A PROCEDURE FOR ESTIMATING MONOFILLED SOLID WASTE LEACHATE COMPOSITION

Technical Resource Document
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DISCLAIMER

This report has received extensive technical review, but the Agency's peer and administrative review process has not yet been completed. Therefore it does not necessarily reflect the views or policies of the Agency. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

FOREWORD

The Environmental Protection Agency was created because of increasing public and governmental concern about the dangers of pollution to the health and welfare of the American people. Noxious air, foul water, and spoiled land are tragic testimony to the deterioration of our natural environment. The complexity of the environment and the interplay between its components require a concentrated and integrated attack on the problem.

The Office of Solid Waste is responsible for issuing regulations and guidelines on the proper treatment, storage, and disposal of hazardous wastes to protect human health and the environment from the potential harm associated with improper management of these wastes. These regulations are supplemented by guidance manuals, technical guidelines, and technical resource documents, made available to assist the regulated community and facility designers in understanding the scope of the regulatory program. Publications like this one provide facility designers with state-of-the-art information on design and performance evaluation techniques.

This technical resource document describes a laboratory procedure for generating aqueous extracts from industrial solid wastes that are candidates for monofill disposal. Although the extracts produced are not necessarily representative of leachate that would be produced in a land disposal environment, the procedure can be used to estimate the quantity of potentially leachable constituents in a solid waste and potential concentrations of these constituents in aqueous leachates produced from the waste at a given solid-to-liquid ratio.

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PREFACE

Subtitle C of the Resource Conservation and Recovery Act (RCRA) requires the Environmental Protection Agency (EPA) to establish a Federal hazardous waste management program. This program must ensure that hazardous wastes are handled safely from generation until final disposition. EPA issued a series of hazardous waste regulations under Subtitle C of RCRA that are published in 40 Code of Federal Regulations (CFR) through 265 and 122 through 124.

Parts 264 and 265 of 40 CFR contain standards applicable to owners and operators of all facilities that treat, store, or dispose of hazardous wastes. Wastes are identified or listed as hazardous under 40 CFR Part 261. The Part 264 standards are implemented through permits issued by authorized states or the EPA in accordance with 40 CFR Part 122 and Part 124 regulations. Land treatment, storage, and disposal (LTSD) regulations in 40 CFR Part 264 issued on July 26, 1982, establish performance standards for hazardous waste landfills, surface impoundments, land treatment units,. and wastepiles.

The Environmental Protection Agency is developing three types of documents for preparers and reviewers of permit applications for hazardous waste LTSD facilities. These types include RCRA technical guidance documents, permit guidance manuals, and technical resource documents (TRD's).

The RCRA Technical Guidance Documents present design and operating specifications or design evaluation techniques that generally comply with or demonstrate compliance with the design and operating requirements and the closure and post-closure requirements of Part 264.

The Permit Guidance Manuals are being developed to describe the permit application information the Agency seeks and to provide guidance to applicants and permit writers in addressing the information requirements. These manuals will include a discussion of each step in the permitting process, and a description of each set of specifications that must be considered for inclusion in the permit.

The Technical Resource Documents present state-of-the-art summaries of technologies and evaluation techniques determined by the Agency to constitute good engineering designs, practices, and procedures. They support the RCRA Technical Guidance Documents and Permit Guidance Manuals in certain areas (e.g., liners, leachate management, closure covers, water balance) by describing current technologies and methods for designing hazardous waste facilities or for evaluating the performance of a facility design. Although emphasis is given to hazardous waste facilities, the information presented in these TRD's may be used in designing and operating nonhazardous waste LTSD facilities as well. Whereas the RCRA Technical Guidance Documents and Permit Guidance Manuals are directly related to the regulations, the information in these TRD's covers a broader perspective and should not be used to interpret the requirements of the regulations.

This document has undergone review by recognized experts in the technical areas covered. Their comments, and recommendations for improvement, have been incorporated whenever possible. The Agency intends to update these TRD's periodically as warranted. Communications on any of the TRD's should be addressed to Docket Clerk, Room S-212(A), Office of Solid Waste (WH-562), U.S. Environmental Protection Agency, 401 M Street, S.W., Washington, D.C. 20460. The document under discussion should be identified by title and number (i.e. "A Procedure for Estimating Monofilled Solid Waste Leachate Composition, SW-924").

ABSTRACT

The monofilled waste extraction procedure (MWEP) is a laboratory procedure developed for generating aqueous extracts from industrial solid wastes. The extracts produced by this procedure are not necessarily representative of the leachate that would be produced in a specific land disposal environment. However, the procedure can be used to estimate the quantity of potentially leachable constituents in a given solid waste and to measure the concentration of these constituents in an extract produced by extracting the waste at a given solid-to-liquid ratio.

The procedure uses a four-step sequential batch extraction of a waste sample to produce data that can be used to construct an aqueous extraction profile of waste constituents. Each step involves extracting the waste with a fresh aliquot of extraction medium for 18 hours at a 10:1 liquid-to-solid ratio. The sequential extractions permit estimation of the total amounts of specific constituents in a waste that may be released into aqueous solution.

The procedure is applicable to all solid wastes containing Appendix VIII constituents except for those containing volatile organics, and has been demonstrated on several wastes, including electroplating sludge and polystill bottoms.

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DEFINITIONS OF KEY TERMS

1. Extract—The liquid derived from a laboratory batch extraction method in which fixed amounts of waste and liquid extraction medium are mechanically mixed to achieve contact for a specified amount of time.

2. Extraction profile—The relative change in concentration of dissolved waste constituents in extracts (or in mass released into solution) as a waste sample is repeatedly extracted. This can be graphically illustrated by fitting a curve to the concentration data for the sequential extracts.

3. Extractable quantity—The total mass of a constituent that can be extracted from a waste using the MWEP.

4. Intensity—The concentration of a constituent in an extract.

5. Leachate—An aqueous mixture of dissolved and/or suspended waste constituents derived by percolating water through a solid waste.

6. Monofill—A landfill in which only one type of waste is disposed, e.g., municipal waste incinerator ash, fly ash, and electroplating sludge, and there is no codisposal with other municipal waste.

1.0 INTRODUCTION

The proper design and operation of waste management facilities often requires an assessment of leachable chemical constituents of the deposited waste. This information may be required to determine the compatibility of liner materials and leachate or to design leachate treatment facilities. In the absence of information on the composition of a waste leachate obtained under actual field conditions, a laboratory-generated extract can be used to simulate this actual leachate. This document describes a laboratory extraction procedure developed for this purpose.

In this document, the term "monofill" describes the disposal of a hazardous waste in an environment isolated from co-disposed materials such as municipal refuse that might alter the leaching chemistry of the primary waste from that resulting from leaching with natural precipitation. Under current legislation the term "monofill" has no regulatory meaning with respect to the disposal of hazardous wastes.

The monofilled waste extraction procedure (MWEP) described in this document should not be confused with the extraction procedure (EP) or the toxicity characteristic leaching procedure (TCLP). The EP (1) and TCLP (2) were developed to assess the potential of a solid industrial waste to produce a toxic leachate when co-disposed with municipal refuse and each has regulatory significance. The MWEP was developed to produce extraction data that may be used, in conjunction with engineering analysis and judgement, to assess leachable constituents of a solid waste disposed of in a monofill facility. The MWEP has no regulatory significance and none is to be implied by the publication of this document.

1.1 BACKGROUND

Difficulties experienced in the past with disposal of hazardous waste can be attributed partially to a lack of knowledge concerning the long-term behavior of various waste forms in various disposal environments. The poor performance of past disposal practices dictates that factors such as waste solubility, and compatibility of facility components with wastes or waste leachates be considered when designing new hazardous waste disposal facilities. Leachate data from long-term field studies of actual hazardous waste containment facilities are not available for most wastes. Therefore, to evaluate waste leachability, it is necessary to generate a representative laboratory extract to approximate field leachate composition.

Several laboratory extraction procedures have been developed for solid hazardous wastes (3,4). Batch extraction procedures involve the mechanical mixing of a fixed volume of extracting medium with a fixed mass of hazardous waste. Advantages of batch extraction procedures over column leaching procedures include ease of operation and lower experimental variation (5).

1.2 PURPOSE OF PROCEDURE

The MWEP provides a standardized laboratory method for the batch extraction of chemical constituents from solid waste. The primary purpose of

the procedure is to assess maximum leachate concentrations and total extractable mass of solid waste constituents by producing an extract using water as the extraction medium. The MWEP specifies a four-step sequential batch extraction of a waste sample with fresh aliquots of extraction medium. This is similar to Procedure R of the Standard Leaching Test (SLT) developed by Ham et al.(6).

The MWEP extract can be used for the following purposes:

o   Designing and evaluating the performance of leachate treatment systems for new and existing hazardous waste facilities.

o   Assessing the compatibility of hazardous wastes with earthen and synthetic liners and with leachate collection system components.

o.   Assessing potential groundwater contamination fate and transport in the case of facility failure.

o   Evaluating waste stabilization techniques (e.g. admixing lime) intended to reduce the solubility of hazardous components in the waste.

MWEP data may be used to construct an aqueous extraction profile of waste constituents; the profile may be used to assess the waste's capacity for releasing specific constituents, and to infer the time-release of leachable constituents if certain conditions are satisfied. Sequential extractions at realistic liquid-solid ratios are suggested in the procedure for estimating leachate composition as a function of time. Guidance is provided for translating the number of repetitive extractions into leaching time for a waste in an idealized field environment.

1.3 LIMITATIONS OF PROCEDURE

The production of leachate from a landfilled solid waste is influenced by numerous variables, such as the amount and chemical composition of precipitation infiltrating into the waste, permeability within the disposal facility, interaction of contaminants with one another, the presence and composition of a common solid waste matrix, and exposure time to the leaching medium. It is impossible to duplicate these and other variables in the laboratory and generate a representative leachate for a specific waste site. This procedure is a compromise intended to produce an aqueous waste extract that reflects the maximum level of extractable constituents that could be leached from the waste. For the MWEP, significant compromises are:

o   A batch extraction procedure cannot reproduce slow changes in the leaching characteristics of waste in a disposal environment.

o   Chemical reactions that result from mixing leachate produced in two or more segregated waste cells within a disposal facility cannot be accurately simulated by a single component extraction procedure. However, the extract generated from one waste type can be used as the extracting medium for a second waste type.

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o   A batch extraction procedure does not address physicochemical
      changes in the waste initiated by long-term microbiological or
      chemical processes.

o   The procedure may be unsuitable for oily wastes that produce
      extracts having a water-immiscible organic liquid layer. This layer
      may influence the precision or accuracy of the procedure.

o   The procedure is not applicable to volatile constituents of waste
      samples, since the extraction vessels are not headspace free.

o   The hydrologic regime of the actual field disposal environment
      (e.g., flow channeling) is not addressed by a batch extraction
      procedure.

Because of these compromises, analysis of the extract from this laboratory
procedure may not agree with results from field leachate analyses.
      This extraction test may be used to obtain data to estimate the long-term
leaching behavior of a waste. However, the procedure does not account for
long-term changes in waste chemistry or physical characteristics (e.g.,
hydraulic conductivity), due to the action of chemical, physical, and
biological processes.

1.4 REGULATORY SIGNIFICANCE

      The MWEP has no regulatory significance and none is to be implied by the
publication of this document. Differences between the MWEP and other EPA
waste extraction procedures used for regulatory purposes are summarized in
Table 1. The Extraction Procedure (EP) is currently used to classify waste as
hazardous or non-hazardous as defined in 40 CFR 261.14. The Toxicity
Characteristic Leaching Procedure (TCLP) will replace the EP under recent
legislation in the reauthorization of RCRA.

