Development and Applications of a Multiple-batch
Leaching Procedure

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DEVELOPMENT AND APPLICATIONS OF A MULTIPLE-BATCH **LEACHING PROCEDURE**

by

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ABSTRACT

Preliminary studies were conducted to determine the test conditions in a multiple-batch leaching procedure used to evaluate waste leachability. The results of applications of this procedure to a variety of wastes are described in this report. The leaching procedure comprised six batch extractions using three different leaching media (distilled water, an acidic solution buffered at pH 4.5, and a synthetic municipal landfill leachate) at two liquid-to-solid ratios (4:1 and 20:1). A wide range of leaching conditions was selected to simplify comparisons with the results of other leaching tests and to indicate potential changes in the leachability of a waste under different test conditions.

RESUME

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Des études préalables ont été effectuées pour déterminer les conditions d'essai d'une méthode de lessivage par lots multiples permettant d'évaluer le rendement du lessivage de divers résidus. Le présent rapport décrit les résultats de l'application de cette méthode à ces résidus. La méthode de lessivage utilisée comprenait six extractions par lots réalisées avec trois agents de lessivage différents (de l'eau distillée, une solution acide tamponnée à pH 4,5, et le lessivat* synthétique d'une décharge municipale), avec deux rapports liquide-solide différents (4/1 et 20/1). Une vaste gamme de conditions de lessivage a été retenue pour simplifier les comparaisons avec les résultats d'autres essais de lessivage et pour déterminer les variations possibles de la capacité de lessivage de divers types de résidus dans différentes conditions d'essai.

^{*} Dans le présent texte, le terme "lessivat" désigne le produit de lessivage.

TABLE OF CONTENTS

vi

LIST OF FIGURES

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vii k

LIST OF TABLES

i,

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CONCLUSIONS AND RECOMMENDATIONS

Conclusions

- 1. Most agitation methods used in existing batch leach tests either fail to provide good liquid-solid contact or are too severe and cause fragmentation of the solid particles. Slow rotation (2 to 3 rpm) of a square bottle laid on its side was found to be a satisfactory method of mixing.
- 2. A mixing time of 24 hours appears to be sufficient for equilibrium to be reached between most wastes and leaching solutions.
- 3. When a waste is leached with distilled water, the equilibrium pH of the leachate is controlled by the alkalinity or acidity of the waste. When a waste is leached with an acidic solution, leachate pH is a function of both the buffering capacity of the waste and the amount of acid available in the leaching medium to react with the waste.
- 4. Increasing the liquid-to-solid ratio when distilled water is used as the leaching medium has little effect on the equilibrium pH of a leachate. Differences in contaminant concentrations at the higher liquid-to-solid ratio are frequently due to dilution.
- 5. Increasing the liquid-to-solid ratio when an acidic solution is used as the leaching medium increases the amount of acid contacting the waste and shifts the equilibrium pH towards the pH of the leaching medium. This can increase or decrease contaminant release depending on the direction of the pH shift and the nature of the contaminants.
- 6. For some contaminants and wastes, good correlations exist between the logarithm of release and final leachate pH, and between the logarithm of release and the amount of acid in the leaching medium. The lack of correlation in some cases appears to be partially due to the apparent ability 01 the synthetic municipal landfill leachate to complex some contaminants and to increase their release above that to be expected from pH-solubility considerations alone.
- 7. The results of the Multiple-Batch Leaching Procedure (MBLP) are useful for comparing the leachability of various wastes under the same test conditions, but cannot be used to predict the long-term leachability of these wastes in field disposal situations.

Recommendations

- 1. The three batch extractions at the 4:1 liquid-to-solid ratio should be excluded from future applications of the MBLP, as they frequently add only marginally more information about waste leachability than do the 20:1 extractions.
- 2. For wastes where codisposal with municipal refuse is not an option, the extraction with the synthetic municipal landfill leachate should be omitted.
- 3. The MBLP (with the modifications noted above) should be used in future leaching studies as a prescreening test to provide information on the leachability of a waste relative to other wastes under the same test conditions.

1 **INTRODUCTION**

Considering the large quantity and variety of industrial wastes that are now being produced, and the large number of widely distributed production sources and disposal sites, it is obvious that a case by case field evaluation of the hazards associated with the disposal of specific wastes in specific landfill sites is impossible. Nevertheless, the potential for environmental contamination dictates that some type of methodology must be used to assess the possible extent of contaminant release in a field disposal situation. This methodology has traditionally been in the form of bench-scale laboratory leaching tests.

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Leaching tests can be conducted for several different purposes. For example, they might be used to determine if a waste is hazardous, to measure the efficiency of a stabilization technique, or to characterize the leachate produced from a waste (for evaluating the potential for groundwater contamination or designing a leachate treatment system). In general, there are two approaches to leaching studies. The choice of the approach to be used depends on the purpose of the test.

In the first approach, a standard test is adopted which allows results between different laboratories to be compared on a common basis and provides a uniform criterion for the evaluation of different wastes. The standard test also permits development of a data base for comparison of laboratory and field data. A precise protocol is used for all wastes irrespective of the disposal situation and all wastes are treated on the same basis. This protocol must be precisely defined and yield reproducible results, and should be rapid, inexpensive, and universally applicable. One example of a standard test is the EPA Extraction Procedure which is used as part of the protocol to classify hazardous wastes (U.S. EPA, 1980).

In the second approach, the test conditions are made as site specific as possible. This approach attempts to evaluate the hazardousness of a waste in the context of an actual field disposal situation. As a result, decisions based on a site specific test are more conClusive than those arising from a standard test. The site specific test, however, is more time consuming and costly, and does not allow for the establishment of a data base or for the comparison of different wastes on the same basis.

A leaching test can be used, therefore, for either waste classification or leachate quality prediction. When a test is used for classification of a waste (e.g., hazardous or non-hazardous), a standard methodology is appropriate since it allows comparison among laboratories and ensures better reproducibility of the results. When a test is used for predicting the quality of the leachate in a field disposal situation, the major concern is to make the test conditions as site specific as possible.

A multitude of leaching test protocols have been proposed, however, there is no uniformity over the test conditions and no general agreement over the adoption of a standard protocol (Lowenback, 1978). There are also many important environmental conditions present in a field disposal situation which cannot be routinely reproduced in a laboratory. The inability to reproduce these conditions is principally due to temporal differences between the two leaching situations. For convenience, many investigators frequently attempt to condense several years of leaching into a short period of time by using a high liquid-to-solid ratio. This temporal contraction precludes reproduction of the kinetics of pollutant release in a landfill, and necessitates neglect of those factors which are time-dependent in a field leaching situation, such as microbial activity, temperature variations and possible interactions among wastes. These limitations of laboratory leaching tests should be recognized and taken into account in the interpretation of results.

Leaching tests can generally be classified as column tests or batch tests. In the former, the waste is placed in a column and an appropriate leaching medium passed through it. Soil attenuation can be modelled by adding a layer of soil at the bottom of the column. Batch tests consist of agitating a waste sample with a predefined quantity of liquid for a specified time. More than one elution can be performed, and either the waste or liquid is replaced at each elution in order to obtain information on the maximum concentration or the maximum release of contaminants, respectively. Although column tests are often considered to be more representative of field conditions than batch, they have been criticized because their duration can range from weeks to months and the results are often difficult to reproduce. The latter problem may arise from channeling, non-uniform packing of the waste, clogging, and biological growth. Batch tests are comparatively rapid and simpler to perform. The results are more reproducible than those from columns since test conditions can be closely controlled over a relatively short period of time. Consequently, most of the recent development work on leaching tests has been done by batch testing.

In 1980, a study was undertaken at the Wastewater Technology Centre (WTC), Burlington, Ontario, to investigate various aspects of batch leaching (Côté and Constable, 1981, 1982, 1983). After a literature review, those conditions that needed further consideration were identified and a series of experiments were performed to examine the effect of various test conditions on contaminant release from wastes. The results were subsequently used as the basis for a batch leaching protocol which is referred to herein as

the Multiple-Batch Leaching Procedure (MBLP). The MBLP comprises six batch extractions using three different leaching media at two liquid-to-solid ratios. A wide range of test conditions was selected to simplify comparisons with the results of other leaching tests, and to indicate potential changes in the leachability of a waste under different test conditions.

The Multiple-Batch Leaching Procedure has been applied to various wastes from across Canada to establish a standardized data base on waste leachability. The data base allows comparisons to be made on the leachability of different wastes under the same test conditions and assists in the comparison of results from other leaching protocols including those proposed by ASTM (1979) and the U.S. EPA (1980).

This report describes the preliminary studies that were conducted to determine the six extraction conditions used in the Multiple-Batch Leaching Procedure, and presents the results of applications of the MBLP to a variety of wastes.

2 **VARIA TIONS IN EXISTING BATCH LEACHING TESTS**

A partial survey of batch leaching tests was done by the U.S. EPA (Lowenbach, 1978). Thirty existing tests representing a large range of test conditions were considered (Table 1). The test conditions and procedures were found to frequently originate from the availability of laboratory material (e.g., shaker, bottles) and from the type of waste being tested. Many tests were developed by modifying an existing procedure to suit particular needs.

2.1 Leaching Medium

The choice of a leaching medium should be dictated by expected landfill conditions and by the type of chemical species to be leached from the waste. For example, most metals will be solubilized by an acidic solution, whereas organic chemicals will leach more readily under basic conditions.

Ham et al. (1979a) identified three landfill situations that could represent extremes in leaching medium composition (Table 2). When waste is landfilled by itself with relatively small amounts of other wastes, the leaching media will consist of rainwater (after passing through the cover layer of soil) in the top layers of waste, and of leachate produced from the waste in the bottom layers. If the waste is co-disposed with municipal refuse, the leaching medium can have the characteristics of municipal refuse sanitary landfill leachate. If the waste is co-disposed with other industrial wastes, the composition of the leaching medium can vary considerably. Depending on the landfill situation, therefore, the appropriate leaching medium may range in composition from a synthetic leachate modeled on actively decomposing municipal sanitary landfill leachate to something approaching distilled water.

One way of accounting for these large variations is to consider the aggressiveness of a leaching medium, which refers its ability to extract chemical constituents from a waste. A non-aggressive leaching medium such as distilled water allows the waste to create its own leaching environment, whereas a strong chemical solution or synthetic leachate essentially controls the leaching environment. For example, a waste containing small amounts of a leachable basic salt will raise the pH of a distilled water leachate, and only materials that are soluble in basic solutions will be found in the leachate. Conversely, use of a heavily buffered acid solution will probably neutralize the basic salt while maintaining an acidic pH. In the first case the waste controls the pH of the solution, while in the second case, the leaching medium is the controlling factor.

