Shaking leaching tests (SLT), landfill-simulator leaching tests, and a field-cell leaching test were performed with ash, municipal solid waste (MSW), and a MSW and ash mixture. The SLTs included a Swedish and a German test designed to assess leaching at different liquid-to-solid ratios.

Comparision of the test results identified factors that influence leaching. These factors are: liquid to solid (L/S) ratio, water withdrawal, recirculation rate, presence or absence of biological processes, size of particles, duration of experiment, temperature and pre-treatment of the waste. The authors found that the leaching patterns of the SLT and simulators are not similar, indicating that "either the L/S is not useful as a predictive tool or that one or more other factors influence leaching to a greater extent than the amount of added water" (Kylefors, et al. 2002). The authors concluded that the presence of biological processes in a landfill has the greatest impact on leaching and is the main reason why shaking leach tests are less useful for long-term predictions.

4.1.2.4 The Use of Alternative Materials in Road Construction (European Commission, 2001)

A collaborative research project entitled ALT-MAT (ALTernative MATerials in road construction) was funded by the European Commission and carried out by a consortium of nine organizations in seven countries. In Europe, many countries have created economic incentives in the form of taxes on landfills and on the use of natural aggregates in order to increase the use of alternative materials in construction applications. Alternative materials evaluated in the study were municipal solid waste incinerator (MSWI) bottom ash, crushed concrete, granulated steel slag and air-cooled steel slag, glass-like slag, and building demolition material. The use of such materials in construction in Europe remains low. This has to do with the perception of such materials as being "waste" and hence inferior, particularly for non-road by-products; partly for economic reasons; and partly because of concerns about the mechanical and environmental performance of the materials (European Commission, 2001).

The objective of the ALT-MAT study was to develop test methods to assess the suitability of alternative materials in road construction, concentrating on unbound granular applications such as road sub-base and capping. To achieve this objective, various activities and tests were carried out to relate laboratory tests and field performance including: a literature review; selection and characterization of alternative and natural reference materials; inspection and monitoring of existing roads constructed with alternative materials; lysimeter tests; climate chamber tests; laboratory tests for mechanical properties, leaching behavior and hydrodynamic properties; and an end-users workshop.

The ALT-MAT report (European Commission, 2001) describes the leaching tests used. The authors note that a number of national standards, pre-standard CEN ("Committee European de Normalization") tests, or Nordtest¹-recommended leaching test methods are available to assess the leaching properties of alternative materials under different circumstances. Several of these tests were selected and applied in this project including the column leach test (Nordtest NT ENVIR 002 [Nordtest, 1998b]) and two-stage serial batch leaching tests (the CEN prEN 12457-3 and the Nordtest NT ENVIR 005 [Nordtest, 1998a]). The authors recommend

¹ Nordtest (Finland) is an institution under the Nordic Council of Ministers and acts as a joint Nordic body in the field of conformity assessment. The emphasis is on the development of Nordic test methods and on Nordic co-operation concerning conformity assessment.

that a number of leaching methods be used to fully characterize the leaching properties of the alternative materials. Comparison of the results of these tests with lysimeter and climate chamber tests indicates that column tests provide the most detailed simulation of the actual leaching behavior of the materials under normal circumstances (European Commission, 2001).

The ALT-MAT report presents selected data and qualitative information providing a comparison of laboratory leaching tests (i.e., the column test and the two-stage batch test) to the lysimeter tests for two materials: air-cooled blast furnace (ACBF) slag and crushed concrete.

ACBF Slag: For the evaluation of ACBF slag, the ALT-MAT report provides limited quantitative data comparing the lysimeter test data to the laboratory leaching tests, however, information is provided on pH and concentrations of cobalt, copper, nickel, vanadium, and S. The pH values in the lysimeter test were lower than found in the leaching tests. The authors believe the low pH value in the lysimeters was caused by the oxidation of sulfides. Such a low pH was not reproduced in any of the laboratory leaching tests. Leached amounts of cobalt, copper, nickel, and zinc were found to be higher in the lysimeter test than in the leaching tests. The authors concluded that the low pH in the lysimeters was most probably the cause of the increased leaching of the potential harmful elements (exceeding reference materials). The authors further concluded [for this material] that "laboratory tests cannot satisfactorily predict leaching in a field situation" (European Commission, 2001).

Crushed Concrete: For the evaluation of crushed concrete, the ALT-MAT report provides a comparison of lysimeter test data to the laboratory leaching test data for pH, chromium, copper, nickel, lead and sulfate. Early in the sequence of tests, the pH value in the lysimeter test was in the same range as the leaching tests. However, the lysimeter pH dropped considerably, from about 12.5 to 9, while the pH in the two-stage batch tests and in one of the column tests remained relatively constant. The authors indicated the change in pH may have been due to carbonation of the crushed concrete. The lysimeters showed lower accumulated leached amounts of trace metals chromium, copper, nickel, and lead than the corresponding column tests. The authors concluded that for crushed concrete, column test would thus be appropriate to use because they would not underestimate the leaching in a field situation for these potentially harmful elements.

The authors provide the following additional conclusions and recommendations regarding the use of leaching tests:

- If there is any chance that the pH of the material might change significantly in the field, for example by carbonation of oxides/hydroxides or oxidation of sulfides, pH-static tests should be used to estimate the effect of the anticipated pH change. Leaching tests at high liquid to solid (L/S) ratios (e.g. availability tests) are of limited value in road construction applications, unless the road does not have a relatively impermeable surface layer (e.g. gravel roads in rural areas).
- Impact assessments and predictions should be based on scenario calculations, i.e., a
 description of the physical lay-out of the site in question and the flow of water through
 the site, in conjunction with a description of the composition of the leachate formed as a

function of L/S or time. The leachate composition should be derived from laboratory or lysimeter leaching tests.

- Among the useful tools for assessment of the environmental properties of alternative materials used in road construction, the authors particularly recommended the following: (1) Draft prENV12920, Methodology Guideline for the Determination of the Leaching Behaviour of Waste under Specified Conditions; (2) Column leaching test NT ENVIR 002; (3) and Batch Compliance test prEN 12457-3. The pH-dependency of the leaching behavior should be investigated, e.g. using a pH-static leaching test. CEN/TC 292 is currently developing most of these leaching procedures as European Standards.
- When effects of climate are to be predicted in larger systems, then lysimeters and climate chamber tests can be used. The conditions for lysimeter tests will resemble natural conditions more closely than laboratory leaching tests, since they will be exposed to the natural climatic conditions.
- Laboratory leaching tests can be used to give a conservative estimate of environmental effects, since laboratory tests in most cases will overestimate actual leaching.

The ALT-MAT report also presents a model that can be used to predict the impact of leaching from alternative materials in road construction on the quality of groundwater.

4.1.2.5 The Leaching Behavior of Cement Stabilized Air Pollution Control Residues: A Comparison of Field and Laboratory Investigations (Baur, et al., 2001)

Baur, et al. (2001) investigated the factors controlling leachate composition of cement stabilized air pollution control (APC) residues from municipal solid waste (MSW) incineration using laboratory tests and a pilot landfill. Results from batch leaching tests and dynamic tank leaching tests were compared to field lysimeter data.

The field lysimeter containing the APC residue was constructed in1990/1991 next to the Teuftal landfill in Frauenkappelen, Switzerland. Leachate sampling was conducted during the life of the lysimeter. During dismantling of the lysimeter, samples of the APC material were obtained and subjected to various batch and tank dynamic leaching tests by Baur, et al. For the batch leaching tests, the material was dried and ground to <0.25 mm and suspensions were prepared at L/S ratios (m/v) of 5, 10, and 20. Samples were shaken at room temperature and subsequently passed through 0.45 μ m filters and analyzed. Thermodynamic modeling of the field and laboratory data was performed to determine the possible solubility-controlling phases.

For the tank dynamic leaching test, blocks of the material were leached in ultrapure water at a L/S ratio (v/v) of 10 without agitation. Leachates were replaced and passed through 0.45 μ m filters on days 1, 3, 7, 14, 28, and 42. According to Bauer, et al. (2001), this test was similar to ANSI/ASI-16.1 (Measurement of Leachability of Solidified Low-Level Radioactive Waste by a Short-Term Test Procedure, American Nuclear Society).

The comparison of field and laboratory investigations showed that the leaching behavior of most major species (calcium, SO₄, aluminum, and Si) could be explained either by solubility controlling phases or by diffusion (Baur, et al., 2001). Determined effective diffusion coefficients for sodium and potassium were used to model field concentrations, and agreement with field data was good. Baur, et al. reported, however, that it is more difficult to explain the leaching behavior of heavy metals. Heavy metal concentrations (cadmium, cobalt, copper, Mn, nickel, molybdenum, lead, tungston, and zinc) in all experiments often were lower in the field leachates than expected from batch experiments (i.e., batch leaching tests over-predicted field concentrations of heavy metals).

4.1.2.6 Modelling of the Source Term for a Predominantly Inorganic Waste Landfill Using Data Obtained From Laboratory-Scale Testing, Lysimeter Studies and Pilot Scale Monitoring (van der Sloot, et al., 2002)

Van der Sloot, et al. (2002) conducted a study to identify factors controlling leachate quality in mixed (inorganic and organic) waste. The study evaluated changes in leachate quality with time as a result of interaction and degradation by applying analysis and testing at different scales - laboratory, lysimeters, and pilot scale. Modeling was conducted to evaluate the behavior of the mixed waste and predict long-term leachate quality.

Various wastes were placed into a controlled pilot cell (12,000 m³) and samples of the same waste were taken for laboratory testing and to fill three lysimeters. Laboratory tests included the percolation test – PrEN 14405 and pH dependence test – PrEN 14429. Lysimeters were of the scale 1 to 1.5 m^3 . The study of waste leaching at the field (pilot cell), lysimeter and laboratory scale represented different time scales through the liquid to solid ratio to which the waste was exposed (i.e., leachate quality (mg/l) plotted as a function of L/S can be related to a time scale using the net infiltration rate and the height and density of the waste).

Van der Sloot, et al. (2002) provided graphic presentations (their Figure 2) of pH dependent leaching test results, percolation test results, leachate analysis results for the lysimeters, and leachate analysis results from the pilot cell for dissolved organic carbon (DOC) and zinc (Zn). The underlying data are not given in van der Sloot, et al., 2002. The authors found "mutual consistency of the data at laboratory, lysimeter and field scale, not only for DOC and zinc but for many other parameters" (van der Sloot, et al., 2002).

4.1.2.7 Quality Assessment of Granular Combustion Residues by a Standard Column Test: Prediction versus Reality (Janssen-Jurkovieova, et al., 1994)

Janssen-Jurkovieova, et al. (1994) evaluated the Dutch Standardization Institute draft standards NEN 7343 (column test) and NEN 7341 (availability test). The column test was designed to simulate leaching of granular materials over a medium-range period of about 30 years. The availability test was designed to determine under natural conditions the maximum amount of elements available from a material for leaching in the very long term under extreme conditions. One objective of the study was to "determine the degree of correspondence between leaching as simulated in the column test and leaching occurring in reality" (Janssen-Jurkovieova, et al., 1994).

The results as presented in the study show that there is a great discrepancy between the leaching of elements in the column test and in a natural environment. Some elements (aluminum, iron, potassium) were less leachable in the column test than in the field lysimeter, while others (calcium, magnesium, molybdenum, sodium, silicon, sulfer) were more leachable in the column test compared to the field lysimeter. Janssen-Jurkovieova, et al. (1994) stated that the time factor was the principal cause of the discrepancy between leaching in the column test and under field conditions. That is, the duration of a standard leaching test is far too short for certain aging processes (such as humification, weathering, and the formation of secondary minerals) to occur that influence leaching. The authors concluded that column test leaching results at various L/S ratios are not sufficient to relate the column test to a time scale under field conditions.

Janssen-Jurkovieova, et al. also concluded that the availability test (NEN 7341) is not adequate to determine the maximum amount of elements available from the ash for leaching. This conclusion was based on the finding that greater amounts of certain elements (arsenic, selenium, and vanadium) were leached from the ash by the column and cascade test than was initially leachable by the availability test, whereas the opposite occurs in nature.

To be able to predict the leaching of ecologically significant trace elements such as As, chromium, selenium, and molybdenum, the authors stated that it is important to be able to accurately predict the leaching behavior of the macro-elements (Si, aluminum, iron, calcium). The authors proposed the development of a quantitative predictive model that accounts for the effects of aging, dissolution kinetics, and adsorption and desorption that can be used for analysis of the correspondence between leaching tests and leaching under field conditions.

4.1.2.8 Leaching of Slags and Ashes – Controlling Factors in Field Experiments Versus in Laboratory Tests (Fällman and Hartlén, 1994)

A study reported by Fällman and Hartlén (1994) focused on the leaching behavior of potentially secondary materials used in construction and compared results of laboratory leaching tests with field lysimeter results. The paper summarized a one-year study on blast furnace slag (BF slag), steel slag, sorted municipal solid waste incineration bottom ash (MSWI BA), and wood ash. The comparison of laboratory leaching experiments with the composition of field leachates focused on the differences in the values of the controlling factors pH, redox and flow and the consequences of these differences for the leachate composition.

Field lysimeters were constructed at the Swedish Geotechnical Institute (SGI) in 1992 and 1993. A total of four lysimeters were constructed, one for each residue. Leachate samples were collected from the field lysimeters as closely as possible to predefined liquid to solid ratios of L/S=0.001, 0.003, 0.01, 0.03, 0.1, 0.3, and 1.0.

Samples of the materials were tested in the laboratory by availability and column tests:

• An availability test was used following a modification of the Dutch draft standard NEN 7341. The test was a two-step batch test with L/S ratio of 100. In the first step, pH is

held constant at 7 for three hours. In the second step, pH is held constant at 4 for four hours by addition of nitric acid.

- An oxidized availability test was used to examine the redox dependency of the leaching process and to estimate the potential for leaching under oxidized conditions. The test is the same as the ordinary availability test but with the addition of peroxide.
- The column tests were conducted on materials crushed to <20 mm under up-flow water saturated conditions at a constant rate of L/S=0.1/day and run until L/S=4 was reached. As leachate, synthetic rainwater was used, consisting of demineralized water acidified with nitric acid to an initial pH of 4. Samples were removed at L/S=0.1, 0.3, 0.7, 1.0, 2.0, and 4.0.
- pH static tests were carried out with a L/S=5 and at pH 4, 6, 8, 10, and 12 for 24 hours.

The leachates were analyzed for pH, redox, conductivity, total dissolved solids, metals, and salts. Total composition analyses also were conducted on materials used in the lysimeters.

The leachate composition of the column tests and lysimeters could not be directly compared because none of the lysimeters had produced enough percolate such that their L/S ratio overlapped. Instead, the authors extrapolated the curve of accumulated amounts from the lysimeter for comparison to values obtained from the column tests.

Leached amounts from the MSWI BA lysimeter and the corresponding column test were generally in agreement, which was expected due to the similar pH and redox conditions observed. However, for the other wastes, extrapolations showed a 10 to more than100 times difference between accumulated leached amounts in the lysimeters compared to the column tests.

Significant differences in pH and redox potential were obtained in field experiments in comparison with values obtained in laboratory column tests due primarily to different controlling conditions in the field compared to laboratory tests. Specifically,

- The pH of the lysimeter leachates was much lower than the column tests for the two slags and the wood ash. This was attributable to the exposure of the material to oxygen and CO₂ (carbon dioxide) and oxidation of sulfur (in the BF slag).
- All lysimeter results showed a trend toward a strong reducing condition over time
- Arsenic, cadmium, chromium, copper, and lead were more soluble under oxidizing conditions
- A reduction in pH was shown to increase the solubility of most metals.

The authors concluded that it should be possible to use laboratory tests for prediction of field behavior if the laboratory tests can be designed to reflect the parameters influencing leaching in the field, such as pH and redox.

4.1.2.9 Leachate From Land Disposal of Coal Fly Ash (Hjelmar, 1990)

A paper by Hjelmar (1990), as also described in Hansen, et al. (2000), presents the results of a large-scale lysimeter test in which two pulverized coal fly ashes were exposed to natural weather conditions for up to seven years and compared with those of laboratory tests. Ash "HF1" was a nearly neutral ash and ash "BF2" was a highly alkaline ash. The ashes were moisture conditioned (15 to 20 percent water) prior to placement in ten large-scale lysimeters (with surface areas between 9 and 20 m²).