1.5 SCOPE OF DOCUMENT

      This document is designed to be a technical guide describing laboratory
methods for generating data on the quality of leachate from solid waste. The
data can be used in disposal facility design and permit review processes.

      The document is designed to be used with the technical resource documents
(TRD's) that have been prepared on various aspects of hazardous waste disposal
for the U.S. EPA-Municipal Environmental Research Laboratory. (MERL). This
series of TRD's provides technical guidance on moisture infiltration, waste
decomposition, waste stabilization, land treatment, leachate generation,
disposal facility liners leachate transport and collection, and contaminant
transport, migration, and attenuation. The documents in this series are:

TRD 1   Evaluating Cover Systems for Solid and Hazardous Waste (SW-867).
          This document presents a procedure for evaluating design of final
          covers on solid and hazardous waste.

TRD 2   Hydrologic Simulation of Solid Waste Disposal Sites (SW-868). This
         document provides a computer package to aid planners and designers
         in predicting runoff, evapo-transpiration, and infiltration through
         landfill covers.

TRD 3   Landfill and Surface Impoundment Performance Evaluation (SW-869).
         This document describes how to evaluate the capability of various
         liner/drain designs to control leachate release from landfills.

TRD 4   Lining of Waste Impoundment and Disposal Facilities (SW-870). This
         document provides information and guidance on the performance,
         selection, and installation of specific liners for various disposal
         situations.

TRD 5   Management of Hazardous Waste Leachate (SW-871). This document
         presents management options for controlling and treating leachate.

TRD 6   Guide to the Disposal of Chemically Stabilized and Solidified Wastes
         (SW-872). This document provides basic information on
         stabilization/solidification of industrial waste to reduce leaching
         and ensure safe burial of waste.

TRD 7   Closure of Hazardous Waste Surface Impoundments (SW-873). This
         document describes the methods, tests, and procedures involved in
         closing a site to minimize potential environmental hazards.

TRD 8   Hazardous Waste Land Treatment (SW-874). This document presents and
         discusses a comprehensive land treatment facility design strategy
         based on sound environmental protection principles.

     This document will complement the TRD series by providing a means for
generating a laboratory extract similar in composition to a waste leachate
generated under field conditions.

TABLE 1. PURPOSE, APPLICATION, REGULATORY SIGNIFICANCE, AND CONDITIONS OF EP, TCLP, AND MWEP

	EP	TCLP	MWEP
Purpose	Classification of wastes as hazardous or nonhazardous for 14 specific constituents based on predetermined hazard levels.	Classification of waste as hazardous or nonhazardous for all Appendix VIII constituents, including volatiles, based on predetermined hazard levels.	Recommended approach/rationale for the laboratory batch extraction of wastes subject to monofilling. All Appendix VIII constituents except volatiles are covered, but no hazard levels are assigned.
Application	Industrial wastes subject to codisposal with municipal wastes in a municipal waste landfill and/or a landfill not properly designed to receive industrial waste.	Same as for EP.	Industrial wastes subject to monofill disposal in a properly engineered facility. Not applicable to other types of facilities. Data could be used in conjunction with other data to demonstrate site suitability for receiving waste, for demonstrating liner compatibility, and for leachate treament system design.

TABLE 1. (continued)

	EP	TCLP	MWEP
Regulatory Significance	40 CFR 261.24	To replace EP in conjunction with ban on landfilling of industrial wastes.	None.
Conditions liquid:solid ratio	20:1	20:1	10:1 per extraction
Extraction medium	0.5N Acetic Acid	0.1N acetate buffer	Distilled/deionized water or other for special site conditions
pH control	5	5	None
Contact time	24 hours	18 hours	18 hours per extraction
Mixing technique	Tumbler	Tumbler; zero headspace for volatiles	Tumbler
Number of extractions	One	One	Four, sequentially

EP   = Extraction procedure.
TCLP = Toxicity characteristic leaching procedure.
MWEP = Monofilled waste extraction procedure.

TABLE 2. MWEP TEST PARAMETERS AND RECOMMENDED EXPERIMENTAL CONDITIONS

Extraction test parameter	Experimental conditions	Reference section number
Sample	Solid waste	2.1
Contact area/particle size	Surface 3.1 cm^2/g or sized to pass a 9.5-mm sieve [unless waste is Monolithic]	2.2
Extraction medium	Distilled/deionized water (or other for special site conditions)	2.3
Temperature	25±1 °C	2.4
Method of mixing	Rotary mixer (tumbler)	2.5
Time of extraction	18 hours	2.6
Number of extractions	Flexible. Four are recommended.	2.7
Liquid to solid ratio	10:1 (10 mL liquid to 1 g solid)	2.8

2.0 EXPERIMENTAL CONDITIONS

Test parameters and experimental conditions for the MWEP are presented in Table 2. The specifications for this procedure accommodate a variety of waste types, and address criteria that enable the widespread use of the extraction procedure for evaluating hazardous waste samples. These criteria include:

o   Applicablity to wastes containing inorganic and semivolatile organic constituents.

o   Reproducibility, cost-effectiveness, and simplicity.

Experimental parameters specified for the MWEP are described below.

2.1 WASTE SAMPLES

Samples of the hazardous waste to be evaluated must be collected and transferred to the laboratory in glass or Teflon® containers. Metal contact with the waste should be avoided to eliminate possible contamination of the sample. Methods for obtaining representative samples of waste can be found in SW-846 (1).

2.2 CONTACT AREA/PARTICLE SIZE

The contact area of the sample must equal 3.1 cm^2/g unless the solid waste is monolithic, i.e., composed of massive solidified material. To achieve this, the sample may be passed through a 9.5-mm standard sieve as specified in EPA Method 1310 (1). The required contact area and particle size were selected to simulate the conditions likely to be encountered in the field disposal environment due to mechanical filling operations and weathering. Any waste passing the structural integrity procedure (1) should be considered to be monolithic and tested as a whole rather than at a reduced particle size, as its monolithic character will be retained in a landfill environment. Such wastes should not have their particle size reduced as this would cause them to be more leachable than they are under field conditions.

2.3 EXTRACTION MEDIUM

Distilled/deionized water is recommended as the standard extracting medium to provide for uniform test conditions and to permit the comparison of data across waste types. The extraction sequence always should be carried out with this water so that the extractability of different waste types can be ranked on an equal basis. The water should conform to one of the grades of reagent water as given in ASTM Method D1193 (7). ————► pH

Method users also have the option of performing the test with a site-specific medium (in addition to deionized water) that replicates expected conditions at the disposal site. This medium may be used with or without first leaching any soil cover material that is to be used on the site. Suggestions for generating and using a site-specific extraction medium are presented in Appendix A. Alternatively, the extract obtained from one waste may be used to extract a second waste if this is likely to occur in the field.

2.4 TEMPERATURE

To permit comparison of test data across laboratories and waste types, it is recommended that all extractions be carried out at 25±1 °C. The user has the option of also conducting the testing at a temperature close to that expected for the site-specific leachate. The following discussion is presented as guidance in the selection of a site-specific temperature.

Temperature is known to affect the solubility and reaction rates of many chemical compounds present in waste. For extractions carried out using site-specific conditions, the temperature used in the laboratory procedure should be close to that expected for the site leachate, unless data are available to show that temperature variances will have little effect on leaching of the waste being studied. Although ambient air temperatures at land disposal sites may range from extreme cold (-40 °C) to very high (45 °C), temperatures for the leachates associated with these sites are likely to be less varied because overlying cover soil and waste layers have a dampening effect on atmospheric temperature variations (8). The temperature of the leachate emerging from the bottom of a disposal site may be that of the waste/soil at the same depth or may be higher due to chemical reactions within the waste. Seasonal fluctuations in waste/soil temperature at various depths can be obtained from historical disposal-site data or may be measured during preliminary site investigations. Leachate temperatures also may be measured in situ.

2.5 METHOD OF MIXING

A rotary extractor is recommended for this procedure, however, any mixing device can be used for this procedure that will impart sufficient agitation to prevent stratification of the sample suspension and bring the waste solids into continuous contact with the extraction medium.

Methods of agitating or mixing the sample suspension have been studied to determine which method performs best in terms of ease of operation and extent of liquid-solid contact. Several problems have been associated with mechanical stirrers: (1) binding of the stirring blade by solid particles; (2) movement of the stirred vessel; (3) stalling of the stirring motor; (4) uneven blade alignment; and (5) sample grinding (9). However, a mechanical stirrer has been designed that in many cases eliminates or reduces these problems (10).

Ham et al. (6) investigated five methods of mixing: mechanical shaking, manual shaking, mechanical stirring, swing shaking (180° swing), and variable pitch rotary mixing. Their study showed comparable results for inorganic constituents using the various methods. However, their recommendation was to use a rotary extractor because visual observations indicated that the other mixing devices occasionally failed to wet the waste uniformly. A study completed for the U.S. EPA comparing the operation of the stirrer and rotary extractor (NBS-design tumbler) indicates the rotary extractor produces greater precision than the stirrer (11). Examples of rotary extractors are shown in Figures 1 and 2.