TABLE 1 SUMMARY OF EXISTING LEACHING TEST CONDITIONS

Leaching Media (Typical: distilled water)

- water (distilled, deionized, rain)
	- water adjusted for pH (with different buffering capacities)
- site specific liquid
- synthetic municipal landfill leachate
- bacterial nutrient media

Liquid/Solid Ratio (Typical: 10:1)

1:1 to 500:1

Time per Elution (Typical: 24 hours)

 \bullet 1/2 hour to 28 days

Number of Elutions (Typical: 1)

1 to 10 \bullet

Solid Preparation (Typical: as received)

- as received
- crushed and sieved
- dried
- vacuum filtered (0.45 µm)

Size of the Solid Sample (Typical: 100 g)

1 to 500 g

Agitation Technique (Typical: shaking)

- none
- stirring
- shaking: reciprocating, circular, wrist action
- gas bubbling

Temperature (Typical: room)

20°C to 33°C

TABLE 2 LEACHING MEDIA FOR VARIOUS TYPES OF LANDFILLS (Ham et al., 1979a)

Several characteristics of the leaching medium contribute to its aggressiveness, inCluding:

- 1) pH: The value of the pH and the buffering capacity that maintains that value are both important. Most leaching media encountered in the environment are acidic. The pH of rain falling in Ontario varies between 4.0 and 6.0 and is controlled by the nitrate, sulphate and carbonate systems. Leachate in an actively decomposing sanitary landfill can reach pH values of 4.0 to 4.5 , and be heavily buffered by volatile acids (Stanforth et al., 1979).
- 2) Redox Potential: Redox potential is a measure of the oxidizing and/or reducing *intensity* of a system. It determines, in part, the aerobic or anaerobic conditions under which the material will be leached. Redox reactions are important in the solubilization of iron and manganese. In a landfill, bacterial activity plays a major role in controlling redox potential.
- 3) Complexation Capacity: Complexation is defined as the formation of a complex from a metal ion with a negative ion. Many different ligands, both organic and inorganic, can complex metals and leach them from wastes. Organic compounds containing nitrogen, oxygen or sulphur in the proper configuration can be very strong complexers.
- 4) Ionic Strength: Ionic strength may affect the leaching of materials in three ways: 1) by increasing the solubility through lowered activity coefficients, 2) by ion exchange processes that replace an ion bound to an ion exchange site with one of the more predominant ions in solution, and 3) by decreasing the size of the double layer around colloidal particles and promoting coagulation.

Not all of these aggressive properties are of equal importance with respect to leaching chemical species from a waste. Synthetic leaching media have been developed that attempt to model one or more of these aggressive properties. For example, synthetic landfill leaching media were developed by researchers at the University of Wisconsin to model co-disposal of a waste in a municipal landfill site (Stanforth et al., 1979). The main advantage of using a synthetic leaching medium is a more realistic representation of the leaching conditions likely to be encountered in a field disposal situation. It may also be argued that, in a short test, it is better to use an aggressive medium that will control the leaching environment. The disadvantages include the increased laboratory work involved, the possible introduction of chemical impurities that could interfere with the results of the test, and the toxicity of the leaching medium of fish, thus preventing the use of bioassays. The chemical compositions of several leaching media are given in Table 3.

2.2 Liquid-to-Solid Ratio

The liquid-to-solid ratio (R) used in batch tests can vary from 1:1 to 500:1, with a typical test using a ratio of 10:1. 'R' values reported in the literature, however, are difficult to compare because of the different interpretations given to the determination of the liquid portion of a waste, and the role of that portion in the ratio.

There are six possible ways of defining R. The liquid portion of a waste sample can be defined as the difference between the wet and dry weights of the sample, or as the amount of water separable by filtration. Furthermore, the liquid portion, however determined, can be considered in the ratio to be (1) part of the waste, (2) part of the leaching medium, or (3) part of neither, as expressed in the following equations:

$$
R = \frac{L}{W+S}
$$
 (1)

$$
R = \frac{L + W}{S} \tag{2}
$$

$$
R = \frac{L}{S}
$$
 (3)

where: $L =$ liquid added as leaching medium

 $W =$ liquid portion of the waste (which may be interpreted in two different ways)

 $R =$ solid portion of the waste (complement of the liquid portion)

In the case where a waste contains a significant amount of liquid (e.g., a wastewater treatment sludge), the method used to compute R may have a significant effect on test results. This effect will become less significant as the value selected for R increases.

Selection of a suitable value for R is influenced by practical considerations, such as the availability of a proper agitation apparatus and the volume of leachate required for analysis. The ratio should be high enough to prevent limiting contaminant release into the leaching medium because of solubility constraints, yet small enough to provide concentrations that are above the detection limit of the analytical apparatus. A review of existing tests suggests that a ratio between 4:1 and 20:1 would satisfy these conditions.

2.3 Time of Elution

The concentration of a chemical species in the leaching medium is a function of the time of elution up to the time where equilibrium is reached. Lee and Plumb (1974) found four release patterns in a 500-day leaching study using taconite tailings (Figure 1).

TIME

Types of release patterns

Examples 1) specific conductance, alkalinity, Ca, Mg, and others

- 2) silica (a slow hydrolysis step needed before Si is solubilized)
- 3) Cu, Zn-Ioss due either to rising pH in solution, or absorption back onto solids
- 4) several species
- FIGURE 1 TYPES OF RELEASE IN LONG-TERM LEACHING TEST ON TALCONITE TAILINGS (Lee and Plum, 1974)

Not only did equilibration times for different parameters vary widely, but for some parameters a series of reactions occurred which produced concentration maxima with subsequent concentration decreases. The variety of release patterns found indicated that no one time of elution is best for all wastes. The results also showed that a time of elution on the order of hours (as opposed to days) should be sufficient to allow most leachable contaminants to approach equilibrium in the leachate.

The choice of an appropriate time of elution is often dictated by practical considerations. It should be long enough to allow rapidly dissolving species to approach equilibrium. A very short test (e.g., less than 2 hours) is not suitable because of the variability in the results induced by laboratory manipulations. The leaching medium and the waste are in contact before and after the agitation period for a certain length of time which depends on the number of test bottles and on the skill of the technician. This period of time can range from a few minutes to an hour; it should not be of the same order as the time of elution. The time of elution should also be convenient to personnel. If multiple elutions are needed, the time of elution should be compatible with working schedules. A practical time of elution would thus seem to be some multiple of one day; many leaching tests use 24 hours.

2.4 Number of Elutions

Performing multiple elutions by replacing either the solid or liquid phase can be done in order to get an indication of the dynamics of the leaching process. Successive elutions can reveal the release pattern of a contaminant over time, and often can give an idea of the factors affecting its release. Successive elutions are particularly important when the release of one contaminant is inhibited by the release of another. For example, trace metals will not be leached from a waste with some acid neutralization capacity until this capacity has been expended by the leaching medium. If the test is ended before this occurs, the potential for trace metal leaching will be overlooked.

When the waste is replaced in successive elutions, information will be generated on the maximum concentration of species in solution. If the liquid is replaced in successive elutions, the maximum release of chemical species under test conditions can be evaluated. The results can be expressed as the ratio of mass of contaminant leached to mass originally present in the waste.

2.5 Surface of Contact

The particle size of the waste controls the surface of contact between the waste and the leaching medium. The surface of contact should not affect the equilibrium concentrations of chemical species in leachate, although it may have profound effects on the kinetics of release. The importance of this factor is determined by the permeability of the waste and test conditions such as the agitation method and the time of elution. Three processes may limit the rate of dissolution of a species: 1) the rate of chemical reaction at the liquid-solid interface, 2) the diffusion of products away from the surface, and 3) the renewal of the species at the liquid-solid interface by diffusion through the waste. If condition I or 3 is the dissolution controlling factor, the ratio of the surface of contact to the volume of the waste then becomes most important.

2.6 Agitation Techniques

The agitation method is important because it determines the degree of contact between the leaching medium and the waste. Existing methods include stirring, shaking or bubbling gas through the test bottle. An efficient agitation method must meet the following conditions: 1) the entire surface area of the waste must be contacted with the leaching medium, 2) the mixing technique must prevent stratification in the test bottle, and 3) the physical structure of the waste must be preserved.

The first condition eliminates the commonly used shaking techniques (e.g., reciprocating shaker and wrist action shaker where the waste remains on the bottom of a bottle and the liquid is agitated over it. It is possible to increase shaking speed to ensure good contact but this may result in violation of the third condition.

2.7 Sample Preparation

A leaching test, in order to be reproducible and widely applicable, should include a sample preparation protocol. This should provide guidance for representative sampling, liquid-solid separation and particle size reduction of monolithic wastes. The need for a liquid-solid separation step before a leaching test depends on the method selected to compute the liquid-to-solid ratio and on the reason for conducting the test. If the purpose of the test is classification of wastes, liquid-solid separation can be performed to improve reproducibility. On the other hand, if the test is to be used to predict leachate quality in a landfill, the liquid portion can be considered to be an integral part of the waste and liquid-solid separation should not be performed.

3 DEVELOPMENT OF THE MULTIPLE-BATCH LEACHING PROCEDURE (MBLP)

3.1 Evaluation of Leaching Test Conditions

A series of five experiments was conducted at the WTC to measure the sensitivity of batch leaching test variables on the release of contaminants from wastes. The results of the experiments were then used to select a set of test conditions that would maximize test reproducibility and sensitivity, and also provide information for a wide range of leaching situations. The five experiments examined the effects of the following on contaminant release:

- 1) nine different methods for agitation;
- 2) a 2^5 factorial screening experiment on five test conditions leaching medium, liquid-to-solid ratio, particle size, time of elution, and number of elutions;
- 3) two different leaching media monitored during a 24-hour leaching period to evaluate the kinetics of release;
- 4) six different leaching media; and
- 5) liquid-solid separation of a waste sample prior to leaching.

A summary of the test conditions used in each experiment is given in Table 4. Two inorganic sludges were used (Row 1), a Basic Oxygen Furnace (BOF) sludge from a steel industry (Experiment 1), and a metal hydroxide (MH) sludge from a metal finishing industry (the other four experiments). In Experiments 2, 3 and 4, the MH sludge was solidified prior to leaching using 20 percent Portland cement and 5 percent lime in order to ensure particle size control. The solidified sludge was air cured for 28 days, then crushed and sieved to the required particle size. The characteristics of the raw sludges are presented in Table 5.

The agitation techniques used in each experiment are indicated in Row 2 of Table 4. Nine different techniques were considered in the first experiment. These techniques are depicted graphically in the first column of Figure 2, and described in Table 6. They included a wrist action shaker (A in Figure 2), a reciprocating shaker at two speeds with two bottle sizes (B, C and D), a circular shaker at two speeds with square and round bottles (E, F and G), a tumbler (H) and a stirrer(I). Based on the results of Experiment 1, the tumbler agitation method (H) was selected for use in subsequent experiments. In this method, the liquid-solid mixture is rotated slowly (2 to 3 rpm) in square bottles so that the waste gently tumbles through the liquid.

TABLE 4 SUMMARY OF EXPERIMENTAL CONDITIONS

* BOF = basic oxygen furnace sludge; MH = metal hydroxide sludge; MH_S = solidified metal hydroxide sludge

** DW = distilled water; AS1, AS2 = acidic solutions; SL1, SL2, SL3 = synthetic leaching media

*** the -1 and +1 indicate the two settings of each parameter in the level 2 factorial experiments

**** pH and conductivity were also measured in every experiment

Row 3 of Table 4 lists the leaching media used in the experiments. Distilled water and one of two acidic solutions were used in most of the experiments. The first acidic solution had a pH of 4.5, and was buffered to 82 meq of acid per litre by the addition of acetic acid and sodium acetate (ASTM, 1979). The second solution had a larger buffering capacity (200 *meq/L),* and was prepared to provide the same amount of acidity as the EPA Extraction Procedure (U.S. EPA, 1980) when the maximum amount of acid is added. Six different leaching media were used in Experiment 4, including distilled

* Basic Oxygen Furnace sludge: average of 2 analyses

Metal Hydroxide sludge: average of $\overline{3}$ analyses

water, the two acidic solutions, and three synthetic leachates (Stanforth et al., 1979; Anon., 1977). The chemical compositions of the media are given in Table 3. All six leaching media were analyzed for Cr, Cu, Mg and Zn. All metal concentrations were below the detection limit except for Mg in the Netherlands synthetic leachate (Anon., 1977). Leaching tests were conducted at liquid-to-solid ratios of 4:1, 10:1 and 20:1 (Row 4). The ratios were computed using Equation 3 (Section 2.2) in the first four experiments, and Equation 1 in Experiment 5.