Laboratory column leaching tests were performed on both ashes and multiple batch leaching tests were performed on fly ash HF1. The leachates from the lysimeters and the laboratory experiments were characterized for pH, conductivity, alkalinity and concentrations of sulfate, fluoride, chloride, nitrate, nitrite, ammonium, phosphate, B, sodium, potassium, calcium, magnesium, arsenic, barium, cadmium, cobalt, chromium, copper, mercury, molybdenum, nickel, lead, selenium, vanadium, and zinc.

The results were interpreted in terms of concentrations of contaminants as functions of the L/S ratio and accumulated release of the contaminants as function of L/S. The results of the accelerated leaching test correlated well with the results of the large-scale lysimeter experiments. The author concluded that laboratory leaching tests are useful tools for predicting the composition of leachate and its variation with time at planned or existing coal fly ash landfill sites (Hjelmar, 1990 and Hansen, et al., 2000).

4.1.2.10 Comparison of Leachate Quality in Foundry Waste Landfills to Leach Test Results (Ham, Boyle, and Blaha, 1986 and Ham, et al., 1986)

Studies of leachate from foundry waste landfills in Wisconsin were report by Ham, Boyle, and Blaha (1986) and Ham, et al. (1986). Foundry process wastes typically consist of molding sands, cores, slags, and various dusts or sludges from air pollution control equipment. In general, the majority of these wastes resemble fine sand.

Ham, Boyle, and Blaha (1986) and Ham, et al. (1986) reported on batch leach testing conducted on auger samples obtained from foundry waste landfills and compared these results to median pore water concentrations obtained from suction lysimeters installed within the landfills. The waste leaching tests included the EPA Extraction Procedure (EP) test and a modification of the EP test in which deionized water is substituted for the pH-5 acetic acid (referred to as the "EP (water) test").

The study found that the EP tests conducted on auger samples successfully predicted what contaminants would be present in the lysimeter pore waters for 84 percent of the samples tested. Tests conducted using the EP (water) test were nearly equally successful at predicting median pore water concentrations (at 83 percent of the samples). The success of either of these test methods to predict pore water concentrations was somewhat less when the laboratory tests were performed on samples composited from waste generated by the foundries.

Ham, Boyle, and Blaha (1986) and Ham, et al. (1986) noted that while the overall success of the EP and EP (water) tests were about the same, the EP (water) method was better at predicting the presence of cadmium and lead while the EP was better at predicting the presence of manganese, zinc, and copper.

4.1.2.11 Environmental Performance Assessment of Coal Combustion Byproducts Use Sites: Road Construction Applications (EPRI, 1995)

EPRI (1995) reported on investigations performed at five road construction sites where fly ash or scrubber ash was used. Investigations were performed at road construction sites in Rincon, Georgia; Pine Bluff, Arkansas; Russellton, Pennsylvania; Wichita, Kansas; and Joseph City, Arizona. The sites were selected to represent a range of climates and hydrogeologic settings. The objective was to gather data for a preliminary assessment of leaching and migration of chemicals in soil, groundwater, and vegetation. In the detailed sampling investigation, ash, soil, vegetation, and ground water samples were collected (at four out of five sites) for analysis. Samples of ash materials also were evaluated by the TCLP and a "saturated paste" method (a method used to determine the availability of plant nutrients). The saturated paste method is believed to be a good representation of field pore water concentrations.

Comparison of field leachate (pore water) concentrations to laboratory leaching tests was not a specified objective of the study, however, study results allow for such a comparison if the extracts from the saturated paste method are considered representative of pore water.

The ratio of the TCLP analysis result to the corresponding saturation extract analysis results provides a simple indicator of whether the TCLP overestimated the pore water concentration (ratio > 1) or underestimated the pore water concentration (ratio < 1) (see **Table 4-3**). For those constituents that were detected in both the soil paste sample and the TCLP, the ratios indicate that the TCLP consistently overestimated the concentration of barium and consistently underestimated the concentration of selenium. The results for other metals (arsenic, chromium, and lead) were mixed but reasonably accurate (differing by no more than one order of magnitude). However, due to the small number of observations these results should not be viewed as representative of all coal combustion byproducts used in road construction applications.

Sites/Test Media		Constituents						
		Ba	As	Cr	Pb	Se		
Arizona	TCLP	2,800			6.5	5		
	Soil Paste	570			3.1	167		
	TCLP/Soil Paste	4.91			2.1	0.03		
Georgia	TCLP	2,800	25		7	12		
	Soil Paste	160	11.2		14	60		
	TCLP/Soil Paste	17.5	2.23		0.5	0.2		
Arkansas	TCLP	600	1.8	230		16		
	Soil Paste	280	2.3	1,200		40		
	TCLP/Soil Paste	2.14	0.78	0.19		0.4		
Pennsylvania	TCLP	880	12.5		6	13		
	Soil Paste	30	5.9		0.9	18		
	TCLP/Soil Paste	29.3	2.12		6.67	0.72		
Kansas	TCLP	1,750						
	Soil Paste	176						
	TCLP/Soil Paste	9.94						

Table 4-3. TCLP and Soil Paste Extract Sample Analysis Results for Coal Combustion Byproducts Used at Road Construction Sites

Concentration units are µg/l.

Empty cells indicate constituent was either not detected in both TCLP and Soil Paste samples or not analyzed.

4.1.2.12 Comparison of Laboratory Batch Methods and Large Columns for Evaluating Leachate from Monofilled Solid Wastes (Jackson and Bisson, 1990)

A study conducted by Jackson and Bisson (1990) compared the concentrations of selected metals in leachates from laboratory batch leaching procedures and large column tests. Industrial solid wastes used in this study included electroplating sludge, electric arc furnace dust, paint incinerator ash, MSW incinerator ash and mine tailings. Aliquots of each waste were subjected to hydraulic conductivity and bulk density determinations as well as total elemental analysis.

The column leaching tests were conducted using large high-density polyethylene tanks and low volumes of water relative to the mass of solid waste to ensure realistic static leaching conditions. Air-dried waste samples were packed and compacted into the tanks to the approximate bulk density determined for each waste and then leached with demineralized water at approximately biweekly intervals. The tank leachates were analyzed for pH, electrical conductivity (EC), aluminum, barium, beryllium, calcium, chromium, iron, potassium, magnesium, sodium, and nickel.

Batch extraction procedures were conducted in duplicate on each of the five wastes used in the column studies. These procedures included the Monofilled Waste Extraction Procedure (MWEP), the U.S. EPA Extraction Procedure (EP), Acetate Buffer Extraction Procedure

(ABEP), the Ham Procedure C (Ham C), and the Saturated Paste Procedure (Paste). Three of these procedures (MWEP, Ham C and Paste) use deionized water as the leaching fluid, while the other two (EP and ABEP) employ a leaching fluid containing acetic acid or sodium acetate buffer. The leachates obtained from the batch experiments were analyzed for pH, EC, chromium, nickel, lead and cadmium.

Leached amounts from the large column and batch extraction procedures were compared on the basis of total metals (mg) extracted or leached per kilogram of wastes. In general, analyte concentrations in column leachates "were related qualitatively to those found in extracts from batch procedures which used deionized water as the extraction medium (MWEP, HAM-C, and PASTE)" (Jackson and Bisson, 1990). No statistical analysis of the data was performed and none of the deionized water extraction tests consistently reflected the analyte concentrations found in the column leachates. On the other hand, the analyte concentrations in the EP and ABEP leachates differed considerably from those found in the column leachates. The authors concluded that "batch extraction procedures that employ acetic acid or acetate buffer are less effective for assessing the leachability of monofilled wastes than extraction methods that use de-ionized water as the extraction fluid" (Jackson and Bisson, 1990).

4.1.2.13 Verification of Laboratory-Field Leaching Behavior of Coal Fly Ash and MSWI Bottom Ash as a Road-based Material (Schreurs, et al., 1997)

Schreurs, et al. (1997) reported on a study conducted to determine the extent to which estimates based on laboratory leaching data can be correlated with field measurements of leachate composition for secondary materials used in road-based applications. The study focused on four road-based applications that employed coal fly ash and MSWI bottom ash as stabilization materials.

To estimate constituent releases based on laboratory leaching results, the authors followed the guidelines presented in the Dutch Building Materials Decree. This document specifies formulas for calculating constituent releases as well as correction factors to account for temperature differences between lab and field, degree of contact with water under field conditions and exposure time. Since no leaching data for the specific materials studied were available, leaching data obtained for similar materials in previous studies were employed to calculate releases. For three of the four applications, a percolation-controlled system was believed to be the dominant leaching mechanism and the corresponding model was employed to estimate constituent release (E_p) .

Constituent release under field conditions was determined by: 1) measuring the concentration decrease of the constituent in the secondary material itself (E_I); 2) determining the concentration increase in the underlying soil and/or collected percolate (E_{II}) or 3) measuring the difference in constituent leaching between the fresh material and the field-exposed or aged material (E_{III}). Concentration profiles were established for each core sample (construction material and underlying sand layer) obtained from the four road-based applications studied (10 cores per application) and used to determine constituent release from the secondary material in the exposure period (10 years).

The authors reported great variability in measured concentrations in the core samples (up to 200% for some elements) as well as large uncertainties for the calculated releases based on laboratory data. Due to these large variations in measured and predicted release values, only a qualitative comparison of the results was possible. For most of the constituents evaluated, the results indicated a good to very good agreement (defined as a difference of less than a factor of 2) between predictions based on laboratory tests and field data. For certain mobile constituents (e.g., B, chromium, molybdenum, vanadium and SO₄), there was a poor correlation between the measured release E_{II} and the estimated release. For these constituents, the level of agreement improved when E_{II} was corrected for constituent losses to ground water. A discrepancy between E_{II} and the estimated release under a diffusion-controlled scenario (E_d) was also observed in the study. The authors attributed the lower value of E_{II} to a diffusion resistance effect at the interface between the material and the unsaturated sand layer, which was not accounted for in the predictive model, and concluded that the E_{II} measurement is the most reliable determination of actual release in this case.

The authors provided the following additional conclusions and recommendations regarding the use of predictive models and field data to estimate or measure constituent releases:

- In a percolation-controlled system, E_{II} (determination of constituent release based on the concentration increase in the underlying soil) is the most appropriate methodology for determining releases of elements that sorb strongly to soil. For diffusion-controlled scenarios, E_{II} is appropriate for all constituents.
- For mobile constituents, E_{III} (constituent release measured by the difference in leaching between fresh and aged material) or E_{II} corrected for losses to ground water provide a good measurement of constituent release.
- Releases based on concentration decreases (E_I) are only useful in the case of homogenous materials.
- For some materials, pH decreases considerably over time due to the release of mobile pH controlling species and/or carbonation. Predicted releases based on laboratory tests (which employ a high pH) can differ significantly from the actual release under field conditions.

4.1.2.14 Characterization of Municipal Waste Combustion Ash, Ash Extracts, and Leachates (USEPA, 1990)

The USEPA and the Coalition on Resource Recovery and the Environment (CORRE) cosponsored a study (USEPA, 1990c), conducted by NUS Corporation, to enhance the database on the characteristics of municipal waste combustion (MWC) ashes, laboratory extracts of MWC ashes, and leachates generated from the land disposal of MWC ash.

To conduct the study, NUS collected combined bottom and fly ash samples from five mass-burn MWC facilities and leachate samples from the corresponding ash disposal facilities. The ash and leachate samples were analyzed for various semivolatile compounds, PCBs, dioxins and furans,

metals, and several miscellaneous conventional compounds. The ash samples were subjected to six laboratory extraction procedures (leaching tests) and the extracts were then analyzed for the same compounds as the ash samples. The following six extraction procedures were used during the study:

- EP-TOX (Extraction Procedure): Uses an L/S ratio of 20:1, 0.5N acetic acid, pH control at 5, extraction time of 24 hours, tumbler agitation, particle size of <9.5 mm, single extraction
- TCLP (Toxicity Characteristic Leaching Procedure): Used an L/S ratio of 20:1, 0.1N acetate buffer, pH control at 5, extraction time of 18 hours, tumbler agitation, particle size of <9.5 mm, single extraction
- Method SW-924 (Monofill Waste Extraction Procedure (MWEP)): Uses an L/S of 10:1 per extraction, distilled/deionized water, no pH control, extraction time of 18 hours, tumbler agitation, particle size of <9.5 mm, up to 4 sequential extractions.
- CO₂ saturated deionized water: Method description not available. (Ecology assumes the method is a modification of the TCLP with a L/S ratio of 20:1, a slightly acidic eluant created by bubbling CO₂ through distilled, deionized water.
- Simulated acid rain (SAR): Method description was not available.

The study found that leaching test extracts from the EP-TOX and TCLP contained generally higher concentrations of metals than the extracts from the other leaching test methods (SW-924, CO₂, and SAR). Extracts from the SW-924, CO₂, and SAR extraction procedures simulated field concentrations of lead and cadmium better than the other three procedures. Few organics were detected in the field leachates and leach test extracts. None of the extracts contained dioxins/furans, confirming that these organics are not readily leached from ash.

4.1.2.15 Concentrations of Hazardous Constituents in Field Leachates from Treated/Land Disposed K088 Hazardous Waste Compared to Concentrations Predicted by the TCLP (USEPA, 1997)

In 1988, the United Stated Environmental Protection Agency (EPA) listed waste generated from the primary reduction of aluminum ("pot liner") as a hazardous waste (K088) under the Resource Conservation and Recovery Act (RCRA). In 1996, EPA proposed restrictions on the land disposal of K088 in the Phase III Land Disposal Restriction rule (61 FR 15566, April 8, 1996). The Phase III LDR prohibited the land disposal of spent potliner unless the waste satisfied EPA's treatment standard for the waste. The treatment standards were set based on TCLP analysis of waste treated using the best available technology. Thus, compliance with the treatment standard was to be measured via TCLP analysis. Standards were established for 25 constituents, including arsenic, cyanide, fluoride, toxic metals, and polycyclic aromatic hydrocarbons.

Subsequent to the establishment of the treatment standards, EPA became aware of actual sampling data from the Reynolds Aluminum monofill at Gum Springs, Arkansas containing

treated K088 (USEPA, 1997). Data characterizing leachate from the leachate collection system showed high concentrations of hazardous constituents in the leachate, exceeding the maximum predicted by the TCLP, the analytical test used to measure performance of the treatment technology for certain hazardous constituents in K088. A comparison of these data is given in **Table 4-4**.

Table 4-4. Comparison of Monofill Leachate Concentrations to Maximum Concentrations
Predicted by the TCLP for Treated/Land Disposed K088 (Gum Springs, Arkansas)

Constituent	Maximum Predicted in Treated K088 by TCLP (mg/L)	Concentrations Found in K088 Monofill Leachate (mg/L)
Arsenic	5.0	6.55 15.7 21.6
Fluoride	48	2,228

EPA determined that for K088 nonwastewaters, treatment effectiveness as measured by the TCLP was not realized under field conditions. EPA noted that the TCLP uses a weakly acidic extractant, whereas actual disposal conditions for K088 are highly alkaline (due to the potliner's alkalinity), and arsenic is more soluble under highly alkaline than weakly acidic conditions. (See 62 FR 1993, January 14, 1997). Specifically, the TCLP uses a weakly acidic extraction fluid (pH 5.0) that, when applied to the alkaline K088 waste, results in a leachate pH of approximately 7.6 and not the observed landfill pH of approximately 12.5, at which arsenic is highly mobile.

In response to judicial review of the LDR standards, the United States Court of Appeals for the District of Columbia Circuit in 1998 vacated the treatment standards of K088 waste indicating that EPA's use of the TCLP as a basis for setting treatment standards for K088 was arbitrary and capricious. In response, EPA promulgated interim replacement standards for K088 (63 FR 51254, September 24, 1998) set at 26.1 mg/kg total (not TCLP) arsenic based on data from an arsenic immobilization technology. In addition, EPA has proposed (USEPA, 2000) testing of fluoride concentrations in K088 nonwastewaters using a version of the TCLP with de-ionized water as the leachate fluid (i.e., ASTM Method D3987–85 (1999a)). With de-ionized water as the leachate test fluid, leachate pH is controlled by the physical properties of the waste (and not the artificial buffering capacity of the test fluid), and more closely correlates with monofill conditions.

