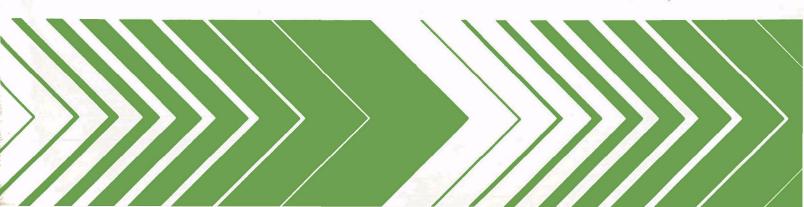
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Research and Development

# Elutriate Test Evaluation of Chemically Stabilized Waste Materials



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# ELUTRIATE TEST EVALUATION OF CHEMICALLY STABILIZED WASTE MATERIALS

Ъу

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#### FOREWORD

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

Research and development is that necessary first step in problem solution and it involves defining the problem, reasuring its impact, and searching for solutions. The Municipal Environmental Research Laboratory develops new and improved technology and systems for the prevention, treatment, and management of wastewater and solid and hazardous waste pollutant discharges from municipal and community sources, for the preservation and treatment of public drinking water supplies, and to minimize the adverse economic, social, health, and aesthetic effects of pollution. This publication is one of the products of that research; a most vital communications link between the researcher and the user community.

This report presents results from the evaluation of the use of a distilled water elutriate or shake test for comparing the effectiveness of a variety of solidification/stabilization (fixation) systems on containing undesirable materials in semisolid wastes (sludges). It provides basic data that will add to our knowledge regarding land disposal of treated wastes and will assist in the development of adequate waste testing techniques.

Francis T. Mayo, Director Municipal Environmental Research Laboratory

#### ABSTRACT

A distilled water shake test, the elutriate test, was developed and tested to provide a fast, simple, procedure for predicting the escape of pollutants from treated and untreated sludges. The preliminary test consisted of subjecting various treated and untreated flue gas desulfurization (FGD) and industrial waste sludges to the elutriate test procedure and measuring the levels of a wide variety of constituents in the elutriate and comparing these with analyses of digested sludges. The resulting data are presented as percent attenuation and a comparison is made between treated and untreated wastes.

The short-term elutriate test results were compared to results of a long-term leaching test using the same treated and untreated sludges. The results suggest that the elutriate test may be useful in predicting the pollutant potential of various treated or untreated wastes. Further research and modifications are suggested to improve the predictive value of the test.

This report is submitted in partial fulfillment of Interagency Agreement No. EPA-IAG-D4-0569 between the U.S. Environmental Protection Agency, Municipal Environmental Research Laboratory, Solid and Hazardous Waste Research Division (EPA, MERL, SHWRD) and the U.S. Army Engineer Waterways Experiment Station (WES). Work for this report was conducted during the period of August 1976 through August 1977.

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# INTRODUCTION

#### BACKGROUND

Recent concern for the environment has brought about an increase in air and water pollution control legislation. This legislation calls for the strict control of pollutant emissions to the environment. The resulting imposed regulations have brought about an increase in the number and efficiency of pollutant removal systems. The typical treatment system discharges pollutants in a concentrated form such as a sludge. This sludge must be disposed so as to result in a negligible impact on the environment. Many sludges must be considered hazardous to the environment because of high concentrations of potentially dangerous pollutants and poor handling of characteristics of the sludges.

The final receptor for sludges is usually the land. In many disposal systems, a physical barrier such as a clay or man-made liner is placed between the sludge and the surrounding environment to prevent the migration of potentially hazardous materials from the disposal site. Leaking of pollutants from the disposal area can occur due to unfavorable reactions between the liner and the inclosed sludge or as a result of deterioration or accidential rupture of the liner.

Another method for reducing the migration of pollutants from the sludge disposal area to the surrounding environment is sludge fixation or solidification/stabilization. At present, the two major types of fixation processes in use are encapsulation and addition of materials that react with the sludge. Encapsulation methods provide a physical barrier against pollutant mobility. Fixation processes admix organic or inorganic materials with the sludges to reduce pollutant mobility generally by altering the chemical and/or physical properties of the sludge. Chemical alterations are aimed at reducing the solubility of the pollutants. Physical alterations are aimed at lowering the permeability of the sludges and decreasing the surface area-to-volume ratio.

There are certain problems associated with sludge solidification/stabilization. The high cost of total encapsulation processes limit their application to very toxic sludges usually with relatively small volumes. In most fixation processes, the admixed materials-to-sludge ratios are difficult to standardize. Since this fixation technique generally depends on a reaction of additives with the sludge, it must be tested in advance to determine whether the desired reaction will take place and the proper conditions for the reaction (additives-to-sludge ratio pH, moisture content, etc.) exist. After fixation, the

material must be tested to determine the rate of leaching of pollutants. The testing procedures generally used involve column leaching and/or an extended surface wash of the material. These tests are usually performed over a relatively long time period to allow prediction of future performance of the fixed sludge for the life of the disposal system.

There is a need for a fast, simple test procedure that can be used to predict the total pollutant migration from a fixed sludge. A test of this type will speed up fixation technology development and provide regulatory agencies with an economical method for evaluating the pollution potential of any fixed sludge. This report summarizes the development and use of a fast, simple testing procedure for determining the total pollutant migration from a raw or fixed sludge.

#### HISTORY OF ELUTRIATE TEST

The standard elutriate test was developed by the Corps of Engineers in an attempt to develop a more technically sound approach toward dredged material disposal. The previous use of bulk chemical composition of the sediment had not provided a useful index of potential environmental quality problems associated with disposal (1). This test was designed to be used as a leaching test using site water. The information obtained from a standard elutriate test is used as a basis for estimating the potential significance of contaminants present in sediment to be dredged.