The sizes (diameters) of the particles used in the first four experiments are indicated in Row 5 of Table 4. The waste was passed through two different sieves and the particles retained on the bottom sieve were used in the experiments (Table 7). The diameters given in Row 5 (Table 4) are the geometric mean of the two sieve sizes used to segregate the particles. Particle size segregation was not performed in Experiment 5.

- Experimental measurement \bullet
- Mean of three measurements
- \rightarrow 95% confidence interval for the mean (Student's t distribution)
	- Minimum difference between means for significance (95 % confidence) according to Tukey's paired comparison procedure.

COMPARISON OF AGITATION METHODS **FIGURE 2**

TABLE 6 DESCRIPTION OF AGITATION METHODS

* The control was limited to one minute of shaking by hand at the beginning and end of the 24-hour elution period.

TABLE 7 PAR TICLE SIZE DEFINITION

* assuming each particle is a spheriod

The time of elution in most experiments was 24 hours (Row 6, Table 4). Experiment 2 also used an elution time of 48 hours, and Experiment 3 used times ranging from 0.5 to 24 hours.

Only one elution was performed in most of the experiments (Row 7, Table 4). A second elution was used in Experiment 2 to compare contaminant release during the first and second contacts of a waste with equal amounts of fresh leaching medium.

Replicates were performed in most of the experiments to estimate error variance and evaluate the statistical significance of the results (Row 8, Table 4).

The parameters measured in the filtered $(0.45 ~\mu m)$ leachates from each experiment are listed in Row 9 (Table 4).

3.2 Results and Discussions

3.2.1 Experiment 1: Agitation Methods. The first experiment was conducted to find a suitable agitation technique for use in all subsequent experiments. Nine agitation methods were evaluated on their ability to satisfy the three conditions listed in Section 2.6, their reproducibility (triplicates were run), and the difference between their results and those of a control ('J' in Table 6). The control was not agitated except for one minute of shaking by hand at the beginning and end of the 24-hour elution period.

The results for four of the parameters measured in each leachate (pH, conductivity, Ca and Mg) are shown in Figure 2. Comparisons of the range of triplicate measurements for each parameter and of the differences between the results obtained from each technique and from the control show that some agitation methods are more reproducible and aggressive than others.

The methods that gave results different from those of the control for most parameters were A, D, H and I. Methods A, D and I, however, were deemed unacceptable because agitation was too severe and caused fragmentation of the waste particles. Method H, a tumbler, was judged to best meet the criteria established for an acceptable agitation method, and was used in all subsequent experiments.

Further work using the tumbler has shown that results are reproducible irrespective of bottle size or liquid level, as long as a square bottle is used. A square bottle allows the waste particles to gently tumble through the leaching medium, providing good liquid-solid contact without particle fragmentation.

3.2.2 Experiment 2: Screening. After determination of a suitable agitation technique, a replicated $2⁵$ factorial experiment was performed to examine the effect of five other test conditions on contaminant release: leaching medium (L), liquid-to-solid ratio (R) , particle size (P) , time of elution (T) and number of elutions (N) . The two levels considered for each test condition were selected to be representative of the typical range of variation encountered in existing leaching tests, and are given in Table 4.

The response measured at each of the experimental settings are given in Table 8. These responses were analyzed using least-squares regression. The 32 (i.e., 2^5) pieces of information generated were the average, 5 main effects, 10 two-factor

RESPONSES OF THE 2^5 FACTORIAL DESIGN SCREENING EXPERIMENT TABLE 8

*52 degrees of freedom

interaction effects, 10 three-factor interaction effects, 5 four-factor interaction effects, and 1 five-factor interaction effect. If the higher order interaction effects are neglected, the results can be represented by the following regression equation:

/

$$
\hat{Y} = \hat{\beta}_0 + \hat{\beta}_L \cdot L + \hat{\beta}_R \cdot R + \hat{\beta}_P \cdot P + \hat{\beta}_T \cdot T + \hat{\beta}_N \cdot N + \hat{\beta}_L R \cdot L \cdot R + \hat{\beta}_L P \cdot L \cdot P + \n\hat{\beta}_L T \cdot L \cdot T + \hat{\beta}_L N \cdot L \cdot N + \hat{\beta}_R P \cdot R \cdot P + \hat{\beta}_R T \cdot R \cdot T + \hat{\beta}_R N \cdot R \cdot N + \hat{\beta}_P T \cdot P \cdot T + \n\hat{\beta}_P N \cdot P \cdot N + \hat{\beta}_T N \cdot T \cdot N
$$
\n(4)

where: \hat{Y} = predicted response (pH, conductivity, etc.) $L, R, P, T, N =$ experimental factors assuming the values -1, 0 or +1
 $\hat{\beta}_0, \hat{\beta}_L, ..., \hat{\beta}_{TN} =$ estimated values of the regression coefficients

The settings of the test conditions corresponding to the -1 and +1 levels of the experimental factors are given in Table 4. The regression coefficients (β 's) that were significantly different from zero (Student t test, 95 percent confidence interval) are presented in Table 9. The first line of the table gives the overall average of the response. The next five lines give the main effects. The rest of the table lists the 2-factor interaction affects.

The importance of each test condition can be evaluated by considering the magnitude of each coefficient in Table 9. The larger the absolute magnitude of a coefficient for a particular measured response, the more influence the corresponding experimental factor had on that response in a leaching test. The sign of each coefficient indicates whether changing an experimental factor from its minus to plus level increased (positive coefficient) or decreased (negative coefficient) the response magnitude.

While all coefficients in Table 9 are statistically significant, most of the variation in response magnitude that is caused by changes in test conditions can be accounted for by considering only those having the largest absolute values. These are indicated by the underlined values in Table 9, and account for at least 90 percent of the variation observed in each response. In order to facilitate the interpretation, the results can be presented as regression equations for each response. The only terms included in each equation are the underlined coefficients in Table 9.

Experimental Factor (Independent Variable)	pH	Conductivity (mS/cm)	Chromium (m/L)	Copper (mg/L)	Magnesium (mg/L)
Average	9.76	4.73	4.97	0.69	29.0
Leaching Medium (L)	0.88	0.13	1.32	0.17	-28.8
Liquid-to-Solid Ratio (R)	-0.27	-1.14	-1.81	-0.26	17.6
Particle Size (P)	-0.15	-0.24	-0.24	-0.07	
Time of Elution (T)	0.13				1.4
Number of Elutions (N)	-0.20	-1.39	-2.08	-0.32	15.5
LR interaction	0.21	-0.44	-0.44	-0.08	-17.7
LP interaction	0.17				
LN interaction	0.16	-0.50	-0.35	-0.11	-15.5
RP interaction		-0.05		-0.05	-4.5
RT interaction	0.03		0.12		1.8
RN interaction	-0.03	0.29	0.53	0.12	6.7
PT interaction	0.04				-1.4

TABLE 9 COEFFICIENTS FROM A REGRESSION ANALYSIS OF THE SCREENING EXPERIMENT

Notes: - the settings of the experimental factors are given in Table 4

PN interaction -0.04 -0.03 -2.2 TN interaction -0.03 0.09 -0.02 0.8

> - only the significant coefficients are included in this table (i.e., those whose 95 percent confidence interval (Student t test) does not include zero)

> - the summation of the sum of squares of underlined coefficients is greater than 90 percent of the total sum of squares

- the LT interaction was confounded with blocks

Note that these regression equations are specific to the waste and the environmental conditions of this experiment. The independent variables are transformed variables which take the values -1 and +1; the test conditions corresponding to these two settings are given in Table 4.

Examination of the response equations indicates that the major experimental factors are the leaching medium, the liquid-to-solid ratio and the number of elutions. The effect of each of these three factors on test results cannot be evaluated independently of the other two because of the relatively large LR and LN interaction coefficients. The significance of these interaction coefficients can be explained by considering the different leachate pH's observed in each test. Leachate pH was found to be essentially independent of Rand N when distilled water was used as the leaching medium. When a buffered acidic solution was used, however, the acidity of the leaching medium resulted in a lower leachate pH than observed with the distilled water leach. Increasing the liquidto-solid ratio or performing a second elution increased the amount of acidity brought into contact with the waste, and resulted in a further decrease in leachate pH.

3.2.3 Experiment 3: Time Release Patterns. Since the results of Experiment 2 indicated that 24-hour leach tests gave approximately the same results as 48-hour leach tests, it was decided to examine the effects on test results of using leaching periods less than 24 hours. Tests were conducted for 0.5 , 1 , 2 , 4 , 8 and 24 hours using two media (distilled water and an acidic solution). Leachates were analyzed for pH, conductivity, Cr, Cu and Mg.

The temporal variations in pH and copper concentrations over a 24-hour leaching period are shown in Figure 3a for the distilled water leach and Figure 3b for the acidic solution leach. It is apparent that the release patterns were considerably different for the two leaching media. With distilled water, both pH and Cu reached or approached their final values within the first half-hour. However, with the acidic solution, pH increased gradually over 24 h, whereas Cu peaked at 4.2 mg/L at 0.5 h and then decreased to reach its final value of 0.32 mg/L at 8 h. The peak occurred because the copper was solubilized in the initial stages when pH was low, but reprecipitated as pH increased. The increase in pH was caused by neutralization of the acidic medium by the waste.

Release patterns for conductivity, Cr and Mg in both leaching media were similar to that shown for pH in Figure 3a (i.e., final values were reached within the first half hour).

3.2.4. Experiment 4: Leaching Media. The results of Experiments 2 and 3 showed that the leaching medium was a major factor, therefore, Experiment 4 was undertaken to compare the effect of six different leaching media on contaminant release. Other experimental conditions were kept the same during all six tests (Table 4). The results are presented in Table 10, and provide further evidence of the importance of the leaching medium in batch tests. All parameters displayed wide variability among tests, particularly the metals. While some of the differences in leached metal concentrations can be explained by solubility considerations, many of the high metal concentrations observed in

b) Acid solution leach

FIGURE 3	TEMPORAL VARIATION OF pH AND COPPER CONCENTRATION IN
	EXPERIMENT 3

 \ast see Table 3

the values shown are the measured conductivities minus the conductivity of the $**$ leaching media

the concentration of Mg in the leachate was lower than the original concentration in the leaching medium

tests using synthetic leaching media are probably due to the complexing agents contained in these media.

3.2.5 Experiment 5: Liquid-Solid Separation. A replicated 2^3 factorial experiment using two leaching media and two liquid-to-solid ratios was conducted to evaluate the importance of performing liquid-solid separation of a waste sample prior to a leaching test.

A raw metal hydroxide sludge (TS = 10%) was used in the runs without liquidsolid separation. In those runs with separation, the sludge was pressure filtered following the U.S. EPA Extraction Procedure (EPA, 1980), and the filter cake (TS = 20%) used in the tests. The filtrate was analyzed and discarded.