4.1.2.16 Environmental Effects of Dredging, Technical Notes - A preliminary Evaluation of Contaminant Release at the Point of Dredging (Havis, 1988)

A study conducted by the U.S. Army Engineer Waterways Experiment Station (Havis, 1988) evaluated the use of the Standard Elutriate Test (SET) as a tool for predicting contaminant release resulting from dredging operations. Specifically, the study focused on determining whether the SET was an adequate test to predict the release of contaminants at the point of dredging. For this purpose, the results of chemical analyses of SET elutriates were compared to dissolved chemical concentrations measured in the water column near the point of dredging.

Water-column samples were collected at four dredging sites. The samples were taken near the bottom of the water column in the vicinity of the dredge and analyzed for dissolved chemical constituents. Sediment samples for SET testing were collected from each of the dredging sites prior to beginning of dredging operations.

The authors concluded that, for most constituents, the SET was a conservative predictor of dissolved concentrations in the water column at the point of dredging in the four sites evaluated. In general, SET results were consistently higher, within an order of magnitude, than the water-column measurements for most constituents. However, SET underpredicted the water-column concentrations of zinc and lead at one site and overestimated concentrations of mercury, manganese, iron, total Kjeldahl nitrogen (TKN), and ammonia at another. The authors noted that modifications to the procedure, such as reducing the solids-to-water ratio, could lead to more accurate predictions of constituent concentrations, especially for hydrophobic chemicals and TKN.

4.1.2.17 Sediment and Water Quality Evaluation Framework for Disposal of Dredged Material in an Upland Rehandling Facility, Portland, Oregon (Thornburg, et al., 2002)

Thornburg, et al. (2002) reported on the development of a tiered evaluation framework for maintenance dredging materials to be disposed of at an upland rehandling facility. The framework, proposed by the Port of Portland in consultation with dredged material management agencies, consists of a decision matrix to streamline the characterization of dredging spoils destined for upland rehandling or disposal. It specifies an initial screening criteria for bulk sediments and includes the use of dredge elutriate (modified elutriate test or MET) and leaching protocols (SPLP), as necessary. Under this framework, initial evaluation of the dredge material is accomplished by bulk analysis of the sediment. If results of sediment bulk analysis exceed criteria (i.e., dredge material screening levels or SLs) either elutriate or leachate testing is performed on the material.

In the development of the framework, dredging spoils generated by two marine terminals of the Port of Portland were evaluated for disposal at the rehandling facility. This facility consisted of a primary basin for offloading of dredged material and a secondary basin for settling and clarification of the dredging elutriate decanted from the primary basin. Three potential pathways for contaminant release at the rehandling facility were identified and investigated in this study:

- Discharge of dredged material effluent to the river through the effluent return flow of the rehandling facility
- Seepage of dredged material leachate under the disposal facility berm and into the river
- Infiltration of leachate into the subsurface soil and underlying aquifer.

Decision criteria and testing requirements were specified for each of the three pathways. Contaminant releases from the dredging materials were evaluated under each of the three pathways through testing protocols, which included bulk sediment chemistry analysis, the modified elutriate test (MET), the column-settling test (CST) and the SPLP. The modified elutriate test was conducted using a one-day settling time and a seven-day settling time to bracket the range of detention times at the rehandling facility. Bulk sediment, elutriate and SPLP leachate were analyzed for priority pollutants, tributyltin (TBT), chlorinated pesticides, PCBs, and semivolatile organics (including PAHs, phenols, phthalates, chlorobenzenes, and other extractables).

Field samples, collected at the point of discharge to the river at the rehandling facility, were analyzed and compared to the one-day and seven-day MET. In general, a good agreement between the effluent concentrations predicted by the one-day and seven-day MET and actual concentrations in the field samples was observed. The MET successfully predicted dissolved metal concentrations in the field elutriates. In addition, TBT concentrations in field samples were well predicted by the MET, although higher concentrations were observed in the field. Chlorinated pesticides, PCBs, and most PAHs were not detected in either of the two MET tests nor were they detected in the field samples. The authors concluded that the MET "accurately predicted effluent water quality" at the Port of Portland's rehandling facility. No comparison of SPLP results and field data was reported.

4.2 Techniques for Evaluating Leaching Test Results and Predicting Long-Term Release

Each class of leaching test is designed to address some specific aspect of leaching. The selection of one or more leaching tests, the manner in which the test(s) is (are) applied, the form in which the result is expressed, and the approach used to evaluate the result must consider the scientific, quality, and regulatory objectives. Ultimately, leaching tests are used to assess leaching behavior of waste, soil, or other materials for regulatory purposes or to predict long-term leaching behavior (e.g., to support risk assessment). Regulatory objectives may include waste classification (e.g., hazardous waste determination), assessment of waste treatment effectiveness, evaluation of management options for wastes or soil, and site characterization and cleanup. Leaching test results are used as source terms in fate and transport models to support human health and ecological risk assessments (e.g., to support both site-specific studies and regulatory development).

Section 4.2.1 describes the sources of variability in leaching test results. Section 4.2.2 describes the outputs of various classes of leaching tests. Section 4.2.3 describes techniques for evaluating leaching test results generated for regulatory purposes. Section 4.2.4 provides an overview of how leaching test results are used as inputs to fate and transport models to support risk assessment and waste management unit design. Section 4.2.5 describes how leaching tests can be selected and the results interpreted using a scenario-based framework. A scenario-based framework employs a suite of leaching tests used in a tiered manner to assess leaching behavior considering material- and site-specific conditions. The approach responds to a growing consensus among researchers that assessing the leaching behavior of a wide variety of materials in a broad range of management scenarios cannot be addressed adequately by one single laboratory leaching test (van der Sloot, 1990; van der Sloot, et al., 1997; Kosson, et al., 2002).

4.2.1 Sources of Variability and Bias in Leaching Test Results

When leaching tests are required to make a decision regarding the use or management of waste, soil, or other material, a data collection process is employed using a series of steps that include planning, sample collection, laboratory analysis, and data assessment. The output of the data collection process then is used to make a decision regarding the management or use of the material. However, as with any sampling and analysis effort, there will be uncertainty in the results of leaching tests, and this measurement uncertainty causes decision uncertainty. Measurement uncertainty is due to factors such as random variability and bias in the sampling and analysis processes. Random variability (measured as variances) and bias (a systematic shift away from the true value) are caused by the inherent heterogeneity of the material and the imprecision and biases of the methods used to collect and analyze samples of the material. Variability and bias collectively are known as "error."

Many factors contribute to the overall error or uncertainty associated with leaching test results. Some of the most influential of these include the following:

- *Inherent heterogeneity of the material*. The greater the heterogeneity of the material (over time and/or space), the greater will be the sample-to-sample variability.
- *Physical size, shape, and orientation of each sample* (known as the "sample support"). The sample support will have an influence on the sample analysis result. Sampling theory (Pitard, 1993; Gy, 1982 and 1998) suggests there is a particle-size sample-mass relationship that controls the degree of error associated with sampling such that larger samples result in smaller sampling error. Particle-size reduction, followed by subsampling, can be used to reduce large field samples to the mass required for laboratory analysis and to control sampling error.
- *Sampling collection bias.* Sampling bias can be introduced due to the use of incorrectly designed sampling devices and due to the loss or gain of constituents during sampling, transport, storage, subsampling, and sample preservation.
- Sample preparation/leaching test bias. Smaller particle sizes produce larger surface areas allowing for increased contact between the solid material and the leaching fluid resulting in increased release of constituents. Many leaching tests (such as the SPLP and TCLP) specify a maximum particle size but do not specify a lower limit on particle size. Thus, using different particle sizes, and an otherwise identical leaching methodology, to evaluate a waste could yield very different results.
- *Analytical error*. Sample analysis errors could occur, for example, due to instrument calibration drift, instrument contamination, or unaccounted for interference.

Van der Sloot, et al. (1997) observed that, since leaching from many materials is controlled by solubility, sensitivity to sampling errors in leach testing is less than will be observed when total composition is measured. Van der Sloot, et al. (1997) state that the major factors controlling the pore water conditions that are responsible for the solubility control of trace constituents are not likely affected by sampling errors. They note that a more critical source of error is related to the final pH of the leachate. For example, a minor change in final pH can result in a relatively large change in analyte concentrations in the leachate (Venner and Lowery, 2002), resulting in errors of 50 to 100 percent (van der Sloot, et al. 1997). Moreover, because the result of a leaching test is solubility-controlled, the concentration in the test leachate will be bounded on the high end by the solubility limit, not necessarily linearly related to the total content of the constituent. Venner and Lowry (2002) argue that due to the non-linear property of the TCLP it is incorrect to use the average of multiple TCLP analysis results to determine the regulatory status of a waste.

4.2.2 Outputs of Leaching Tests

Leaching test results typically are expressed as leachate concentrations in mg/L for comparison to regulatory thresholds and to study the solubility of constituents in different leaching solutions (e.g., at various pH conditions). Leaching test results also may be expressed as constituent released (mg/kg), where "release" is defined as the mass of contaminant dissolved divided by the mass of material subjected to leaching (van der Sloot, et al., 1997). Mass release data are necessary for comparison of data obtained at different liquid-to-solid (L/S) ratios and for determination of availability (van der Sloot, et al., 1997). Availability refers to the maximum potential for release under anticipated environmental conditions, without consideration of the time frame for release to occur (Kosson, et al., 2002).

In general, batch tests such as the SPLP and TCLP use a relatively high L/S ratio that corresponds to a relatively long timeframe (3 to 10 years) of leaching (Kimmell and Friedman, 1986). Column tests can be run at much lower L/S ratios (<1) thus providing a means to evaluate leaching under conditions approaching field conditions.

For "flow-around" leaching tests performed on monolithic or compacted granular materials (e.g., NEN 7345 Tank Leach Test), the test results are expressed in cumulative mass released on a surface area basis (e.g., mg/m^2). This class of leaching tests measures releases that occur at the exterior boundary of the material.

Table 4-5 provides a summary of the outputs of the various leaching tests identified in Section 3.2 and excerpts from each method regarding interpretation of the results.

Table 4-5.	Outputs of	Common	Leaching Tests
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Leaching Test	Form of Resulting Data	Evaluation/Interpretation of Results (as specified in the method)
ASTM D 3987-85 - Standard Method for Shake Extraction of Solid Waste with Water	Generally expressed as mg/L or µg/L. Method does not specify units.	The procedure is "not intended to provide an extract that is representative of the actual leachate produced from a waste in the field or to produce extracts to be used as the sole basis of engineering design."
ASTM D 6234-98 - Standard Test Method for Shake Extraction of Mining Waste by the Synthetic Precipitation Leaching Procedure	Generally expressed as mg/L or µg/L. Method does not specify units.	The procedure is "not intended to provide an extract that is representative of the actual leachate produced from a waste in the field or to produce extracts to be used as the sole basis of engineering design." The procedure has "not been demonstrated to simulate actual disposal site leaching conditions."
Synthetic Precipitation Leaching Procedure (SPLP) 1312	Generally reported in mg/L	The method indicates that the user should "compare the analyte concentrations in the 1312 extract with the levels identified in the appropriate regulations." There are no Federal regulations that require use of the SPLP.
Toxicity Characteristic Leaching Procedure (TCLP) 1311	Generally reported in mg/L	The method indicates that the user should "compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations."
Dredge Elutriate Test (DRET)	Generally expressed as mg/L or µg/L. Method does not specify units.	No information provided in the method.
Standard Elutriate Test (SET)	Generally expressed as mg/L or µg/L. Method does not specify units.	No information provided in the method.
NEN 7341, Availability Test	Release in mg/kg	Total availability is obtained by multiplying the test result (mg/L) by the test L/S ratio (L/kg)

Table 4-5. Outputs of Common Leaching Tests

Leaching Test	Form of Resulting Data	Evaluation/Interpretation of Results (as specified in the method)
EN 12457/1-4, Compliance Test for Granular Waste Materials and Sludges (European Union)	Release in mg/kg	Measure release at specified L/S ratios for comparison to regulatory level (European Union).
ASTM D 4793-93 Standard Method for Sequential Batch Extraction of Waste with Water	mg/L (as indicated in the method)	The procedure is "not intended to provide an extract that is representative of the actual leachate produced from a waste in the field or to produce extracts to be used as the sole basis of engineering design." The procedure has "not been demonstrated to simulate actual disposal site leaching conditions."
ASTM D 5744-96, Standard Test Method for Accelerated Weathering of Solid Materials Using a Modified Humidity Cell	Effluent concentrations are expressed in μg/g (as indicated in the method) Release rates are expressed in μg/g/week (as indicated in the method)	The procedure is "intended for use to meet kinetic testing regulatory requirements for mining wastes and ores." The procedure is "not intended to provide an extract that is representative of the actual leachate produced from a waste in the field or to produce extracts to be used as the sole basis of engineering design." The procedure has "not been demonstrated to simulate actual disposal site leaching conditions." Analytical results for the effluent can be used to classify a material's tendency to produce acidic, alkaline or neutral effluent. The method includes formulas to calculate release rates for constituents of interest under the conditions of the test.
Sequential Batch Leachate Test (SBLT)	Generally expressed as mg/L, µg/L, or ng/L. Method does not specify units.	The SBLT provides two types of information: (1) Maximum leachate concentration for a particular contaminant, which may be compared to drinking water and surface water standards to provide an indication of potential leachate problems. (2) Contaminant partitioning between sediment and water - Results can be used to calculate distribution coefficient to make extrapolations when evaluating sediments of the same type with different contaminant concentrations
NEN 7345 Tank Leach Test (The Netherlands)	mg/m ²	Test provides a mechanism for evaluation of surface area-related (diffusion) release.

Table 4-5. Outputs of Common Leaching Tests

Leaching Test	Form of Resulting Data	Evaluation/Interpretation of Results (as specified in the method)
ASTM D 4874-95, Standard Test Method for Leaching Solid Material in a Column Apparatus	Generally expressed as mg/L or µg/L. Method does not specify units. Method also suggests measurement of pH, conductivity, redox, and total solids.	The procedure is "intended to provide an aqueous leaching of a material in a dynamic partitioning manner." The test "does not produce results that can be used as the sole basis for (1) engineering design of a disposal site, or (2) the characterization of wastes based on their leaching characteristics."
Pancake Column Leach Test (PCLT)	Generally expressed as mg/L or µg/L. Method does not specify units.	Test results may be used with mass transport modeling to estimate the long-term water quality impact and contaminant flux in a confined disposal site.
NEN 7343 Column Test (The Netherlands)	Results are expressed in mg/kg (as indicated in the method)	Column test results in conjunction with L/S ratios can be used to estimate "the time-dependence of the leaching of a material under practical conditions."
PrEN 14405 Upflow percolation test (European Union)	Plot leachate concentration (e.g., mg/L or mol/L) as a function of L/S ratio.	To determine the rate of contaminant leaching as a function of liquid to solid ratio, particularly at the low L/S ratios prevailing in disposal scenarios.

4.2.3 Evaluating Leaching Test Results Against Regulatory Standards

Federal regulations promulgated under the Resource Conservation and Recovery Act (RCRA) call for waste handlers to conduct sampling and analysis of waste to make a hazardous waste determination, determine if a waste is subject to treatment and, if so, if the waste has been adequately treated under the Land Disposal Restrictions (LDR) program. In addition, some states require sampling and analysis of soils to determine compliance with soil cleanup programs and some states have beneficial re-use programs that require sampling and analysis. These programs involve the use of leaching tests to measure compliance with the regulatory standards. The following sections summarize how these tests are used and how the results are evaluated.

