Part I

Getting Started

Chapter 2

Characterizing Waste

Contents

Characterizing Waste

This chapter will help you:

- *Understand the industrial processes that generate a waste.*
- *Determine the waste's physical and chemical properties.*
- *Estimate constituent leaching to facilitate ground-water risk analysis.*
- *Quantify total constituent concentrations to facilitate air emissions analysis.*

Understanding the physical and chemical properties of a waste using sampling and analysis techniques is the cornerstone upon which subsequent steps in the Guide are built. It is necessary for gauging what risks a waste might pose to surface water, ground water, and air and drives waste management unit design and operating decisions. Knowing the composition of the waste is also necessary when determining the constituents for which to test. And, as discussed in Chapter 3–Integrating Pollution Prevention, knowledge of the physical and chemical properties of the waste is crucial in identifying pollution prevention opportunities.

In many instances, you can use knowledge of waste generation processes, analytical testing, or some combination of the two to estimate waste generation rates and waste constituent concentrations. To the extent that the waste is not highly variable, the use of process knowledge can be a sound approach to waste characterization and can prove more reasonable and cost effective than frequent sampling of the waste. It is important to note, however, that owners or operators using process knowledge to characterize a waste in lieu of testing are still responsible for the accuracy of their determinations. No matter what approach is used in characterizing a waste, the goal is to maximize the available knowledge that is necessary to make the important decisions described in later chapters of the Guide. Also, as changes are made to the industrial processes or waste management practices, it might be necessary to recharacterize a waste in order to accurately make waste management decisions and evaluate risk.

This chapter will help you address the following questions:

- How can process knowledge be used to characterize a waste?
- Which constituent concentrations should be quantified?
- Which type of leachate test should be used?

In considering the use of process knowledge or analytical testing, it is important to note that the ground water and air emissions models that accompany the Guide use constituent concentrations to estimate risk. Input requires specific concentrations which cannot be precisely estimated solely by knowledge of the processes that generate the waste. Further, when wastes are placed in a waste management unit, such as a landfill or surface impoundment, they are subjected to various physical, chemical, and biological processes that can result in the creation of new compounds in the waste, changes in the mass and volume of the waste, and the creation of different phases within the waste and within the landfill or impoundment. In order to accurately predict the concentration of the contaminants in the leachate, these changes must be accounted for.

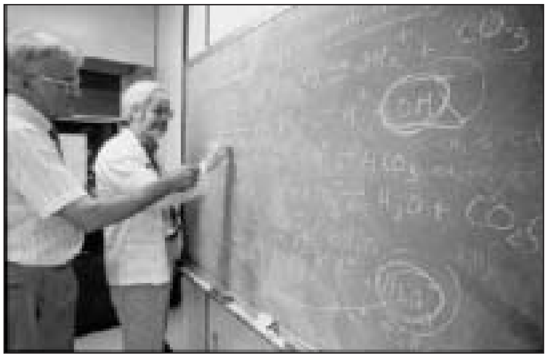
Accurate waste management unit constituent characterization is also necessary for input to the modeling tools provided in the Guide. Because model input requires specific data, model output will be based on the accuracy of the data input. Process knowledge alone (unless based on previous testing) might not be sufficiently accurate to yield reliable results. Leachate testing (discussed later in this chapter), for example, will likely give you a more precise assessment of waste constituent concentrations than process knowledge. Also note that whether you are using process knowledge, testing, or a combination of both, sources of model input data must be well documented so that an individual evaluating the modeling results understands the background supporting the assessment.

I. Waste Characterization Through Process Knowledge

A waste characterization begins with an understanding of the industrial processes that generate a waste. You must obtain enough information about the process to enable proper characterization of the waste, for example, by reviewing process flow diagrams or plans and determining all inputs and outputs. You should also be familiar with other waste characteristics such as the physical state of the waste, the volume of waste produced, and the general composition of the waste. In addition, many industries have thoroughly tested and characterized their wastes over time, therefore it might be beneficial to contact your trade association to determine if waste characterizations have already been performed and are available for processes similar to yours. Additional resources can assist in waste characterization by providing information on waste constituents and potential concentrations. Some examples include:

- Chemical engineering designs or plans for the process, showing process input chemicals, expected primary and secondary chemical reactions, and products.
- Material Safety Data Sheets (MSDSs) for materials involved. (Note that not all MSDSs contain information on all constituents found in a product.)
- Manufacturer's literature.
- Previous waste analyses.
- Literature on similar processes.
- Preliminary testing results, if available.

A material balance exercise using process knowledge can be useful in understanding where wastes are generated within a process and in estimating concentrations of waste constituents particularly where analytical test data



are limited. In a material balance, all input streams, such as raw materials fed into the processes, and all output streams, such as products produced and waste generated, are calculated. Flow diagrams can be used to identify important process steps and sources where wastes are generated. Characterizing wastes using material balances can require considerable effort and expense, but can help you to develop a more complete picture of the waste generation process(es) involved.

Note that a thorough assessment of your production processes can also serve as the starting point for facility-wide waste reduction, recycling, or pollution prevention efforts. Such an assessment will provide the information base to explore many opportunities to reduce or recycle the volume or toxicity of wastes. Refer to Chapter 3–Integrating Pollution Prevention for ideas, tools, and references on how to proceed.