The responses measured at each of the experimental settings are given in Table 11. These responses were analysed using least-squares regression. The significant main and interaction effects (at a 95 percent confidence level) for pH, conductivity, Cu and Mg are given in Table 12. The largest effects arose from changes in the liquid-tosolid ratio and the leaching medium. Performing liquid-solid separation on the waste prior to leaching had relatively little effect on Cu and Mg concentrations and pH, and no significant effect on conductivity.

Analyses of the filtrate from the liquid-solid separation step revealed that the pH of the free liquid was 12.0. This value is close to the average pH of 11.1 obtained in the tests when distilled water was the medium. The conductivity of the filtrate varied from 10.5 to 14.0 mS/cm, while the Cu concentration was 1 mg/L and Mg was nondetectable.

Based on the concentration of Cu found in the filtrate, the percent of Cu in the leachate originally present in the free water of the sludge was computed for the extractions where no liquid-solid separation had been performed (Table 13). It can be seen that when distilled water was the leaching medium, a significant fraction of the Cu found in the leachate (37 percent and 50 percent depending on the liquid-to-solid ratio) came from the free water associated with the sample, the rest being extracted from the solid. With the acidic solution, these values dropped significantly to 0.10 percent and 0.01 percent due to a higher release of Cu from the solid fraction of the waste. Thus when distilled water was the leaching medium, a considerable amount of the Cu found in the leachate was not extracted from the solid portion of the waste, but originated from the free liquid portion.

TABLE 11 RESPONSES OF THE 2³ FACTORIAL DESIGN LIQUID-SOLID SEPARATION EXPERIMENT

* 8 degrees of freedom

TABLE 12 COEFFICIENTS FROM REGRESSION ANALYSIS OF THE LIQUID-SOLID SEPARATION EXPERIMENT

1) The settings of R and L are given in Table 4. The -1 and +1 levels of S were without and with liquid-solid separation, respectively.

2) Only the significant coefficients are included in this table (i.e., those whose 95 percent confidence interval (Student t test) does not include zero).

TABLE 13 PERCENT OF COPPER IN LEACHATE THAT WAS ORIGINALLY PRESENT IN THE FREE LIQUID PORTION OF THE SLUDGE

3.3 The Multiple-Batch Leaching Procedure.

Based on the literature survey and the results of the laboratory work previously described, a leaching procedure involving six batch extractions was defined which would allow comparisons of the leachability of various wastes and indicate potential changes in the leachability of a waste under different test conditions. In selecting the test conditions to be used in the leaching procedure, a choice had to be made between single and multiple elutions. Multiple elutions are useful for evaluating the kinetics of contaminant release, particularly when the leaching medium has some buffering capacity; however, they add considerably to the time and effort required to complete a leaching test. It was decided to restrict the procedure to single elutions, but to use a variety of test conditions to provide a wider data base for examination of the effects of different conditions on waste leachability. The test conditions were chosen in order to maximize test sensitivity and reproducibility, and to envelop the range of test conditions used in the standard leaching tests proposed by ASTM (1979) and the U.S. EPA (1980). Consideration was also given to developing a relatively inexpensive test by using available laboratory equipment and attempting to minimize the amount of training required to perform the procedure.

The MBLP comprises a set of six batch extractions (Table 14) using three leaching media (distilled water, an acidic solution buffered at pH 4.5 (ASTM, 1979), and a synthetic municipal landfill leachate (Stanforth et al., 1979)), at two liquid-to-solid ratios (4:1 and 20:1). The chemical compositions of the latter media are given in Table 3 (Numbers 2 and 5).

The two liquid-to-solid ratios of $4:1$ and 20:1 correspond to those used in the proposed ASTM (1979) and U.S. EPA (1980) leaching tests. The batch extractions are performed in square plastic bottles that are rotated slowly (2 to 3 rpm) for 24 hours. The leachates are then decanted, filtered $(0.45~\mu m)$, and analyzed for the parameters of

TABLE 14 IDENTIFICATION OF THE SIX EXTRACTIONS USED IN THE MULTIPLE-BATCH LEACHING PROCEDURE

*the numbers in brackets indicate the meq of $(H⁺)$ available per g of waste

interest. A detailed step-by-step protocol for conducting the MBLP is given in Appendix I.

3.4 Comparison With Other Leaching Tests

Some extractions of the MBLP are similar to the proposed ASTM leaching test (ASTM, 1979) and the Extraction Procedure (U.S. EPA, 1980).

The proposed ASTM leaching test consists of two extractions, one with distilled water (Method A) and one with an acidic solution (Method B). The ASTM tests are similar to MBLP Extractions DW4 and AS4 except for three conditions: the ASTM test uses a reciprocating table for agitation, the ASTM protocol does not include any particle size reduction, and the time of elution is 48 hours. The laboratory work described in Section 3.2 showed leaching test results were not markedly changed by those conditions. The two ASTM tests and Extractions DW4 and AS4, therefore, should give comparable results.

The MBLP extractions DW20 and AS20 are similar to the U.S. EPA Extraction Procedure (EP) except for liquid-solid separation and the amount of acid added to the waste sample. In the EP, provision is made for liquid-solid separation before the test. The solid portion is then extracted, and the extract mixed with the liquid portion prior to analysis for the contaminants of interest. The amount of acid added in the EP is variable. The pH is continuously monitored and adjusted to 5.0 up to a maximum acid addition of 2 meq of H+ per gram of waste (the amount of acid added is dependent on the buffering capacity of the waste). In the MBLP, no acid is added in the DW20 extraction, whereas 1.63 meq/g is added in the AS20 extraction. Thus these two extractions can be

interpreted approximately as the lower and upper extremes of acid addition in the EPA Extraction Procedure.

4 RESULTS OF MBLP APPLICATIONS

The Multiple-Batch Leaching Procedure has been applied to a number of wastes, including bottom and fly ashes from coal-fired power generating stations, metal finishing and chemical plant residues, mine tailings and industrial wastewater treatment sludges. Some of the wastes have also been subjected to the U.S. EPA Extraction Procedure (U.S. EPA, 1980) and the ASTM proposed leaching tests (ASTM, 1979).

The wastes that were tested are listed in Table 15. The results of these tests are given in Appendix II, and are expressed in three formats:

1) Concentration - the ratio of the mass of chemical species X in a leachate (M_{XL}) to the volume of leachate (V_L) :

$$
C_{\text{XL}} = \frac{M_{\text{XL}}}{V_{\text{L}}}
$$
 (10)

2) Release - the ratio of the mass of chemical species X in a leachate (M_{XI}) to the mass of the leached sample (M_S) ; it is also equal to the product of the liquid-to-solid

ratio (R) and the concentration of chemical species X in the leachate (C_{XL}):
REL_X =
$$
\frac{MXL}{M_S} = \frac{MXL}{M_S} \times \frac{VL}{VL} = \frac{V_L}{M_S} \times \frac{MXL}{VL} = R.C_{XL}
$$
 (11)

3) Efficiency - the ratio of the mass of chemical species X in a leachate (M_{XL}) to the

mass of that species in the leached sample (M_{XS}) expressed as a percentage:

$$
EFF_X = \frac{MXL}{MXS} \times 100\%
$$
 (12)

4.1 Observed Trends

Some of the trends that have been observed on the effects that different leaching media and liquid-to-solid ratios have on test results are described in subsequent sections.

4.1.1 The Effect of Leaching Medium on Leachate pH. One of the major conclusions of the background study leading to the development of the MBLP was that the release of contaminants from a waste was largely governed by the final pH of the leachate. This is to be expected as leachate pH has a major influence on contaminant solubility. Leachate pH, in turn, is a function of the alkalinity of the waste, and of the amount of acid available in the leaching medium to react with the waste. The amount of acid in the six batch extractions used in the MBLP varies from none for distilled water to 3.14 meq/g

 \sim μ

TABLE 15 SAMPLES SUBJECTED TO THE MULTIPLE-BATCH LEACHING PROCEDURE

* indicates samples were also subjected to EPA's Extraction Procedure and/or ASTM's batch leaching tests

of waste for the synthetic municipal landfill leachate at the 20:1 liquid-to-solid ratio (Table 14). For comparison, the U.S. EPA Extraction Procedure specifies a maximum acid addition of 2.0 meq/g of waste (U.S. EPA, 1980).

To examine the influence of the leaching medium on leachate pH, the pH data from the 51 MBLP applications listed in Table 15 were compared both with respect to the volume of leaching medium (i.e., the liquid-to-solid ratio), and the acidity of the medium. The results are shown in Figure 4.

When distilled water was used as the leaching medium, final leachate pH was controlled by the acidity or alkalinity of the waste and was largely independent of liquidto-solid ratio, as shown by the coincidence of most of the data points in Figure 4a with the diagonal. Replicates were run on three wastes to measure the reproducibility of the test. The standard deviation of the pH measurements was 0.32 (11 degrees of freedom); the largest difference was 0.55.

When the acidic solution or the synthetic municipal landfill leachate was used as the leaching medium (Figures 4b and 4c), the spread in the leachate pH data decreased. The leachate pH's were shifted towards pH 4.5, the value at which the leaching media were buffered. As expected, shifts were larger in the synthetic leachate extractions than in the acidic solution extractions since the former medium contained almost twice as much acid (157 meg/L versus 82 meg/L). Not only was the range decreased, but the data was skewed to the right (i.e., leachate pH's were lower at the higher liquid-to-solid ratio). This occurred because more acid was available to react with the waste at the higher ratio.

4.1.2 The Effect of Liquid-to-Solid Ratio on Contaminant Release. The effect of the liquid-to-solid ratio 'R' on test results was examined by comparing contaminant release at R = 20:1 to that at R = 4:1. This value, denoted as $REL_{20}/4$, is simply the ratio of the mass of a contaminant in the 20:1 leachate to that in the 4:1 leachate:

$$
REL_{20}/4 = \frac{REL_{20}}{REL_{4}} = \frac{M_{XL20/MS}}{M_{XL4/MS}} = \frac{M_{XL20}}{M_{XL4}}
$$
 (13)

REL $20/4$ ratios were calculated for the results of the 51 MBLP applications listed in Table 15. Ratios could only be calculated for those cases where species were detectable at both liquid-to-solid ratios. A total of 317 and 310 REL $20/4$ ratios could be calculated for the acidic solution and synthetic leachate extractions, respectively, whereas only 292 ratios could be calculated for the distilled water extractions, indicating the more aggressive leaching characteristics of the former media. Furthermore, for the distilled water extractions, 58 percent of the measured species were above detection limits at the

4:1 liquid-to-solid ratio but only 48 percent at the 20:1 ratio, because dilution at the higher ratio decreased contaminant concentrations below detection limits. Approximately the same percentage of species, however, were detected at the two ratios when the other leaching media were used (60 percent for the acidic solution and 64 percent for the synthetic municipal landfill leachate).