4.2.3.1 Hazardous Waste Determination

Federal regulations (40 CFR § 261.3) define hazardous waste in two ways. First, solid wastes are hazardous wastes if the U.S. EPA lists them as hazardous wastes in 40 CFR Part 261, Subpart D. Second, EPA regulations at 40 CFR § 261.10 also identify wastes as hazardous if they exhibit any of the following four characteristics of a hazardous waste: ignitability, corrosivity, reactivity, or toxicity (based on the results of the Toxicity Characteristic Leaching Procedure, or TCLP). Washington State's Dangerous Waste Regulations (WAC 173-303) use similar criteria to define hazardous wastes.

When a waste handler conducts testing to determine if the waste exhibits a hazardous waste characteristic, he or she must obtain a representative sample (within the meaning of a representative sample given at § 260.10) using the applicable sampling method specified in Appendix I of Part 261 or alternative method (per § 261.20(c)). The waste handler must test the representative sample for the hazardous waste characteristics of interest.

When a waste is tested for the toxicity characteristic, the regulations at 40 CFR § 261.24 (and at WAC 173-303-090(8)(c)) indicate that the TCLP analysis results (in mg/L) must be compared to the concentration limits in the table of "Maximum Concentration of Contaminants for the Toxicity Characteristics." If the TCLP analysis result contains any of the contaminants listed in the table at the concentration equal to or greater than the respective value given in that table, then the waste exhibits the toxicity characteristic.

Note that the regulations for the hazardous waste characteristics do not specify the number of samples required, nor do they require the use of any statistical test to interpret the sample analysis results. Historical guidance on sampling and data evaluation is provided in Chapter Nine (September 1986) of EPA's *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, EPA Publication SW-846. The guidance advises users to collect a number of samples (determined using a sample size equation that accounts for the expected variability, desired margin of error, and desired confidence level), analyze the samples for the constituent(s) of interest, and compare the upper confidence limit on the mean of the sample analysis results to the TC regulatory threshold. If the upper confidence limit on the mean is less than the applicable regulatory threshold, then the waste is considered nonhazardous. Otherwise, the opposite conclusion must be reached.

In 2002, EPA published new sampling and data assessment guidance to update the information provided in Chapter Nine of SW-846. The new guidance, entitled the *RCRA Waste Sampling Draft Technical Guidance* (USEPA, 2002), recognizes that the toxicity characteristic regulatory levels specified in Table 1 of 40 CFR § 261.24 represent "maximum" concentrations that cannot be equaled or exceeded; otherwise, the solid waste must be classified as hazardous. The guidance suggests that the maximum sample analysis results be used to make a hazardous waste determination. This guidance is based, in part, on the notion that RCRA does not allow for *de minimis* amounts of a hazardous waste to fall outside of the RCRA subtitle C (hazardous waste) regulations, thus even a single sample of a solid waste exhibiting a hazardous waste characteristic is indicative of hazardous waste.

4.2.3.2 Land Disposal Restrictions Treatment Standards

The LDR program regulations found at 40 CFR § 268 require that a hazardous waste generator determine if the waste has to be treated before it can be land disposed. This is done by determining if the hazardous waste meets the applicable treatment standards at § 268.40, § 268.45, or §268.49. EPA expresses treatment standards either as required treatment technologies that must be applied to the waste or as contaminant concentration levels that must be met. For most nonwastewaters, the treatment standards are expressed in mg/L, TCLP.

Determining the need for waste treatment can be made by either of two ways: testing the waste or using knowledge of the waste (see § 268.7(a)). If testing is performed, the data evaluation must show that no portion of the waste exceeds the applicable treatment standard, otherwise, there is evidence that the standard is not met (see 63 FR 28567, March 26, 1998). Importantly, statistical variability is "built in" to the LDR standards (USEPA, 1991a).

In addition to the LDR treatment standards for hazardous process wastes, EPA promulgated alternative LDR treatment standards for contaminated soils that contain a hazardous waste or exhibit a hazardous waste when generated (i.e., excavated) from remedial sites. The standards require hazardous soils destined for land disposal to be treated to reduce concentrations of hazardous constituents by 90 percent or meet hazardous constituent concentrations that are 10 times the Universal Treatment Standard (10 x UTS). If the treatment process achieves the 90percent reduction standard, or the treatment reduces constituent concentrations to levels that achieve the standard of 10 x UTS, then EPA does not require further treatment. Under 40 CFR 268.49(c), treatment for non-metals must achieve 90-percent reduction in total constituent concentrations. Treatment for metals must achieve 90-percent reduction as measured in leachate from the treated soil (testing according to the TCLP) when a metal stabilization treatment technology is used, and as measured in total constituent concentrations when a metal removal technology is used. Detailed guidance on measuring compliance with the alternative LDR treatment standards for contaminated soil can be found in *Guidance on Demonstrating* Compliance With the Land Disposal Restrictions (LDR) Alternative Soil Treatment Standards (USEPA 2001).

4.2.3.3 Waste Site Cleanup Programs

Across the nation, contamination exists at hundreds of thousands of active and inactive (e.g., abandoned) sites as a result of various industrial and government activities that resulted in releases of hazardous constituents to the environment. At many of these sites, contaminants have been released into the soil and groundwater potentially threatening human or ecological receptors, affecting property use and value, and resulting in long-term liabilities to owners or operators. The identification and cleanup of contaminated sites is addressed under various Federal and state regulatory and non-regulatory programs depending on the nature of the threat and the operating, ownership, and financial status of the site or facility. Examples of these cleanup programs include the following:

- **RCRA Corrective Action Program** addresses solid waste management units at active industrial facilities with RCRA permits.
- Brownfields a program in which States, communities, and other stakeholders in economic development work together to clean up and sustainably reuse brownfield sites. Brownfield sites are real property at which the expansion, redevelopment, or reuse may be complicated by the presence or potential presence of a hazardous substance, pollutant, or contaminant.
- **Superfund** an EPA Federal program to locate, investigate, and clean up the worst sites nationwide.
- Underground Storage Tanks (USTs) The nation's UST program is implemented by the States to cleanup more than 400,000 confirmed releases from USTs containing petroleum products and hazardous substances (USEPA, 2003a).
- Cleanup of Federal Facilities The Federal Government is engaged in cleanup activities at thousands of sites across the country contaminated with hazardous waste, unexploded ordnance, radioactive waste, fuels, and a variety of other toxic contaminants. These federal facilities include many different types of sites, such as abandoned mines, nuclear weapons production plants, fuel distribution areas, and landfills.
- State Programs Most states have some type of regulatory or non-regulatory program to address contamination problems at sites that are not addressed by Federal regulations (such as industrial waste landfills and certain special wastes). Many state programs include the use of soil cleanup standards and the use of leaching tests to ensure the soil cleanup is protective of groundwater or surface water.

A detailed description of all waste site cleanup programs is beyond the scope of this report; however, under many of these programs, establishing remediation goals and determining attainment of cleanup can involve the use of leaching tests (e.g., in connection with soil remediation). The following sections briefly outline how leaching tests can be used in (1) setting cleanup standards or remediation goals and (2) determining attainment of cleanup standards.

4.2.3.3.1 Establishing Soil Cleanup Levels Protective of Ground Water Under the Model Toxics Control Act

Site-specific cleanup level for soil that are protective of groundwater can be back-calculated from acceptable groundwater concentrations (corresponding to an acceptable level of risk) taking into consideration site-specific fate and transport parameters (see also Section 4.2.4).

Under Washington Department of Ecology Model Toxics Control Act (MTCA) Cleanup Regulation, Chapter 173-340 WAC, soil cleanup levels are established using methods that include two fate and transport models (the 3-phase and 4-phase equilibrium partitioning models), alternative fate and transport models, leaching tests, or the use of site-specific empirical data (WAC 173-340-747). If a leaching test is used, the leaching test effluent concentration must meet the criteria specific at WAC 173-340-747(7). These criteria specify that the test effluent must be less than or equal to 10 times the applicable groundwater cleanup level for cadmium, lead, and zinc at WAC 173-340-720 and less than or equal to the applicable groundwater cleanup level established for arsenic, total chromium, hexavalent chromium, copper, mercury, nickel, and selenium at WAC 173-340-720. The regulation does not specify the number of tests or statistical methods for evaluating leaching test data, nor does the regulation provide specific criteria for the use of leaching tests to evaluate other metals and organic constituents.

4.2.3.3.2 Determining Attainment of Cleanup Standards

Once site remediation activities have been completed (e.g., via removal of contaminated soil), sampling typically is required to confirm attainment of the cleanup standard. Statistical methods can be used to confirm, with a desired level of confidence, that predefined areas (e.g., exposure units) attain the standard. Where leaching of contaminants from the soil to groundwater is a concern, leaching tests can be used to confirm that leachate produced from the soil will not exceed groundwater protection or cleanup standards (such as Maximum Contaminant Levels (MCLs)).

An example of this approach is used in Texas. The Texas Commission on Environmental Quality (TCEQ) implements the Texas Risk Reduction Program (TRRP). TRRP regulates the cleanup of hazardous wastes released into the environment from regulated commercial and industrial facilities (TCEQ, 2003). Under the risk reduction standards (§§335.551 - 335.569, TCEQ, 2003), attainment of the soil-to-groundwater cross-media protection standards can be demonstrated using the Synthetic Precipitation Leaching Procedure (SPLP), SW-846 Method 1312. The demonstration must show that the concentration in soil does not produce a leachate in excess of the risk-based groundwater standard as established in the regulation. The regulation allows for the use of other leaching test methods that more accurately simulate conditions at the facility, subject to prior approval by the State.

Under the Washington Department of Ecology Model Toxics Control Act (MTCA) Cleanup Regulation (WAC 173-340), soil sampling is required to determine if the soil cleanup level has been met at the designated point of compliance. Individual soil samples may be compared directly with the cleanup level if there is documented, reliable information that the soil samples have been taken from locations where the worst soil contamination is likely to be found. Otherwise, statistical methods must be used to determine if the soils have met the cleanup level. If the leaching pathway is a concern at a site, groundwater monitoring may also be required to verify that the soil cleanup level is adequately protecting groundwater (Ecology, 2001).

4.2.3.4 Beneficial Use Determinations

Many States have regulatory or non-regulatory programs that allow for the beneficial use of nonhazardous, industrial solid wastes in lieu of disposal. A beneficial use determination may be made where it is determined that a waste or soil can be used in a manner that will present no threat to human health or the environment. A recent survey (ASTSWMO, 2000) of the States requested information on testing methods used in making beneficial use determinations, specifically regarding tests related to chemical analysis and leaching characteristics of the materials being evaluated. Of the 30 States and 2 Territories that responded to the survey, 28 States require total metals testing, 24 require total organics analyses, 24 require the TCLP, 10 require the SPLP, and 3 require ASTM Method 3987.

A comprehensive review of all state beneficial use programs is beyond the scope of this report, however, a review of several state programs that employ leaching tests in their beneficial use determinations was conducted to identify methods used to evaluate leaching test results.

Florida - Beneficial Use of Ash Generated From Waste to Energy Facilities

Under Florida statute (Florida Administrative Procedure Act, Section 403.7045(5)), the Florida Department of Environmental Protection (DEP) has authority for approving the beneficial use of ash generated from waste-to-energy (WTE) facilities. Beneficial use approvals must provide that public health is protected and applicable Department standards and criteria are not violated. WTE facility owner/operators or third party recyclers or operators who wish to beneficially use WTE ash rather than dispose of it must demonstrate to the Department that the proposed use will satisfy these requirements.

Florida DEP published a guidance document (Florida DEP, 2001) for the regulated community and the Department to assist in the preparation of acceptable beneficial use demonstrations. The document includes guidance on conducting sampling (including the number and type of samples to obtain) and data evaluation. The guidance established a minimum number of samples required, but states that the number of samples should be determined using the procedures described in EPA publication SW-846, Chapter Nine.

The DEP guidance outlines specific goals for the beneficial use demonstration:

- a) The ash management must not cause violations of applicable air, groundwater, or surface water standards and criteria
- b) The use of the ash must not pose increased health risks to humans via inhalation, ingestion, and dermal contact with the ash in its proposed use

- c) The ash must have chemical or physical properties similar to the raw material it is replacing or its use must have enhancing qualities to the final product that would distinguish that use from disposal, and
- d) The use of the ash must not create a public nuisance.

The demonstration process includes a "baseline analysis" of the ash (1) to determine total and leachable concentrations of chemicals for use in a risk assessment or for direct comparison to Florida's Reuse Target Levels (RTLs); (2) to determine if the ash or product will leach constituents at concentrations greater than the Florida DEP's groundwater or surface water standards; and (3) to identify the chemicals of concern (COCs) that should be monitored during routine analysis. COCs for leachability are identified by first comparing total concentrations to the State's RTL leachability values. If the total concentration of a chemical exceeds its RTL leachability value, then the applicant may choose to conduct testing using the Synthetic Precipitation Leaching Procedure (SPLP), EPA Method 1312, for that chemical to further evaluate its leachability. In that case, the chemical is considered a COC for leachability when the upper 95 percent confidence limit on its mean SPLP concentration exceeds the RTL leachability value. If total analysis exceeds the RTL leachability standard and the risk assessment indicates the risks are within acceptable limits, then the SPLP test shall be the determining factor.

As an alternative to the SPLP, the DEP guidance allows potential groundwater or surface water impacts to be evaluated using column leaching tests (lysimeters), and the groundwater impact evaluation can be supplemented using computerized groundwater modeling or longer term studies of smaller field demonstration projects.

Michigan – Regulation R 299.4116, Criteria for Designating Inert Materials Appropriate For Reuse at a Specific Location

Under Rule 116 of Michigan's Solid Waste Management Act Administrative Rules, Part 115 of the Natural Resources and Environmental Protection Act (Michigan DEQ, 1999) a person may petition the director of the State to designate a solid waste as an inert material that is appropriate for reuse at a specific property. The director may approve such a petition if (a) the material does not pose a threat to groundwater, and (b) the material will not otherwise result in an unacceptable risk.

A solid waste shall be considered to not pose a threat to groundwater if the concentration of each hazardous substance in the leachate of the waste is less than one of the following:

- a) The leachate concentration generated by background soil
- b) The method detection limit for the substance in question
- c) All of the following concentrations: risk-based concentration levels calculated using the method specified in the State's regulation, secondary maximum contaminant levels (if available), and taste or odor thresholds and concentrations

d) A concentration that is otherwise authorized pursuant to the provisions of the Michigan Water Resources Commission Act.

The petition to designate a material as inert at a specific location must include a determination of the leaching potential of the constituents using any of the following:

- a) The TCLP, EPA test method 1311
- b) The SPLP, EPA test method 1312, or
- c) Other test methods which are approved by the Michigan DEP and which more accurately simulate conditions at the site.

If a hazardous substance is reported to be present in a sample at concentrations above the waste classification criteria, the petitioner may demonstrate that the data are not statistically significant, using one of the statistical methods used in the State groundwater monitoring program at R 299.4908.

4.2.4 Evaluating Risks Associated with Leaching

Leachate released from a waste or fill material can enter the environment via transport to ground water or via direct discharge to surface water. The potential for the leachate to impact human or ecological receptors depends on the specific contaminants and their concentrations, release and transport mechanisms, the nature of the receiving media, and the presence of potentially exposed populations or receptors. The risk evaluation is performed via one of two different mechanisms (Townsend, et al., 2003b). In one (Section 4.2.4.1), a leaching test is performed and the contaminant concentration in the test leachate is compared to an applicable water quality or risk-based standard after accounting for dilution that can occur in ground water. The second mechanism (Section 4.2.4.2) for assessing leaching risk involves comparing the total concentration in the waste, fill, or soil material to a risk-based leaching level that represents the total concentration that would produce leachate exceeding a applicable ground water or surface water standard (Townsend, et al, 2003b). The leaching level is determined by means of a soil/water partition equation. These methods are described in more detail in the following sections.