The standard elutriate test procedure as published in the Federal Register (2,3) involves the mixing of sediment and disposal site water in a ratio of 1:4 on a volume-to-volume basis. The mixture is shaken for one-half hour, allowed to settle for one hour, and then filtered or centrifuged prior to analysis. The analysis of the elutriate water is compared to the analysis of the site water. A significant increase in the concentration of a particular contaminant signals a potential problem with the contaminant in the dredging process.

# PURPOSE AND OBJECTIVES

The U. S. Army Engineer Waterways Experiment Station (WES) has undertaken, through an interagency agreement with the U. S. Environmental Protection Agency, a study directed toward examining the potential for success of fixation processes as applied to various sludges to yield products environmentally acceptable for disposal. Several sludges associated with different industrial processes and with flue gas desulfurization (FGD) systems have been used in the study. The objectives of the study are as follows:

- a) To assess on a laboratory scale the pollution potential, leachability and physical durability of selected hazardous industrial sludges, FGD sludges, and fixed sludges from these two categories.
- b) To verify the laboratory data by field studies. The study has been divided into three phases, the first of which is sludge characteriza-

tion and experimental design. This phase has been completed and is summarized in an earlier project report (4). The second phase involves laboratory testing and includes the elutriate test work. This phase has been partially completed and summarized in an earlier project report (5). The elutriate test work was not included in that report. Work is continuing in the second phase of the study and should soon be completed. Work on the third phase, which involves field testing, has been recently completed.

The elutriate test was developed to provide a fast, simple test procedure that can be used to predict the total pollutant migration from a sludge. The objectives of the elutriate test study were as follows:

- a) To assess the applicability of the elutriate test in predicting pollutant migration from various hazardous sludges.
- b) To assess the correlation between the elutriate test and long-term leaching tests.
- c) To evaluate the performance of various fixation techniques in limit ing the migration of pollutants from certain hazardous sludges.

This report summarizes the work completed in the elutriate test study. Work is continuing in the verification of the elutriate test as data becomes available from other phases of the overall study.

#### CONCLUSIONS

Experimental studies involving the elutriate test have indicated that such a test can be a very effective aid in evaluating pollutant migration from various hazardous sludges, but careful interpretation and application of the results is required.

The elutriate test is not a true analog of a disposal site because no provisions are made for modeling the attenuation of pollutants by soil or the dilution of pollutant concentrations by disposal site groundwater, but the elutriate test does provide a fast, simple technique for comparing raw and/or fixed sludges on the basis of pollutant migration to the elutriate.

The pollutant concentrations found in the elutriate from the elutriate test are mainly dependent on the physical durability of the sludge sample being tested and on the solubility of the pollutants in the sludge sample.

The fixation processes were only partially successful in limiting the escape of pollutants from the hazardous sludges in this study. Cadmium and chromium were found to be the most difficult metals to contain. Success rates for metal containment by fixation were lower for the industrial sludges than for the FGD sludges.

A review of the fixation processes used in the study identified several factors that could contribute to the poor success of the fixation processes in limiting metal migration. These factors include changes in sludge permeability, pH control, and the composition of the process additives.

# RECOMMENDATIONS

Verification of the elutriate test should be continued as data become available from other phases of the overall study.

Although the elutriate test appears to have some potential for evaluating pollutant migration from sludges, other promising techniques for predicting pollutant migration should be investigated, including modifications of the elutriate test. Two such modifications that should be investigated are as follows: 1) repetitive testing of the same sludge sample to determine how migration varies with elutriate volume, and 2) reducing the degree of agitation to a point where the sludges can maintain their physical integrity throughout the test.

#### MATERIALS AND METHODS

#### SLUDGES

The sludges used in this study are from both industrial processes and flue gas desulfurization processes. The selection of the sludges was principally directed by EPA personnel and was based on availability and composition. The sludges are identified only by code numbers in this report as has been done in other reports related to the project (5).

A description of the sludges used in the study along with the identifying numerical code for each sludge is presented in Table 1. Chemical and physical properties of the sludges have been presented in an earlier project report (5).

#### FIXATION

The fixation processes used in this study have been discussed in detail in an earlier project report (5). A brief description of each fixation process, along with its identifying alphabetical code, is presented in Table 2. Elutriate test evaluations were not made on fixed sludge specimens prepared using process D (total encapsulation). This process involves encapsulating the sludge in a 0.64 centimeter plastic jacket. Previous testing had indicated very little potential for pollutant migration from sludges fixed with this process.

#### ELUTRIATE TEST PROCEDURE

The elutriate test conducted on the sludges is based on a standard elutriate test (1). The methodology for the elutriate test used on the raw sludges is as follows:

- a) Place 200 ml of sludge in a 1000-ml graduated Erlenmeyer flask and fill to 1000 ml with deionized water.
- b) Shake vigorously for 30 minutes.
- c) Centrifuge at 2500 rpm for 20 minutes.
- d) Filter centrifugate through a 0.45-micron membrane filter.

TABLE 1. IDENTIFICATION OF SLUDGES

Code Number	Description
100	Flue gas desulfurization, lime process, eastern coal
200	Electroplating
300	Nickel/cadmium battery
400	Flue gas desulfurization, limestone process, eastern coal
500	Flue gas desulfurization, double alkali process, eastern coal
600	Flue gas desulfurization, lime process, western coal
700	Inorganic pigment production
800	Chlorine production
900	Calcium fluoride
1000	Flue gas desulfurization, double alkali process, western coal

TABLE 2. IDENTIFICATION OF FIXATION PROCESSES

Process Code	Description
A	Fly ash and a lime additive, pozzolan product
В	Two additives, soil-like material
C	Organic resin, other additives, and pH adjustment, rubber-like material
D	Plastic encapsulation
E	Two commercially available additives, concrete-like material
F	Patented additive and pH adjustment, clay-like material
G	Waste product additive and pH adjustment, clay-like material

The methodology used on the fixed sludges involved using a 200 g cured sample and 800 ml of deionized water. The subsequent steps were identical to the raw sludge elutriate test procedure. All tests were conducted in triplicate at a constant temperature. After filtering, the centrifugate was split into two samples. One sample was preserved with nitric acid for metal analysis and the other sample was preserved by cooling to 4°C.