If it is assumed that 1) equilibrium between the leaching medium and the waste is achieved within the 24 hour elution period, 2) contaminant solubilities are dependent only on final leachate pH, and 3) the final leachate pH's (and thus contaminant solubilities) are the same in extractions with the same leaching medium but different liquid-to-solid ratios, then the theoretical range for $REL_{20/4}$ is 1 to 5. The lower limit of unity will occur when the same mass of contaminant is released at the two liquid-to-solid ratios. This suggests that there is a limited mass of contaminant available for leaching, and that adding 5 times more leaching medium simply dilutes the concentration by a factor of 5. The upper limit of 5 will be achieved when five times as much mass of contaminant is leached at the higher ratio. This will occur if there is a sufficient amount of contaminant available for leaching to achieve saturation at both ratios. Intermediate values between 1 and 5 will be obtained when there is sufficient contaminant available to fully saturate the leachate at the 4:1 liquid-to-solid ratio, but not enough to produce saturation at the 20:1 ratio.

The REL₂₀/4 ratios from the distilled water and the acidic solution extractions are shown in Figure 5 in the form of relative frequency histograms. The histogram for the synthetic municipal landfill leachate extractions is similar to the acidic solution histogram. It is interesting to note that there is a peak at $REL_{20}/4 = 1$ when distilled water was used as the leaching medium, indicating that concentration differences between the two liquid-to-solid ratios were simply due to dilution. The effect of dilution was also apparent when the acidic solution and synthetic leachate were used, but was not as pronounced. Eighty-two percent of the $REL_{20}/4$ ratios fell within the theoretical range of 1 to 5 when distilled water was used as the leaching medium (Figure 5a) but only 70 percent and 77 percent of the ratios were within this range when the acidic solution / and synthetic leachate, respectively, were used (Figure 5b). The poorer fit of the acidic solution and synthetic leachate release ratios between the theoretical limits is to be expected because leachate pH's at the two liquid-to-solid ratios were frequently different {Figure 4d; violating the third assumption in the development of the theoretical limits. It also appears that the synthetic leachate complexes some metals, which would increase metal solubilities (and thus their release) above those expected from pH considerations alone.

FIGURE 5 RELATIVE FREQUENCY HISTOGRAMS OF THE RATIO OF CONTAMINANT RELEASE AT R = 20:1 TO THAT AT R = 4:1

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4.1.3 Leachability of Selected Priority Pollutants under Various Test Conditions. Concentrations of Cd, Cr, Cu, Pb, Zn and cyanide in leachates are given in the tabular portion of Figures 6 to 11 for several of the residues subjected to the MBLP. The concentration of the contaminant in the waste sample, expressed on a dry weight basis, is also given (C_w) . Interpretation of leaching data in this form is very difficult because of the effects of the different leaching media and liquid-to-solid ratios on contaminant concentrations. Data evaluation can be simplified by considering contaminant release (Eq. 11) rather than contaminant concentration, and by plotting the logarithm of release versus final leachate pH. The resulting graphs are shown in Figures 6 to 11. The U.S. EPA Extraction Procedure (U.S. EPA, 1980) and the ASTM proposed leaching tests a and b (ASTM, 1979) were also performed on several of the residues. Releases from these tests are indicated in Figures 6 to 11 by the symbols EP, ASTMa and ASTMb.

For cadmium, lead, zinc and cyanide, a strong linear trend existed between the logarithm of contaminant release and final leachate pH over the pH range experienced in the leachates. There was also good correlation between the relative degree of release and the amount of acid available in the leaching medium to react with the waste. The latter values indicate that the order of increasing contaminant release to be expected, assuming release to be solely dependent on the amount of acid contacting the waste, would be DW4 and DW20 with the same amount of release, followed by AS4, SL4, AS20 and SL20 (Table 14). Examination of Figures 6 to 9 show this progression to be true in all cases except the reversed order of SL4 and AS20 in Figure 7a.

The results suggest that it would be possible, in some cases, to predict the release of these contaminants into a different leaching medium and/or at a different L/S ratio if the pH of the leachate could be evaluated from knowledge of the interaction that occurs between the waste and leaching medium (i.e., through a titration curve of the waste). This would be very helpful in trying to compare the results from different batch leaching tests. This supposition is supported by some of the release data obtained from application of the Extraction Procedure (EP) to Sample 14. Acid was added in the EP of Sample 14 at a rate of 2 meq/g of waste. From Table 14, it would be expected that release from the EP at this acid addition rate would fall between releases from the AS20 and SL20 extractions. This was indeed the case for cadmium and zinc (Figures 6 and 8). This linear trend, however, was not observed for all contaminants. No readily discernable pattern was apparent for the. release-pH curves for chromium (Figure 10) and copper

FIGURE 7 LEAD CONCENTRATIONS AND RELEASES FOR SAMPLES 13 AND 20

FIGURE 8 ZINC CONCENTRATIONS AND RELEASES FOR SAMPLES 14 AND 20

FIGURE 9 CYANIDE CONCENTRATIONS AND RELEASES FOR SAMPLE 14

FIGURE 10 CHROMIUM CONCENTRATIONS AND RELEASES FOR SAMPLES 2, 14 AND 15

 $\frac{2}{9}$

FIGURE 11 COPPER CONCENTRATIONS AND RELEASES FOR SAMPLES 1, 15 AND 20

 $\overline{5}$

(Figure 11). Comparison of chromium and copper releases from the ASTM tests and the MBLP provides further evidence that it may be possible to predict contaminant release from knowledge of a leaching medium's acid content. ASTM Method A is similar to the distilled water extraction at the 4:1 liquid-to-solid ratio, except that a different mixing procedure is used and the medium is contacted with the waste for 48 hours instead of 24 hours. Chromium release in Sample 2 (Figure lOa) and copper release in Sample 1 (Figure lla) were identical for both methods. Similarly, ASTM Method B is similar to the acidic solution extraction at the 4:1 liquid-to-solid ratio. The same leaching medium is used in both extractions, but the mixing procedure and contact time differ as in ASTM Method A. As shown in Figure lOa, chromium releases in Sample 2 were similar for ASTM Method Band MBLP Extraction AS4.

It appears from Figures 10 and 11 that the synthetic municipal landfill leachate may have complexed chromium and copper since releases were higher than those of the acidic solution, even though final leachate pH's were similar at equalliquid-to-solid ratios. The ability of some leaching media to complex chemical species complicates the task of predicting contaminant release under different leaching conditions. More work is required to determine if the effects of complexing on release can be predicted from a knowledge of the chemical composition of the leaching medium and the waste.

4.2 Summary

When distilled water was used as the leaching medium, the final pH of the leachate was controlled by the alkalinity or the acidity of the waste. Changing the liquidto-solid ratio had little effect on leachate pH. The difference in the concentrations observed at the two liquid-to-solid ratios were the result of dilution in a large number of cases.

When an acidic solution or synthetic leachate was used as the leaching medium, final leachate pH appeared to be a function of the interaction between the waste pH and the amount of acid available in the leaching medium to react with the waste. Increasing the liquid-to-solid ratio increased the amount of acid contacting the waste and shifted the leachate pH towards the pH of the leaching medium. This decreased or increased contaminant release depending on the direction of the pH shift.

Use of a synthetic leachate appeared to complex some contaminants and increase their release above that to be expected from pH - solubility considerations alone.

For some contaminants and wastes, there were strong linear relationships between the logarithm of release and final leachate pH, and a good correlation between the logarithm of release and the amount of acid in the leaching medium. This linear relationship, although not evident in all cases, suggests that it may be possible to predict release under different test conditions simply by knowing the final pH of the leachate.

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APPENDIX I THE MULTIPLE-BATCH LEACHING PROCEDURE ł,

 $\sim 10^{11}$ km $^{-1}$

APPENDIX I THE MUL TIPLE-BA TCH LEACHING PROCEDURE

The Multiple-Batch Leaching Procedure comprises a set of six batch extractions using three leaching media (distilled water, an acidic solution buffered at pH 4.5, and a synthetic municipal landfill leachate) at two liquid-to-solid ratios (4:1 and 20:1). The extractions are performed in square plastic bottles that are rotated slowly for 24 hours. The leachates are then decanted, filtered (0.45 µm) and analyzed for the parameters of interest. A flowchart of the steps involved in the procedure is presented in Figure I.1. A description of each of these steps follows.

1.1 Sampling (after ASTM, 1979)

- 1. Obtain a representative sample of the waste to be tested (e.g., ASTM standard technique for sampling wastes). Two kilograms of waste are necessary for each set of the six extractions included in the Leaching Procedure.
- 2. Samples should be kept in closed containers prior to testing. Biologically active samples should be stored at 4°C and their extraction should be started within eight hours if possible.

1.2 Particle Size Reduction

1. The waste is crushed, cut or ground so that it passes through a 9.5 mm (0.375 inch) sieve.

1.3 Structural Integrity Procedure (after U.S. EPA, 1980)

If the waste sample is monolythic, this procedure should be followed for each of the 6 extractions of Step 1.5.

- 1. A Structural Integrity Tester having a 3.18 cm (1.25 in) diameter hammer weighing 0.33 kg (0.73 lb) and having a free fall of 15.24 cm (6 in) shall be used. This device is available from various suppliers (Millipore, Nuclepore) or it may be fabricated to meet the specifications shown in Figure 1.2.
- 2. Fill the elastomeric sample holder with the material to be tested. If the sample of waste is a large monolithic block, cut a portion from the block having the dimensions of a 33 mm (1.3 in) diameter x 7.1 cm (2.8 in) long cylinder. For a chemically fixed waste, samples may be cast in the form of a 33 mm (1.3 in) diameter x 7.1 cm (2.8 in) cylinder and allowed to cure in a 100 percent humidity environment for 7 days prior to further testing.

FIGURE I.1 FLOW CHART OF THE MULTIPLE-BATCH LEACHING PROCEDURE

***ELASTOMERIC SAMPLE HOLDER FABRICATED OF MATERIAL FIRM ENOUGH TO SUPORT THE SAMPLE**

FIGURE 1.2 STRUCTURAL INTEGRITY TESTER (U.S. EPA, 1980)

- 3. Place the sample into the Structural Integrity Tester. Raise the hammer to its maximum height and release it. Repeat the hammering procedure fifteen times.
- 4. Remove the material from the sample holder, weigh it, and proceed to Extraction Step 1.5.

1.4 Subsampling (after ASTM, 1979)

This procedure is applicable to free-flowing particles. It is used for subsampling from the main sample (Steps 1.1 or 1.2) to obtain samples for the extractions (Step 1.5). The sample is quartered on an impermeable sheet of glazed paper, oil cloth, or other flexible material as follows:

- 1. Empty the sample container into the centre of the sheet.
- 2. Flatten out the sample gently with a suitable straight edge until it is spread uniformly to a depth approximately equal to its particle size.
- 3. Remix the sample by lifting a corner of the sheet and drawing it across, low down, to the opposite corner in a manner that the material is made to roll over and over and does not merely slide along. Continue operation with each corner proceeding in a clockwise direction. Repeat this operation ten times.
- 4. Lift all four corners of the sheet towards the centre. Holding all four corners together, raise the entire sheet into the air to form a pocket for the sample.
- 5. Repeat Step 2.
- 6. With a straight edge at least as long as the flattened mound of sample (such as a thin-edged yard stick), gently divide the sample into quarters. An effort should be made to avoid using pressure on the straight edge sufficient to cause damage to the particles.

1.5 Batch Extractions

The extractions are done in square closed bottles. Polyethylene bottles are used when the contaminants of interest are inorganic while glass bottles are used for organics.

The amount of waste and leaching medium added in each test bottle is computed on a weight basis (wet weight of the waste) according to the calculations given below. Instructions for preparing the acidic and synthetic leaching media are given in Table I.!.