While the use of process knowledge is attractive because of the cost savings associated with using existing information, you must ensure that this information accurately characterizes your wastes. If using process descriptions, published data, and documented studies to determine waste characteristics, the data should be scrutinized carefully to determine if there are any differences between the processes in the studies and the waste generating process at your facility, that the studies are acceptable and accurate (i.e., based on valid sampling and analytical techniques), and that the information is current.

If there are discrepancies, or if you begin a new process or change any of the existing processes at your facility (so that the documented studies and published data are no longer applicable), you are encouraged to consider performing additional sampling and laboratory analysis to accurately characterize the waste and ensure proper management. Also, if process knowledge is used in addition to, or in place of, sampling and analysis, you should clearly document the information used in your characterization assessment to demonstrate to regulatory agencies, the public, and other interested parties that the information accurately and completely characterizes the waste. The source of this information should be clearly documented.

What is process knowledge?

Process knowledge refers to detailed information on processes that generate wastes. It can be used to partly, or in many cases completely, characterize waste to ensure proper management. Process knowledge includes:

- Existing published or documented waste analysis data or studies conducted on wastes generated by processes similar to that which generated the waste.
- Waste analysis data obtained from other facilities in the same industry.
- Facility's records of previously performed analyses.

II. Waste Characterization Through Leachate Testing

Although sampling and laboratory analysis is not as economical and might not be as convenient as using process knowledge, it does have advantages. The resulting data usually provide the most accurate information available on constituent concentration levels.

Incomplete or mis-characterization of waste can lead to improper waste management, inaccurate modeling outputs, or erroneous decisions concerning the type of unit to be used, liner selection, or choice of land application methods. Note that process knowledge allows you to eliminate unnecessary or redundant waste testing by helping you focus on which constituents to measure in the waste. Again, thorough documentation of both the process knowledge used (e.g., studies, published data), as well as the analytical data is important.

The intent of leachate and extraction testing is to estimate the leaching potential of constituents of concern to water sources. It is important to estimate leaching potential in order to accurately estimate the quantity of chemicals that could potentially reach ground- or surface-water resources (e.g., drinking water supply wells, waters used for recreation). The Industrial Waste Management Evaluation Model (IWEM) developed for the Guide uses expected leachate concentrations for the waste management units as the basis for liner system design recommendations. Leachate tests will allow you to accurately quantify the input terms for modeling.

If the total concentration of all the constituents in a waste has been estimated using process knowledge (which could include previous testing data on wastes known to be very similar), estimates of the maximum possible concentration of these constituents in leachate can be made using the dilution ratio of the leachate test to be performed.

For example, the Toxicity Characteristic Leachate Procedure (TCLP) allows for a total constituent analysis in lieu of performing the test for some wastes. If a waste is 100 percent solid, as defined by the TCLP method, then the results of the total compositional analysis may be divided by twenty to convert the total results into the maximum leachable concentration[1]. This factor is derived from the 20:1 liquid to solid ratio employed in the TCLP. This is a conservative approach to estimating leachate concentrations and does not factor in environmental influences, such as rainfall. If a waste has filterable liquid, then the concentration of each phase (liquid and solid) must be determined. The following equation may be used to calculate this value:[2]

$$\frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + 20V_2}$$

Where:

V_1 = Volume of the first phase (L)

C_1 = Concentration of the analyte of concern in the first phase (mg/L)

V_2 = Volume of the second phase (L)

C_2 = Concentration of the analyte of concern in the second phase (mg/L)

Because this is only a screening method for identifying an upper-bound TCLP leachate concentration, you should consult with your state or local regulatory agency to determine whether process knowledge can be used to accurately estimate maximum risk in lieu of leachate testing.

A. Sampling and Analysis Plan

One of the more critical elements in proper waste characterization is the plan for sampling and analyzing the waste. The sampling plan is usually a written document that describes the objectives and details of the individual tasks of

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[1] This method is only appropriate for estimating maximum constituent concentration in leachate for non-liquid wastes (e.g., those wastes not discharged to a surface impoundment). For surface impoundments, the influent concentration of heavy metals can be assumed to be the maximum theoretical concentration of metals in the leachate for purposes of input to the ground-water modeling tool that accompanies this document. To estimate the leachate concentration of organic constituents in liquid wastes for modeling input, you will need to account for losses occurring within the surface impoundment before you can estimate the concentration in the leachate (i.e., an effluent concentration must be determined for organics).

[2] Source: Office of Solid Waste Web site at **<www.epa.gov/sw-846/sw846.htm>**.

a sampling effort and how they will be performed. This plan should be carefully thought out, well in advance of sampling. The more detailed the sampling plan, the less opportunity for error or misunderstanding during sampling, analysis, and data interpretation.

To ensure that the sampling plan is designed properly, a wide range of personnel should be consulted. It is important that the following individuals are involved in the development of the sampling plan to ensure that the results of the sampling effort are precise and accurate enough to properly characterize the waste:

- An engineer who understands the manufacturing processes.
- An experienced member of the sampling team.
- The end user of the data.
- A senior analytical chemist.
- A statistician.
- A quality assurance representative.

It is also advisable that you consult the analytical laboratory to be used when developing your sampling plan.