# TOTAL DIGEST PROCEDURE

A total digest was conducted on samples of the raw and fixed sludges to develop bulk analysis information. The methodology used in the total digest procedure is as follows:

- a) Place 2 g (wet weight) of sludge in a covered Teflon beaker.
- b) Digest at 175°C with a solution of 15 ml of hydrofluoric acid and 10 ml of concentrated nitric acid.
- c) Evaporate to near dryness, and dissolve the residue in hot 6  $\underline{N}$  hydrochloric acid.
- d) Dilute to 100 ml with deionized water in a volumetric flask. All tests were conducted in triplicate. Metal analyses were conducted on all samples.

### CHEMICAL ANALYSIS

The methods adopted for chemical analysis were selected by the WES and reviewed by the USEPA. These methods have been discussed in detail in an earlier report (5). Table 3 contains a list of the chemical analyses (with references) conducted on the elutriate samples. Metal analysis methods used on samples from the total digest test were identical to the methods used on the elutriate samples.

An extensive quality control program was conducted within the analytical program. This program included internal, and interlaboratory procedures. A discussion of the quality control program was included in an earlier report (5).

TABLE 3. CHEMICAL ANALYSIS

Cations	Anions
Arsenica Berylliuma,b Borona,b Cadmiuma,b Calciuma,b Chromiuma,b Cobalta,b Coppera,b Leada,b	Bicarbonate a Carbonate a Carbonate a Chloride a b Cyanide a b Nitrate a b Nitrite a b Sulfate a b Sulfite
Lead, Magnesium A,b Manganese Mercury Molybdenum Nickel Potassium Selenium Sodium Tin Vanadium Vanadium Zinc **  Manganese  Mercury  Molybdenum  Nickel  Nicke	Descriptive  pH <sup>a,b</sup> Conductivity <sup>a</sup> Organic  Chemical oxygen demand <sup>a,b</sup>

<sup>&</sup>lt;sup>a</sup> Standard Methods for the Examination of Water and Wastewater, 13th Edition. American Public Health Association, Washington, D. C., 1971.

b Methods for Chemical Analysis of Water and Wastes. EPA-625/6-74-003, U. S. Environmental Protection Agency, Cincinnati, Ohio, 1974.

<sup>&</sup>lt;sup>c</sup> Cyanide in Water and Wastewater. Technicon Industrial Method No. 315-74W, Technicon Company, Tarrytown, New York, 1974.

# ELUTRIATE TEST

#### ADAPTATION FOR SLUDGES

A modification of the standard elutriate test was chosen as a potential technique for ascertaining the migration of pollutants from sludges. The major modification involved the use of deionized water rather than site water. Also, each elutriate sample was centrifuged to aid in filtering. These modifications resulted in a more reproducible test.

#### FACTORS AFFECTING THE ELUTRIATE TEST

Studies conducted on the standard elutriate test for dredged materials have established several factors that could affect the test (6). Those factors which could affect the elutriate test for sludges include solids-liquid ratio, pH, time of contact, particle size, and solid-liquid separation. A discussion of these factors follows.

# Solids-Liquid Ratio

A very high or low solids-liquid ratio could result in the elutriate test yielding low estimates of available contaminants in a sludge sample. For high solids-liquid ratios, the release of contaminants from the sludge may be limited by saturation concentrations. If a particular contaminant reaches the saturation level, a higher solids-liquid ratio would not produce a proportionate concentration increase in the elutriate. For low solids-liquid ratios, the amount of contaminants released to the elutriate may not produce a concentration above the minimum detection limits of standard analytical techniques. Each contaminant has an optimum release rate corresponding to some particular solids-liquid ratio due to the concentration of the contaminant in the sludge. The elutriate test procedure used on the sludges resulted in a solids-liquid ratio of approximately 1:8 (on a weight basis). This ratio proved to be a good compromise between the optimum ratios for each contaminant.

# pН

The pH of the elutriate is an important controlling factor in the release of contaminants from a sludge. Generally, solubilities increase with decreasing pH. In the elutriate test, pH is controlled by the buffer capacity of the water and sludge. Deionized water used in the test has very little buffer capacity. Most of the sludges in the study were generated in processes where pH control is used to minimize metal solubility. The additives used to control

the pH usually result in the sludges having a very high buffer capacity, and therefore the sludges controlled the pH of the resultant elutriate. This is also true for the fixed sludges. This difference in buffer capacities in the elutriate test probably best represents the field situation and therefore no attempt was made to control the pH of the elutriate during the test.

# Time of Contact

The time of contact between the elutriate and sludge is an important parameter in the elutriate test because the release and migration of contaminants is not an instantaneous process. The elutriate test procedure allows for 30 minutes of contact along with agitation to promote interaction between the water and sludge. Longer shaking periods were tried but no significant increases in contaminant concentrations were found in the elutriate.

# Particle Size

Particle size associated with each sludge is an important parameter in the elutriate test because solution is dependent on surface area. Smaller size particles have a greater surface area per unit weight than do larger size particles. Therefore, a greater migration of contaminants would be expected from smaller size particles. In the elutriate test procedure, several chunks of a particular fixed sludge (approximately 3/4 inch in diameter) were placed in an Erlenmeyer flask. This procedure allowed all of the fixed sludges to start the test with similar surface areas. The raw sludges were placed in flasks in a thick slurry form resulting in a high surface areato-volume ratio. All of the fixed sludges tested deteriorated with many of them falling completely apart. This situation probably represents the worst possible field condition. Any attempt at particle size modification such as grinding and sizing of the sludges would greatly increase the time needed for conducting the elutriate test. Such a procedure would not represent the conditions in a disposal site.