4.2.4.1 Using Leach Test Results to Evaluate Leaching Risks

The formation of leachate within fill materials results in the dissolution of constituents from the solid phases in the fill material into the pore water within the fill. When leachate exits the bottom of the fill (i.e., at the fill-subsurface interface, see **Figure 2-1**), the leachate is transported through the unsaturated zone (or immediately enters the saturated zone where the fill is emplaced in or on the water table) to the saturated zone and transported in the subsurface. As the leachate moves through fill, unsaturated zone, and groundwater, contaminant concentrations are attenuated by adsorption and degradation. In the aquifer, dilution by clean groundwater further reduces concentrations before contaminants reach receptor points such as drinking water wells or

surface water. This reduction in concentration can be expressed by a dilution attenuation factor (DAF), defined as the ratio of leachate concentration to receptor point concentration. The lowest possible DAF is 1, corresponding to the situation where there is no dilution or attenuation of a contaminant (i.e., when the concentration in the receptor well or surface water is equal to the fill leachate concentration). In situations where leachate is released directly from fill material into surface water, a DAF of 1 would the most protective approach. On the other hand, high DAF values correspond to a large reduction in contaminant concentration from the fill to the receptor well or surface water.

Fate and transport models are used to estimate DAFs to support site-specific risk assessments (e.g., for Superfund or RCRA Corrective Action cleanups), select waste management unit designs that are protective of groundwater, and in regulatory development. Fate and transport models typically require inputs of waste- and site-specific information including the expected leachate concentration. Leachate concentration can be estimated using a mathematical model such as a soil/water distribution equation or via laboratory leaching tests on the actual material.

For example, under EPA's industrial waste initiative (USEPA, 2003b), EPA suggests the use of a fate and transport model to determine the appropriate liner design for a new industrial waste management unit. The model, known as the Industrial Waste Management Evaluation Model (IWEM), requires users to provide a source term leachate concentration in mg/L for each waste constituent expected to be in the leachate that will infiltrate into the soil underneath a waste management unit. EPA's guidance suggests the leachate source term be obtained from the analysis of waste samples by means of a TCLP, SPLP, or other tests.

4.2.4.2 Evaluating Risks from Leaching Using Partition Coefficients

A method available for assessing leaching risk involves comparing the total concentration in the waste, fill, or soil material to a risk-based leaching level that represents the total concentration that would produce leachate exceeding an applicable ground water or surface water standard (Townsend, et al, 2003b). The methodology incorporates a standard linear equilibrium soil/water partition equation to estimate constituent release in soil leachate and a water-balance equation to calculate a DAF in the aquifer.

The acceptable total concentration level in the fill is back calculated from acceptable ground water concentrations (i.e., MCLs). First, the acceptable ground water concentration is multiplied by the DAF to obtain a target leachate concentration. For example, if the DAF is 10 and the acceptable ground water concentration is 1 mg/L of the constituent of concern, then the target fill leachate concentration would be 10 mg/L. The partition equation is then used to calculate the total fill concentration corresponding to this leachate concentration.

An example equation for determining the leaching level is taken from EPA's Soil Screening Guidance (USEPA, 1996b):

$$C_{t} = C_{w} \left[K_{d} + \left(\frac{\theta_{w} + \theta_{a} H'}{\rho_{b}} \right) \right]$$
(1)

where	•	=	acceptable total concentration in soil or fill (mg/kg)
	C_w	=	target leachate concentration (mg/L) (e.g., MCL x DAF)
	K _d	=	soil-water partition coefficient
	θ_{w}	=	water-filled soil porosity (L _{water} /L _{soil})
	θ_{a}	=	air-filled soil porosity (Lair/Lsoil)
	H'	=	dimensionless Henry's Law constant
	$ ho_{\scriptscriptstyle b}$	=	dry soil bulk density (kg/L).

The equation (1) relates the concentration of the contaminant adsorbed to the soil or fill to leachate concentrations in the zone where the soil or fill is emplaced. Use of the equation relies are the input of various measured values or assumptions including the partition (or distribution) coefficient, K_d . K_d is a measure of sorption of contaminants to soils and is defined as the ratio of a chemical concentration in soil to the concentration of the chemical in water when the system is at equilibrium.

There are five general methods used to measure K_d values (USEPA, 1999b): laboratory batch method, in-situ batch method, laboratory flow-through (or column) method, field modeling method, and K_{oc} method. Of particular interest for this study is the use of laboratory batch methods and flow-through methods. A detailed description of each method is given in EPA 1999b.

Batch methods include the ASTM "24-hour Batch-Type Measurement of Contaminant Sorption by Soils and Sediments" (ASTM, 1987) [replaced by "D 5285-92(1997) Standard Test Method for 24-Hour Batch-Type Measurement of Volatile Organic Sorption by Soils and Sediments"]; the USEPA batch adsorption test (USEPA, 1991c, page 161-162); and methods given in Roy, et al. (1991). These methods are used to determine K_d by either the measurement of adsorption of a constituent in the aqueous phase to the solid phase or by measurement of desorption of the contaminant from the solid phase to the liquid phase. The methods involve a series of batch tests that vary the ratios of solid to liquid and/or the liquid composition.

Laboratory flow-through (column) methods also are used for determining K_d . A solution containing known amounts of contaminant is introduced into a column of packed soil of known bulk density and porosity. The effluent concentration is monitored as a function of time. A known amount of nonadsorbing tracer may also be introduced into the column and its time-varying concentration provides information about the pore-water velocity. The resulting data are plotted as a break-through curve and used to calculate the constituent residence time, retardation factor, and K_d (USEPA, 1999b). The methods determine K_d by the measurement of adsorption of a constituent in the aqueous phase to the solid phase, but can be used to measure desorption of the contaminant from the solid phase to the liquid phase.

4.2.5 Systematic Scenario-Based Approaches for Evaluating Leaching

In the last decade, methodologies have emerged that address leaching of materials using a framework to define the question to be answered, specify the disposal or use scenario, identify relevant parameters influencing leaching, perform appropriate tests for those parameters, and model leaching behavior to simulate and forecast release under the specified time and use

scenario. A methodology guideline entitled ENV 12920:1997 E (CEN, 1997) developed by the European Committee for Standardization CEN/TC 292 is such a scenario-based approach. The methodology is an extension of the approach used in the Dutch Building Materials Decree (MinVROM 1999, 2000). In the United States, Kosson, et al. (2002) recently proposed a similar framework. The framework of Kosson, et al. (2002) was developed in part to respond to many of the criticisms of the TCLP (USEPA 1991b, 1999) and the EPA Science Advisory Board's call for a more flexible, case-specific, tiered testing scheme that addresses the most important parameters affecting leaching (USEPA, 1999a). The framework of Kosson, et al. (2002) is outlined below with emphasis on the techniques used to evaluate the leaching test results.

The framework is based on the notion that waste or materials testing should provide information about potential contaminant release in the context of the anticipated disposal or reuse conditions, rather than testing using a fixed, predetermined scenario that may not apply to the material in its actual disposal environment and over the long term. The framework evaluates leaching using a series of four steps:

- 1. Define the management scenarios and controlling release mechanisms
- 2. Measure intrinsic leaching parameters over a range of leaching conditions
- 3. Use release models incorporating measured leaching parameters to estimate release fluxes and long-term cumulative release, and
- 4. Compare release estimates to accepted criteria.

The framework is presented as the three-tier testing program in which each successive tier provides leaching data (via multiple tests) that is more specific to the material tested and the anticipated disposal conditions than the previous tier. Kosson, et al. (2002) propose six new leaching protocols most of which are similar to existing standard tests. The protocols are designed only for application to inorganic species, however, the general framework would have application to both inorganic and organic species (Kosson, et al., 2002).

4.2.5.1 Testing Protocols

Tier 1 – Screening Tests - When time or economics dictate, first-tier screening tests (the availability test) can be used to provide the most conservative estimate of release over a broad range of anticipated environmental conditions. An example of an availability test is the NEN 7341 in which finely ground samples are leached at a high L/S ratio (50:1, v/m) at pH ranges of 4 and 7. Kosson, et al. (2002) propose a similar method in which the pH range is 4 to 8 and the L/S ratio is 100 mL/g. They also propose an alternative test using ethylenediamine-tetraacetic acid (EDTA) to chelate metals of interest into solution in a single extraction. The test determines the maximum quantity of inorganic constituents in a solid matrix that potentially can be released in presence of a strong chelating agent.

The availability can be determined for each constituent on a dry sample mass basis by multiplying the constituent concentration in the leachate by the test-specific L/S:

$$AVL = C \bullet LS \tag{2}$$

where AVL is the constituent availability (mg/kg dry), C is the constituent concentration in the leachate (mg/L), and LS is the liquid to solid ratio (L/kg).

An example calculation (modified from Sanchez, et al., 2001) can be demonstrated for analysis of arsenic-contaminated soil. Analysis for total content of arsenic was 20,000 mg/kg. Availability leaching tests were performed at both pH 4.0 (HNO₃) and 8.0 (KOH) using a L/S ratio of 100 L/kg. The maximum arsenic concentration in the test leachate was 23.4 mg/L. The total arsenic available for release via leaching was calculated as:

$$AVL = C \bullet LS = 23.4 \text{ mg/L} \bullet 100 \text{ L/kg} = 2,340 \text{ mg/kg}$$

or 11.7 percent of the total content.

Tier 2 – Solubility and Release as a Function of pH – The objective of this testing is to determine the acid/base titration buffering capacity of the test material and the liquid-solid partitioning equilibrium of the constituents of concern (Kosson, et al., 2002). Kosson, et al. (2002) proposed that the testing be performed using up to eleven parallel solubility extractions on size-reduced material using a L/S ratio of 10 mL/g and pH endpoints distributed between 3 and 12. An abbreviated version using just three points can be used, however, the range of pH examined should bracket the extreme values anticipated under actual field conditions. Leaching behavior can then be evaluated by plotting the pH of the sample analyzed as a function of the leachate, a liquid-solid partitioning (LSP) curve can be generated for each constituent of concern by plotting the concentration as a function of solution pH. **Figure 4-2** is an example of such a curve for a hypothetical case of arsenic-contaminated soil. The curve indicates the equilibrium concentration of the constituent of interest at the L/S ratio of 10 mL/g over a pH range of approximately 2 to 11.

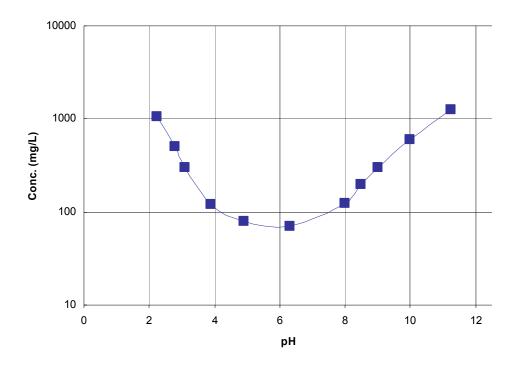


Figure 4-2. Constituent release as a function of pH (hypothetical example for arsenic, modified from Sanchez, et al., 2001)

Tier 2 – Solubility and Release as a Function of L/S Ratio – The objective of this test is to estimate initial pore water conditions and initial leachate compositions in many percolation scenarios. In this test, the pH and redox conditions are dictated by the sample matrix. The test obtains solubility and release data as a function of L/S ratio over a range of L/S ratios (Kosson, et al., 2002). The test objective is accomplished by a series of parallel extractions (with deionized water) of multiple aliquots of the test material at a range of L/S ratios. Kosson, et al. (2002) proposed a batch test protocol, however, they indicate a column test such as prEN 14404 or the NEN 7343 may be used. The filtered extracts are analyzed for constituents of interest, pH, conductivity and other parameters. Constituent concentrations (mg/L) and cumulative amount released (mg/kg) can be plotted as a function of L/S ratio. **Figure 4-3** is an example of such a curve for a hypothetical evaluation of an inorganic constituent. Note that as the L/S ratio increases (which increase with time), the cumulative amount released will asymptotically approach the total amount available for release (e.g., as measured in an availability test).

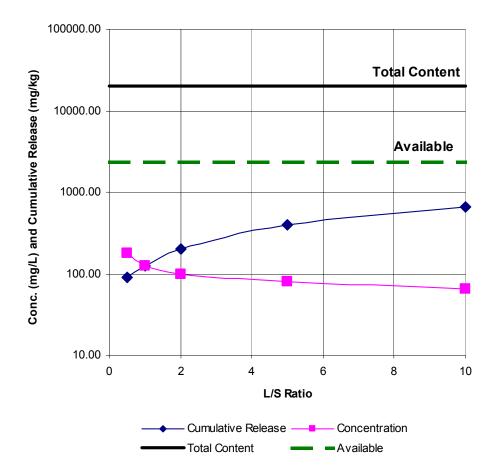


Figure 4-3. Constituent concentration and cumulative release as a function of L/S ratio (hypothetical example for arsenic-contaminated soil)

Tier 3 – Mass Transfer Rate – For wastes or materials that are monolithic or compacted granular material, release to the liquid phase is controlled by mass transfer or diffusion, rather than by percolation. Mass transfer-controlled scenario occurs when infiltrating water is diverted around a low permeability fill or monolithic materials. The information required to estimate constituent release during such a scenario are the (a) field geometry, (b) field density, (c) initial leachable content, and (d) observed diffusivity of the species of concern. Mass transfer rate tests are used to measure the release of constituents from monolithic materials or compacted granular material. The output of the test is an estimate of observed diffusivity (measured in m²/s) which expresses cumulative release as a function of time. Kosson, et al. (2002) recommends methods that are analogous to NEN 7345, the Dutch Tank Leach Test.

Assessment of Release – Using the scenario-based approach proposed by Kosson, et al. (2002), release estimates may be obtained using either (1) site- and waste-specific information, or (2) a default scenario where detailed site-specific information is not available or where conservative assumptions are more appropriate.

The evaluation process first requires application of the steps outlined previously: (1) define the management scenarios and controlling release mechanisms (percolation-controlled or mass-transfer rate controlled), (2) measure intrinsic leaching parameters over a range of leaching conditions, (3) use release models incorporating measured leaching parameters to estimate release fluxes and long-term cumulative release, and (4) compare release estimates to accepted criteria.

4.2.5.2 Evaluating a Percolation-Controlled Scenario

Percolation-controlled release occurs when water flows through a permeable fill with low infiltration and low liquid-to-solid ratio (Kosson, et al., 2002). For this leaching scenario, an estimate of constituent release first requires an estimate of the site-specific liquid-solid ratio (LS_{site}), which can be determined according to:

$$LS_{site} = 10 \frac{\inf \cdot t_{year}}{\rho \cdot H_{fill}}$$
(3)

where LS_{site} is the site-specific liquid-solid ratio (in L/kg); *inf* is the anticipated infiltration rate (cm/year); t_{year} is the estimated time period (years); ρ is the fill density (kg/m³); H_{fill} is the fill depth (m); and 10 is a conversion factor (10 L/cm-m²).

The cumulative mass release per unit mass of material for the specified time period (M_t) can then be obtained using the anticipated site-specific L/S ratio and the constituent solubility at the anticipated field pH ($S_{\text{field pH}}$) according to:

$$M_{t} = (LS_{site})(S_{field \, pH}) \tag{4}$$

An example calculation (adopted from Sanchez, et al., 2001) to estimate the site-specific L/S ratio and cumulative mass released is demonstrated for a site with arsenic-contaminated soil. The site-specific infiltration rate is 20 cm/yr, the fill density is 1.2 g/cm^3 (1,200 kg/m³), and the fill geometry (depth) is H = 1 m. From testing, it is known that the natural pH of the fill is 6.3 and the solubility of arsenic at that pH is 70 mg/L (as determined by the equilibrium-based testing to characterize release as a function of pH). The time interval of interest is 100 years. Using equation (2), the site-specific liquid-solid ratio (LS_{site}) is determined as follows:

$$LS_{site} = 10 \frac{20 \text{ cm/yr} \cdot 100 \text{ yr}}{1.2 \text{ g/cm}^3 \cdot 1 \text{ m}} = 17 \text{ L/kg}$$

The cumulative mass release for the 100-year time period (M_t) can then be obtained as follows:

$$M_{100 \text{ vears}} = (17 \text{ L/kg})(70 \text{ mg/L}) = 1,190 \text{ mg/kg}$$

or about 6 percent of the total content.