Background information on the processes that generate the waste and the type and characteristics of the waste management unit is essential for developing a sound sampling plan. Knowledge of the unit location and situation (e.g., geology, exposure of the waste to the elements, local climatic conditions) will assist in determining correct sample size and sampling method. Sampling plan design will depend on whether you are sampling a waste prior to disposal in a waste management unit or whether you are sampling waste from an existing unit. When obtaining samples from an existing unit, care should be taken to avoid endangering the individuals collecting the samples and to prevent damaging the unit itself. Reasons for obtaining samples from an existing unit include, characterizing the waste in the unit to determine if the new waste being added is compatible, checking to see if the composition of the waste is changing over time due to various chemical and biological breakdown processes, or characterizing the waste in the unit or the leachate from the unit to give an indication of expected concentrations in leachate from a new unit.

The sampling plan must be correctly defined and organized in order to get an accurate estimation of the characteristics of the waste. Both an appropriate sample size and proper sampling techniques are necessary. If the sampling process is carried out correctly, the sample will be representative and the estimates it generates will be useful for making decisions concerning proper management of the waste and for assessing risk.

In developing a sampling plan, accuracy is of primary concern. The goal of sampling is to get an accurate estimate of the waste's characteristics from measuring the sample's characteristics. The main controlling factor in deciding whether the estimates will be accurate is how representative the sample is (discussed in the following section). Using a small sample increases the possibility that the sample will not be representative, but a sample that is larger than the minimum calculated sample size does not necessarily increase the probability of getting a representative sample.

As you are developing the sampling plan, you should address the following considerations:

- Data quality objectives.
- Determination of a representative sample.
- Statistical methods to be employed in the analyses.
- Waste generation and handling processes.

- Constituents/parameters to be sampled.
- Physical and chemical properties of the waste.
- Accessibility of the unit.
- Sampling equipment, methods, and sample containers.
- Quality assurance and quality control (e.g., sample preservation and handling requirements).
- Chain-of-custody.
- Health and safety of employees.

Many of these considerations are discussed below. Additional information on data quality objectives and quality assurance and quality control can be found in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods—SW-846* (U.S. EPA, 1996e), *Guidance for the Data Quality Objectives Process* (U.S. EPA, 1996b), *Guidance on Quality Assurance Project Plans* (U.S. EPA, 1998a), and *Guidance for the Data Quality Assessment: Practical Methods for Data Analysis* (U.S. EPA, 1996a).[3]

A determination as to the constituents that will be measured can be based on process knowledge to narrow the focus and expense of performing the analyses. Analyses should be performed for those constituents that are reasonably expected to be in the waste at detectable levels (i.e., test method detection levels). Note that the Industrial Waste Management Evaluation Model (IWEM) that accompanies this document recommends liner system designs, if necessary, or the appropriateness of land application based on calculated protective leachate thresholds (Leachate Concentration Threshold Values or LCTVs) for various constituents that are likely to be found in industrial waste and pose hazards at certain levels to people and the environment. The constituents that are evaluated are listed in Table 1.2 of the *Industrial Waste Management Evaluation Model Technical Background Document* (U.S. EPA 2002). The LCTV tables also are included in the *IWEM Technical Background Document* and the model on the CD-ROM version of this Guide, and can be used as a starting point to help you determine which constituents to measure. It is not recommended that you sample for all of the organic chemicals and metals listed in the tables, but rather use these tables as a guide in conjunction with knowledge concerning the waste generating practices to determine which constituents to measure.

1. Representative Waste Sampling

The first step in any analytical testing process is to obtain a sample that is representative of the physical and chemical composition of a waste. The term "representative sample" is commonly used to denote a sample that has the same properties and composition in the same proportions as the population from which it was collected. Finding one sample which is representative of the entire waste can be difficult unless you are dealing with a homogenous waste. Because most industrial wastes are not homogeneous, many different factors should be considered in obtaining samples. Examples of some of the factors that should be considered include:

- **Physical state of the waste.** The physical state of the waste affects most aspects of a sampling effort. The sampling device will vary according to whether the sample is liquid, solid, gas, or multiphasic. It will also vary according to whether the liquid is viscous or free-flowing, or whether the solid is hard, soft, powdery, monolithic, or clay-like.
- **Composition of the waste.** The samples should represent the average concentration and variability of the waste in time or over space.

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[3] These and other EPA publications can be found at the National Environmental Publications Internet site (NEPIS) at **<www.epa.gov/ncepihom/nepishom/>**.

- **Waste generation and handling processes.** Processes to consider include: if the waste is generated in batches; if there is a change in the raw materials used in a manufacturing process; if waste composition can vary substantially as a function of process temperatures or pressures; and if storage time affects the waste's characteristics/composition.
- **Transitory events.** Start-up, shut-down, slow-down, and maintenance transients can result in the generation of a waste that is not representative of the normal waste stream. If a sample was unknowingly collected at one of these intervals, incorrect conclusions could be drawn.

You should consult with your state or local regulatory agency to identify any legal requirements or preferences before initiating sampling efforts. Refer to Chapter 9 of the EPA's SW-846 test methods document (see side bar) for detailed guidance on planning, implementing, and assessing sampling events.