Although the reproducibility of test results is enhanced by the mixing action of the rotary extractor, its use may cause excess particle size reduction and thus release an unrealistically high level of Appendix VIII
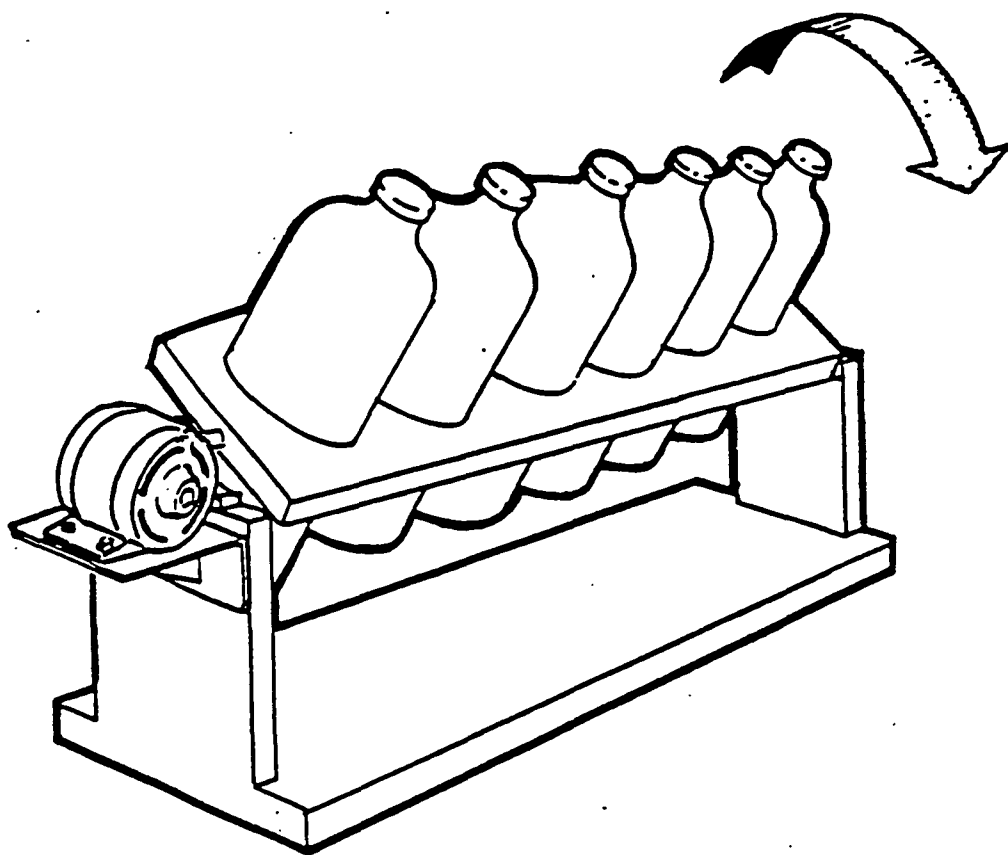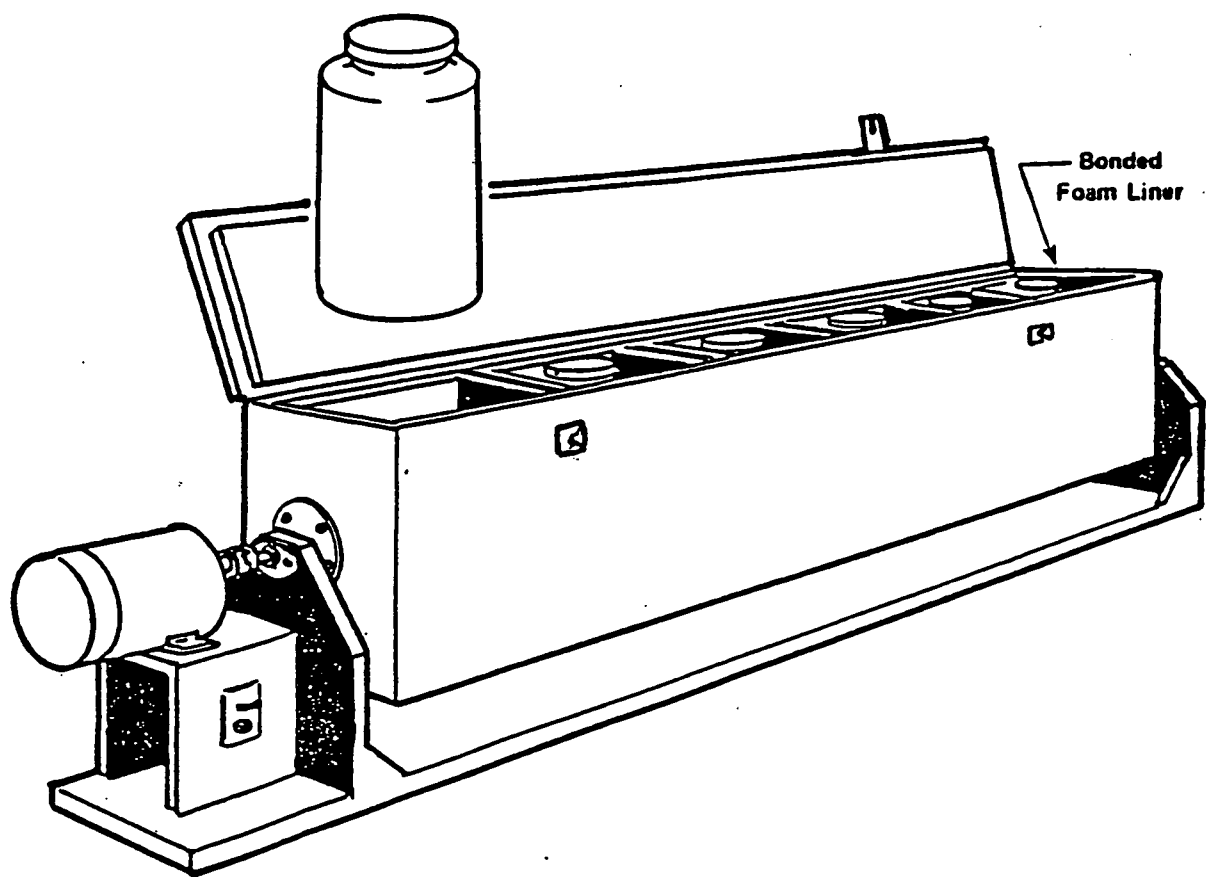
Figure 1. NBS - design rotary extractor.

Figure 2. EPRI/Acurex rotary extractor.

constituents relative to leachate produced in an actual waste monofill. However, the possible sacrifice in method accuracy is necessary to achieve a higher precision, a primary criterion for the procedure.

2.6 TIME OF EXTRACTION

The recommended time per extraction is 18 hours. This mixing time was selected out of consideration for convenience and cost-effectiveness; it allows samples to be placed on the extractor on an afternoon and removed the following morning. The reader should be aware that this time is not necessarily long enough for solute equilibrium to be reached. Equilibrium times can be expected to vary from waste to waste and from constituent to constituent due to differences in dissolution rates; thus it is not practical to try to define unique extraction periods for each waste type or constituent. If attaining equilibrium is necessary, a series of extractions can be conducted to determine the optimum extraction time.

2.7 NUMBER OF EXTRACTIONS

Four sequential extractions of each waste sample with fresh extraction media are recommended to determine an extraction profile over a liquid to solid ratio of 40:1. The solid waste used for the four extractions is retained pending the analyses of the filtrates and interpretation of the results. In the event the results warrant further extractions, the extraction cycle can be resumed with the saved sample and be continued for as many repetitions as desired.

2.8 LIQUID-TO-SOLID RATIO

The recommended ratio of liquid to solid used for each sequential extraction is 10:1 (10 mL/g waste). This ratio should provide both a sufficient amount of extracting medium to wet the sample and enough excess to allow sufficient liquid for proper mixing and subsequent analyses. Ham et al. (6) recommended a 10:1 liquid-to-solid ratio for the standard leaching test based on extensive comparative studies using liquid to solid ratios ranging from 5:1 to 20:1.

The solid-to-liquid ratio that a solid waste will experience in situ is highly site-dependent and very difficult to forecast precisely. In most cases, the ratio will be one of a large amount of solid per unit volume of leachate. The ratio specified for this procedure does not truly reflect likely field conditions; rather it is a workable minimum amount that will still allow sufficient liquid for proper mixing and constituent analysis. A 2:1 ratio might give excellent information on the concentration (of constituents) that can be generated by the waste, i.e., the intensity function, but would probably provide a poor picture of the extractable quantities of the waste constituents since solubilities of many constituents might be exceeded early in the extraction. Likewise, a 20:1 or higher ratio would give less information on intensity and more on extractable quantities. The 10:1 ratio is an effective compromise ratio that can provide both intensity (single extractions) and extractable quantity (multiple extractions) information.

3.0 TEST PROTOCOL

3.1 APPARATUS AND MATERIALS

The apparatus and materials used in the monofill waste extraction procedure must be free from chemical species that might bias the determination of extracted constituents. The apparatus and materials used in the procedure must be selected to avoid potential interactions between the laboratory equipment and the waste-extract solution. Examples of interactions to be avoided are:

o   Dissolution of the extract vessel or other equipment by the extract, as might happen with a hydrofluoric acid-containing waste placed in a glass vessel.

o   Preferential sorption of constituents out of the extract by the sample container or filtration unit, as might happen with some organic compounds contained in polypropylene vessels.

o   Contamination of the extract by constituents leached from the sample container, as might happen with waste extract to be analyzed for nickel and chromium contained in stainless steel vessels.

The following discussion suggests appropriate equipment and materials for use in the procedure. Specification of a particular manufacturer or model is for purposes of guidance only. Addresses of suppliers referenced in this section can be found in Appendix C.

3.1.1 Agitation Apparatus and Extraction Bottles

An agitation apparatus must be designed to avoid stratification of the sample of solid waste and soil and the leaching medium, which would inhibit adequate contact between the sample and leachate. The type of extraction apparatus recommended for this procedure is the rotary extractor or tumbler (Figures 1 and 2). The extractor consists of a rack or box device to hold the sample containers, which are rotated through 360° at approximately 30 revolutions per minute.

A six-place tumble extractor derived from a design by the National Bureau of Standards is illustrated in Figure 1. This equipment may be fabricated by the investigator or obtained commercially from Associated Design and Manufacturing Company (Model No. 3740-4-BRE [four-place tumbler] or Model No. 3740-6-BRE [six-place tumbler]). A second type of six-place tumbler, shown in Figure 2, may be fabricated by the investigator or obtained commercially from Acurex Corporation (no model number available).

Extraction bottles sized to fit the tumbling apparatus may be made of glass, plastic, or Teflon®. Plastic should not be used to extract wastes containing organic compounds. Heavy-gauge glass bottles are available from Associated Design and Manufacturing Co. (Cat. N. 37402GB).

3.1.2 Separation Apparatus

Separation of the solid and liquid layers following extraction of the waste sample can be accomplished by a combination of settling and filtering. Details on performing these operations can be found in Section 3.4, which gives step-by-step instructions for the procedure.

Filter Holder

The filter holder must be capable of supporting a 0.45-micrometer membrane filter and must withstand the pressure needed to accomplish separation. These units may be simple vacuum units (Millipore model No. XX10-047-00 Nuclepore model No. 410400 or equivalent). However, units capable of being pressurized up to 75 psi may be needed for many solid wastes (Millipore model No. YT30-142-HW, Nuclepore model No. 420800, or equivalent).

Filter Pads

A glass-fiber prefilter, nylon fine-mesh screen spacer, and a membrane filter are suggested for use in all filtrations of inorganic waste constituents. Suggested vendors and model numbers are:

o   Coarse glass-fiber prefilter pad (Millipore model No. AP 25-042-00 or No. AP 25-127-50 or equivalent)

o   Fine-mesh screen spacer (Millipore model AP32-124-50 or equivalent)

o   0.45-micrometer nitrocellulose membrane filter (Millipore model No. HAWP-047-00 or No. HAWP-142-50 or equivalent)

A 0.6-0.8 μm glass micro fiber filter (Whatman Grade GF/F) should be used in place of the nitrocellulose membrane filter for filtration of semivolatile organic waste constituents.

3.1.3 General Labware

Sample Bottles

The sample bottles used for containing wastes or extracts should be of suitable materials, such as glass or Teflon® for organic analysis or polypropylene for inorganic analysis should have a screw cap with an inert liner such as Teflon®.

3.2 REAGENTS

3.2.1 Extraction Medium

The extracting medium selected for general use in the MWEP is reagent water (9). This water must be of sufficient quality, that is, free of organic and inorganic interferences at the minimum levels of interest for the subsequent extractions and extract analyses.

3.2.2 Nitric Acid

Trace-element-analysis-grade nitric acid (J. T. Baker No. 9598 or equivalent) should be used to preserve extracts prior to analysis of inorganics (see Section 3.3.2).

3.3 SAMPLING

3.3.1 Sample Collection and Handling

Samples of the solid wastes and soil cover materials to be tested should be collected using the methods described in reference (1). It is particularly important that the sample be representative of the solid waste as it occurs in a landfill environment.

A minimum sample of 1 kg should be collected and sent to the laboratory in a sealed container or containers. The containers must be of a material that will not react with the waste (see Section 3.1.3).

3.3.2 Sample Preservation

Preservatives must not be added to waste or soil samples. Samples known to be stable with regard to biological or chemical change may be shipped and stored at room temperature. Samples that might undergo significant biological or chemical change at room temperature must be maintained at 0-5 °C during shipping and storage. If the stability of the waste or soil is uncertain or unknown, shipping and storage of the waste at 0-5 °C is recommended.

Extraction of samples should be initiated within 1 week of sample collection to minimize changes in the sample with storage time. Longer storage times may be used if the waste or soil samples are known to be chemically and physically stable.

MWEP extracts should be analyzed as soon as possible following extraction. If they need to be stored, even for a short period of time, storage must be at 4 °C.

When inorganic constituents are to be determined, trace-metals-analysis-grade concentrated nitric acid should be added to the extract after separation until the mixture has a pH of less than 2. This prevents precipitation of trace metals prior to analysis.

3.4 EXTRACTION PROCEDURE

The overall flow scheme for the MWEP is shown in Figure 3. Detailed laboratory procedures are given below.