# Solid-Liquid Separation

Solid-liquid separation in the elutriate test refers to the removal of solid material from the elutriate. This solids removal is achieved by centrifugation followed by filtration. Centrifugation speeds up the filtering operation by removing most of the solids. The elutriate test filtration procedure specifies a 0.45 micron membrane filter which is readily available at a moderate cost. These filters can pass particulate matter in the lower colloidal size range which will be measured as "soluble" species. Although particles larger than colloidal size are often found in leachate from sludge disposal sites, the leachate can usually be filtered before analysis. The filtering procedure results in a better comparison of the data from the elutriate test and the analysis of leachate from disposal sites.

Obviously, in any elutriate test procedure, a standard filter type must be specified and used where results of a series of tests are to be compared.

#### PRACTICABILITY OF THE ELUTRIATE TEST

The elutriate test was developed to provide a fast, simple testing procedure at a reasonable cost for evaluating pollutant migration from sludges. The elutriate test usually requires two days for completion; two to three hours for the testing procedure and twelve to fourteen hours for the chemical analysis (depending on the number of parameters). The test can be performed by technically trained personnel and equipment normally available in analytical laboratories. Costs for collection and transportation of sludge samples are low due to the small amount of material needed for the test. On a cost basis, the elutriate test is a good alternative for evaluating pollutant migration from sludges.

There are certain problems concerning the practicability of the elutriate test that must be addressed in any testing program. The nonhomogeneity of certain sludges can result in nonreproducible results. The concentrations of various contaminants in a sludge can vary over a period of time due to operational changes in the system producing the sludge. In some cases, sludge is placed in a disposal area along with other materials that can release contaminants (i.e., bottom ash, domestic wastes, etc). It is important to obtain a representative sample of sludge if the elutriate test is to provide meaningful results.

Another major problem concerning the practicability of the elutriate test is the interpretation and application of the results. These results must be correlated with long-term pollutant migration in a field situation. This problem will be discussed in detail in the next subsection.

# INTERPRETATION AND APPLICATION OF ELUTRIATE TEST RESULTS

The elutriate test was developed for use in ascertaining the migration of pollutants from sludges. The modeling of actual field conditions at a disposal site is a difficult and time-consuming process. Since the elutriate test is not a true analog of a disposal site, various problems arise in the interpretation of elutriate test results.

It should be noted that the elutriate test is a simple shake test. No attempt is made in the elutriate test procedure to prevent the physical deterioration of the sludges. This means that the deionized water used in the elutriate test is permitted to come in contact with a larger surface area than it would if the sludges did not fall apart. The elutriate test therefore gives an indication of the solubility of the pollutants in a particular sludge. In comparing raw and/or fixed sludges, the elutriate test results will indicate the success of chemical alterations by the fixation processes aimed at reducing the solubility of the pollutants.

Many of the fixation processes attempt to limit migration of pollutants from sludges by chemically altering the physical properties of the sludges in order to decrease the permeability and surface area-to-volume ratio. These physical alterations are severely tested in the elutriate test due to the agitation used in the procedure. In an actual disposal site, the fixed sludge would usually be placed in such a manner that the permeability of the

fixed sludge mass would be much less than that of the surrounding soil. In this situation, the percolating water would travel through the soil until it contacted the sludge mass. Then the water would flow down the surface of the fixed sludge mass without deep penetration due to the difference in permeabilities of the sludge and the surrounding soil. The elutriate test represents a "worst case" condition for this type of sludge disposal, that is, a situation where the sludge mass is fractured or deteriorated in some manner so as to allow the penetration of percolating water. If the sludge mass does deteriorate over a period of years, the pollutants will become more mobile unless their solubility has been reduced by chemical alteration during the fixation process. The elutriate test can provide an indication of the success of fixation processes in reducing the solubility of these pollutants.

The elutriate test is a "one time" test and not a continuous flow test. The concentrations of pollutants in the elutriate are not necessarily the concentrations that would be found in leachate from a disposal site. Pollutant concentrations in leachate can vary due to dilution by surrounding groundwater. Concentrations can also change due to attenuation of certain pollutants by the soil surrounding the disposal site. The elutriate test procedure has no provisions for modeling these factors. The pollutant concentrations found in the elutriate are dependent on the physical durability of the sludge sample and on the solubility of the pollutants in the sludge sample.

The potential of the elutriate test is in its ability to provide a fast, simple technique for comparing raw and/or fixed sludges. Elutriate test results provide an indication of the relative effectiveness of different fixation processes on limiting the migration of pollutants. Elutriate test data can also indicate which pollutants will be particularly mobile. The elutriate test can be a very effective aid in evaluating sludge performance but careful interpretation and application of the results is required.

# PRESENTATION OF ELUTRIATE TEST RESULTS

# CHEMICAL PROPERTIES

The results of the analysis of the elutriate from the elutriate tests conducted on the raw and fixed sludges are presented in Tables 4 and 5. All values except for pH and conductivity are expressed in terms of mg/kg on a dry weight basis. Conductivity is expressed in terms of micromhos/cm. The conversion of concentrations of parameters to a dry weight basis provides a common base for comparison of the raw and fixed sludges. This conversion also results in the multiplication of any error associated with chemical analysis. The sludges are identified in terms of a three or four digit number which indicates the source of the sludge (Table 1) and a letter which indicates either a raw sludge (R) or a fixation process (Table 2). The different combinations of sludges and fixation processes are the result of fixation processors declining to work with certain sludges.

Table 6 contains the bulk analysis results (metals only) from the total digest conducted on each sludge. All values have been corrected for blanks and are expressed in terms of mg/kg on a dry weight basis. The bulk analysis data along with the elutriate test data was used to calculate the percent solubilization of certain metals during the elutriate test. This information is contained in Table 7.

Many of the high concentrations of contaminants reported for the elutriate test and the total digest test can be correlated with sludge generation processes or fixation techniques. Since the exact source of each sludge and the nature of the fixation additives cannot be disclosed in this report, correlation between raw and fixed sludge generation and high contaminant concentrations can be discussed only in general terms.