1. The six extraction bottles containing the waste and the leaching medium are filled to approximately 90 percent of their capacity.

TABLE I.1 LEACHING MEDIA PREPARATION

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Calculate the amount of waste to be added in each bottle using the following approximate formula, neglecting specific gravity considerations:

$$
W_W = \frac{0.9 V}{R + 1}
$$

where: $V = volume of the bottle (mL)$

minimum: 500 mL if $R = 4:1$ 1000 mL if $R = 20:1$ $R =$ liquid to solid ratio W_W = weight of waste (g)

- 2. Add the calculated amount of waste to each of the six bottles.
- 3. Add each leaching medium to the test bottles according to the formula:

 $W_L = R W_W$

where: W_{L} = weight of leaching medium (g)

4. Agitate the bottles using an apparatus similar to the one illustrated in Figure 1.3 for $24 h + 0.5 h$ at room temperature. Set the speed of rotation between 2 and 3 rpm.

1.6 Leachate Analysis

- 1. After the period of agitation, separate the bulk of the aqueous phase from the solid phase by decantation, centrifugation or filtration through filter paper as appropriate. Vacuum filter the aqueous phase through a 0.45μ m membrane filter. For oily wastes, use a $8 \mu m$ filter (ASTM, 1979).
- 2. Measure the pH and conductivity of the filtrate.
- 3. Transfer the filtrate to sample bottles and preserve in a manner consistent with the chemical analyses to be performed.

1.7 Waste Analysis

- 1. Determine the total solids content of the waste as follows:
	- dry dishes at $104 \div 2^{\circ}$ C
	- cool a dish in a dessicator and weigh it (W_0)
	- put approximately 50 g of waste into the dish and weigh (W_1)
	- dry at $104 + 2$ °C for 24 h
	- cool to room temperature in a dessicator and reweigh (\mathbb{W}_2)
	- calculate the total solids content (S_T) from:

$$
S_T = \frac{w_2 - w_0}{w_1 - w_0}
$$

- 2. Determine the fixed solids content as follows:
	- muffle sample and dish at 550°C for 1 hour
	- air cool briefly, then place in a dessicator to cool
	- weigh (W_3)
	- calculate the fixed solids content (S_F) from:

$$
SF = \frac{W_3 - W_0}{W_2 - W_0}
$$

3. When possible, analyse the waste for the contaminants of interest in the leachate.

FIGURE 1.3 ILLUSTRATION OF TUMBLER

1.8 Report

- 1. The report should include a description of the waste:
	- source, \overline{a}
	- observed physical characteristics (e.g., colour, odour, particle size, homogeneity, phases),
	- total solids and fixed solids contents, and
	- chemical composition on a dry weight basis.
- 2. The results of the leachate analyses can be expressed using the three following formats (the chemical composition of the waste must be available to compute leaching efficiency):

a) Concentration - the ratio of the mass of chemical species X in a leachate (MXL) to the volume of leachate (V_L) :

$$
C_{\mathbf{XL}} = \frac{M_{\mathbf{XL}}}{v_{\mathbf{L}}}
$$

b) Release - the ratio of the mass of chemical species X in a leachate (M_{XL}) to the mass of the leached sample (MS); it is also equal to the product of the liquid-to-solid ratio (R) and the concentration of chemical species X in the leachate (CXL):

$$
REL_X = \frac{M_{XL}}{M_S} = \frac{M_{XL}}{M_S} \times \frac{V_L}{V_L} = \frac{V_L}{M_S} \times \frac{M_{XL}}{V_L} = R.C_{XL}
$$

c) Efficiency - the ratio of the mass of chemical species X in a leachate (M_{XL}) to the mass of that species in the leached sample (MxS) expressed as a percentage:

$$
EFFX = \frac{MXL}{MXS} \times 100\%
$$

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APPENDIX II LEACHING TEST RESULTS

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SAMPLE NO.: 1 WASTE TYPE: Raw Metal Finishing Sludge

DATE TESTED: June 1981

COMMENTS: Green, si It-sized particles

SOLID PHASE TOTAL SOLIDS: 9.7% VOLATILE SOLIDS: 9.8%

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SAMPLE NO.: 1 WASTE TYPE: Raw Metal Finishing Sludge

DATE TESTED: June 1981

COMMENTS: Continued from previous page. Acid addition in EPA extraction procedure = 68.8 mL = 0.737 meq H⁺/g of waste.

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SAMPLE NO.: 2 WASTE TYPE: Dewatered Metal Finishing Sludge

DATE TESTED: May 1981

C04MENTS: Green, particle size = 3 to 50 mm

SOLID PHASE TOTAL SOLIDS: 20.5% VOLATILE SOLIDS: 15.0%

MULTiPlE-BATCH LEACHING PROCEDURE

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SAMPLE NO.: 2 WASTE TYPE: Dewatered Metal Finishing Sludge

DATE TESTED: May 1981

COMMENTS: Continued from previous page. Acid addition in EPA extraction procedure = 242 mL = 1.03 meq H⁺/g waste.

EPA EXTRACTION PROCEDURE AND ASTM LEACHING TESTS

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SAMPLE NO.: 3 WASTE TYPE: Foundry Sand

DATE TESTED: June 1981

C<J4MENTS: Black

SOLID PHASE TOTAL SOLIDS: 92.0% **VOLATILE SOLIDS: 1.5%**

MULTIPLE-BATCH LEACHING PROCEDURE

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SAMPLE NO.: 3 WASTE TYPE: Foundry Sand

DATE TESTED: June 1981

COMMENTS: Continued from previous page. Acid addition in EPA extraction procedure = 11 mL = 0.054 meq H^+/g waste.

EPA EXTRACTION PROCEDURE AND ASTM LEACHING TESTS

SAMPLE NO.: 4 WASTE TYPE: Solidified Pickling Liquor and Baghouse Dust

DATE TESTED: June 1981

 $\texttt{COMMENTS:} \qquad$ Reddish brown, particle size = 30 to 100 mm, solidified by addition of silicates and lime

SOLID PHASE TOTAL SOLIDS: 82.8% VOLATILE SOLIDS: 7.6%

MULTIPLE-BATCH LEACHING PROCEDURE

SAMPLE NO.: 4 WASTE TYPE: Solidified Pickling Liquor and Baghouse Dust

DATE TESTED: June 1981

COMMENTS: Continued from previous page. Acid addition in EPA extraction procedure = 400 mL = 2.0 meq H⁺/g waste.

EPA EXTRACTION PROCEDURE AND ASTM LEACHING TESTS

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SAMPLE NO.: 5 WASTE TYPE: Fly Ash from a Coal Fired Generating Station

DATE TESTED: June 1981

COMMENTS: Grey, particle size: 75 to 100 µm

SOLID PHASE TOTAL SOLIDS: 99.5% VOLATILE SOLIDS: 0.2%

MULTIPLE-BATCH LEACHING PROCEDURE

DATE TESTED: June 1981

C(J4MENTS: Continued from previous page

SOLID PHASE TOTAL SOLIDS: 99.5% VOLATILE SOLIDS: 0.2%

MULTIPLE-BATCH LEACHING PROCEDURE

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SAMPLE NO_.: 5 WASTE TYPE: Fly Ash

EPA EXTRACTION PROCEDURE AND ASTM LEACHING TESTS

DATE TESTED: June 1981

COMMENTS: Continued from previous page. Acid addition in EPA extraction procedure = 77 mL = 0.39 meq H^+/g waste.

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SAMPLE NO.: 6 WASTE TYPE: Bottom Ash from a Coal Fired Generating Station

DATE TESTED: September 1981

COMMENTS: Black, particle size = 0.075 to 10 mm

SOLID PHASE TOTAL SOLIDS: 99.1% VOLATILE SOLIDS: 2.2%

MULTIPLE-BATCH LEACHING PROCEDURE

SAMPLE NO.: 6 WASTE TYPE: Bottom Ash

DATE TESTED: September 1981

C<M4ENTS: continued from previous page

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 $\sim 10^{11}$

SOLID PHASE TOTAL SOLIDS: VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING FROCEDURE

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SAMPLE NO_.: 6 WASTE TYPE: Bottom Ash

DATE TESTED: September 1981

COMMENTS: Continued from previous page. Acid addition in EPA extraction procedure = 0 mL = 0 meq H⁺/g waste.

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SAMPLE NO.: 7 WASTE TYPE: Bottom Ash from a Coal Fired Generating Station

DATE TESTED: September 1981

COMMENTS: Brown-grey.

SOLID PHASE TOTAL SOLIDS: 60.1% VOLATILE SOLIDS: 8.0%

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MULTIPLE-BATCH LEACHING mOCEDlRE

SAMPLE NO.: 7 WASTE TYPE: Bottom Ash

DATE TESTED: September 1981

C<M4ENTS: Continued from prev lous page.

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SAMPLE NO.: 7 WASTE TYPE: Bottom Ash

DATE TESTED: September 1981

COMMENTS: Continued from previous page.

SOLID PHASE TOTAL SOLIDS: VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING PROCEDURE

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SAMPLE NO.: 8 WASTE TYPE: Fly Ash from a Coal Fired Generating Station

DATE TESTED: September 1981

C04MENTS: Light grey

SOLID PHASE TOTAL SOLIDS: 100% VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING PROCEDURE

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SAMPLE NO.: 8 WASTE TYPE: Fly Ash

DATE TESTED: September 1981

COMMENTS: Continued from previous pages.

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DATE TESTED: September 1981

C04MENTS: Continued from previous page.

SAMPLE NO.: 9 WASTE TYPE: Fly Ash from a Coal Fired Generating Station

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DATE TESTED: September 1981

C04MENTS: Light grey-brown.

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SOLID PHASE TOTAL SOLIDS: 99.6% VOLATILE SOLIDS: 3.2%

MULTIPLE-BATCH LEACHING PROCEDURE

SAMPLE NO.: 9 WASTE TYPE: Fly Ash

DATE TESTED: September 1981

COMMENTS: Continued from previous page.

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SAMPLE NO.: 10 WASTE TYPE: Bottom Ash from a Coal Fired Generating Station

MTE TESTED: September 1981

COMMENTS: Black.

SOLID PHASE TOTAL SOLIDS: 99.6% VOLATILE SOLIDS: 3.5%

MULTIPLE-BATCH LEACHING FROCEDURE

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SAMPLE NO.: 10 WASTE TYPE: Bottom Ash

DATE TESTED: September 1981

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COMMENTS: Continued from previous page.

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SAMPLE NO.: 11 WASTE TYPE: Gold Mine Tailings

DATE TESTED: August 1981

COMMENTS: Blanket cyanide treatment. Mercury used to form amalgam. Arseno-pyrite ore.

SOLID PHASE TOTAL SOLIDS: 86.1% VOLATILE SOLIDS: 0.7%

MULTIPLE-BATCH LEACHING FROCEDURE

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SAMPLE NO.: 12 WASTE TYPE: Anaerobically Digested WWTP Sludge

DATE TESTED: July 1981

C<M4ENTS: From an activated sl udge treatment plant with anaerobic digestion. The fol lowing organic compounds were detected In the sludge: phenanthrene at 17 µg/g, flourene at 3 µg/g, di-n-butylphthalate at 116 µg/g, and bis-(2-ethylhexyl) phthalate at 408 pg/g. None of these were detected in the leachates, except for di-n-butylphthalate at 0.01 mg/L In the OW 20:1 leachate

SOLID PHASE TOTAl SOLIDS: 3.2% **VOLATILE SOLIDS:** 46.0%

MULTIPLE-BATCH LEACHING PROCEDURE

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SAMPLE NO.: 12 WASTE TYPE: Anaerobically Digested WWTP Sludge

DATE TESTED: July 1981

C**OMMENTS:** Continued from previous page. Acid addition in EPA extraction procedure = 61 mL = 1.10 meq H⁺/g waste.