3.4.1 Extraction 1

Step 1 Mixture Preparation

Using a separate aliquot of the waste, determine the percent solids content and compensate for differences in wet weight by extracting an amount of waste equal to 100 g dry weight of sample. Compensate for the amount of extraction medium added to maintain a 10:1 liquid-to-solid ratio.
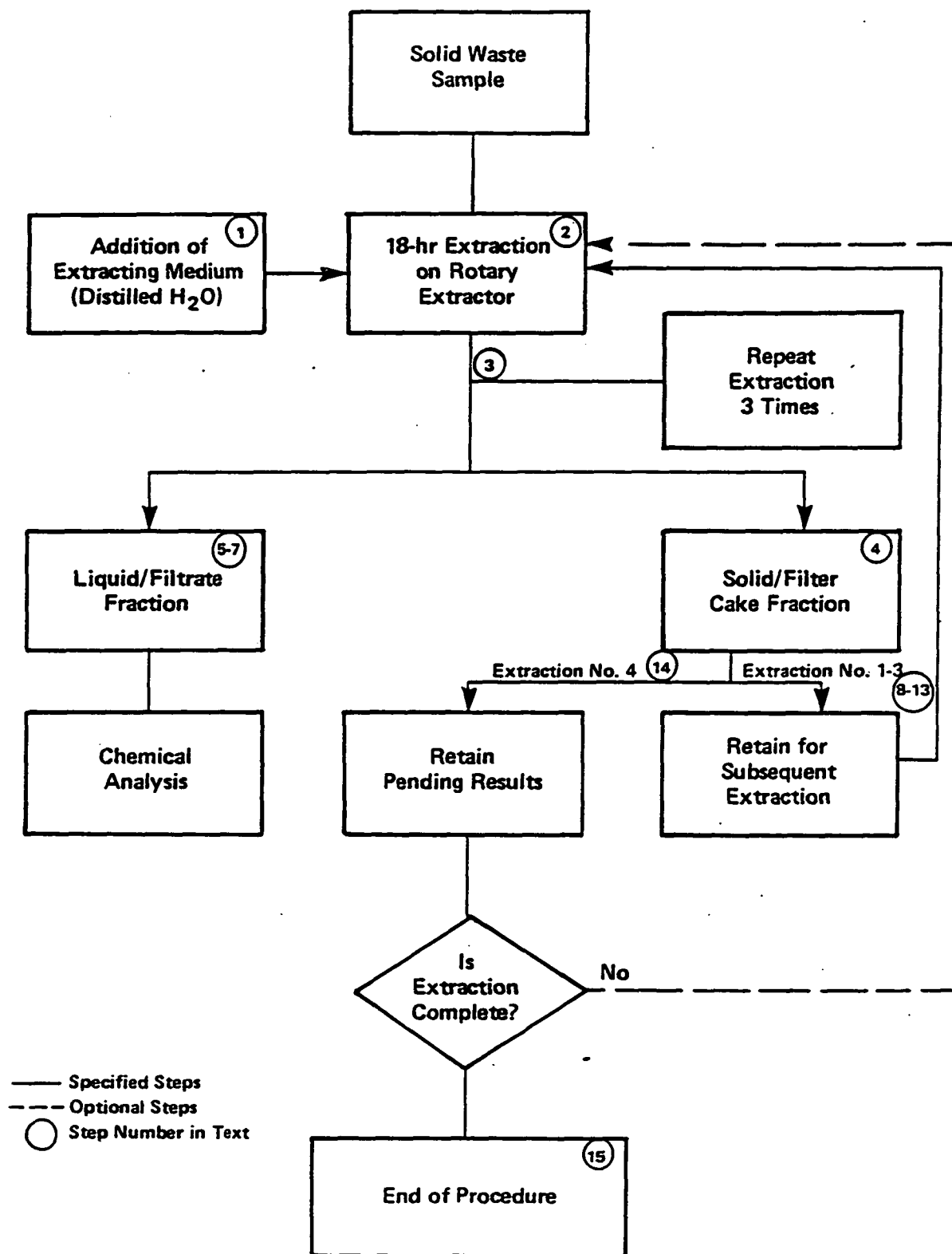
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Figure 3. Schematic diagram of the monofill waste extraction procedure (MWEP).

Place a 100-g (dry-weight basis) representative sample of the solid waste that has been prepared for testing (see comments in Section 2.2) in an extraction vessel (tumbler bottle or equivalent container). Add 1 L of extraction medium (Section 2.8) to the extraction vessel. (Note: Different amounts of waste and extraction medium may be used as long as the 10:1 liquid-to-solid ratio is achieved).

Step 2 Tumbling

Tighten the cap on the vessel and mix by tumbling, using the rotary extractor. Tumble for 18 ($\pm$2) hours at room temperature. Stop the rotary extractor, remove the extraction vessel, and allow the mixture to settle for 15 minutes.

If a discrete water-immiscible layer is present, withdraw the layer using a syringe with a wide-bore needle. .Transfer the layer to a tared sample container of suitable material, such as glass. Determine the mass of the layer and analyze it separately.

Step 3 Separation

Assemble the filter holder and filter pads following the manufacturer's instructions. Place the 0.45-micrometer nitrocellulose membrane or the glass microfiber filter pad on the support screen of the filter holder. Place the nylon mesh screen and then the coarse.glass fiber prefilter pad on top of the membrane pad so that the coarse pad will be in contact with the filter cake.

After assembling the filtration apparatus, wet the uppermost filter pad with a small portion of the liquid phase of the extraction mixture. Transfer the remainder of the extraction liquid to the filtration unit. Take care to avoid transferring much of the solid from the extraction vessel, because substantial amounts of solid can clog the filter pads. Apply vacuum or gentle pressure (10 to 15 psi) until all liquid passes through the filter.

Stop the filtration when all the liquid has passed through the filter pads. If this point is not reached under vacuum or using gentle pressure, then increase the pressure in 10-psi increments to a final maximum pressure of 75 psi.

If liquid remains above the filter pads after 30 minutes of filtration at 75 psi, halt the filtration by slowly venting the pressurizing gas. Be certain to follow the manufacturer's instructions for venting a pressurized filtration apparatus. Some liquid may be trapped in the vent port and may be released. Care must be taken to direct the vent port away from laboratory personnel. After venting, decant the liquid above the filter pads into a suitable container. Place the topmost (coarse) prefilter pad plus any solid/filter cake into a suitable container, such as the extraction vessel for use in the next extraction. Replace the filter pads, placing the fresh pads on the unit in the correct order, and resume filtering the decanted liquid.

Repeat the process of replacing the filter pad as often as necessary until all of the liquid has been filtered. In each process, retain the topmost (coarse) prefilter pad along with any solid/filter cake.

After completing the filtration, return the unit to atmospheric pressure by either carefully breaking the vacuum or slowly venting the filtration apparatus.

Step 4   Solid/Filter Cake

Retain the solid/filter cake after filtration for use in the next extraction or pending the interpretation of the results obtained from the procedure. Include the topmost (coarse) prefilter pad from all filtrations, as stated in Step 3. Use tweezers made of or coated with an inert material to break apart the filter pads prior to beginning the next extraction.

Step 5 Sample for pH and Inorganic Constituents Analyses

Transfer an aliquot of the liquid/filtrate from Step 4 to a suitable container, such as a beaker. Determine the pH. If an analysis for inorganic constituents is needed, add a minimum volume of nitric acid (see Section 3.2.2) to lower the pH to less than 2. Transfer the acidified sample to a suitable container, such as a screw-cap polypropylene bottle. Store at room temperature prior to analysis. Label the sample container including the date, extraction sequence number, and an appropriate sample identification number.

Step 6 Sample for Semivolatile Organic Constituents Analysis

Transfer an aliquot of the liquid filtrate from Step 4 to be used for semivolatile organic constituents analysis to a suitable container, such as a glass bottle with an Teflon® lined screw-cap. Store the sample at 0-5 °C prior to analysis. Label the sample container properly, including the date, extraction sequence number, and an appropriate sample identification number.

Step 7 Remainder of Filtrate

Retain any remaining filtrate after samples have been removed for analysis. After analyses are complete, dispose of the filtrate in accordance with approved laboratory procedures for disposal of potentially hazardous liquids.

3.4.2   Extraction 2

Step 8 Mixture Preparation

Place the solid/filter cake from Extraction 1 (Step 5) in an extraction vessel. Add 1 L of fresh extracting medium to the extraction vessel.

Step 9

Repeat Steps 2 through 8 using the previously extracted solid waste plus fresh extracting medium mixture.

3.4.3 Extraction 3

Step 10 Mixture Preparation

Place the solid/filter cake from Extraction 2 in an extraction vessel. Add 1 L of fresh extracting medium to the extraction vessel.

Step 11

Repeat Steps 2 through 8 using the previously extracted solid waste plus fresh extracting medium mixture.

3.4.4 Extraction 4

Step 12 Mixture Preparation

Place the solid/filter cake from Extraction 3 in an extraction vessel. Add 1 L of fresh extracting medium to the extraction vessel.

Step 13

Repeat Step 2 through 8 using the previously extracted solid waste plus fresh extracting medium.

Step 14 Retention of Solid/Filter Cake

Retain the solid/filter cake from Extraction 4 pending a decision on the need for further extractions.

3.4.5 Further Extractions

The need for further extractions is determined based on interpretation of the results. Depending on the amount of solid waste that is dissolved during each extraction, the extraction of the same solid waste sample with fresh extracting medium can be repeated as many times as needed to satisfy the objectives of the testing or until solute concentrations of interest are no longer detectable.

Step 15 Disposal of the Solid/Filter Cake

After all laboratory testing has been completed, the solid/filter cake be discarded in accordance with approved laboratory procedures for disposal of potentially hazardous waste.

3.5 ANALYSIS

The samples collected for analysis should be analyzed for the Appendix VIII constituents of concern by one of the EPA-approved or EPA-proposed methods. These methods can be found in a variety of references (1,12,13). The extracts should be analyzed within 7 days after generation.

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4.0 QUALITY CONTROL

To ensure that the data generated using the MWEP are of acceptable quality for the purposes for which they will be used, procedural blanks and replicate parallel extractions are carried out.

4.1 PROCEDURAL BLANK

The procedural blank extraction is carried out by extracting 1,000 mL of distilled/deionized water or site-specific extraction medium in an extraction vessel as if a sample were present. The water that is filtered at the conclusion of 18 hours of extraction is then analyzed for the Appendix VIII constituents of interest using the appropriate sample collection, preservation, preparation, and analysis procedures. The results of the procedural blank are used to determine if contamination and/or memory effects are occurring.

Unacceptable contaminant levels are determined based on the intended use of the data. If unacceptable levels of Appendix VIII constituents are found in the final filtered extract of the procedural blank, the individual steps of the procedure should be investigated to determine the source of the contamination. For example, prewashing of the prefilters and 0.45-μm filters may be required if contamination from these sources appears to be a problem.

A clean procedural blank must be obtained prior to any waste testing. A minimum of one procedural blank extraction per five waste sample extractions per extractor system·per unique extraction medium is to be carried out. If less than five samples are extracted, then one blank extraction is to be performed.

4.2 PARALLEL EXTRACTIONS

To assess the extraction precision, three parallel extractions of each waste are to be carried out. The results are to be reported in terms of the relative standard deviation (RSD) of the extract levels of the constituents of interest.

4.3 ASSESSMENT OF ACCURACY

Analyses of each extract for specific constituents should be assessed for accuracy by means of standard additions, spiking, and analyses of replicates as called for in the analytical procedures.

At present, no standard solid waste reference material is available by which the accuracy of the entire procedure can be assessed. At best, accuracy can be assessed by extraction of a sample for which a historical data base is available.