# General Analysis

A general analysis of each elutriate sample was conducted to provide information concerning pH, organic content, and anion migration from the sludges (Table 4).

# COD--

The COD (chemical oxygen demand) is a measure of the oxygen equivalent of the material in a sample that can be oxidized by a strong chemical oxidant. Although the COD test is designed to reflect the concentration of organic material in a sample, the oxidation of inorganics such as chloride

TABLE 4. ANALYSIS OF ELUTRIATE SAMPLES (GENERAL)

			Par	rameters		
Sludge	COD	so <sub>4</sub>	so <sub>3</sub>	No <sub>2</sub> -N	NO3-N	CN
Identification	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)
100R	230	7200	6.8	4.6	0.60	BDLe
100A	45 <u>.</u>	6000	$\mathtt{BDL}^{\mathtt{b}}$	4.6 BDL	0.08	$\mathtt{BDL}$
100B	$\mathtt{BDL}^\mathbf{a}$	11000	BDL	0.36	0.78	$\mathtt{BDL}$
100E	$\mathtt{BDL}$	720	BDL	$\mathtt{BDL}$	BDL	$\mathtt{BDL}$
100F	110	31.00	160	0.42	BDL	3.8
200R	210	17000	6.1	0.22	1.8	BDL
200A	80.	8800	100	0.10	$\mathtt{BDL}$	2.2
200B	310	22000	140	0.81	$\mathtt{BDL}$	11.
200C	18000	25000	97.	BDL	1.0	0.30
300R	820	11000	12.	5200	88000	2.8
300A	240	3800	87.	2.4	480	$\mathtt{BDL}$
300B	450	280	160	220	3900	0.95
400R	500	17000	14.	BDL	6.2	BDL
400A	74.	6600	55.	0.49	320	$\mathtt{BDL}$
400B	160	7200	410	0.20	BDL	$\mathtt{BDL}$
400E	61.	2900	78.	BDL	BDL	BDL
500R	120	17000	930	1.1	0.30	0.10
500A	85.	8800	68.	BDL	BDL	BDL
500B	130	16000	260	0.80	0.30	0.30
500E	68.	1700	6.4	BDL	BDL	BDL
500G	BDL	11000	BDL	BDL	BDL	BDL

TABLE 4. (CONTINUED)

		<del></del>	Par	ameters		
Sludge	COD	so <sub>l</sub>	SO <sub>3</sub>	NO2-N	NO3-N	CN
Identification	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)
600R	190	42000	33.	BDL	23.	BDL
600A	BDL	5000	6.0	BDL	0.23	$\mathtt{BDL}$
600В	BDL	4800	11.	BDL	0.45	$\mathtt{BDL}$
600E	BDL	630	6.7	BDL	BDL	$\mathtt{BDL}$
600F	62.	5100	5.8	0.37	0.21	BDL
700R	58 <b>.</b>	2800	3.3	11.	12.	0.74
700C	16000	29000	7.0	0.12	3.0	BDL
800R	1400	8000	1.8	0.16	0.16	0.10
A008	97.	4900	BDL	1.2	1.4	0.46
800B	1100	12000	7.3	0.73	0.26	BDL
900R	57•	3200	4.4	1.7	0.30	0.10
900A	280	5200	8.2	0.24	0.24	0.24
900B	720	2900	14.	2.1	0.13	0.59
1000R	240	21000	1500	0.10	1.7	0.15
1000A	130	9100	5.9	$BD\Gamma$	0.50	$\mathtt{BDL}$
1000B	180	29000	440	0.82	1.4	BDL
1000E	44.	3100	8.1	0.08	BDL	BDL

TABLE 4. (CONTINUED)

	Parameters								
Sludge	Cl	CO3	HCO <sub>3</sub>	рН	Conductivity				
Identification	(mg/kg)		as CaCO3)		(X10 <sup>3</sup> micrombos/cm)				
100R	730	NA <sup>g</sup>	NA	7.0	1.6				
100A	50.	$\mathtt{BDL}^\mathbf{n}$	1300	7.0	2.5				
100B	570 BDL	$\mathtt{BDL}$	210	8.1	2.9				
100E	$\mathtt{BDL}^\mathtt{1}$	$\mathtt{BDL}$	720	7.4	1.5				
100F	240	BDL	980	7.1	2.2				
200R	380	NA	NA	7.2	3.0				
200A	130	$\mathtt{BDL}$	1400	7.5	3.2				
200B	700	$\mathtt{BDL}$	3200	7.8	5.4 6.2				
200C	80.	$\mathtt{BDL}$	3200 BDL	5.2	6.2				
300R	4100	NA	NA	11.4	3.1				
300A	72.	$\mathtt{BDL}$	480	7.0	2.0				
300B	360	3000	BDL	11.6	4.9				
400R	70.	<b>N</b> A	NA	7.9	1.6				
400A	$\mathtt{BDL}$	$\mathtt{BDL}$	500	7.3	2.4				
400B	BDL	570	$\mathtt{BDL}$	12.1	7.3				
400E	BDL	BDL	290	6.9	1.4				
500R	1400	NA	NA	12.6	7.0				
500A	710	$\mathtt{BDL}$	2700	6.7	3.6				
500B	BDL	BDL	BDL	12.1	13.5				
500E	110	BDL	2200	9.8	2.6				
500G	450	BDL	2500	6.9	5.4				