To ensure that the chemical information obtained from waste sampling efforts is accurate, it must be unbiased and sufficiently precise. Accuracy is usually achieved by incorporating some form of randomness into the sample selection process and by selecting an appropriate number of samples. Since most industrial wastes are heterogeneous in terms of their chemical properties, unbiased samples and appropriate precision can usually be achieved by simple random sampling. In this type of sampling, all units in the population (essentially all locations

More information on Test Methods for Evaluating Solid Waste, Physical/Chemical Methods—SW-846

EPA has begun replacing requirements mandating the use of specific measurement methods or technologies with a performance-based measurement system (PBMS). The goal of PBMS is to reduce regulatory burden and foster the use of innovative and emerging technologies or methods. The PBMS establishes what needs to be accomplished, but does not prescribe specifically how to do it. In a sampling situation, for example, PBMS would establish the data needs, the level of uncertainty acceptable for making decisions, and the required supporting documentation; a specific test method would not be prescribed. This approach allows the analyst the flexibility to select the most appropriate and cost effective test methods or technologies to comply with the criteria. Under PBMS, the analyst is required to demonstrate the accuracy of the measurement method using the specific matrix that is being analyzed. SW-846 serves only as a guidance document and starting point for determining which test method to use.

SW-846 provides state-of-the-art analytical test methods for a wide array of inorganic and organic constituents, as well as procedures for field and laboratory quality control, sampling, and characteristics testing. The methods are intended to promote accuracy, sensitivity, specificity, precision, and comparability of analyses and test results.

For assistance with the methods described in SW-846, call the EPA Method Information Communication Exchange (MICE) Hotline at 703 676-4690 or send an e-mail to mice@cpmx.saic.com.

The text of SW-846 is available online at: **<www.epa.gov/sw-846/main.htm>**. A hard copy or CD-ROM version of SW-846 can be purchased by calling the National Technical Information Service (NTIS) at 800 553-6847.

or points in all batches of waste from which a sample could be collected) are identified, and a suitable number of samples is randomly selected from the population.

The appropriate number of samples to employ in a waste characterization is at least the minimum number of samples required to generate a precise estimate of the true mean concentration of a chemical contaminant in a waste. A number of mathematical formulas exist for determining the appropriate number of samples depending on the statistical precision required. Further information on sampling designs and methods for calculating required sample sizes and optimal distribution of samples can be found in Gilbert (1987), Winer (1971), and Cochran (1977) and Chapter 9 of EPA SW-846.

The type of sampling plan developed will vary depending on the sampling location. Solid wastes contained in a landfill or waste pile can be best sampled using a three-dimensional random sampling strategy. This involves establishing an imaginary three-dimensional grid or sampling points in the waste and then using random-number tables or random-number generators to select points for sampling. Hollow-stem augers combined with split-spoon samplers are frequently appropriate for sampling landfills.

If the distribution of waste components is known or assumed for liquid or semi-solid wastes in surface impoundments, then a two-dimensional simple random sampling strategy might be appropriate. In this strategy, the top surface of the waste is divided into an imaginary grid and grid sections are selected using random-number tables or random-number generators. Each selected grid point is then sampled in a vertical manner along the entire length from top to bottom using a sampling device such as a weighted bottle, a drum thief, or Coliwasa.

If sampling is restricted, the sampling strategy should, at a minimum, take sufficient samples to address the potential vertical variations in the waste in order to be considered representative. This is because contained wastes tend to display vertical, rather than horizontal, non-random heterogeneity due to settling or the layering of solids and denser liquid phases. Also, care should be taken when performing representative sampling of a landfill, waste pile, or surface impoundment to minimize any potential to create hazardous conditions. (It is possible that the improper use of intrusive sampling techniques, such as the use of augers, could accelerate leaching by creating pathways or tunnels that can accelerate leachate movement to ground water.)

To facilitate characterization efforts, consult with state and local regulatory agencies and a qualified professional to select a sampling plan and determine the appropriate number of samples, before beginning sampling efforts. You should also consider conducting a detailed waste-stream specific characterization so that the information can be used to conduct waste reduction and waste minimization activities.

Additional information concerning sampling plans, strategies, methods, equipment, and sampling quality assurance and quality control is available in Chapters 9 and 10 of the SW-846 test methods document. Electronic versions of these chapters have been included on the CD-ROM version of the Guide.

2. *Representative Waste Analysis*

After a representative sample has been collected, it must be properly preserved to maintain the physical and chemical properties that it possessed at the time of collection. Sample types, sample containers, container preparation, and sample preservation methods are critical for maintaining the integrity of the sample and obtaining accurate results. Preservation and holding times are also important factors to consider and will vary depending on the type of constituents being measured (e.g., VOCs, heavy metals, hydrocarbons) and the waste matrix (e.g., solid, liquid, semi-solid).

The analytical chemist then develops an analytical plan which is appropriate for the sample to be analyzed, the constituents to be analyzed, and the end use of the information required. The laboratory should have standard operating procedures available for review for the various types of analyses to be performed and for all associated methods needed to complete each analysis, such as instrument maintenance procedures, sample handling procedures, and sample documentation procedures. In addition, the laboratory should have a laboratory quality assurance/quality control statement available for review which includes all key personnel qualifications.

The SW-846 document contains information on analytical plans and methods. Another useful source of information regarding the selection of analytical methods and quality assurance/quality control procedures for various compounds is the Office of Solid Waste Methods Team home page at **<www.epa.gov/sw-846/index.htm>**.