5.0 CONVERSION AND INTERPRETATION OF EXPERIMENTAL DATA

5.1 DATA CONVERSION

Conversion of the experimental data obtained in the extraction experiments is necessary to estimate the waste extraction characteristics under laboratory conditions. For interpretative purposes, experimental data can be converted to concentration and mass of released waste constituents or, with pertinent assumptions, to constituents released into the extract as a function of time. Statistical treatment of these data is described in Appendix B.

5.1.1 Concentration and Mass Release Conversions

The experimental data generated by the MWEP can be converted to (1) concentration of constituents in the extracts, (2) the mass of constituents released during each extraction, and (3) the cumulative mass of released constituents. The following discussion describes methods for converting data into each of the above forms.

5.1.1.1 Calculation of Concentration in Extract

The data obtained using the MWEP can be used directly in terms of the concentration of the constituent that was found on analysis of the extract solution. The general method of calculating this concentration is given in Equation (1):

$$C(x)_i = C(anal)_i \times DF \qquad (1)$$

where:

$C(x)_i$ = the concentration of constituent x in the extracted solution from extraction sequence number $\underline{i}$

$C(anal)_i$ = the concentration of x that was found on analysis

DF = the dilution factor or concentration factor for the analysis; the dilution factor is the extent to which the extracted solution was diluted or concentrated prior to analysis.

5.1.1.2 Calculation of Mass Per Gram of Waste Released Per Gram of Water Per Extraction

The mass of the constituent released from the solid waste sample per gram of waste for each extraction can be calculated by the method given in Equation (2):

$$M(x)_i = \frac{C(x)_i}{S:L} \qquad (2)$$

where:

$M(x)_i$ = the mass of constituent x that was released from the solid waste sample during extraction sequence number $\underline{i}$ per gram of waste

S:L = the solid-to-liquid ratio used in the extraction. The volume in the denominators of both $C(x)_i$ and S:L must be in the same units, such as liters or milliliters, that these units will cancel. The term $M(x)_i$ will then have the dimensions of mass of x released per unit mass of solid waste, such as mg of x per g of waste.

5.1.1.3 Calculation of Cumulative Mass Released Per Gram of Waste

MWEP data can be used to calculate the cumulative mass of a constituent released from the solid waste during a series of extractions. The general method for calculating cumulative mass released is given in Equation (3).

$$M(x)_{cum} = \sum_{i=1}^{n} M(x)_i \tag{3}$$

where:

$M(x)_{cum}$ = the cumulative mass of constituent x per gm of waste that was released over the number of extractions (n).

5.1.1.4 Compilation of Concentration and Mass Release Calculations

A data set of concentration and mass release calculations for a series of four extractions on one waste sample is presented in Table 3. These data are representative of data obtained during experimental development of this procedure.

TABLE 3. CONVERSION OF EXPERIMENTAL DATA

Extraction sequence number	$C(anal)$ $(mg/L)^i$	DF	$C(x)_i$ (mg/L)	$M(x)$ (mg/g)	$M(x)_{cum}$ (mg/g)
1	4.6	2.0	9.2	0.092	0.092
2	3.8	1.0	3.8	0.038	0.130
3	2.8	1.0	2.8	0.028	0.158
4	2.0	0.5	1.0	0.010	0.168

The results calculated above can also be presented graphically showing the extraction sequence number versus the concentration of constituent in the extracted solution. Examples of graphed results are shown in Figure 4, which presents the data from Table 3. The area under the histograms is approximately equal to the total mass released.

An alternate way of estimating the releasable mass of constituent is to fit the data to a curve and integrate the area under the curve. For purposes of illustration, the data in Figure 4 have been connected by a curve. Details on one approach to mathematical curve fitting of such data are described in Jackson et al. (14)

The curve-fitted extraction profiles may be used to estimate the quantities and concentrations of constituent released at higher liquid-to-solid ratios than used in the MWEP. For example, in Figure 4 the curve can be extended beyond the fourth extraction sequence to show the estimated release concentration and quantity of constituent at a 50:1 liquid-to-solid ratio. These estimates are valid only if the extraction at these higher liquid-to-solid ratios is governed by the same factors as for the measured extractions.

Similarly, extractable quantity can be estimated by extending the curve to intersect the x-axis at concentration = 0. This can be useful in allowing total extractable quantity to be estimated using a minimum number of extractions.

Note that whereas extrapolation to higher liquid-to-solid ratios is possible, extrapolation to lower ratios is not, since the MWEP assumes that all of the concentrations and mass released by lower ratios are integrated into the first extraction sequence.

5.1.2 Extraction Profile Analysis

Extraction profiles provide an indication of the concentration level of analyte in the extract as well as an indication of the buffering capacity of the waste for maintaining a given constituent concentration in the extract. Examples of extraction profiles are presented in Figures 5 through 10. These examples are reproduced from Jackson et al. (14).

In this investigation, four wastes were evaluated: electroplating sludge, fly ash, filter cake, and polystill bottoms. Figure 5 demonstrates the high buffering capacity of the electroplating waste at an approximate pH of 8. Figures 6 and 7 illustrate the effect of repetitive leaching on the solubility of calcium and copper in the electroplating sludge.

Figure 8 shows the neutralization of fly ash extract as sequential extractions are made. This effect is due to acidifying SO_2 sorbed onto fly ash surfaces, which results in a relatively low pH in the first extraction. Subsequent extractions reflect the buffering capacity of calcium and magnesium oxides and silicates, which are associated with the structure of the fly ash.

Aluminum in an organic filter cake was found to increase in extract concentrations with repetitive extraction (Figure 9). This effect may be due

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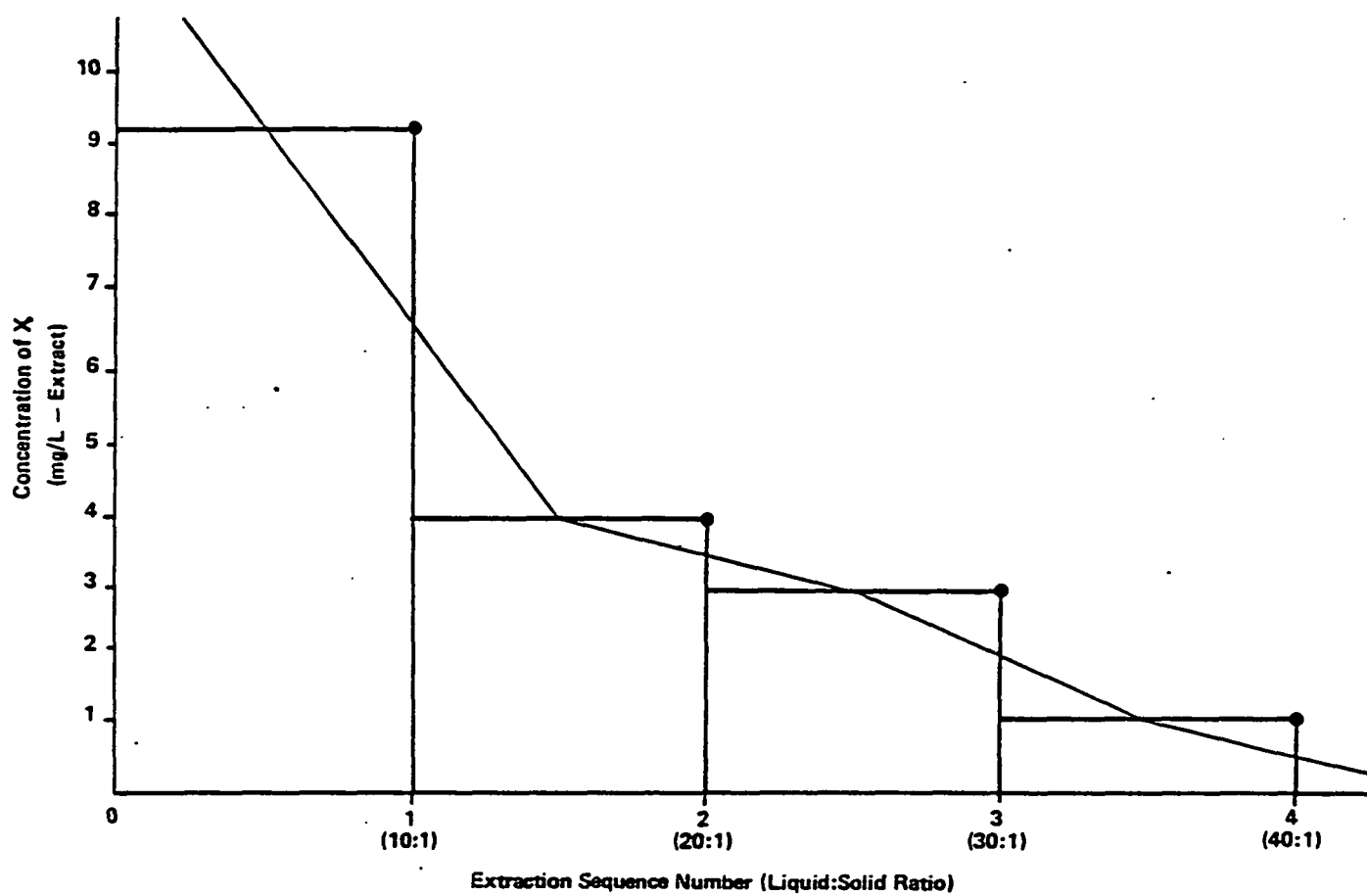Figure 4. Extraction profile for constituent data in Table 3.

pH

9
8.5
8
7.5
7
6.5
6
5.5
5
4.5
4
3.5
3
2.5
2
1.5
1
0.5
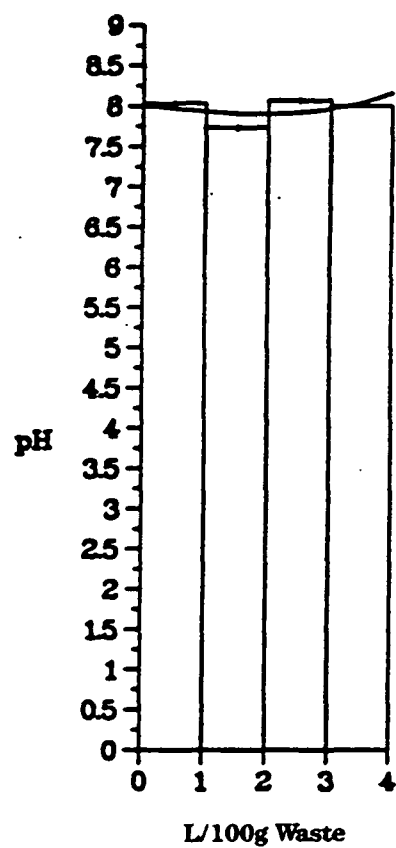0

0   1   2   3   4

L/100g Waste

Figure 5. pH of extract versus liters of water per 100 g of waste for electroplating sludge.

Figure 6. Calcium concentration in extract versus liters of water per 100 g of waste for electroplating sludge.

Figure 7. Copper concentration in extract versus liters of water per 100 g of waste for electroplating sludge.