TABLE 4. (CONTINUED)

				ameters	
Sludge	Cl	CO3	HCO3	pН	Conductivity
Identification	(mg/kg)		as CaCO3)		(X10 <sup>3</sup> micromhos/am)
600R	3100	NA	NA	7.3	1.8
600A	97.	BDL	1100	6.8	2.4
600в	730	$\mathtt{BDL}$	3000	6.7	2.0
600E	BDL	$\mathtt{BDL}$	1300	6.8	0.9
600F	330	$\mathtt{BDL}$	1700	7.1	3.1
700R	$\mathtt{BDL}$	NA	NA	7.3	1.7
700C	98.	BDL	ήήO	6.0	6.5
800R	24000	NA	NA	8.2	2.1
800A	5500	$\mathtt{BDL}$	1900	7.2	5.8
800В	44000	130	3400	8.6	24.8
900R	BDL	NA	NA	7.1	1.3
900A	BDL	$\mathtt{BDL}$	1600	7.1	2.4
900B	BDL	BDL	2100	7.3	1.4
1000R	660	NA	NA	9.1	8.3
1000A	89	$\mathtt{BDL}$	2700	6.9	<b>3.</b> 6
1000B	$\mathtt{BDL}$	1100	$\mathtt{BDL}$	12.4	15.3
1000E	77.	1100	$\mathtt{BDL}$	10.2	2.2

**a** <5

g Not available

b <1 c <0.01 d <0.01 h <10 i <10

e <0.01

f <5

TABLE 5. ANALYSIS OF ELUTRIATE SAMPLES (METALS)

				Met	als			
Sludge Identification	As (mg/kg)	B (mg/kg)	Be (mg/kg)	Ca (mg/kg)	Cd (mg/kg)	Cr (mg/kg)	Cu (mg/kg)	Mg (mg/kg)
100R	1.3	NDA <sup>b</sup>	BDLc	4900	0.11	0.08	0.33	$^{\mathrm{BDL}}^{\mathrm{d}}$
100A	1.3 BDL	9.6	0.03	1400	0.16	0.25	0.81	BDL
100B	0.42	5.2	0.06	2400	0.06	0.75	0.63	$\mathtt{BDL}$
100E	0.09	1.4	0.01	440	0.01	0.03	0.04	$\mathtt{BDL}$
100F	0.06	8.0	0.02	2000	0.01	0.008	0.39	$\mathtt{BDL}$
200R	BDL	NDA	0.15	2700	0.39	0.69	16.	BDL
200A	0.08	23.	0.04	2400	0.38	3.9	4.7	$\mathtt{BDL}$
200B	0.008	2.3	0.07	2600	0.59	59.	13.	$\mathtt{BDL}$
200C	0.06	8.8	5.2	630	25.	21.	1200	$\mathtt{BDL}$
300R	0.03	NDA	$\mathtt{BDL}$	74.	0.75	0.20	0.61	$\mathtt{BDL}$
300A	0.01	20.	0.007	1100	220	0.21	0.06	$\mathtt{BDL}$
300B	0.009	0.26	0.002	28.	1.0	0.13	0.11	BDL
400R	0.09	NDA	BDL	9600	0.01	0.14	0.29	BDL
400A	0.02	20.	0.004	2200	0.13	0.40	0.19	$\mathtt{BDL}$
400B	0.04	0.14	0.003	510	0.008	0.43	0.10	$\mathtt{BDL}$
400E	0.07	1.6	0.002	1200	0.03	0.18	0.02	BDL
500R	0.03	NDA	BDL	4800	0.008	0.11	0.13	BDL
500A	0.19	14.	0.006	2900	0.07	0.17	0.06	$\mathtt{BDL}$
500B	0.02	0.70	0.005	1900	0.008	0.42	0.23	$\mathtt{BDL}$
500È	0.15	0.82	0.003	220	0.003	0.29	0.03	$\mathtt{BDL}$
500G	0.31	37.	0.009	3100	0.009	0.009	0.07	$\mathtt{BDL}$

TABLE 5. (CONTINUED)

	Metals								
Sludge Identification	As (mg/kg)	B (mg/kg)	Be (mg/kg)	Ca (mg/kg)	Cd (mg/kg)	Cr (mg/kg)	Cu (mg/kg)	Hg (mg/kg)	
600R	0.12	NDA	BDL	19000	0.06	0.40	0.05	BDL	
600A	0.01	7.8	0.009	1600	0.28	0.21	0.05	0.001	
600В	0.04	4.6	0.009	2200	0.45	0.42	0.24	BDL	
600E	0.009	1.3	0.009	600	0.01	0.13	0.01	$\mathtt{BDL}$	
600F	0.87	10.	0.01	3600	0.003	0.21	0.02	0.001	
700R	BDL	NDA	BDL	580	2.1	0.08	0.11	BDL	
700C	BDL	43.	0.01	1100	140	1.7	4.5	BDL	
800R	BDL	NDA	BDL	1100	0.05	0.48	0.33	0.03	
800A	0.11	23.	0.009	2900	150	0.23	0.39	0.003	
800В	0.04	0.32	0.008	930	0.18	1.7	0.22	0.004	
900R	BDL	NDA	BDL	1300	0.004	0.007	0.004	BDL	
900A	0.07	25.	0.007	3300	0.005	0.20	1.4	BDL	
900B	0.02	1.0	0.004	1600	0.03	0.20	26.	BDL	
1000R	0.03	NDA	BDL	390	0.02	0.03	0.10	BDL	
1000A	0.02	11.	0.004	2400	0.02	0.21	0.10	BDL	
1000B	0.03	2.1	0.01	220	0.03	0.66	0.47	BDL	
1000E	0.09	0.69	0.002	280	0.01	0.52	0.36	$\mathtt{BDL}$	

TABLE 5. (CONTINUED)