EPA EXTRACTION PROCEDURE AND ASTM LEACHING TESTS

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SAMPLE NO.: 13 WASTE TYPE: Mine Tailings

DATE TESTED: July 1981

C04MENTS: Grey

SOLID PHASE TOTAL SOLIDS: 16.2% VOLATILE SOLIDS: 1.3%

MULTIPLE-BATCH LEACHING FROCEDURE

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SAMPLE NO.: 13 WASTE TYPE: Mine Tailings

DATE TESTED: July 1981

COMMENTS: Continued from previous page. Acid addition in EPA extraction procedure = 31.0 mL = 0.26 meq H⁺/g of waste.

EPA EXTRACTION PROCEDURE AND ASTM LEACHING TESTS

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SAMPLE NO.: 14 WASTE TYPE: Matal Finishing Sludge

DATE TESTED: September 1981

C04MENTS: Gold-brown, cake from pressure f I Iter.

SOLID PHASE TOTAL SOLIDS: 29.6% VOLATILE SOLIDS: 35.2%

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MULTIPLE-BATCH LEACHING PROCEDURE

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SAMPLE NO.: 14 WASTE TYPE: Metal Finishing Sludge

DATE TESTED: September 1981

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COMMENTS: Continued from previous page. Acid addition in EPA extraction procedure = 400 mL = 2.00 meq H⁺/g waste.

EPA EXTRACTION PROCEDURE AND ASTM LEACHING TESTS

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SAMPLE NO.: 15 WASTE TYPE: Caustic Metal Cleaning Waste

DATE TESTED: July 1981

COMMENTS: Grey. Neutralized spent caustic metal cleaner, solidified with Portland cement.

SE TOTAL SOLIDS: 87.7% VOLATILE SOLIDS: 6.7%

WTC LEACHING PROCEDURE

SAMPLE NO.: 15 WASTE TYPE: Caustic Metal Cleaning Waste

DATE TESTED: July 1981

C04MENTS: Continued from previous page. Acid addition In EPA extraction procedure = 400 mL = 2.00 meq H+/g of waste.

EPA EXTRACTION PROCEDURE AND ASTM LEACHING TESTS

MEASURED TEST CHARACTERISTIC | CONDITION Concentration EPA (mg/U ASTM OW ASTM AS Release FPA $(\mu g/g)$ ASTM DW ASTM AS Efficiency | EPA $(\%)$ ASTM DW ASTM AS pH (Units) Conductivity (mS/cm)

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SAMPLE NO.: 16 WASTE TYPE: Halifax Harbour Dredge Spoils

DATE TESTED: July 1981

C04MENTS: Black. Dredge spoils disposed in bermed site on DND property In Spring, 1980.

SAf4>LE NO.: 18 **WASTE TYPE:** Leaded Refinery Organic Fuel Tank Sludge

DATE TESTED: August 1981

C04MENTS: Black, gasoline odour.

SOLID PHASE TOTAL SOLIDS: 76.7% VOLATILE SOLIDS: 12.3%

MULTIPLE-BATCH LEACHING FROCEDURE

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SAMPLE NO.: 19 WASTE TYPE: Fresh Mining and Smelting Tailings

DATE TESTED: February 1982

C04MENTS: From copper/lead/zinc mine/mi II/smelter.

SOLID PHASE TOTAL SOLIDS: 74.5% VOLATILE SOLIDS: 4.4%

MULTIPLE-BATCH LEACHING PROCEDURE

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SAMPLE NO.: 20 WASTE TYPE: Mining and Smelting Tailings

DATE TESTED: August 1981

COMMENTS: See Sample 19. Tailings approximately two years old

SOLID PHASE **TOTAL SOLIDS: 89.9%** VOLATILE SOLIDS: 3.8%

MULTIPLE-BATCH LEACHING PROCEDURE

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DATE TESTED: March 1982

COMMENTS: See Sample 19. Tailings Approximately 6 to 10 years old

SOLID PHASE TOTAL SOLIDS: 92.5% VOLATILE SOLIDS: 10.4%

MULTIPLE-8ATCH LEACHING PROCEDURE

SAMPLE NO.: 22 WASTE TYPE: Mining and Smelting Tailings

DATE TESTED: August 1981

C04MENTS: See Samp Ie 19. Tall ings approximately 15 years old.

SOLID PHASE TOTAL SOLIDS: 77.8% VOLATILE SOLIDS: 3.6%

MULTIPLE-BATCH LEACHING PROCEDURE

SAMPLE NO.: 23 WASTE TYPE: Zinc Sulphide Residue

DATE TESTED: August 1981

COMMENTS: Excess residue from zinc plant (acid leach solids)

SOLID PHASE TOTAL SOLIDS: 75.2% **VOLATILE SOLIDS:** 0.8%

MULTIPLE-BATCH LEACHING PROCEDURE

SAMPLE NO.: 24 WASTE TYPE: Zinc Hydroxide Sludge

DATE TESTED: August 1981

COMMENTS: Originated from floor washings, overflow from zinc oxide pond, etc., which is mixed with slurried lime to precipitate zinc for recovery.

SOLID PHASE **TOTAL SOLIDS: 14.3%** VOLATILE SOLIDS: 8.7%

MULTIPLE-BATCH LEACHING PROCEDURE

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SAMPLE NO.: 25 WASTE TYPE: High Arsenic Residue from Flotation/Cyanidation Process

DATE TESTED: September 1981

C04MENTS: High arsenic concentrations Interfered with gold recovery. Therefore residue was stockpiled until technology could be developed to extract the gold.

SOLID PHASE **TOTAL SOLIDS: 87.8% VOLATILE SOLIDS:** 9.8%

MULTIPLE-BATCH LEACHING PROCEDURE

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SAMPLE NO.: 29 WASTE TYPE: Phosphate Process Slag

DATE TESTED: September 1981

COMMENTS: Grey, particle size = 5 to 10 cm

SOLID PHASE TOTAL SOliDS: 99.9% VOLATILE SOlIDS: 0.1%

MULTIPLE-BATCH LEACHING PROCEDURE

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SAMPLE NO.: 32 WASTE TYPE: Equilibrium Fluid Catalyst from Catalytic Cracker Unit

DATE TESTED: September 1981

COMMENTS: Grey. From oil refinery.

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SOLID PHASE TOTAL SOLIDS: 94.4% VOLATILE SOLIDS: 1.4%

MULTIPLE-BATCH LEACHING PROCEDURE

SAMPLE NO.: 33 WASTE TYPE: Refinery Sludge

DATE TESTED: October 1981

C04MENTS: Dark-brown.

SOLID PHASE **TOTAL SOLIDS: 92.9% VOLATILE SOLIDS:** 15.6%

MULTIPLE-BATCH LEACHING FROCEDURE

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SAMPLE NO.: 40 WASTE TYPE: Filter Cake Solids from a Chemical Plant

DATE TESTED: December 1981

C04MENTS: Dark-grey.

SOLID PHASE **TOTAL SOLIDS: 76.0% VOLATILE SOLIDS:** 32.0%

MULTIPLE-BATCH LEACHING FROCEDURE

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SAMPLE NO.: 41 WASTE TYPE: Neutral Filter Cake Solids from a Chemical Plant

DATE TESTED: December 1981

C04MENTS: Dark-grey.

SOLID PHASE TOTAL SOLIDS: 76.0% VOLATILE SOLIDS: 30.0%

MULTIPLE-BATCH LEACHING mOCEDlRE

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SAWLE NO.: 41 **WASTE TYPE:** Neutral Filter Cake Solids

DATE TESTED: December 1981

COMMENTS: Continued from previous page. Acid addition in EPA extraction procedure = 0 mL = 0.0 meq H⁺/g waste.

EPA EXTRACTION PROCEDURE AND ASTM LEACHING TESTS

SAMPLE NO.: 42 WASTE TYPE: Aluminum Production Red Mud

DATE TESTED: February 1982

COMMENTS: Rust red.

SOLID PHASE TOTAL SOLIDS: 56.8% VOLATILE SOLlOS: 8.4%

MULTIPLE-BATCH LEACHING PROCEDURE

DATE TESTED: February 1982

C<J4MENTS: Rust-red.

SOLID PHASE TOTAL SOLIDS: 59.1% **VOLATILE SOLIDS:** 6.8%

MULTIPLE-BATCH LEACHING PROCEDURE

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SAMPLE NO.: 45 WASTE TYPE: Decontaminated Oliy Refinery Sand

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DATE TESTED: February 1982

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C04MENTS: Light brown.

SOLID PHASE TOTAL SOLIDS: 92.5% VOLATILE SOLIDS: 0.4%

MULTIPLE-BATCH LEACHING PROCEDURES

SAMPLE NO.: 47 WASTE TYPE: Bed Material from an FBC Coal Fired Generating Station

DATE TESTED: January 1982

COMMENTS: White and brown.

SOLID PHASE TOTAL SOLIDS: 99.9% VOLATILE SOLIDS:

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MULTIPLE-BATCH LEACHING PROCEDURE

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SAMPLE NO.: 47 WASTE TYPE: FBC Bed Material

DATE TESTED: January 1982

COMMENTS: Continued from previous page.

 \overline{E} TOTAL SOLIDS: VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING PROCEDURE

SAMPLE NO.: 47 WASTE TYPE: FBC Bed Material

DATE TESTED: January 1982

COMMENTS: Continued from previous page.

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SAMPLE NO.: 47 WASTE TYPE: FBC Bed Material

DATE TESTED: January 1982

COMMENTS: Continued from previous page.

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SOLID PHASE **TOTAL SOLIDS:** VOLATILE SOLIDS:

MUTIPLE-BATCH LEACHING PROCEDURE

SAMPLE NO_s: 48 WASTE TYPE: Baghouse Material from FBC Coal Fired Generating Station

DATE TESTED: January 1982

C04MENTS: Brown.

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SOLID PHASE TOTAL SOLIDS: 95.7% VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING PROCEDURE

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SAMPLE NO.: 48 WASTE TYPE: FBC Baghouse Material

DATE TESTED: January 1982

CCM4ENTS: Continued from previous page.

SOLID PHASE TOTAL SOLIDS: VOLATILE SOLIDS:

MULTIPE-BATCH LEACHING mOCEDlRE

SAMPLE NO.: 48 WASTE TYPE: FBC Baghouse Material

DATE TESTED: January 1982

C<M4ENTS: Continued from previous page.

SAMPLE NO.: 49 WASTE TYPE: Bed Material from FBC Coal Fired Power Generating Station

DATE TESTED: January 1982

COMMENTS: White and brown.

SOLID PHASE TOTAL SOLIDS: 99.1% VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING FROCEDURE

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SAMPLE NO.: 49 WASTE TYPE: FBC Bed Material

DATE TESTED: January 1982

C04MENTS: Continued from previous page.