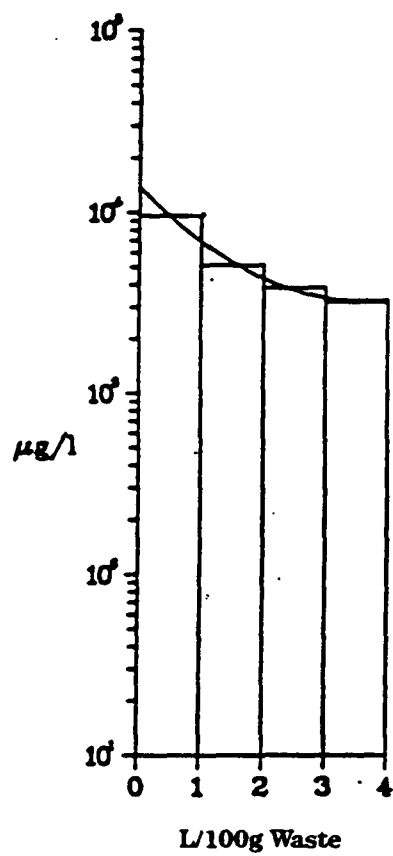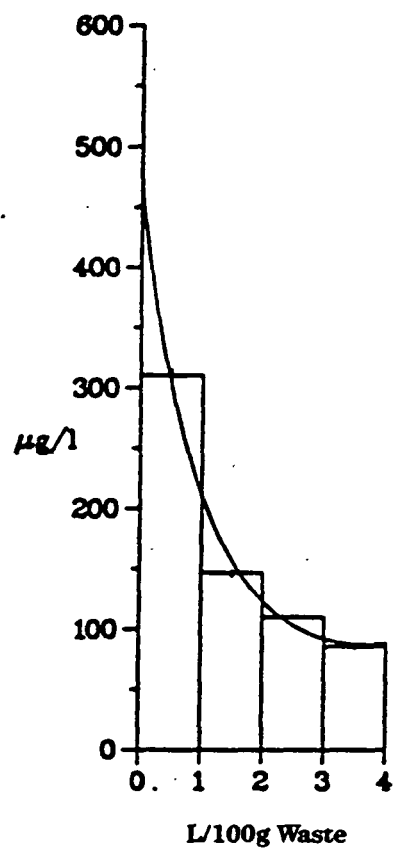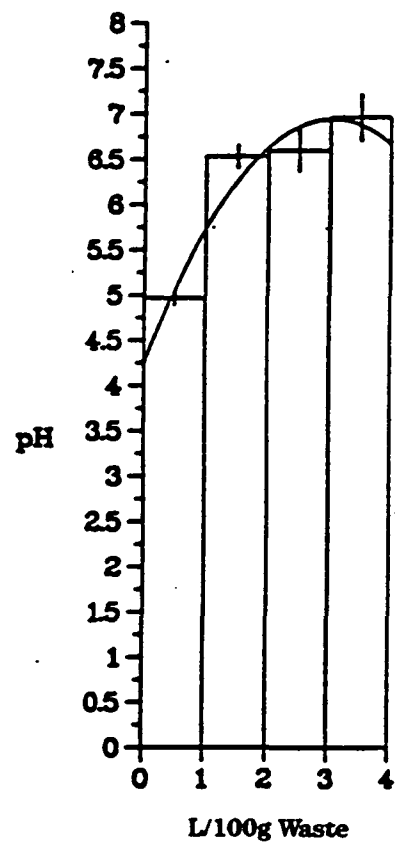**Figure 8. pH of extract versus liters of water per 100 g of waste for fly ash.**
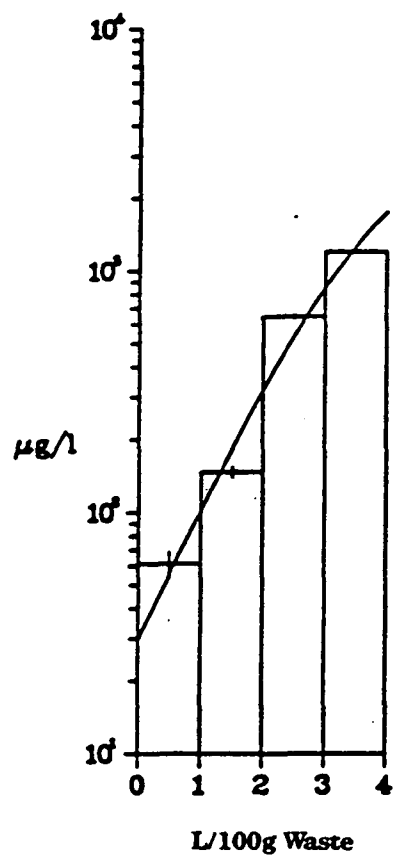
Figure 9. Aluminum concentration in extract versus liters of water per 100 g of waste for filter cake.
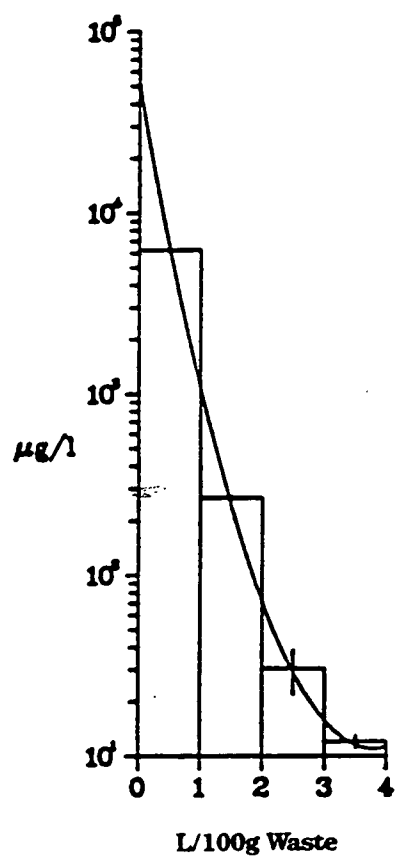
Figure 10. Copper concentration in extract versus liters of water
per 100 g waste for polystill bottoms.

to the particle size diminution of the material as well as to increased wetting due to agitation of the waste.

Copper concentrations in extracts of a polystill bottom waste (Figure 10) illustrate a constituent that is in a relatively high concentration initially, but does not have high buffering capacity. The copper concentration dropped approximately an order of magnitude with each succeeding extraction.

5.2 INTERPRETATION

The utilization of extraction profile analysis for estimating the total extractable quantity of a waste constituent is shown in the data presented in Figure 11. The results of 10 sequential extractions of a waste are plotted. The extraction procedure used was not the MWEP but was a similar procedure developed by EPA in which a synthetic acid rain mixture was used as extractant (15,16). In this procedure, waste samples that have been previously extracted according to the extraction procedure toxicity test, EP (Method. 1310), are sequentially extracted nine times using a synthetic acid rain extraction fluid (60/40 weight percent sulfuric acid and nitric acid, pH 3.0 ± 0.2). Each extraction requires 24 hours and is carried out at a liquid-to-solid ratio of 20:1. The extraction sequence and conditions are designed to simulate the leaching a waste will undergo due to repetitive precipitation of acid rain on an improperly designed sanitary landfill. The repetitive extractions reveal the highest concentration and quantity of each constituent that is likely to leach in a natural environment. If the concentrations of any of the listed constituents of concern increase from the seventh or eighth extraction to the ninth extraction, the procedure is repeated until the concentrations decrease.

A sequence of 10 extractions requires at least 10 days to complete. If it could be shown from the extraction profile that the extraction of a particular constituent was following a trend well before the 9th or 10th extraction, then considerable time and effort could be saved.

The data in Figure 11 are from a delisting petition submitted to EPA by General Motors Corporation (GMC) for exclusion of the treatment residue generated from the use of the Chemfix® treatment process on electroplating operations. The residue was contained in onsite surface impoundments. The results of the sequential extractions for cadmium, hexavalent chromium, and nickel indicated that the treatment residue exhibited long-term stability by extracting nonhazardous levels of these elements after multiple synthetic acid rain extractions. However, the extraction profile in Figure 11 indicates that after the 4th extraction, a trend is clearly established that allows estimation of the total extractable mass of chromium without the need for performing these additional extractions. While this type profile will not always be obtained, the user of the MWEP should take advantage of this data presentation method to minimize the effort required to obtain the needed extraction information.

The profile illustrated in Figure 4 may be encountered when the constituent is steadily depleted from the solid waste. Each subsequent extraction releases more of the constituent, although the mass released per extraction decreases with each extraction. Other profiles that might be encountered would show such trends as (1) a steady release where each extraction releases essentially the same amount and (2) a delayed release. where initially little or none of the constituent was released, followed by
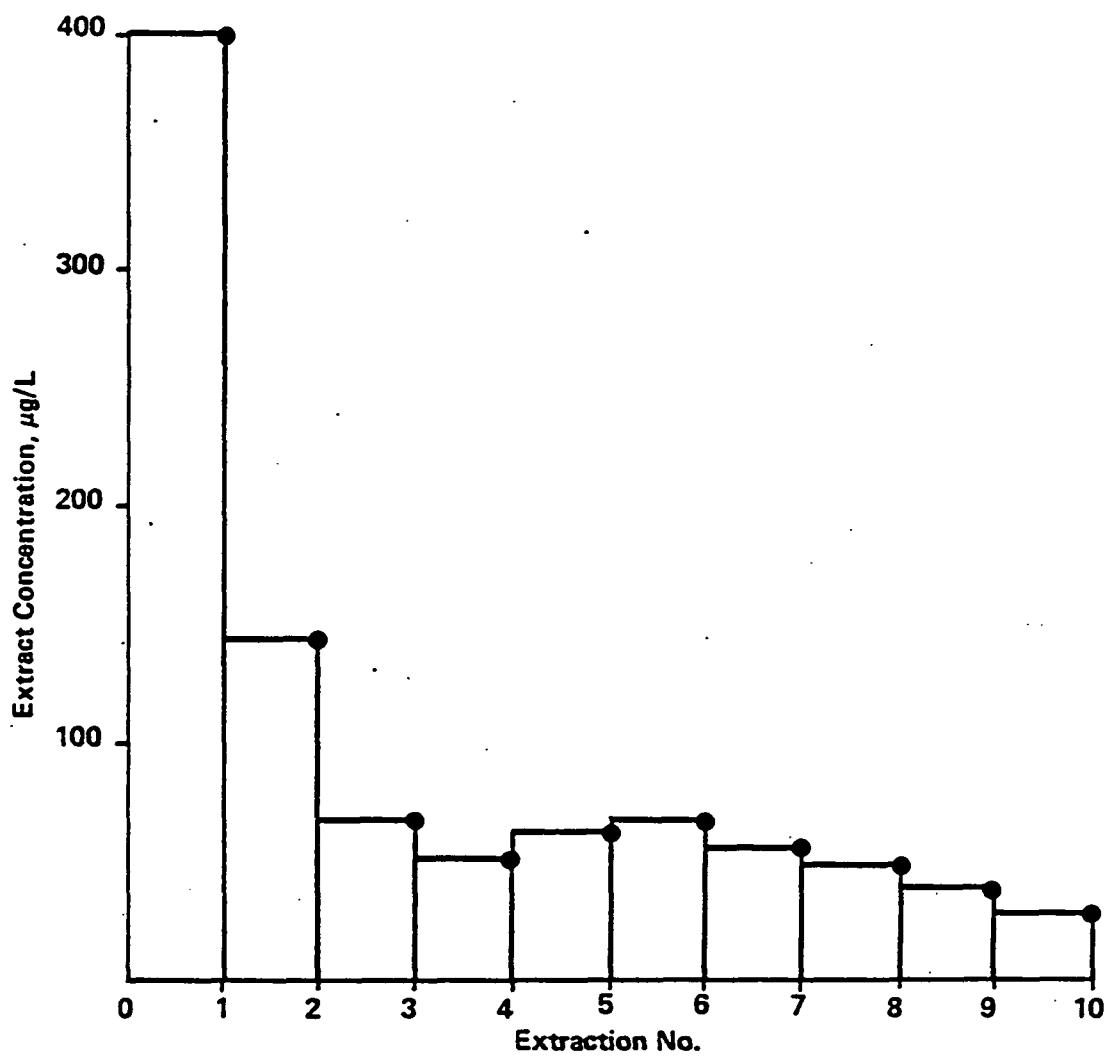
Figure 11. Extraction profile of Cr from electroplating sludge.

large increases in the amount released when alkalinity is leached and pH decreases, allowing metals to go into solution.