Sludge Identification	Metals									
	K (mg/kg)	Mg (mg/kg)	Mn (mg/kg)	Mo (mg/kg)	Na (mg/kg)	Ni (mg/kg)	Pb (mg/kg)	Se (mg/kg)	Zn (mg/kg)	
100R	NDA	74.	0.73	NDA	NDA.	2.2	0.04	0.05	0.21	
100A	47.	i3.	0.72	0.93	1500	0.19	0.07	0.10	0.50 <sub>h</sub> BDL	
100B	900	12.	0.72	1.3	2000	0.26	0.04	0.14	$^{\mathrm{BDL}^{\mathbf{n}}}$	
100E	360	12.	0.12	0.26	100	0.03	0.01	0.09	$\mathtt{BDL}$	
100F	28.	29.	2.8	0.85	140	0.18	0.04	0.08	$\mathtt{BDL}$	
200R	NDA	630	4.1	NDA	NDA	4.1	0.02	0.05	7.8	
200A	140	16.	0.99	1.3	1300	0.56	0.25	0.07	6.2	
200B	220	11.	1.5	1.2	7000	0.84	0.74	0.12	5.0	
200C	12.	510	11.	0.66	4700	120	$\mathtt{BDL}^{\mathbf{g}}$	0.09	330	
300R	NDA	1.9	$_{\mathrm{BDL}}^{\mathbf{e}}$	NDA	NDA	2.4	0.03	0.10	0.26	
300A	150	11.	0.26	0.62	890	12.	$\mathtt{BDL}$	0.08	0.39	
300B	210	0.55	0.10	BDLf	5200	2.6	0.02	0.10	BDL	
400R	NDA	97.	0.55	NDA	NDA	0.70	0.15	0.10	7.3	
400A	89.	7.4	0.36	2.1	300	0.45	0.03	0.05	BDL	
400B	690	0.81	0.37	BDL	5200	0.22	0.02	0.09	BDL	
400E	330	10.	0.22	0.61	180	0.05	0.04	0.05	$\mathtt{BDL}$	
500R	NDA	0.83	0.04	NDA	NDA	0.18	0.02	0.10	BDL	
500A	340	6.8	0.71	2.3	2700	0.23	0.03	0.08	0.4	
500B	720	0.20	0.03	1.6	13000	0.08	0.03	0.43	BDL	
500E	360	2.2	0.02	BDL	1600	0.02	0.02	0.07	BDL	
500G	2600	270	6.1	0.98	2300	0.19	0.02	0.09	BDL	

TABLE 5. (CONTINUED)

Sludge Identification	Metals									
	K (mg/kg)	Mg (mg/kg)	Mn (mg/kg)	Mo (mg/kg)	Na (mg/kg)	Ni (mg/kg)	Pb (mg/kg)	Se (mg/kg)	Zn (mg/kg)	
600R	NDA	820	19.	NDA	NDA	2.5	BDL	0.37	1.4	
600A	26.	20.	0.23	1.2	190	0.15	0.02	0.22	$\mathtt{BDL}$	
600B	210	15.	1.7	1.5	1600	0.19	0.20	0.14	BDL	
600E	93.	7.0	0.17	$\mathtt{BDL}$	130	0.04	0.02	0.06	0.44	
600F	160	28.	2.2	.87	140	0.11	$\mathtt{BDL}$	0.07	$\mathtt{BDL}$	
700R	NDA	420	5.4	NDA	NDA	0.14	0.51	0.01	$\mathtt{BDL}$	
700C	15.	1300	150	$\mathtt{BDL}$	5600	3.7	30.	0.11	16.	
800R	NDA	33.	0.37	NDA	NDA	0.05	0.01	0.04	BDL	
800A	150	12.	0.14	1.4	2600	6.0	0.02	0.09	$\mathtt{BDL}$	
800B	250	3.0	0.24	BDL	31000	0.20	0.07	0.16	$\mathtt{BDL}$	
900R	NDA	130	0.67	NDA	NDA	0.62	0.02	0.01	0.10	
900A	130	42.	0.67	2.1	2100	0.51	BDL	0.11	0.58	
900В	260	17.	0.22	BDL	780	7.3	0.10	0.14	0.46	
1000R	NDA	89.	BDL	NDA	NDA	0.07	0.03	0.08	0.14	
1000A	55.	31.	0.38	1.3	2100	0.12	5.5	0.12	BDL	
1000B	470	0.21	0.03	$\mathtt{BDL}$	20000	0.23	0.22	0.17	BDL	
1000E	270	4.2	0.07	BDL	1500	0.16	0.01	0.10	0.28	

f <0.03

a <0.002 b No data available

g <0.001

c <0.001

h <0.05

d <0.0002

e <0.003

TABLE 6. ANALYSIS OF TOTAL DIGEST (METALS)

	Metals									
Sludge Identification	As (mg/kg)	Be (mg/kg)	Ca. (mg/kg)	Cd (mg/kg)	Co (mg/kg)	Cr (mg/kg)	Cu (mg/kg)	Hg (mg/kg)		
100R	170	12.	63000	2.4	47.	99.	88.	0.82		
100A	60.	8.5	34000	BGS	29.	85.	68.	0.31		
100B	56.	4.3	43000	8.0	27.	90.	51.	0.50		
100E	25.	2.5	60000	7.5	21.	60.	35.	0.16		
100F	2.1	1.6	84000	5.2	11.	27.	26.	0.05		
200R	24.	840	75000	1800	120	150000	95000	0.53		
200A	29.	98.	58000	320	45.	24000	12000	0.20		
200B	13.	320	45000	730	66.	85000	55000	0.26		
200C	2.6	72.	20000	130	14.	20000	9800	0.04		
300R	BGS <sup>€</sup>	$\mathtt{BDL}^{\mathbf{b}}$	37000	520000	150	180	1300	9.8		
300A	30.	7.8	24000	110000	37.	89.	130	0.69		
300B	0.20	BDL	47000	490000	180	110	360	14.		
400R	35.	6.1	160000	13.	35.	91.	170	1.9		
400A	44.	6.9	63000	26.	22.	63.	75.	0.39		
400B	13.	BDL	42000	20.	11.	36.	45.	0.44		
400E	41.	1.9	110000	4.4	16.	48.	33.	0.33		
400G	5.4	0.78	180000	BGS	16. BDL <sup>c</sup>	21.	14.	0.08		
500R	150	3.9	69000	22.	22.	54.	92.	1.6		
500A	73	9.0	93000	BGS	28.	62.	78.	0.83		
500B	120	BDL	190000	43.	14.	34.	50.	1.8		
500E	19.	2.5	46000	6.9	19.	53.	46.	0.16		
500G	38.	1.1	150000	BGS	BDL	14.	16.	0.24		