SOLID PHASE **TOTAL SOLIDS:** VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING PROCEDURE

SAMPLE NO.: 49 WASTE TYPE: FBC Bed Material

DATE TESTED: January 1982

C04MENTS: Continued from previous page.

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DATE TESTED: January 1982

C04MENTS: Continued from previous page.

SOLID PHASE TOTAL SOLIDS: VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING PROCEDURE

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SAMPLE NO.: 50 WASTE TYPE: Baghouse Material from FBC Coal Fired Generating Station

DATE TESTED: January 1982

COMMENTS:

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SL 20:1 | | | 1.08 | 1.37 | 59.3 | 1.28 | 13.3 | 39.8

SAMPLE NO.: 50 WASTE TYPE: FBC Baghouse Material

DATE TESTED: January 1982

COMMENTS: Continued from previous page.

SAMPLE NO_.: 50 WASTE TYPE: FBC Baghouse Material

DATE TESTED: January 1982

COMMENTS: Continued from previous page.

SAMPLE NO.: 50 WASTE TYPE: FBC Baghouse Material

DATE TESTED: January 1982

COMMENTS: Continued from previous page.

SOLID PHASE TOTAL SOLIDS: VOLATILE SOLIDS: \sim

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MULTIPLE-BATCH LEACHING PROCEDURE

SAMPLE NO.: 51 WASTE TYPE: 40:60 Mix of Baghouse: Bed Material from FBC Coal-Fired Generating Station

DATE TESTED: January 1982

COMMENTS:

SOLID PHASE TOTAL SOLIDS: 97.6% VOLATILE SOLIDS:

MULTIPLE-BATOH LEACHING PROCEDURE

SAMPLE NO.: 51 WASTE TYPE: FBC Baghouse: Bed Mixture

DATE TESTED: January 1982

COMMENTS: Continued from previous page

SAMPLE NO.: 51 WASTE TYPE: FBC Baghouse: Bed Mixture

DATE TESTED: January 1982

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COMMENTS: Continued from previous page

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DATE TESTED: January 1982

COMMENTS: Continued from previous page

DATE TESTED: March 1982

COMMENTS: Combined primary and waste activated sludge from leather tannery, dewatered on centrifuge. Grey.

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SAMPLE NO.: 53 WASTE TYPE: Copper Wool Scrap

SL 4:1 | 0.68 | 25.2 SL 20:1 \vert 0.74 \vert 37.9

DATE TESTED: February 1982

COMMENTS: Green. From a phosphoric acid plant. Cu is used to remove As in the production of phosphoric acid

SOLID PHASE TOTAL SOLIDS: 66.5% VOLATILE SOLIDS: 12.5% PARAMETER | As | Cu Concentration (ppm) 106 000. 206 000. MULTIPLE-BATCH LEACHING PROCEDURE MEASURED **I** TEST CHARACTERISTIC | CONDITION | As | Cu Concentration \vert DW 4:1 | 100. | 5557. (mg/l) 0tI 20: 1 20. 1111. AS $4:1$ 80. 6761. AS 20:1 | 18. | 1852. SL $4:1$ | 120. | 8613. SL 20:1 \vert 26. \vert 2593. Release $\begin{array}{|c|c|c|c|c|c|c|c|c|} \hline \text{Rel} & \text{DM} & \text{4:1} & \text{400.} & \text{22 228.} \hline \end{array}$ $(\mu g/g)$ 0W 20:1 400. 22 220. AS $4:1$ 320. 27 044. AS $20:1$ 360. 37 040. SL $4:1$ | 480. | 34 452. SL 20:1 \parallel 520. 51 860. Efficiency \vert DW 4:1 \vert 0.57 16.2 (\$) \vert DW 20:1 \vert 0.57 \vert 16.2 AS 4:1 | 0.45 | 19.8 AS 20:1 $\begin{vmatrix} 0.51 & 27.1 \end{vmatrix}$ pH (Units) 3.30 3.70 3.50 3.95 3.40 4.15 Conductivity (mS/cm)

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16.9 4.4 17.2 6.4 18.5 9.1

SAM='LE NO.: 53 **WASTE TYPE:** Copper Wool Scrap

DATE TESTED: February 1982

COMMENTS: Continued from previous page. Acid addition in EPA extraction procedure = 0.0 mL = 0.0 meq H+/g waste

SAMPLE NO.: 54 WASTE TYPE: Sodium Chlorate Sludge (unwashed)

DATE TESTED: February 1982

OOMMENTS: Black. Sludge from the production of sodium chlorate.

SOLID PHASE TOTAL SOLIDS: 58.8% VOLATILE SOLIDS: 25.4%

MULTIPLE-BATCH LEACHING FROCEDURE

SNf'LE NO.: 54 **WASTE TYPE:** Sodium Chlorate Sludge (unwashed)

DATE TESTED: February 1982

COMMENTS: Continued from previous page. Acid addition in EPA extraction procedure = 356 mL = 2.00 meq H+/g waste

SAMPLE NO_.: 55 WASTE TYPE: Sodium Chlorate Sludge (washed)

DATE TESTED: February 1982

COMMENTS: Black. Lagoon sl udge from production of sodium chlorate.

SOLID PHASE TOTAL SOLIDS: 36.5% **VOLATILE SOLIDS:** 10.4%

MULTIPLE-BATCH LEACHING PROCEDURE

SAMPLE NO.: 55 **WASTE TYPE:** Sodium Chlorate Sludge (washed)

DATE TESTED: February 1982

COMMENTS: Continued from previous page. Acid addition in EPA Extraction procedure = 426 mL = 2.00 meq H+/g waste.

SAMPLE NO.: 56 **WASTE TYPE:** Cyanide Leach Tai lings

DATE TESTED: March 1982

COMMENTS: Tail ings produced from cyanide leach of flotation concentrate. Note: SW = local river water, with As 1.0 mg/L, Cr 0.01 mg/L , Cu = 0.01 mg/L , Fe = 0.73 mg/L , Pb 0.01 mg/L , Zn = 0.02 mg/L and TCN = 0.02 mg/L . Concentrations shown for SW are minus the concentrations found in the site water leaching medium.

SOLID PHASE **TOTAL SOLIDS: VOLATILE SOLIDS:**

MULTIPLE-BATCH LEACHING PROCEomE

SAMPLE NO_®: 57 WASTE TYPE: Bulk Sulphide Flotation Tank Tailings

DATE TESTED: March 1982

C04MENTS: Brown. Note: SW = local river water, with As 1.0 mg/L, Cr 0.01 mg/L, Cu = 0.01 mg/L, Fe = 0.73 mg/L, Pb 0.01 mg/L, Zn=0.02 mg/L and TCN=0.02 mg/L. The concentrations shown for SW leachates are minus the concentrations In the site water leaching medium.

SOLID PHASE TOTAL SOLIDS: VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING PROCEDURE

SAMPLE NO.: 58 WASTE TYPE: Dewatered Sludge from an FGD Coal-Fired Generating Station

DATE TESTED: March 1982

C04MENTS: Grey

SOLID PHASE TOTAL SOLIDS: 68.5% VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING PROCEDURE

SAMPLE NO.: 58 WASTE TYPE: FGD Sludge

DATE TESTED: March 1982

COMMENTS: Continued from prev I ous page

SNoPLE NO.: 58 **WASTE TYPE:** FGD Sludge

DATE TESTED: March 1982

COMMENTS: Continued from previous page.

SOLID PHASE TOTAL SOLIDS:

PARAMETER | Co \angle Concentration (ppm) <5 Th \leq 1 Zr 58.3

VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING FROCEDURE

SNFLE NO.: 61 WASTE TYPE: Bottom Ash from an FGD Coal-Fired Generating Station

DATE TESTED: March 1982

COMMENTS: Rust brown

SOLID PHASE TOTAL SOLIDS: 88.8% VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING FROCEDURE

SAMPLE NO.: 61 WASTE TYPE: FGD Bottom Ash

DATE TESTED: March 1982

COMMENTS: Continued from previous page.

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SNflLE NO.: 61 **WASTE TYPE:** FGD Bottom Ash

DATE TESTED: March 1982

COMMENTS: Continued from previous page.

SOLID PHASE **TOTAL SOLIDS: VOLATILE SOLIDS:**

MULTIPLE-BATCH LEACHING PROCEDURE

SAMPLE NO.: 62 WASTE TYPE: Fly Ash Material from an FGD Coal-Fired Generating Station

DATE TESTED: March 1982

COMMENTS: Brown, particle size <150 μ m

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SOLID PHASE TOTAL SOLIDS: 99.8% VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING FROCEDURE

SAMPLE NO.: 62 WASTE TYPE: FGD Fly Ash

DATE TESTED: March 1982

COMMENTS: Continued from previous page.

SAMPLE NO.: 62 WASTE TYPE: FGD Fly Ash

DATE TESTED: March 1982

COMMENTS: Continued from prev lous page.

SOLID PHASE TOTAL SOLIDS: VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING PROCEDURE

SAMPLE NO.: 63 WASTE TYPE: Sludge Material from an FGD Coal-Fired Generating Station

DATE TESTED: April 1982

COMMENTS: Biege

SOLID PHASE TOTAL SOLIDS: 72.3% VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING PROCEDURE

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SAMPLE NO.: 63 WASTE TYPE: FGD Sludge

DATE TESTED: April 1982

COMMENTS: Continued from previous page.

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DATE TESTED: April 1982

COMMENTS: Continued from prey I ous page.

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SAMPLE NO.: 64 WASTE TYPE: Cyanide and Arsenic Tailings from Gold Mine Effluent Treatment

DATE TESTED: September 1982

COMMENTS: Brown. Note: SW = holding pond water, $pH = 7.8$, contains 0.16 mg/L TCN

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SOLID PHASE TOTAL SOLIDS: 13.8% VOLATILE SOLIDS: 9.1%

MULTIPlE-BATCH LEACHING mOCEDLRE

SNPLE NO.: 67 WASTE TYPE: Saghouse Sludge from an FGD Coal-Fired Generating Station

DATE TESTED: February 1983

COMMENTS: Grey

SOLID PHASE TOTAL SOLIDS: 99.8% VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING PROCEDURE

SAMPLE NO.: 67 WASTE TYPE: FGD Baghouse Sludge

DATE TESTED: February 1983

COMMENTS: Continued from previous page

SOLID PHASE TOTAL SOLIDS: VOLATILE SOLIDS:

MULTIPLE-8ATCH LEACHING mOCEDlRE

SAMPLE NO.: 68 WASTE TYPE: Cured Sludge from an FGD Coal-Fired Generating Station

SOLID PHASE TOTAL SOLIDS: 69% VOLATILE SOLIDS:

DATE TESTED: February 1983

COMMENTS: Grey

SAMPLE NO.: 68 WASTE TYPE: Cured FGD sludge

DATE TESTED: February 1983

COMMENTS: Continued from previous page

SOLID PHASE TOTAL SOLIDS: VOLATILE SOLIDS:

MULTIPLE-BATCH LEACHING FROCEDURE

SAMPLE NO.: 70 WASTE TYPE: Impregnated Charcoal

DATE TESTED: August 1983

COMMENTS: Spent charcoal from gas mask cartridges, R.M.S. Kingston

SOLID PHASE TOTAL SOLIDS: 92.3% VOLATILE SOLIDS:

 $*$ Aqua-regia leach and nitric acid $*$ H₂O₂ digestion

MULTIPLE-BATCH LEACHING FROCEDURE