B. Leachate Test Selection

Leaching tests are used to estimate potential concentration or amount of waste constituents that can leach from a waste to ground water. Typical leaching tests use a specified leaching fluid mixed with the solid portion of a waste for a specified time. Solids are then separated from the leaching solution and the solution is tested for waste constituent concentrations. The type of leaching test performed can vary depending on the chemical, biological, and physical characteristics of the waste; the environment in which the waste will be placed; as well as the recommendations or requirements of your state and local regulatory agencies.

When selecting the most appropriate analytical tests, consider at a minimum the physical state of the sample, the constituents to be analyzed, detection limits, and the specified holding times of the analytical methods.[4] It might not be cost-effective or useful to conduct a test with detection limits at or greater than the constituent concentrations in a waste. Process knowledge can help you predict whether the concentrations of certain constituents are likely to fall below the detection limits for anticipated methods.

After assessing the state of the waste, assess the environment of the waste management unit in which the waste will be placed. For example, an acidic environment might require a different test than a non-acidic environment in order to best reflect the conditions under which the waste will actually leach. If the waste management unit is a monofill, then the characteristics of the waste will determine most of the unit's conditions. Conversely, if many different wastes are being co-disposed, then the conditions created by

Which leaching test is appropriate?

Selecting an appropriate leachate test can be summarized in the following four steps:

1. Assess the physical state of the waste using process knowledge.
2. Assess the environment in which the waste will be placed.
3. Consult with your state and/or local regulatory agency.
4. Select an appropriate leachate test based on the above information.

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[4] There are several general categories of phases in which samples can be categorized: solids, aqueous, sludges, multiphase samples, ground water, and oil and organic liquid. You should select a test that is designed for the specific sample type.

the co-disposed wastes must be considered, including the constituents that can be leached by the subject waste.

A qualified laboratory should always be used when conducting analytical testing. The laboratory can be in-house or independent. When using independent laboratories, ensure that they are qualified and competent to perform the required tests. Some laboratories might be proficient in one test but not another. You should consult with the laboratory before finalizing your test selection to make certain that the test can be performed. When using analytical tests that are not frequently performed, additional quality assurance and quality control practices might need to be implemented to ensure that the tests are conducted correctly and that the results are accurate.

A brief summary of the TCLP and three other commonly used leachate tests is provided below (procedures for the EPA test methods are included in SW-846 and for the ASTM method in the *Annual Book of ASTM Standards*). These summaries are provided as background and are not meant to imply that these are the only tests that can be used to accurately predict leachate potential. Other leachate tests have been developed and might be suitable for testing your waste. The table in the appendix at the end of this chapter provides a summary of over 20 leachate tests that have been designed to estimate the potential for contaminant release, including several developed by ASTM.[5] You should consult with state and local regulatory agencies and/or a laboratory that is familiar with leachate testing methods to identify the most appropriate test and test method procedures for your waste and sample type.

1. Toxicity Characteristic Leaching Procedure (TCLP)

The TCLP[6] is the test method used to determine whether a waste is hazardous due to its characteristics as defined in the Resource Conservation and Recovery Act (RCRA), 40 CFR Part 261. The TCLP estimates the leachability of certain toxicity characteristic hazardous constituents from solid waste under a defined set of laboratory conditions. It evaluates the leaching of metals, volatile and semi-volatile organic compounds, and pesticides from wastes. The TCLP was developed to simulate the leaching of constituents into ground water under conditions found in municipal solid waste (MSW) landfills. The TCLP does not simulate the release of contaminants to non-ground water pathways. The TCLP is most commonly used by EPA, state, and local agencies to classify waste. It is also used to determine compliance with some land disposal restrictions (LDRs) for hazardous wastes. The TCLP can be found as EPA Method 1311 in SW-846.[7] A copy of Method 1311 has been included on the CD-ROM version of the Guide.

For liquid wastes, (i.e., those containing less than 0.5 percent dry solid material) the waste after filtration through a glass fiber fil-

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[5] EPA has only reviewed and evaluated those test methods found in SW-846. The EPA has not reviewed or evaluated the other test methods and cannot recommend use of any test methods other than those found in SW-846.

[6] EPA is undertaking a review of the TCLP test and how it is used to evaluate waste leaching (described in the Phase IV Land Disposal Restrictions rulemaking, 62 Federal Register 25997; May 26, 1998). EPA anticipates that this review will examine the effects of a number of factors on leaching and on approaches to estimating the likely leaching of a waste in the environment. These factors include pH, liquid to solid ratios, matrix effects and physical form of the waste, effects of non-hazardous salts on the leachability of hazardous metal salts, and others. The effects of these factors on leaching might or might not be well reflected in the leaching tests currently available. At the conclusion of the TCLP review, EPA is likely to issue revisions to this guidance that reflect a more complete understanding of waste constituent leaching under a variety of management conditions.

[7] The TCLP was developed to replace the Extraction Procedure Toxicity Test method which is designated as EPA Method 1310 in SW-846.

ter is defined as the TCLP extractant. The concentrations of constituents in the liquid extract are then determined.

For wastes containing greater than or equal to 0.5 percent solids, the liquid, if any, is separated from the solid phase and stored for later analysis. The solids must then be reduced to particle size, if necessary. The solids are extracted with an acetate buffer solution. A liquid-to-solid ratio of 20:1 by weight is used for an extraction period of 18 ± 2 hours. After extraction, the solids are filtered from the liquid through a glass fiber filter and the liquid extract is combined with any original liquid fraction of the wastes. Analyses are then conducted on the liquid filtrate/leachate to determine the constituent concentrations.