4.2.5.3 Evaluating a Mass Transfer-Controlled Scenario

Mass transfer-controlled scenario occurs when infiltrating water is diverted around a low permeability fill or monolithic material (Kosson, et al., 2002). For this leaching scenario, an estimate of cumulative mass of constituent released (M_{mass}^t) relies in part on an analytical solution to the Fickian diffusion model:

$$M_{mass}^{t} = 2 \cdot C_{o} \cdot \frac{S}{V} \cdot \left(\frac{D^{obs} \cdot t}{\pi}\right)^{1/2}$$
(5)

where M_{mass}^{t} is the cumulative mass (mg/kg) of the constituent released at time *t*; C_{o} is the initial leachable content (i.e., available or total elemental content); *S* is the fill surface area (m²); *V* is the fill volume (m³); *t* is the time interval (s); D^{obs} is the observed diffusivity of the species of concern (m²/s).

An example calculation (adopted from Sanchez, et al., 2001) to estimate the constituent release for a mass-transfer-controlled scenario is given for a site with arsenic-contaminated soil. From testing, the total arsenic content is 20,000 mg/kg and the observed diffusivity (D_{obs}) is 1.8 x 10-15 m²/s. The fill geometry is a cube 1-meter on each site for a total surface area (S) of 6 and a volume (V) of 1 m³. As before, the release interval is 100 years. Constituent release under the mass-transfer controlled scenario is estimated as follows:

$$M_{mass}^{t} = 2 \cdot 20,000 \, mg \, / \, kg \cdot \frac{6 \, \text{m}^{2}}{1 \, \text{m}^{3}} \cdot \left(\frac{1.8 \cdot 10^{-15} \, \text{m}^{2}/\text{s} \cdot 100 \, \text{years}}{\pi}\right)^{1/2} = 322 \, \text{mg/kg}$$

or about 1.6 percent of the total content.

A detailed example application of the Kosson, et al. (2002) framework is given by Sanchez, et al. (2001) for mercury-bearing wastes.

5.0 GAPS IN LEACHING TEST METHODOLOGY

5.1 Overview of Leaching Scenarios for Fill Material

SSB 5787 required Ecology to "assess whether [the] list of leaching tests provides appropriate methods for analyzing water quality impacts for all types of projects and in all circumstances" where fill material is used. "Fill material" could include any material that (1) is not a dangerous or extremely hazardous wastes and (2) is exempt from the State's solid waste permitting requirements. The principal scenario in which fill material is used is to bring an area to grade or to expand an existing grade, e.g., in connection with construction projects. Expansion of an existing grade could include placement of fill in the following situations, as permitted under applicable regulations and/or permits: an upland setting above the saturated zone, in a wetland, in a freshwater environment, or in a marine environment.

5.2 Potential Gaps in Existing Leaching Test Methods in Evaluating Potential Impacts to Water Quality From Fill Material

The coverage and gaps of the sixteen leaching test methods were evaluated by identifying the major factors that influence leaching behavior, identifying the aspect of leaching each test was designed to model or simulate, and evaluating the potential suitability of each test for assessing the leaching characteristics of fill materials. The evaluation focused in particular on the extent to which each test is appropriate for evaluating fill materials placed in the following environments:

- Upland setting above the saturated zone, where the leaching fluid will originate primarily from rainwater
- Wetland where the leaching fluid will come from rainwater, surface water and groundwater
- Freshwater environment, where the leaching fluid will be rainwater and surface water, and there will be a high liquid to solid ratio (L/S), and
- Marine environment, where the leaching fluid will be saltwater, and there will be a high L/S.

Sections 2.3.1 through 2.3.3 discussed in general terms the major factors that influence leaching behavior and the various types of leaching tests used to evaluate leaching. Section 2.3.4 categorized the sixteen leaching tests evaluated in this report. **Table 5-1** presents a matrix linking these test methods to the aspects of leaching the test is intended to simulate or model. Section 5.2.1 provides a description of the coverage and gaps of each test method. Section 5.2.2 provides a discussion of which leaching test(s) best simulate the leaching behavior of fill materials.

Table 5-1.	Summary	of Key	Features	of Leaching Tests	
		,			

Test		Leaching Fluid		Particle Size Reduction	Disposal Conditions		
	L/S pH condition duration)		Reduction	Aerobic	Anaerobic	Cost	
Single Extraction Leaching Te	sts						
ASTM D 3987 Standard Test Method for Shake Extraction of Solid Waste with Water	20:1 (v/m)	Reagent water; self- adjusting pH for duration of test.	18 hours	None required	Х		\$85 - \$400
ASTM D 6234 Standard Method for Shake Extraction of Mining Waste by the SPLP	20:1 (m/m)	Reagent water acidified to pH 4.2 or 5.0, with HNO ₃ and H_2SO_4 . The pH is selected by user to reflect site-specific conditions. The pH is controlled by the material being leached	18 hours	9.5 mm	Х		\$100
SPLP Synthetic Precipitation Leaching Procedure	20:1 (m/m)	Reagent water acidified to pH 4.2 or 5.0, with HNO ₃ and H_2SO_4 . The pH is selected by user to reflect site-specific conditions. The pH is controlled by the material being leached	18 hours	9.5 mm	Х		\$45 - \$100

Test		Leaching Fluid		Particle Size Reduction	Disposal Conditions		
	L/S	pH condition	Contact Time (test duration)	Reduction	Aerobic	Anaerobic	Cost
TCLP Toxicity Characteristic Leaching Procedure	20:1 (m/m)	Acetic acid at pH 2.9 used for very alkaline wastes, or acetic acid buffered at pH 4.9 with NaOH for other wastes. The leaching fluid typically controls the pH.	18 hours	9.5 mm	Х		\$45 - \$100
DRET Dredge Elutriate Test	226:1 (v/v)	Site-specific water used. The pH is controlled by the material being leached.	2 hours	None required	Х		\$125 - \$1,200
SET Standard Elutriate Test	4:1 (v/v)	Site-specific water used. The pH is controlled by the material being leached.	2 hours	None required	Х		\$125 - \$1,200
NEN 7341 Availability Test	50:1 (v/m)	Reagent water acidified with HNO ₃ to pH 4 and 7. pH controlled at constant value throughout test.	3 hours per step	<125 μm	Х		No data
EN 12457/1-4 Compliance Test for Granular Waste Materials and Sludges	2 L/kg and 10 L/kg	Reagent water. pH controlled by material being leached.		< 4 mm and < 10 mm	Х		No data

Table 5-1. Summary of Key Features of Leaching Tests

Table 5-1.	Summary	of Key	Features	of Leaching Tests	

Test		Leaching Fluid		Particle Size Reduction	Disposal Conditions		
	L/S	pH condition	Contact Time (test duration)	Reduction	Aerobic	Anaerobic	Cost
Serial Batch Tests							
ASTM D 4793 Standard Test Method for Sequential Batch Extraction of Waste with Water	20:1 (v/m) (Corrected for the mass of moisture present in the sample)	Reagent water. pH controlled by material being leached	18 hours	None required	Х		~\$100/cycle multiple cycles required
ASTM D 5744 Standard Test Method for Accelerated Weathering of Solid Materials Using a Modified Humidity Cell	(L/S = 0.5:1 or 1:1, v/m), refreshed repeatedly	Reagent water. pH controlled by material being leached	1 hour per cycle, multiple cycles	150 μm	Х		no data
SBLT Sequential Batch Leachate Test	4:1 (m/m)	Reagent water. pH controlled by material being leached	24 hours	None required	Х	Х	\$150 - \$250/cycle multiple cycles required
Flow-around Test	1		-				
NEN 7345 Tank Leach Test	5:1 (v/v)	Reagent water acidified to pH 4 with HNO ₃ pH controlled during test by material being leached.	8 hours and 1, 2, 4, 9, 16, 36, and 64 days	Monolith > 40 mm	Х		No data

Test	Leaching Fluid			Particle Size Reduction	Disposal	Conditions	
	L/S	pH condition	Contact Time (test duration)	Reduction	Aerobic	Anaerobic	Cost
Flow-through Tests							
ASTM D 4874 Standard Test Method for Leaching Solid Waste in a Column Apparatus	N/A	Reagent water. pH controlled by material being leached	days to months	10 mm	Х		\$1,000 per 30 pore volumes
PCLT Pancake Column Leachate Testing	N/A	Water (unspecified source). pH controlled by material being leached.	Weeks	None required		Х	\$10,000 - \$30,000 per 30 pore volumes
NEN 7343 Column Test	Seven leachate fractions with L/S range of 0.1 to 10 L/kg	Reagent water acidified to pH 4 with HNO ₃ . pH subsequently controlled by material being leached	21 days	95% < 4 mm			No data
prEN 14405 Upflow percolation test	0.1 to 10 (v/m)	Acidified water		< 4 mm			No data

"X" indicates the test is designed to address the disposal condition.

5.2.1 Coverage and Gaps

This section discusses the coverage and gaps of each leaching test with respect to assessing the leaching behavior of fill materials. Issues evaluated include the following:

- Implementability (availability, location and cost of testing),
- Accuracy of the testing at assessing leachability (completeness of the constituents addressed, testing under anaerobic and aerobic conditions, comparison to field leachate),
- Assessment of concentration- and time-dependent factors,
- Reproducibility of testing, and
- Inclusion of external environmental factors (acid precipitation and high ionic strength disposal conditions).

5.2.1.1 ASTM D 3987, Standard Test Method for Shake Extraction of Solid Waste with Water

Coverage

- The test is relatively inexpensive to perform (\$85 \$400 per test cycle).
- Multiple experienced laboratories are available, including at least one laboratory in the Northwest.
- Particle size reduction is not required; therefore, tested particles will more closely
 resemble the particle size and surface area of those under field conditions.

Gaps

- The test is limited to non-volatile, inorganic constituents.
- The test is conducted under aerobic conditions only.
- The leaching fluid is reagent water, which does not account for the acidity of precipitation, nor does it account for the ionic strength of saltwater found in marine and estuarine environments.
- The test is not intended to provide a leachate that is representative of the actual leachate produced from a solid waste in the field, nor is it intended to produce extracts that will serve as the sole basis of engineering design (ASTM, 1999a).
- This short-term test may not simulate long term leaching.

• No reproducibility data are available.

5.2.1.2 ASTM D 6234, Standard Method for Shake Extraction of Mining Waste by the SPLP

Coverage

- The test is inexpensive to perform (\$45 \$100 per test).
- At least one lab with equipment and experience is available in the Northwest.
- The initial pH of the extraction fluid is dictated by the expected pH of the precipitation in the geographic region in which the material is to be disposed, accounting for the influence of acid precipitation. The final pH of the leachate will typically be controlled by the material being leached.
- Particle size reduction required, so test particles may not be reflective of the size (and surface area) of fill particles.
- Precision data are available for multiple elements from an interlaboratory study.

<u>Gaps</u>

- The test is applicable to inorganic, non-volatile constituents only.
- The extraction fluid is pH-adjusted reagent water, and does not account for the ionic strength of marine and estuarine waters.
- The test is conducted under aerobic conditions only.
- This short-term test may not simulate long term leaching.

5.2.1.3 SPLP, Synthetic Precipitation Leaching Procedure

Coverage

- The test is inexpensive to perform (\$45 \$100 per test).
- Many laboratories with equipment and experience are available in the Northwest.
- The test addresses both organic and inorganic constituents, including volatile organics.
- The extraction fluid is pH-adjusted reagent water, using nitric and sulfuric acids to simulate the acid rain resulting from airborne nitric and sulfuric oxides. The initial pH of the extraction fluid is dictated by the expected pH of the precipitation in the geographic

region in which the material is to be disposed, accounting for the influence of acid precipitation. The final pH of the leachate will typically be controlled by the material being leached.

- Precision data are available.
- Field studies indicate the SPLP can provide conservative to accurate results for selected metals and wastes (sodium, potassium, calcium, and magnesium in ash [Townsend, Jang, and Tolaymat, 2003a] and arsenic, lead, and zinc in soil [Lackovic, et al., 1997] and has been shown to under-predict leaching of chromium in soils (Lackovic, et al., 1997).

<u>Gaps</u>

- The extraction fluid is pH-adjusted reagent water, and does not account for the ionic strength of marine and estuarine waters.
- The initial pH of the test may not reflect actual field pH conditions.
- If the sample contains particles larger than 9.5 mm, then particle size reduction is required. A particle-size-reduced sample may not reflect the size and surface area of the material under field conditions.
- The test is conducted under aerobic conditions only, and therefore may not be a good predictor of leaching under anaerobic conditions.
- This short-term test may not simulate long term leaching.

5.2.1.4 TCLP, Toxicity Characteristic Leaching Procedure

Coverage

- The test is inexpensive to perform (\$45 to \$100 per test).
- Many laboratories with equipment and experience are available in the Northwest.
- The test addresses both inorganic and organic constituents, including volatile organics.
 Volatile organic are addressed by using a special pressurized container for leaching called the Zero-Headspace Extractor.
- Precision data are available.
- The test is intended to mimic acidic leaching conditions and may be useful for predicting impacts not only in co-disposal situations, but also in other situations where there is acidic leachate resulting from biological decomposition.

 A field study of fly ash leaching showed that the TCLP consistently over-predicted concentrations of barium in leachate and under-predicted concentrations of selenium. Results for other metals (arsenic, chromium, and lead) differed by no more than one order of magnitude (EPRI, 1995).

<u>Gaps</u>

- The test was developed for RCRA waste classification and assumes co-disposal of granular solid waste in a municipal waste landfill (with significant organic matter), and may not be appropriate for other disposal scenarios.
- Particle size reduction may be required, so test particles may not be reflective of the size (and surface area) of fill particles.
- The initial pH of the test may not reflect actual field pH conditions.
- This short-term test may not simulate long term leaching.
- The test is conducted under aerobic conditions only.
- Field data indicate the TCLP can under-predict leachate concentrations (e.g., for arsenic, and fluoride) if field conditions do not match the test conditions (USEPA, 1997).

5.2.1.5 DRET, Dredge Elutriate Test

Coverage

- The test is relatively inexpensive to perform, although cost estimates vary (\$125 per test to \$1,200 per 20 L batch).
- Multiple labs with equipment and experience are available in the Northwest.
- The test is conducted with a high L/S ratio (226:1, v/v), which is consistent with belowsurface disposal in marine and estuarine environments.
- The test is applicable to both non-volatile organics and inorganic constituents.
- The leaching fluid is water from the site, accounting for the acidity and ionic strength of field conditions.

<u>Gaps</u>

• The test specifically addresses undisturbed sediment from a dredging site and may not be appropriate for other disposal scenarios.

- The composition of the site water, used as the leaching fluid, may cause the leaching fluid to be chemically unstable during the leaching test, resulting in increased variability of test results.
- The test is conducted under aerobic conditions only.
- The test is conducted with a high L/S ratio (226:1, v/v), which is not consistent with disposal in upland environments.
- This short-term test may not simulate long term leaching.
- No precision data are available.

5.2.1.6 SET, Standard Elutriate Test

Coverage

- The test is relatively inexpensive to perform, although cost estimates vary (\$125 per test to \$1,200 per 20 L batch).
- Multiple labs with equipment and experience are available in the Northwest.
- The leaching fluid is water from the site, accounting for the acidity and ionic strength of field conditions.
- Field data indicate the SET tends to be a conservative predictor of inorganic constituents in the water column (Havis, 1988)

<u>Gaps</u>

- The test specifically addresses undisturbed sediment from a dredging site, and may not be appropriate for other disposal scenarios.
- The composition of the site water, used as the leaching fluid, may cause the leaching fluid to be chemically unstable during the leaching test, resulting in increased variability of test results.
- The test is conducted under aerobic conditions only.
- This short-term test may not simulate long term leaching.
- No precision data are available.
- Experiments have shown that the test overestimates the expected release for some contaminants (DiGiano, et al., 1995).

5.2.1.7 NEN 7341, Availability Test

<u>Coverage</u>

- Two serial extractions, each at different pH values, conducted at high L/S and large surface area of solid material, provide an estimate of the maximum amount of inorganic constituents that can leach.
- L/S ratio of 50:1 (v/m) may be reflective of disposal below surface in marine and estuarine environments.
- While the L/S ratio of 50:1 (v/m) is not reflective of disposal in upland environments, it is consistent with the goal of this test to determine the maximum leachable amount of the constituent of interest.