A decision on the need for further extractions will depend on the profiles obtained for the initial sequence of extractions plus the specific needs of the research being conducted. The application of the results to events in a landfill environment depends on site-specific information such as infiltration rate for rainfall and the density of the waste.

In general, the interpretation of the results involves comparisons of the concentration of mass-released profiles of the various constituents that have been examined. The technical resource documents (TRD's) discussed in Section 1 provide guidance on leachate flow rate calculations (see in particular TRD2, "Hydrologic Simulation on Solid Waste Disposal Sites [SW-868]," TRD3, "Landfill and Surface Impoundment Performance Evaluation [SW-869]," and TRD5, "Management of Hazardous Waste Leachate [SW-871]." These documents plus the studies on leachate generation by Houle and Long (17, 18) are additional sources for information on calculations and interpretation of leachate test data.

5.3 CALCULATION OF TIME REQUIRED FOR FIELD LEACHING

A simple model was developed to infer a time dimension to the extraction profile generated by the MWEP. A hypothetical field environment was assumed for the purpose of determining how many years of constant leaching would be required for the extractable constituents, as indicated by the MWEP, to be released into the environment. The model is based on a "worst-case" scenario and assumes that the waste constituents in the leachate reflect the same concentrations as those produced by the sequential leaching of the MWEP.

The amount of time required in the field to collect a volume of leachate comparable to that in each extraction sequence can be computed from the experimental data assuming the following:

o   Primary leachate volume in the waste is disregarded.[1] This assumption is necessary because primary leachate is mixed with the extraction media in the first sequential extraction of the MWEP.

o   Depth of waste is fixed and known.

o   The concentration of solutes in the waste is assumed to follow the extraction profile produced by the MWEP as the waste is leached over time.

o   Waste is a monofill and is saturated with water at the time of disposal. This assumption was made to simplify the analytical solution of the model.

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[1]Some wastes, as disposed, contain interstitial liquids having high concentrations of solutes that may be displaced as undiluted leachates early in the leaching process. This leachate has been classified as primary in contrast to secondary, which is reflective of solutes dissolved in percolating water of external origin, such as precipitation (19).

Based on these assumptions, the equation for calculating the time corresponding to each leaching fraction is presented below:

$$LT = (DW \cdot \rho_{waste} \cdot LSR \ (I \cdot \rho_{water}) \qquad (4)$$

where:

LT = Leaching time (years to exceed extraction sequence, i.e., to exceed 1:10, 1:20, 1:30 ratio, etc.)

DW = Depth of fill (cm)

ρ_{waste} = Density of waste (g/cm^3)

LSR = Liquid-to-solid ratio of MWEP

I = Infiltration rate (cm/s)

ρ_{water} = Density of water (g/cm^3)

A representative case has been devised to illustrate use of this equation. Let:

DW = 610 cm

ρ_{waste} = 1.3 g/cm^3

LSR = 10:1

I = 1 x 10^{-7} cm/s

ρ_{water} = 1.0 g/cm^3

LT = (610 cm $\cdot$ 1.3 g/cm^3 $\cdot$ 10)/(1 x 10^{-7}cm/s $\cdot$ 1.0 g/cm^3)
= 2,517 years

Thus, the leaching time in the field corresponding to the first leaching sequence is 2,517 years using the 1 x 10^{-7} cm/s infiltration rate.

The usefulness of this computation is illustrated in Table 4. Calculations are presented for four leaching sequences and five infiltration rates. For example, the time corresponding to the first extraction sequence ranges from 25 years to 250,000 for infiltration rates of 10^{-5} cm/s and 10^{-9} cm/s, respectively, at a depth of waste fill of 6.1 m. The results of these time calculations, as shown in Table 4, demonstrate the extremely long time (in terms of landfill life) associated with each extraction sequence. In most cases, the initial extraction sequence will provide an estimate of the leachate fraction of prime interest. These calculations presume that the infiltration rate determines the flow of leachate through the fill.

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TABLE 4. TIME REQUIRED TO EXCEED SPECIFIC EXTRACTION SEQUENCE
FOR SELECTED CAP INFILTRATION RATES (DEPTH OF WASTE = .610 cm)

Extraction sequence number	Time corresponding to sequence at selected infiltration rates (years)				
	10^{-5} cm/s	10^{-6} cm/s	10^{-7} cm/s	10^{-8} cm/s	10^{-9} cm/s
1	25	250	2,500	25,000	250,000
2	50	500	5,000	50,000	500,000
3	75	750	7,500	75,000	750,000
4	100	1,000	10,000	100,000	1,000,000

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APPENDIX A

GENERATION OF SITE-SPECIFIC EXTRACTION MEDIUM

Objective - to generate a site-specific extraction medium.

The approach used in generation of the site specific extraction medium is shown in Figure A-1.

The primary extraction medium should simulate the typical precipitation falling on the site. Rainfall collected at the site is inappropriate for this purpose. It is likely to be unrepresentative of the long-term rainfall (over a period of years). For example, although brief rainstorms may produce little rain, the rain that is produced may be much more acidic then storms of longer duration which would dissolve and rain out the same ions but in a much greater volume of water.

A synthetic precipitation mixture should be prepared based on historical data, which can be obtained from the EPA Acid Deposition System, a computerized data base of acid precipitation data from several long-term monitoring networks (1). The synthetic precipitation sample can be prepared by dissolving high-purity salts and acids in high-purity distilled/deionized water as described by Deardorff et al. (2). This mixture will contain the average levels of cations and anions reported in the historical monitoring data for the general area around the proposed landfill. The mixture will also have a pH based on these data. It is likely that only the acidity of the synthetic precipitation sample will have a significant impact on the leaching of the waste, and this may be minimal if the soil cover that is leached first has a buffer capacity sufficient to neutralize the precipitation acidity. If soil is to be used in the fill as cover material, it is extracted first and the extract used as the solution to carry out the first extraction of the waste. If layers of soil are placed on the waste intermittently, waste extracts should be used to extract the soil in a sequence that simulates the field situations.

This soil should be of the same type and be present in the same proportion as that encountered at the disposal site. The ability of soils to alter the mobility of species in leachates is well known and has been studied extensively for various types of soils and wastes. For example, the extent to which trace inorganic constituents of leachates are attenuated is related to the amounts and types of clay and iron and manganese hydrous oxides in the soil (3, 4); and attenuation of organic compounds such as PCB's is directly related to the organic carbon content of the soil (5). Furthermore, soil type influences complexing agents and colloidal constituents present in a leachate and soil mixture. Where soil is to be mixed with the waste and not used to cover it forming distinct layers, then this admixture should be extracted. Where the solid waste disposal plan precludes the use of soil during active filling, no soil is used in the extraction procedure. Where appropriate, both the intermittent cover material and the material proposed for site closure can be tested along with the waste.
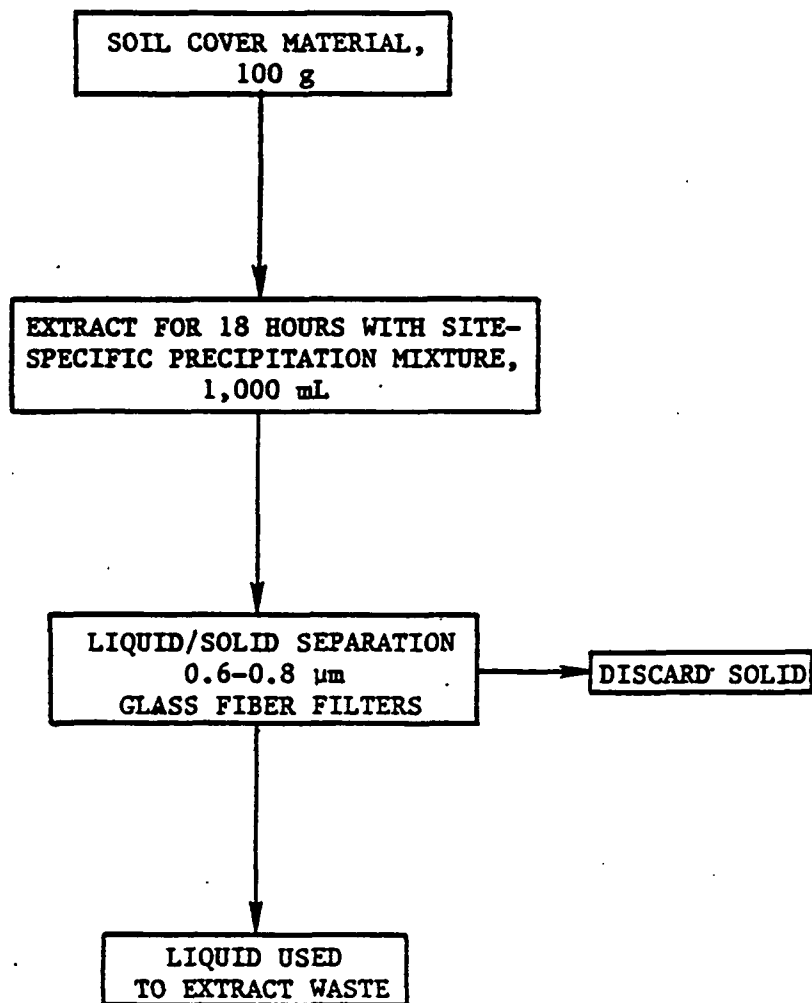
Figure A-1. Generation of site-specific extraction medium.

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APPENDIX B

STATISTICAL EVALUATION OF MONOFILLED SOLID WASTE EXTRACT DATA

A statistical evaluation of data generated from the MWEP is needed to ensure the quality of interpretive information obtained from this procedure. As part of the development of the MWEP, a statistical model was developed for the purpose of analyzing the various components of error associated with the procedure.

Central to the concept of statistical evaluation of a leachate generation method is the analysis of an extraction "profile." It is likely that leachate produced from a waste disposal site changes in composition over time. This process is a function representing the concentration of a chemical species. The MWEP was designed to produce this profile, which would take several years in the field environment, by accelerating the release of chemical species as a function of repetitive extractions with distilled water. Thus, in the laboratory procedure the extraction profile consists of a plot of constituent concentration versus liquid-to-solid ratio or the number of repetitive extractions.

An extraction profile is characterized using the MWEP based on a series of batch extractions. This laboratory extraction procedure involves subsampling from a grab sample obtained from the waste, extracting the sample of waste, filtering to obtain a liquid filtrate and a solid filter cake, and finally analyzing the concentration of important analytes in the filtrate. The leaching process is repeated several times using the filter cake obtained from the previous trial to obtain a characteristic extraction profile for a given waste sample.

There are several important sources of experimental variation that can affect the results of this type of leachate generation method. Significant variation may be associated with (1) subsampling the grab sample of the waste, (2) the extraction procedure, and (3) the chemical analysis process. Sampling of the original waste is recognized as probably being the major source of experimental variation. However, for the purpose of this discussion, it will be assumed that a representative grab sample of the waste was collected. The grab sample usually can be carefully mixed so that sub-sampling variance is small.

Objectives of the statistical analysis of data collected from an extraction method include:

o   Estimating the extraction profile function;

o   Comparing the extraction profile functions across different types of waste;

o   Estimating components of variation associated with waste sampling and extraction and chemical analysis.

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These objectives can be accomplished through the use of a statistical model that characterizes the data generated by the extraction experiments. The statistical model is based on the assumption that a function $\beta(k)$ exists that, apart from uncontrollable experimental variation, estimates the amount of analyte extracted from a sample with each successive extraction (k).