To determine if a waste is hazardous because it exhibits the toxicity characteristic (TC), the TCLP method is used to generate leachate under controlled conditions as discussed above. If the TCLP liquid extract contains any of the constituents listed in Table 1 of 40 CFR Part 261 at a concentration equal to or greater than the respective value in the table, the waste is considered to be a TC hazardous waste, unless exempted or excluded under Part 261. Although the TCLP test was designed to determine if a waste is hazardous, the importance of its use for waste characterization as discussed in this chapter is to understand the parameters to be considered in properly managing the wastes.

You should check with state and local regulatory agencies to determine whether the TCLP is likely to be the best test for evaluating the leaching potential of a waste or if another test might better predict leaching under the anticipated waste management conditions. Because the test was developed by EPA to determine if a waste is hazardous (according to 40 CFR 261.24) and focused on simulating leaching of solid wastes placed in a municipal landfill, this test might not be appropriate for your waste because the leaching potential for the same chemical can be quite different depending on a number of factors. These factors include the characteristics of the leaching fluid, the form of the chemical in the solids, the waste matrix, and the disposal conditions.

Although the TCLP is the most commonly used leachate test for estimating the actual leaching potential of wastes, you should not automatically default to it in all situations or conditions and for all types of wastes. While the TCLP test might be conservative under some conditions (i.e., overestimates leaching potential), it might underestimate leaching under other extreme conditions. In a landfill that has primarily alkaline conditions, the TCLP is not likely to be the optimal method because the TCLP is designed to replicate leaching in an acidic environment. For materials that pose their greatest hazard when exposed to alkaline conditions (e.g., metals such as arsenic and antimony), use of the TCLP might underestimate the leaching potential. When the conditions of your waste management unit are very different from the conditions that the TCLP test simulates, another test might be more appropriate. Further, the TCLP might not be appropriate for analyzing oily wastes. Oil phases can be difficult to separate (e.g., it might be impossible to separate solids from oil), oily material can obstruct the filter (often resulting in an underestimation of constituents in the leachate), and oily materials can yield both oil and aqueous leachate which must be analyzed separately.[8]

2. *Synthetic Precipitation Leaching Procedure (SPLP)*

The SPLP (designated as EPA Method 1312 in SW-846) is currently used by several state agencies to evaluate the leaching of con-

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[8] SW-846 specifies several procedures that should be followed when analyzing oily wastes.

stituents from wastes. The SPLP was designed to estimate the leachability of both organic and inorganic analytes present in liquids, soils, and wastes. The SPLP was originally designed to assess how clean a soil was under EPA's Clean Closure Program. Even though the federal hazardous waste program, did not adopt it for use, the test can still estimate releases from wastes placed in a landfill and subject to acid rain. There might be, however, important differences between soil as a constituent matrix (for which the SPLP is primarily used) and the matrix of a generated industrial waste. A copy of Method 1312 has been included on the CD-ROM version of the Guide.

The SPLP is very similar to the TCLP Method 1311. Waste samples containing solids and liquids are handled by separating the liquids from the solid phase, and then reducing solids to particle size. The solids are then extracted with a dilute sulfuric acid/nitric acid solution. A liquid-to-solid ratio of 20:1 by weight is used for an extraction period of 18±2 hours. After extraction, the solids are filtered from the liquid extract and the liquid extract is combined with any original liquid fraction of the wastes. Analyses are then conducted on the liquid filtrate/leachate to determine the constituent concentrations.

The sulfuric acid/nitric acid extraction solution used in the SPLP was selected to simulate leachate generation, in part, from acid rain. Also note that in both the SPLP and TCLP, some paint and oily wastes might clog the filters used to separate the liquid extract from the solids prior to analysis, resulting in under reporting of the extractable constituent concentrations.

3. *Multiple Extraction Procedure (MEP)*

The MEP (designated as EPA Method 1320 in SW-846) was designed to simulate the leaching that a waste will undergo from repetitive precipitation of acid rain on a landfill to determine the highest concentration of each constituent that is likely to leach in a real world environment. Currently, the MEP is used in EPA's hazardous waste delisting program. A copy of Method 1320 has been included on the CD-ROM version of the Guide.

The MEP can be used to evaluate liquid, solid, and multiphase samples. Waste samples are extracted according to the Extraction Procedure (EP) Toxicity Test (Method 1310 of SW-846). The EP test is also very similar to the TCLP Method 1311. A copy of Method 1310 has been included on the CD- ROM version of the Guide.

In the MEP, liquid wastes are filtered through a glass fiber filter prior to testing. Waste samples containing both solids and liquids are handled by separating the liquids from the solid phase, and then reducing the solids to particle size. The solids are then extracted using an acetic acid solution. A liquid- to-solid ratio of 16:1 by weight is used for an extraction period of 24 hours. After extraction, the solids are filtered from the liquid extract, and the liquid extract is combined with any original liquid fraction of the waste.