Gaps

- Test is conducted under aerobic conditions only.
- Leaching fluid is pH-adjusted reagent water, which does not account for the ionic strength of saltwater found in marine and estuarine environments.
- The test is limited to inorganic constituents.
- No laboratories found in the US that conduct this test.
- Cost data are not available.
- Precision data are not available.
- Janssen-Jurkovieova, et al. (1994) report that the NEN 7341 availability test is not adequate to determine the maximum amount of elements available to leach from ash.

5.2.1.8 EN 12457/1-4, Compliance Test for Granular Waste Materials and Sludges

Coverage

- The test provides protocols at four different L/S, based on solids content and particle size, and may, therefore, be more reflective of field conditions.
- When more than one of the test protocols is used, the analyst may estimate a relative timeframe for contaminant release when compared with availability for leaching.

Gaps

- Particle size reduction may be required, so test particles may not be reflective of the size (and surface area) of fill particles.
- The leaching fluid is reagent water, which does not account for the acidity of precipitation, nor does it account for the ionic strength of saltwater found in marine and estuarine environments.
- The test is conducted under aerobic conditions only.
- The test is limited to inorganic constituents.
- Precision data (within and between laboratories) were limited because of sample hetereogeneity issues.
- No laboratories have been identified in the U.S. that conduct this test.
- Cost data are not available.
- In a field evaluation of blast furnace slag, the method under-predicted leaching of metals (European Commission, 2001).

5.2.1.9 ASTM D 4793, Standard Test Method for Sequential Batch Extraction of Waste with Water

<u>Coverage</u>

- Multiple laboratories are available in the Northwest with suitable equipment and facilities, and with direct experience with similar test protocols.
- The test is relatively inexpensive to perform (\$100 \$400 per test cycle, 10 test cycles = \$1,000 \$4,000 per complete test). These cost estimates are based on laboratory experience with similar test protocols.
- The test includes10 serial extractions, which may provide information about time- and concentration-dependence of leaching behavior.
- Particle size reduction is not required; therefore, tested particles will more closely
 resemble the particle size and surface area of those under field conditions.

<u>Gaps</u>

• No laboratories with direct experience could be found, however the test protocol is sufficiently similar to others that this should not be a problem.

- The test is limited to inorganic constituents.
- The test is conducted under aerobic conditions only.
- The leaching fluid is reagent water, which does not account for the acidity of precipitation, nor does it account for the ionic strength of saltwater found in marine and estuarine environments.
- A limited amount of precision data are available from a collaborative study, however, the estimated precision of the procedure varied with the concentration of each constituent of interest, and no specific trend could be identified.
- The method is not intended for use as the sole basis for engineering design of a disposal site or waste characterization based on its leaching characteristics (ASTM, 1999b).

5.2.1.10 ASTM D 5744, Standard Test Method for Accelerated Weathering of Solid Materials Using a Modified Humidity Cell

<u>Coverage</u>

• The test is intended to accelerate the natural weathering rate of a solid material.

<u>Gaps</u>

- No experienced labs are available.
- Test equipment is not available.
- No cost data are available.
- The test is only applicable to non-volatile inorganic constituents.
- The test is conducted under aerobic conditions only.
- The test may not be suitable for testing materials containing plastics, polymers, or refined materials.
- Elapsed time for completion of the test is long (20 weeks).
- Particle size reduction is required, (very small particle size = 150μ m), so test particles are probably not reflective of the size (and surface area) of fill particles.
- Leaching fluid is reagent water, which does not account for the acidity of precipitation, nor does it account for the ionic strength of saltwater found in marine and estuarine environments.

- No precision data are currently available for this method.
- The test is not intended to simulate site-specific leaching conditions, nor is it intended to
 produce leachates that will serve as the sole basis of engineering design (ASTM, 2001c).

5.2.1.11 SBLT, Sequential Batch Leachate Test

<u>Coverage</u>

- Test protocols are available providing for both aerobic and anaerobic conditions, approximating the conditions found in marine and freshwater sediments, as well as those found in wetland and upland disposal scenarios.
- The test is applicable to both non-volatile organic and inorganic constituents.
- The test is relatively inexpensive to perform (approximately \$125 to \$250 per cycle, multiple cycles required per test).
- Multiple labs with equipment and experience are available in the Northwest.
- The SBLT has been recommended by the US Army Corps of Engineers for leach testing of freshwater sediments (Brannon, Myers, and Tardy 1994; USACE, 2003).
- A minimum of four serial extractions are required, which may provide information about time- and concentration-dependence of leaching behavior.
- Particle size reduction is not required, so test particles may accurately reflect the size and surface area of fill material.

Gaps

- The leaching fluid is reagent water, which does not account for the acidity of precipitation, nor does it account for the ionic strength of saltwater found in marine and estuarine environments.
- This short-term test may not simulate long term leaching.
- No precision data are available.

5.2.1.12 NEN 7345, Tank Leach Test

Coverage

- The test method was developed to test the leachability of inorganic constituents from building materials, monolithic waste and stabilized waste materials, similar to what might be found in fill material.
- The test results allow the user to identify the controlling mechanism of leaching: either dissolution, erosion, or diffusion.

<u>Gaps</u>

- The test is conducted under aerobic conditions only.
- The test is limited to inorganic constituents.
- The leaching fluid is pH-adjusted reagent water, which does not account for the ionic strength of saltwater found in marine and estuarine environments.
- This short-term test may not simulate long term leaching.
- No laboratories have been found in the US that conduct this test.
- Cost data are not available
- Precision data are not available.

5.2.1.13 ASTM D 4874, Standard Test Method for Leaching Solid Waste in a Column Apparatus

Coverage

- There is at least one laboratory with experience available in the Northwest.
- The test is applicable to low concentration semi-volatile and nonvolatile organic compounds, as well as inorganic constituents.
- The procedure allows for selection of specific operating conditions for the column in order to meet the objectives of individual studies.
- Precision data are available (within-laboratory RSD = 33%, between-laboratory RSD = 54%).

Gaps

- The test is relatively expensive to perform (\$1,000 per test).
- The maximum particle size for the column procedure is 10-mm; although particle size reduction is not recommended.
- The leaching fluid is reagent water, which does not account for the acidity of precipitation, nor does it account for the ionic strength of saltwater found in marine and estuarine environments.
- The test is conducted under aerobic conditions only.
- The test is not meant to be used to determine leachability of volatile compounds.
- The method is not intended to be used as the sole basis for engineering design of a disposal site or waste characterization based on its leaching characteristics (ASTM, 2001b).

5.2.1.14 PCLT, Pancake Column Leachate Testing

Coverage

- The test is performed under anaerobic conditions, approximating the conditions found in marine and freshwater sediments.
- Multiple labs with equipment and experience are available in the Northwest.

<u>Gaps</u>

- The test is very expensive to perform (\$10,000 to \$36,000 per 30 pore volumes).
- The elapsed time required for testing is large (30 weeks or longer).
- The leaching fluid is reagent water, which does not account for the acidity of precipitation, nor does it account for the ionic strength of saltwater found in marine and estuarine environments.
- No precision data are available.

5.2.1.15 NEN 7343, Column Test

Coverage

- The test is based on disposal of solid earthy and stony materials and wastes, similar to what might be expected in fill material.
- The test should provide information on short, medium, and long term leaching behavior by relating contaminant release to the L/S ratio (Sorini, 1996).
- Precision data indicate that the test is relatively precise (within-laboratory repeatability of 11%, and between-laboratory reproducibility of 21%).

<u>Gaps</u>

- The size of particles used in the test is controlled (< 4mm), so the test particles may not be reflective of the size (and surface area) of fill particles.
- The leaching fluid is pH-adjusted reagent water, which does not account for the ionic strength of saltwater found in marine and estuarine environments.
- The test is limited to inorganic constituents.
- No laboratories have been found in the US that conduct this test.
- Cost data are not available.

5.2.1.16 prEN 14405, Upflow Percolation Test

Coverage

• The test is intended to determine the rate of contaminant leaching as a function of liquid to solid ratio, particularly at the low L/S ratios prevailing in disposal scenarios.

Gaps

- No laboratories have been found in the US that conduct this test.
- The test is limited to inorganic constituents.
- This short-term test may not simulate long term leaching.
- Cost data are not available.
- Precision data are not available.

6.0 SUMMARY OF CONCLUSIONS REGARDING THE LEACHING TEST LIST AND THE LIST OF METHODOLOGY GAPS

Washington State Legislature Substitute Senate Bill (SSB) 5787 added new parts to the Revised Code of Washington (RCW). New part RCW 90.48.531 directed Ecology to prepare and submit this report to the legislature to identify leaching tests used for evaluating the potential impacts to water quality in situations where fill material is imported, assess whether the tests provide appropriate methods for analyzing water quality impacts for all types of projects and in all circumstances where fill material is imported, and identify any gaps in leaching test methodology.

A search of regulatory and non-regulatory programs of federal agencies, states, and other countries identified a variety of leaching tests used to support activities such as waste classification (e.g., hazardous waste determination), assessment of waste treatment effectiveness, evaluation of management options (e.g., beneficial use) for wastes or soil, site characterization and cleanup, and risk assessment. Other than the TCLP, the most common test specified by other states for their beneficial use programs is the SPLP, however, states contacted provided little or no justification or supporting data for use of the test.

A list of sixteen leaching tests was identified based on their capabilities to evaluate impacts to water quality from fill materials. These tests fall into one of two categories: (1) single extraction/batch tests carried out in the form of leaching tests on a single portion of material using a single portion of leaching fluid (with no renewal of leaching fluid), or (2) multiple extraction/flow-around and flow-through leaching tests.

An assessment of the leaching test methods was performed to evaluate their suitability for analyzing water quality impacts for all types of projects and in all circumstances where fill material is imported. To evaluate the extent to which leaching tests accurately predict leaching, a literature search was conducted to identify studies in which leaching test results were compared to actual field leachates. Within existing time and resource constraints, relatively few studies were found that provided comparisons of laboratory leaching test results to actual field data. Much of the work reported in the literature has focused on leaching of inorganic constituents rather than organic constituents. Of those studies reviewed, the results were mixed in that some leaching tests over-predicted the field leaching, some under-predicted field leaching, and others provided ambiguous results. This was due, in part, to data variability both spatially and over time. Another factor is that batch tests such as the TCLP and SPLP were designed to screen or categorize wastes or materials based on a single disposal scenario -- not to predict constituent concentrations in leachate on a site-specific basis. Thus, the results of batch leaching tests are not expected to match field leachates except where there is a reasonable match between field and laboratory test conditions (such as pH and liquid-to-solid ratio).

Given the limitations in correlating laboratory leaching test results with actual field data, research also was conducted to identify the various techniques available for evaluating and interpreting leaching test results. The appropriate method to evaluate leaching data should be determined after consideration of the technical objectives and regulatory requirements. For

example, leaching test data may be generated to classify a waste as hazardous or nonhazardous (e.g., via the TCLP); determine the effectiveness of a waste treatment process (e.g., via the TCLP); estimate a source term or end point for risk assessment purposes; or assess the release potential under specified reuse or disposal conditions. Data evaluation methods typically involve either (1) comparison of leaching test results to regulatory standards or risk-based levels (e.g., to classify a waste or soil), or (2) use of the leaching test result to predict the constituent release under an anticipated field scenario (e.g., by taking into account site-specific release mechanisms, pH, and liquid-to-solid ratio over a specified timeframe).

Finally, an assessment was conducted to identify any gaps in leaching test methodology. The assessment was conducted as a coverage and gaps analysis to evaluate the extent to which each of the sixteen tests is appropriate for evaluating fill materials placed in the following environments: an upland setting above the saturated zone, wetlands, surface water, groundwater, freshwater environments, and marine environments. The assessment evaluated each of the tests against criteria such as implementability, accuracy, reproducibility, and ability of the test to address scenario-specific factors (e.g., aerobic vs. anaerobic conditions, saturated vs. unsaturated environments). The findings of the assessment are consistent with the growing consensus among researchers: Evaluating the leaching behavior of a wide variety of materials in a broad range of management scenarios cannot be addressed adequately by one single laboratory leaching test.

6.1 Summary of Gaps and Limitations in Leaching Test Methodologies

Leaching tests designed to simulate leaching of constituents under single management scenario are of limited use where the assumptions of the test do not match the field scenario or where the test does not provide sufficiently conservative results. Even use of an "aggressive" (low pH) leaching test (such as the TCLP) may not be protective because some constituents (such as arsenic) can have increased leachability under high pH conditions.

Other gaps in leaching test are related to their ability to address leaching under anaerobic conditions, the lack of tests designed to address organic constituents, and the fact that some of the more promising tests are not commercially available in the United States.

Another gap in the leaching tests reviewed is that they are, for the most part, short-term tests that do not adequately mimic field conditions that develop over time (such as the release of metals due to biological degradation processes or the physical and chemical changes brought about by weathering). While the TCLP attempts to mimic biological degradation processes by artificially creating similar conditions, other tests do not. Because the TCLP was developed specifically for use in a co-disposal scenario with a significant organic component, it is probably not an appropriate choice for testing a monofill with limited organic composition, as would be the case with use of fill material. However, other leaching tests with low pH fluids may be appropriate to use where low pH conditions are anticipated or a potential concern (such as with acid mine drainage).

6.2 Recommendations

Based on a review of regulatory and non-regulatory programs of states, the federal government, and other countries, and a review of the scientific literature, a large number of leaching tests are available to evaluate leaching of fill material. Unfortunately, no single test is completely satisfactory for assessing water quality impacts for all types of projects in all environmental circumstances where fill material could be used. For example, the solubility of most metals is highly sensitive to pH. Where the pH of the fill disposal scenario is not known, then use of a leaching test that assumes a certain pH condition may not provide accurate results. Instead, use of a leaching test that employs a range of pH conditions could be used to evaluate leaching as a function of pH. If the pH conditions of a fill situation are known or can be anticipated, then a test should be selected that best simulates those conditions. Use of an "aggressive" (low pH) leaching test based on a single management scenario (such as the TCLP) may not necessarily be protective because some constituents (such as arsenic) can have increased leachability under high pH conditions.

A relatively small suite of tests (or modifications to existing tests) may be suitable to assess potential impacts to water quality from emplacement of fill material in a saturated or unsaturated environment. Some of the more promising tests are discussed below.

- Of the tests reviewed, the Sequential Batch Leachate Test (SBLT) has many advantages relative to testing the leachability of fill materials in upland, wetland, freshwater and marine environments. The SBLT is one of the few tests that specifically provides for testing under anaerobic conditions such as those found in submerged freshwater and marine environments. Additionally, test conditions are available for the aerobic testing that may be appropriate where such conditions are likely to exist. The test is applicable to both organic and inorganic constituents, unlike many of the other tests that are limited to inorganic constituents. Unlike many of the other tests evaluated, the SBLT does not require particle size reduction so test particles are likely to accurately reflect the size and surface area of the fill material. The SBLT has been specifically recommended by the U.S. Army Corps of Engineers for leach testing of freshwater sediments (Brannon, Myers, and Tardy 1994; USACE, 2003), and has been used for leach testing of estuarine and marine sediments. The test is relatively inexpensive to perform, and can be performed by experienced laboratories in Washington State. The major drawback to the SBLT with respect to this testing scenario is that the un-adjusted reagent water leaching fluid does not account for the acidity of precipitation, nor does it account for the ionic strength of saltwater found in marine and estuarine environments. Only two of the tests evaluated account for the ionic strength of marine and estuarine waters (the DRET and the SET). Many of the tests evaluated accounted for the acidity of precipitation by adjusting the pH of the leaching fluid.
- The Availability Test (NEN 7341) also has advantages relative to testing the leachability of fill materials in upland, wetland, freshwater and marine environments. The test is aggressive, providing an estimate of the maximum amount of inorganic constituents that can leach. The high L/S ratio is consistent with disposal in marine and estuarine environments, where large volumes of water are in contact with the solid materials.

Unfortunately, there is little to no commercial availability of this test in the United States, however, it is relatively easy to perform.