The function $\beta(k)$ describes only the deterministic mechanism affecting the leaching process at successive extraction number k. The complete statistical model would also include the error structure describing the important sources of experimental variability, such as subsampling, extractions, and chemical analysis. The level of detail required to completely describe the development of the appropriate form for $\beta(k)$ and the error structure is too complex to describe in this appendix.

A simplified version of the complete statistical model is a mixture of fixed and random effects and is given by:

$$X_{ikn} = \beta(k) + S_i + Y_{ik} + \epsilon_{ikn}$$

where

X_{ikn} = the observation recorded for the nth analysis for the kth extraction of the ith subsample of the waste

S_i = the random error due to subsampling

Y_{ik} = the random error due to extraction

ϵ_{ikn} = the random error due to chemical analysis.

The usual distribution assumptions for the random model components are:

$$S_i \sim N(0, \sigma_S^2)$$

$$Y_{ik} \sim N(0, \sigma_L^2)$$

and

$$\epsilon_{ikn} \sim N(0, \sigma_\epsilon^2)$$

where $X \sim N(\mu, \sigma^2)$ indicates a random variable distributed normally with mean μ and variance σ^2.

Standard statistical techniques can be used to estimate the profile function $\beta(k)$ and the variance components $\sigma_S^2 \ \sigma_L^2 \ \sigma_\epsilon^2$. Because the total

variance of the observed data Y_{ikn} is given by:

$$\sigma_Y^2 = \sigma_S^2 + \sigma_L^2 + \sigma_\varepsilon^2 ,$$

the relative percent contribution of each source of variability to the total variability in Y can be estimated. For example, the percent relative contribution of subsampling to the total variation is given by:

$$RV \text{ (Subsampling)} = \sigma_S^2 \Big/ \sigma_Y^2 \times 10$$

Figure B-1 and Table B-1 provide examples of the type of information that is obtained from the statistical analysis of a leaching method. Figure B-1 displays data obtained on the level of chromium contained in the leachate obtained from a 100-percent chromate waste for six successive extractions. The smooth curve superimposed on the data is the estimated extraction profile function $\beta(k)$.

Figure B-2 compares the leachate profiles for the analyte chromium extracted from a landfill soil and a chromate waste. Table B-1 presents the estimates for the variance components and their relative contribution to the total variability for the two types of waste and for eight constituents.
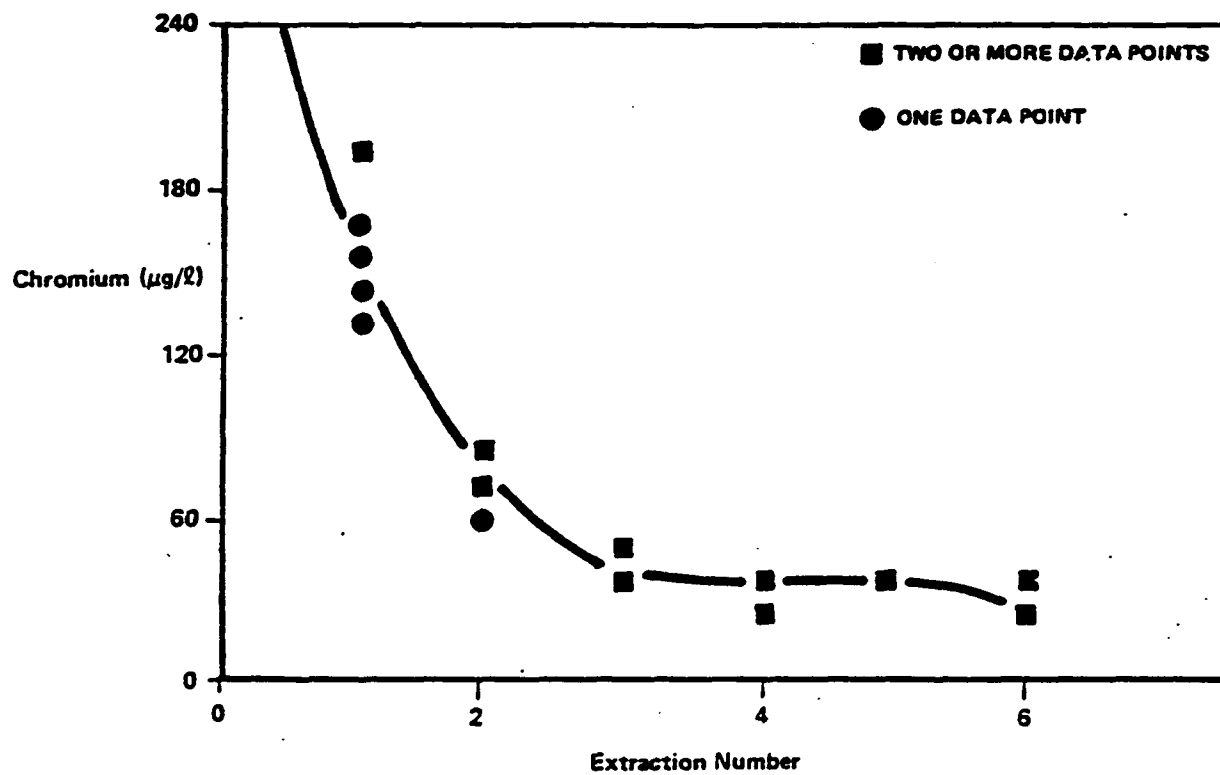
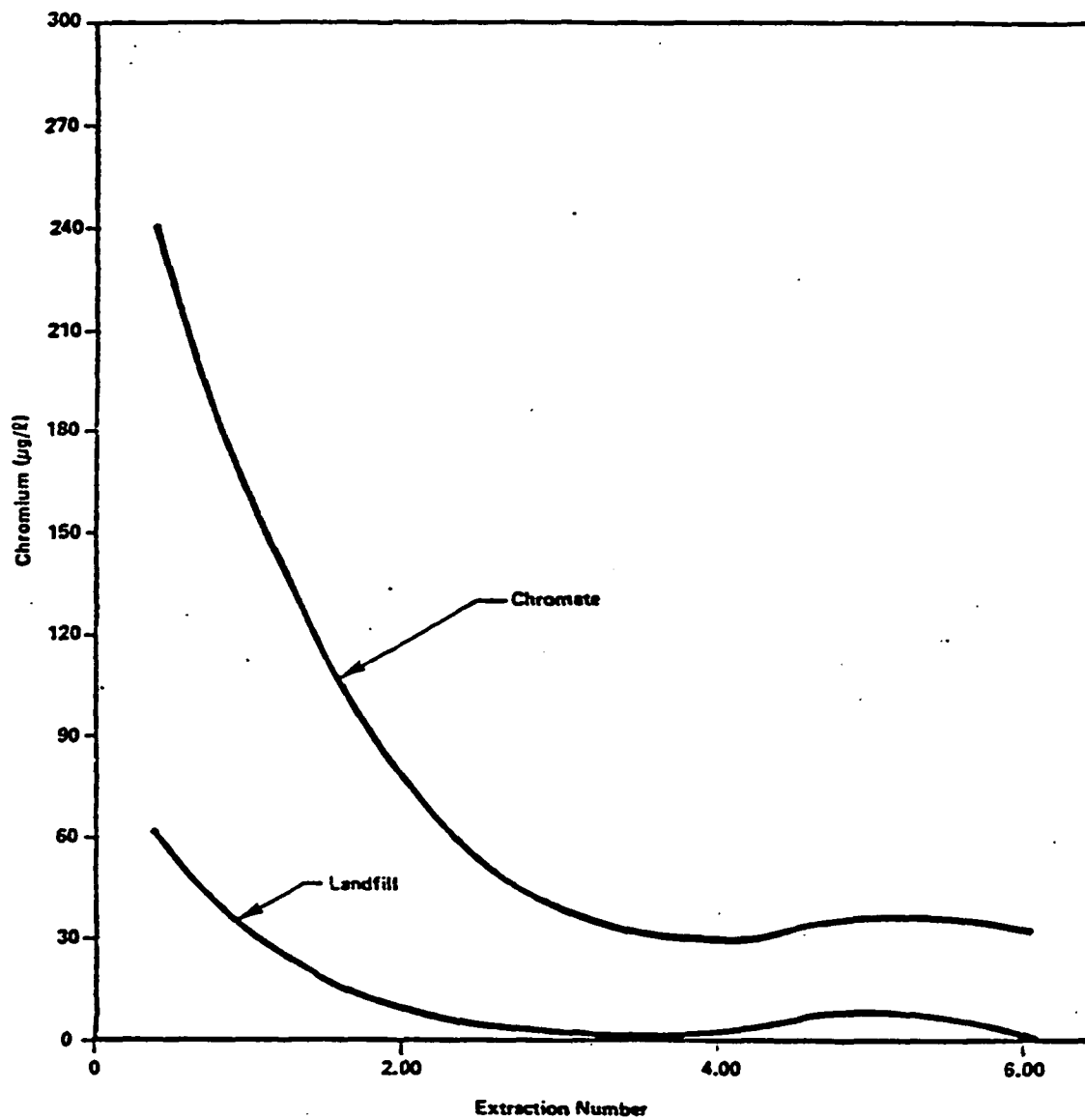Figure B-1. Extraction profile with data for 100% chromate waste.

Figure B-2. Comparison of 100% landfill soil
and 100% chromate waste profiles.

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TABLE B-1. ESTIMATES OF THE PERCENT RELATIVE VARIATION FOR THE
LANDFILL AND 100% CHROMATE WASTES

Analyte	100% Landfill Soil			100% Chromate Waste		
	PRV_s	PRV_L	PRV_E	PRV_s	PRV_L	PRV_E
Boron	0	58	42	6	43	51
Barium	4	95	1	0	100	0
Calcium	0	91	9	37	62	1
Chromium	9	47	44	4	56	40
Magnesium	0	97	3	38	61	1
Sodium	4	61	35	0	81	19
Strontium	0	97	3	36	61	4
Zinc	0	78	22	0	78	22
Mean	2	78	20	15	68	17.

PRV_s = percent relative variation due to laboratory subsampling

PRV_L = percent relative variation due to laboratory analyses

PRV_E = percent relative variation due to extraction

This information is valuable in making comparisons between waste types
and analytes. For example, from Figure B-2 it is clear that the extraction
profiles for the landfill soil and chromate wastes have similar shapes, but
the level of chromium extracted from the landfill soil is much less than from
the chromate waste. Results in Table B-1 indicate that the greatest source of
variability is the extraction procedure for both landfill soil and chromate
waste. This result may be due to the potential error associated with
performance of the sequential extractions, such as filtering and mixing of the
waste. In addition, the contribution of the subsampling to the total
variation in the data appears to be greater for the chromate waste than for
the landfill soil, indicating the soil is more uniformly mixed.

Specific comparisons can also be made. For example, results shown in
Table B-1 indicate that the percent relative variation associated with the
chemical analysis ($\sigma^2 \epsilon$) for boron in the chromate waste is significantly
greater (51 percent on a relative basis) than that for calcium (1 percent on a
relative basis).

APPENDIX C

SUPPLIERS

Item	Suppliers
1. NBS Tumbler	Associated Design and Manufacturing Company 814 North Henry Street Alexandria, VA 22314 (703) 549-5999
2. Membrane Filters	Millipore Corporation Ashby Road Bedford, MA 01730 (800) 225-1380 Nuclepore Corporation 7035 Commerce Circle Pleasanton, CA 94566 (415) 462-2230
3. Glass Fiber Filters	Gelman Sciences, Inc. 600 South Wagner Road Ann Arbor, MI 48106