The solids portion of the sample that remains after application of Method 1310 are then re-extracted using a dilute sulfuric acid/nitric acid solution. As in the SPLP, this acidic solution was selected to simulate leachate generation, in part, from acid rain. This time a liquid-to-solid ratio of 20:1 by weight is used for an extraction period of 24 hours. After extraction, the solids are once again filtered from the liquid extract, and the liquid extract is combined with any original liquid fraction of the waste.

These four steps are repeated eight additional times. If the concentration of any constituent of concern increases from the 7th or 8th extraction to the 9th extraction, the procedure is repeated until these concentrations decrease.

The MEP is intended to simulate 1,000 years of freeze and thaw cycles and prolonged exposure to a leaching medium. One advantage of the MEP over the TCLP is that the MEP gradually removes excess alkalinity in the waste. Thus, the leaching behavior of metal contaminants can be evaluated as a function of decreasing pH, which increases the solubility of most metals.

4. Shake Extraction of Solid Waste with Water or Neutral Leaching Procedure

The Shake Extraction of Solid Waste with Water, or the Neutral Leaching Procedure, was developed by the American Society for Testing and Materials (ASTM) to assess the leaching potential of solid waste and has been designated as ASTM D-3987-85. This test method provides for the shaking of an extractant (e.g., water) and a known weight of waste of specified composition to obtain an aqueous phase for analysis after separation. The intent of this test method is for the final pH of the extract to reflect the interaction of the liquid extractant with the buffering capacity of the solid waste.

The shake test is performed by mixing the solid sample with test water and agitating continuously for 18±0.25 hours. A liquid-to-solid ratio of 20:1 by weight is used. After agitation the solids are filtered from the liquid extract, and the liquid is analyzed.

The water extraction is meant to simulate conditions where the solid waste is the dominant factor in determining the pH of the extract. This test, however, has only been approved for certain inorganic constituents, and is not applicable to organic substances and volatile organic compounds (VOCs). A copy of this procedure can be ordered by calling ASTM at 610 832-9585 or online at <**www.astm.org**>.

III. Waste Characterization of Volatile Organic Emissions

To determine whether volatile organic emissions are of concern at a waste management unit, determine the concentration of the VOCs that are reasonably expected to be emitted. Process knowledge is likely to be less accurate for determining VOCs than measured values. As discussed earlier in this chapter, modeling results for waste management units will only be as accurate as the input data. Therefore, sampling and analytical testing might be necessary if organic concentrations cannot be estimated confidently using process knowledge.

Table 2 in Chapter 5–Protecting Air Quality can be used as a starting point to help you determine which air emissions constituents to measure. It is not recommended that you sample for all of the volatile organics listed in Table 2, but rather use Table 2 as a guide in conjunction with process knowledge to narrow the sampling effort and thereby minimize unnecessary sampling costs. A thorough understanding of process knowledge can help you determine what is reasonably expected to be in the waste, so that it is not necessary to sample for unspecified constituents.

Many tests have been developed for quantitatively extracting volatile and semi-volatile organic constituents from various sample matrices. These tests tend to be highly dependent upon the physical characteristics of the sample. You should consult with state and local regulatory agencies before implementing testing. You can refer to SW-846 Method 3500B for guidance on the selection of methods for quantitative extraction or dilution of samples for analysis by one of the volatile or semi-volatile determinative methods. After performing the appropriate extraction procedure, further cleanup of the sample extract might be necessary if analysis of the extract is prevented due to interferences coextracted from the sample. Method 3600 of SW-846 provides additional guidance on cleanup procedures.

Following sample preparation, a sample is ready for further analysis. Most analytical methods use either gas chromatography (GC), high performance liquid chromatography (HPLC), gas chromatography/mass spectrometry (GC/MS), or high performance liquid chromatography/mass spectrometry (HPLC/MS). SW-846 is designed to allow the methods to be mixed and matched, so that sample preparation, sample cleanup, and analytical methods can be properly sequenced for the particular analyte and matrix. Again, you should consult with state and local regulatory agencies before finalizing the selected methodology.

Waste Characterization Activity List

To determine constituent concentrations in a waste you should:

- ☐ Assess the physical state of the waste using process knowledge.
- ☐ Use process knowledge to identify constituents for further analysis.
- ☐ Assess the environment in which the waste will be placed.
- ☐ Consult with state and local regulatory agencies to determine any specific testing requirements.
- ☐ Select an appropriate leachate test or organic constituent analysis based on the above information.

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Appendix: Example Extraction Tests (Draft 9/30/97)