- The Compliance Test for Granular Waste Materials and Sludges (EN 12457/1-4) has the advantage of estimating the relative timeframe for contaminant release when compared with availability for leaching. This capability may overcome the limitation of other tests to short-term prediction only. Both the Compliance Test and the Availability Test are widely used in Europe, and are similar to tests being proposed by researchers in the United States (Kosson, et. al., 2002). However, no laboratories in the United States currently offer these tests on a commercial basis.
- Batch tests such as the SPLP or TCLP may be adequate for screening or categorizing
 materials where the test is known to produce sufficiently conservative (environmental
 protective) results. Batch tests also can be used to predict constituent concentrations in
 leachate for selected constituents where the anticipated field conditions (e.g., pH, liquidto-solid ratio) match the laboratory test conditions.

An alternative to the use of single scenario batch tests is to use a framework to define the question to be answered, specify the disposal or use scenario, identify relevant parameters influencing leaching, perform tests from a suite of leaching tests for those parameters, and model leaching behavior to simulate and forecast release under the specified time and use scenario. This step-wise approach is used in Europe (CEN, 1997) to evaluate materials for disposal and beneficial reuse, and a similar framework has been proposed in the United States (Kosson, et al., 2002) in response to criticisms of the TCLP. A common theme of these leaching frameworks is the use of a hierarchy of leaching tests in which the type and number of tests employed is scaled to the amount of leaching information required by the user.

• It is recommend that Ecology explore the use of a tiered framework for the selection of leaching tests. A framework could include the use of a small number of existing (or modified) leaching tests, combined with the use of mathematical modeling, to address a broad range of management scenarios for fill material (e.g., see Kosson, et al., 2002 and CEN, 1997).

Finally, several suggestions for future work are given below.

- Where leaching tests are used for determine the suitability of a material for use at a specific site, post-emplacement monitoring of ground water and surface water is recommended to detect any releases of constituents due to leaching.
- There is considerable variability in leaching test results. This variability is due to sample variability, leaching test variability and analytical variability. Accounting for variability in leaching test is more complex than it is when total content analyses are performed. Additional research is required to develop procedures to take this variability into account when making decisions about the suitability of a material for fill.

Many leaching tests are designed specifically to address the leaching of inorganic substances only. Additional research may be warranted to explore how leaching tests designed primarily to assess inorganic substances can be used to address leaching of organic constituents. For example, if the tests are to be used for wastes or soil contaminated with organic materials, the materials used for the test may need to be changed and the procedures for sample preparation, leaching and solid/liquid separation may need to be adjusted, especially if volatile substances are contaminants of concern.

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APPENDICES

Appendix A

Substitute Senate Bill (SSB) 5787

CERTIFICATION OF ENROLLMENT{PRIVATE }

SUBSTITUTE SENATE BILL 5787

Chapter 210, Laws of 2003

58th Legislature 2003 Regular Session

LEACHING TESTS

EFFECTIVE DATE: 5/9/03

{PRIVATE }Passed by the Senate April 23, 2003 YEAS 38 NAYS 10

BRAD OWEN President of the Senate

Passed by the House April 18, 2003 YEAS 61 NAYS 25

FRANK CHOPP Speaker of the House of Representatives

Approved May 9, 2003.

CERTIFICATE

I, Milton H. Doumit, Jr., Secretary of the Senate of the State of Washington, do hereby certify that the attached is SUBSTITUTE SENATE BILL 5787 as passed by the Senate and the House of Representatives on the dates hereon set forth.

MILTON H. DOUMIT JR. Secretary

FILED

May 9, 2003 - 4:26 p.m.

GARY F. LOCKE Governor of the State of Washington Secretary of State State of Washington

SUBSTITUTE SENATE BILL 5787

AS AMENDED BY THE HOUSE Passed Legislature - 2003 Regular Session

State of Washington58th Legislature2003 Regular SessionBySenate Committee on Natural Resources, Energy & Water (originally
sponsored by Senators Morton, Prentice, Hale, Jacobsen, Kohl-Welles,
Hewitt, Doumit and Horn)

READ FIRST TIME 03/03/03.

Protecting

water

quality.

AN ACT Relating to the use of a leaching test in state water quality certifications; adding new sections to chapter 90.48 RCW; and declaring an emergency.

BE IT ENACTED BY THE LEGISLATURE OF THE STATE OF WASHINGTON:

<u>NEW SECTION.</u> Sec. 1. A new section is added to chapter 90.48 RCW to read as follows:

(1) In order to ensure that construction projects involving the use of fill material do not pose a threat to water quality, the department may require that the suitability of potential fill material be evaluated using a leaching test included in the soil clean-up rules adopted by the department under chapter 70.105D RCW in any water quality certification issued under section 401 of the federal clean water act and in any administrative order issued under this chapter, where such certification or administrative order authorizes the placement of fill material, some or all of which will be placed in waters of the state. Any such requirement imposed by the department in a water quality certification or administrative order issued prior to the effective date of this section is ratified and approved by the legislature as a valid and reliable method for determining concentrations of chemical constituents that can be present in fill material without posing an unacceptable risk of violating water quality standards, and shall be in effect as imposed by the department for all work not completed by June 1, 2003.

(2) Nothing in this section limits, in any way, the department's authority under this chapter.

<u>NEW SECTION.</u> Sec. 2. A new section is added to chapter 90.48 RCW to read as follows:

The department shall identify the leaching tests utilized for evaluating the potential impacts to water quality in situations where fill material is imported. The tests may include those identified in the soil clean-up rules adopted by the department under chapter 70.105D RCW. Within existing resources, the department shall assess whether this list of leaching tests provides appropriate methods for analyzing water quality impacts for all types of projects and in all circumstances where fill material is imported. The department shall also identify any gaps in leaching test methodology. The department shall report both the leaching test list and the list of test methodology gaps to the appropriate committees of the legislature by December 31, 2003.

<u>NEW SECTION.</u> Sec. 3. This act is necessary for the immediate preservation of the public peace, health, or safety, or support of the state government and its existing public institutions, and takes effect immediately. Passed by the Senate April 23, 2003. Passed by the House April 18, 2003. Approved by the Governor May 9, 2003. Filed in Office of Secretary of State May 9, 2003.

Appendix B – Comparison of Major Variables of Leaching Tests

TEST METHOD	LEACHING FLUID	L/S RATIO	MAXIMUM PARTICLE SIZE	NUMBER OF EXTRACTIONS	DURATION OF EXTRACTIONS	APPLICABLE CONSTITUENT GROUP(S)
ASTM D 3987-85 (1999) Standard Method for Shake Extraction of Solid Waste with Water	Reagent Water	20:1 (v/m)	N/A	1	18 hours	Non-volatile, inorganic constituents
ASTM D 4793-93 Standard Method for Sequential Batch Extraction of Waste with Water	Reagent water	20:1 (v/m) (Corrected for the mass of moisture present in the sample)	N/A	10	18 Hours	Inorganic constituents only
ASTM D 4874-95 (2001) Standard Test Method for Leaching Solid Material in a Column Apparatus	Reagent Water	N/A	10 mm	Up-flow through the column		Low concentration semi-volatile and nonvolatile organic compounds as well as inorganic constituents
ASTM D 5744- 96 (2001) Standard Test Method for Accelerated Weathering of Solid Materials Using a Modified Humidity Cell	Reagent Water	(L/S = 0.5:1 or 1:1, v/m), refreshed repeatedly	150 μm	Weekly leaching	1 hr	Non-volatile inorganic constituents

TEST METHOD	LEACHING FLUID	L/S RATIO	MAXIMUM PARTICLE SIZE	NUMBER OF EXTRACTIONS	DURATION OF EXTRACTIONS	APPLICABLE CONSTITUENT GROUP(S)
ASTM D 6234-98 (2002) Standard Test Method for Shake Extraction of Mining Waste by the Synthetic Precipitation Leaching Procedure	The pH of the extraction fluid is dictated by the expected pH of the precipitation in the geographic region in which the waste is to be disposed.	20:1 (m/m)	9.5 mm	1	18 hours	Non-volatile, inorganic constituents
Toxicity Characteristic Leaching Procedure (TCLP) 1311	Acetic Acid 0.1 N acetic acid solution, pH 2.9, for alkaline wastes 0.1 N sodium acetate buffer solution, pH 5.0, for non-alkaline wastes	20:1 (m/m)	9.5 mm	1	18±2 Hours	Toxicity Characteristic (TC) constituents in 40 CFR §261.24 including 20 volatile organics, 16 semi- volatile organics, and two pesticides
Synthetic Precipitation Leaching Procedure (SPLP) 1312	 #1 Reagent Water to pH 4.2 with nitric and sulfuric acids (60/40) #2 Reagent Water to pH 5.0 with nitric and sulfuric acids (60/40) 	20:1 (m/m)	9.5 mm	1	18±2 hours	Organic and inorganic constituents
Standard Elutriate Test (SET)	Dredging-site water	4:1 (v/v)	N/A	1	Agitation time: 1 hr Settling time: 1 hr	Not specified in method
Dredge Elutriate Test (DRET)	Dredging-site water	226:1 (v/v)	N/A	1	Aeration time: 1 hr Settling time: 1 hr	Non-volatile organics and inorganic constituents

TEST METHOD	LEACHING FLUID	L/S RATIO	MAXIMUM PARTICLE SIZE	NUMBER OF EXTRACTIONS	DURATION OF EXTRACTIONS	APPLICABLE CONSTITUENT GROUP(S)
Pancake Column Leachate Testing (PCLT)	Water (unspecified)	N/A	N/A	1		Not specified in method
Sequential Batch Leachate Test (SBLT)	Deionized water	4:1 (m/m)	N/A	4	24 hours	Non-volatile organics and inorganics
NEN 7341 Availability Test (The Netherlands)	Demineralized water at pH 7 and pH 4 using HNO ₃	50 (v/m)	<125 μm	2	3 Hours/step	"Inorganic components"
NEN 7343 Column Test (The Netherlands)	Demineralized acidified water, pH = 4	Seven leachate fractions with L/S range of 0.1 to 10 L/kg	95% < 4 mm	Seven consecutive	21 days	"Inorganic components"
NEN 7345 Tank Leach Test (The Netherlands)	Demineralized acidified water, pH = 4	5:1 (v/v)	Monolith > 40 mm	8	8 hours and 1, 2, 4, 9, 16, 36, and 64 days	"Inorganic components"
prEN 14405 Upflow percolation test (European Union)	Acidified water	0.1 to 10 (v/m)	< 4 mm			Inorganic constituents
EN 12457/1-4, Compliance Test for Granular Waste Materials and Sludges (European Union)	Deionized water, pH not controlled	2 L/kg and 10 L/kg	< 4 mm and < 10 mm	Up to 4		Inorganic constituents

Blank cells indicate information was not available

Laboratory	Location	Contact
AAA Lab, Inc.	Cheney, WA	Cheryl Blake
ACZ Laboratories, Inc.	Steamboat Springs, CO	Kristen Russell
Alpha Analytical Labs	Westborough, MA	Laboratory Technical Director
AMRO Environmental Laboratories, Inc.	Merrmack, NH	Nicole Borduz
AMTest, Inc.	Redmond, WA	Kathy Fugiel
Analytical Resources, Inc.	Tukwila, WA	Dave Mitchell
Ana Laboratories	Spokane, WA	Rusty Turner
Anatek Labs, Inc.	Spokane, WA	Kathleen Sattler
APPL, Inc.	Fresno, CA	Diane Anderson
Aquatic Research, Inc.	Seattle, WA	Steven Lazoff
Ardaman and Associates, Inc.	Orlando, FL	Ernest Cox
Avocet Environmental Testing	Bellingham, WA	Madell Briggs
B & P Laboratories, Inc.	Seattle, WA	Victor Broto
Brooks Rand, LLC	Seattle, WA	Colin Davies
Calscience Environmental Laboratories, Inc.	Garden Grove, CA	Laboratory Technical Director
Cascade Analytical, Inc.	Wenatchee, WA	Laura Mrachek
CCI Analytical Laboratories, Inc	Everett, WA	Chuck Rancatti
Center for Laboratory Sciences	Pasco, WA	Rich Westberg
Certes Environmental Laboratories, L.L.C	Houston, TX	Laboratory Technical Director

Appendix C – Laboratories Contacted to Determine Capabilities to Perform Leaching Tests

Laboratory	Location	Contact
CH2M Hill Applied Sciences Laboratory	Corvallis, OR	Ginger Collins
Clayton Services Group	Seattle, WA	Laboratory Technical Director
Coffey Laboratories, Inc.	Portland, OR	Laboratory Technical Director
Columbia Analytical Services	Kelso, WA	Lee Wolf
Columbia Analytical Services	Jacksonville, FL	Joe Wiegel
Curtis & Tompkins, Ltd.	Berkeley, CA	Mike Pearl
DataChem Laboratories Inc.	Salt Lake City, UT	Brent Stephens
Edge Analytical, Inc.	Burlington, WA	Lawrence Henderson
EMAX Laboratories, Inc.	Torrance, CA	Jim Carter
Energy Northwest Environmental Laboratory	Richland, WA	Terry Northstrom
Environmental Testing and Consulting, Inc.	Memphis, TN	Nathan Pera
Everett Environmental Laboratory	Everett, WA	Christiane Khairzada
Frontier Analytical Laboratories	El Dorado Hills, CA	Nial Maloney
Harbor Branch Environmental	Fort Pierce, FL	Kathleen Cooney
Keck Geoenvironmental Laboratory	Hooboken, NJ	Dimitris Dermatas
Keeco Analytical Laboratory	Butte, MT	Marcee Cameron
Kemron Environmental Servives	Marietta, OH	Mike Cochran
Kinnetic Laboratories, Inc.	Santa Cruz, CA	Mr. Kinney
Kuo Testing Labs, Inc.	Othello, WA	Eugene Kuo
Lancaster Laboratories, Inc.	Lancaster, PA	Laboratory Technical Director
Laucks Testing Laboratories, Inc.	Seattle, WA	Harry Romberg

Laboratory	Location	Contact
Mountain States Analytical, Inc.	Salt Lake City, UT	Rolf Larsen
MSE Technology Applications, Inc.	Butte, MT	Neal Egan
MWH Laboratories	Los Angeles, CA	Nilda Cox
NEL Laboratories, Inc.	Las Vegas, NV	Lydia Coleman
North Creek Analytical	Bothell, WA	Dave Wunderlich
North Creek Analytical	Spokane, WA	Emily Carfioli
Northwest Agricultural Consultants, Inc.	Kennewick, WA	Bob Sickles
Oilab Environmental	Oklahoma City, OK	Javier Gamarra
Pace Analytical Services, Inc.	Minneapolis, MN	Sarah Cherney
Pacific Northwest National Laboratory	Richland, WA	Laboratory Technical Director
Paragon Analytics, Inc.	Fort Collins, CO	Laboratory Technical Director
PSC Analytical Services	Renton, WA	Thomas Zhu
Sequoia Analytical Laboratories	Morgan Hill, CA	Tim Costello
Severn Trent Laboratories	Richland, WA	Larry Penfold
Severn Trent Laboratories	Tacoma, WA	Terri Howard
Severn Trent Laboratories	Baltimore, MD	Tara Martz
Soil Technology, Inc.	Bainbridge Island, WA	Tony Parkins
Soiltest Farm Consultants, Inc.	Moses Lake, WA	Brent Thyssen
Specialty Analytical	Tualatin, WA	Marty French
Spectra Laboratories, Inc.	Tacoma, WA	Gary Allen
Spectrum Laboratories, Inc.	Ft. Lauderdale, FL	Donald McCorquodale

Laboratory	Location	Contact
SPL, Inc.	Houston, TX	Laboratory Technical Director
SVL Analytical, Inc,	Kellogg, ID	Paul Duerkson
TestAmerica Analytical Testing, Corp.	Nashville, TN	Mary Louise Linn
Triangle Laboratories, Inc.	Durham, NC	Lauren Tochacek
TriMatrix Laboratories, Inc.	Grand Rapids, MI	Jay Bernarding
Twiss Analytical Laboratories, Inc.	Poulsbo, WA	Steve Twiss
Valley Environmental Laboratory	Yakima, WA	Dara Russel
Versar, Inc	Springfield, VA	Ted Prociv
Water Management Laboratories, Inc.	Tacoma, WA	Chris Mueller