Test Method	Leaching Fluid	Liquid:Solid Ratio	Maximum Particle Size	Number of Extractions	Time of Extractions	Comments
			I. Static Tests			
			A. Agitated Extraction Tests			
Toxicity Characteristic Leaching Procedure (1311)	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	9.5 mm	1	18 ±2 hours	Co-disposal scenario might not be appropriate; no allowance for structural integrity testing of monolithic samples
Extraction Procedure Toxicity Test (1310)	0.5 N acetic acid (pH-5.0)	16:1 during extraction 20:1 final dilution	9.5 mm	1	24 hours	High alkalinity samples can result in variable data
ASTM D3987-85 Shake Extraction of Solid Waste with Water	ASTM IV reagent water	20:1	As in environment (as received)	1	18 hours	Not validated for organics
California WET	0.2 M sodium citrate (pH- 5.0)	10:1	2.0 mm	1	48 hours	Similar to EP, but sodium citrate makes test more aggressive
Ultrasonic Agitation Method for Accelerating Batch Leaching Test[9]	Distilled water	4:1	Ground	1	30 minutes	New—little performance data
Alternative TCLP for Construction, Demolition and Lead Paint Abatement Debris[10]	TCLP acetic acid solutions	20:1	<9.5	1	8 hours	Uses heat to decrease extraction time
Extraction Procedure for Oily Waste (1330)	Soxhlet with THF and toluene EP on remaining solids	100g:300mL 20:1	9.5 mm	3	24 hours (EP)	
Synthetic Precipitation Leaching Procedure (1312)	#1 Reagent water to pH 4.2 with nitric and sulfuric acids (60/40) #2 Regent water to pH 5.0 with nitric and sulfuric acids (60/40)	20:1	9.5 mm	1	18±2 hours	ZHE option for organics
Equilibrium Leach Test	Distilled water	4:1	150 mm	1	7 days	Determines contaminants that have been insolubilized by solidification

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Test Method	Leaching Fluid	Liquid:Solid Ratio	Maximum Particle Size	Number of Extractions	Time of Extractions	Comments
B. Non-Agitated Extraction Tests						
Static Leach Test Method (material characteristic centre- 1)	Can be site specific, 3 standard leachates: water, brine, silicate/bicarbonate	VOL/surface 10 cm	40 mm^2 surface area	1	>7 days	Series of optional steps increasing complexity of analysis
High Temperature Static Leach Tests Method (material characterization centre-2)	Same as MCC-1 (conducted at 100°C)	VOL/Surface 10 cm	40 mm^2 Surface Area	1	>7 Days	Series of optional steps increasing complexity of analysis
C. Sequential Chemical Extraction Tests						
Sequential Extraction Tests	0.04 m acetic acid	50:1	9.5 mm	15	24 hours per extraction	
D. Concentration Build-Up Test						
Sequential Chemical Extraction	5 leaching solutions of increasing acidity	Varies from 16.1 to 40.1	150 mm	5	Varies 3 or 14 days	Examines partitioning of metals into different fractions or chemicals forms
Standard Leach Test, Procedure C (Wisconsin)	DI water SYN Landfill	10:1, 5:1, 7.5:1	As in environment	3	3 or 14 days	Sample discarded after each leach, new sample added to existing leachate
II. Dynamic Tests (Leaching Fluid Renewed)						
A. Serial Batch (Particle)						
Multiple Extraction Procedure (1320)	Same as EP TOX, then with synthetic acid rain (sulfuric acid, nitric acid in 60:40% mixture)	20:1	9.5 mm	9 (or more)	24 hours per extraction	
Monofill Waste Extraction Procedures	Distilled/deionized water or other for specific site	10:1 per extraction	9.5 mm or monolith	4	18 hours per extraction	
Graded Serial Batch (U.S. Army)	Distilled water	Increases from 2:1 to 96:1	N/A	>7	Until steady state	
Sequential Batch Ext. of Waste with Water ASTM D-4793-93	Type IV reagent water	20:1	As in environment	10	18 hours	

Test Method	Leaching Fluid	Liquid:Solid Ratio	Maximum Particle Size	Number of Extractions	Time of Extractions	Comments
Use of Chelating Agent to Determine the Metal Availability for Leaching Soils and Wastes[11]	Demineralized water with EDTA, sample to a final pH of 7±0.5	50 or 100	<300 µm	1	18, 24, or 48 hours	Experimental test based on Method 7341
B. Flow Around Tests						
IAEA Dynamic Leach Test (International Atomic Energy Agency)	DI water/site water	N/A	One face prepared	>19	>6 months	
Leaching Tests on Solidified Products[12]	0.1N acetic acid	20:1 (Procedure A) 2:1 (6 hrs.) & 10:1 (18 hrs.) (Procedure B)	0.6 µm-70µm	1	24 hours	S/S technologies most valid when applied to wastes contaminated by inorganic pollutants
DLT	DI water	N/A	Surface washing	18	196 days	
C. Flow Through Tests						
ASTM D4874-95 Column Test	Type IV reagent water	One void volume	10 mm	1	24 hours	
III. Other Tests						
MCC-5s Soxhlet Test (material characteristic center)	DI/site water	100:1	Out and washed	1	0.2 ml/min	
ASTM C1308-95 Accelerated Leach Test[13]						Only applicable if diffusion is dominant leaching mechanism
Generalized Acid Neutralization Capacity Test[14]	Acetic acid	20:1	Able to pass through an ASTM No. 40 sieve	1	48 hours	Quantifies the alkalinity of binder and characterizes buffering chemistry
Acid Neutralization Capacity	HNO_3, solutions of increasing strength	3:1	150 mm	1	48 hours per extraction	

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[11] Garrabrants, A.C. and Koson, D.S.; Use of Chelating Agent to Determine the Metal Availability for Leaching from Soils and Wastes, unpublished.

[12] Leaching Tests on Solidified Products; Gavasci, R., Lombardi, F., Polettine, A., and Sirini, P.

[13] C1308-95 Accelerated Leach Test for Diffusive Releases from Solidified Waste and a Computer Program to Model Diffusive, Fractional Leaching from Cylindrical Wastes.

[14] Generalized Acid Neutralization capacity Test; Isenburg, J. and Moore, M.