TABLE 6. (CONTINUED)

		Metals									
Sludge Identification	As (mg/kg)	Be (mg/kg)	Ca (mg/kg)	Cd (mg/kg)	Co (mg/kg)	Cr (mg/kg)	Cu (mg/kg)	Hg (mg/kg)			
600R	29.	27.	200000	18.	83.	130	280	0.14			
600A	7.0	4.2	67000	BGS	16.	24.	51.	0.22			
600В	19.	1.9	54000	7.6	29.	43.	77.	0.45			
600E	8.4	1.7	57000	7.2	16.	33.	25.	0.06			
600F	49.	4.2	50000	4.7	15.	67.	30.	0.10			
600G	17.	0.68	180000	BGS	BDL	52.	14.	0.03			
700R	100.	$\mathtt{BDL}$	40000	9700	13.	110000	7600	38.			
700C	.58	$\mathtt{BDL}$	3900	1100	BDL	12000	670	6.5			
800R	BGS	BDL	27000	2.7	BDL	9.3	260	67.			
800A	52.	5.4	110000	BGS	19.	38.	160	38.			
800B	1.6	BDL	120000	9.8	BDL	.65	220	74.			
900R	BGS	BDL	46000	5.5	23.	34.	670	0.06			
900A	14.	6.3	120000	BGS	23.	59.	220	0.24			
900B	0.73	$\mathtt{BDL}$	170000	7.3	9.0	20.	480	0.31			
1000R	13.	BDL	59000	2.3	BDL	25.	38.	0.08			
1000A	7.2	3.0	69000	BGS	12.	27.	50.	0.17			
1000B	4.1	BDL	120000	5.1	27.	14.	24.	0.31			
1000E	18.	1.4	57000	21.	16.	55.	35.	0.10			
1000G	17.	$\mathtt{BDL}$	180000	BGS	$\mathtt{BDL}$	12.	12.	0.09			

TABLE 6. (CONTINUED)

Sludge Identification	Metals									
	Mg (mg/kg)	Mn (mg/kg)	Mo (mg/kg)	Na (mg/kg)	Ni (mg/kg)	Pb (mg/kg)	Sn (mg/kg)	V (mg/kg)	Zn (mg/kg)	
100R	5500	160	24.	17000	140	84.	26.	270	170	
100A	2300	120	43.	20000	68.	61.	38.	180	110	
100B	350	640	36.	19000	76.	52.	25.	120	110	
100E	550	370	25.	4400	32.	18.	6.2	80.	59.	
100F	2100	250	37.	5900	21.	34.	7.7	40.	46.	
200R	5700	390	77.	49000	6100	1400	22000	82.	18000	
200A	3400	150	62.	28000	1100	450	3500	210	2600	
200B	4400	690	21.	19000	3600	880	10000	62.	10000	
200C	1200	BGS	26.	8600	670	240	2500	15.	2000	
300R	6000	370	15.	29000	390000	590	390	20.	3800	
300A	3200	110	51.	23000	37000	70.	28.	200	260	
300B	1000	550	15.	20000	360000	180	150	9.9	850	
400R	18000	250	150	40000	220	100	39.	290	330	
400A	5400	120	53.	25000	74.	51.	44.	190	110	
400B	4800	590	78.	16000	50.	28.	21.	47.	90.	
400E	3800	360	35.	8200	27.	30.	BGS	62.	71.	
400G	3100	140	9.4	20000	19.	89.	BGS	95•	94.	
500R	9700	170	99.	28000	120	160	28.	140	230	
500A	5400	150	71.	31000	66.	91.	26.	200	140	
500B	6800	460	23.	12000	83.	74.	29.	28.	160	
500E	690	290	17.	5600	43.	14.	BGS	97.	73.	
500G	2700	100	19.	20000	12.	64.	BGS	47.	89.	

TABLE 6. (CONTINUED)

					Metals				
Sludge Identification	Mg (mg/kg)	Mn (mg/kg)	Mo (mg/kg)	Na (mg/kg)	Ni (mg/kg)	Pb (mg/kg)	Sn (mg/kg)	V (mg/kg)	Zn (mg/kg)
600R	34000	600	140	100000	210	380	84.	530	340
600A	7800	110	68.	26000	32.	61.	3.8	140	80.
600В	1400	920	67.	15000	60.	85.	48.	110	100
600E	270	320	33.	5400	30.	18.	7.7	50.	€9.
600F	2200	270	41.	7600	40.	22.	11.	100	75.
600G	2200	150	8.5	23000	11.	72.	BGS	61.	66.
700R	60000	3400	5700	20000	700	160000	14000	120	3800
700C	3200	310	390	5200	71.	150	1400	20.	410
800R	1700	82.	15.	11000	30.	110	16.	38.	78.
800A	2900	110	55.	26000	50.	85.	BGS	140	85.
800В	160	250	40.	11000	22.	70.	3.5	12.	91.
900R	7800	220	BGS	25000	880	370	98.	67.	560
900A	7700	160	65.	23000	270	170	24.	180	280
900В	53000	350	31.	22000	690	330	60.	33.	520
1000R	5200	44.	5.7	19000	50.	93.	19.	69.	67.
1000A	8800	88.	45.	29000	25.	51.	BGS	140	77.
1000B	3200	360	51.	15000	20.	49.	10.	35.	47.
1000E	1300	260	31.	6300	48.	15.	5.5	49.	45.
1000G	2600	120	24.	26000	8.8	61.	BGS	59.	56.

a Blank greater than sample

b <0.01

c <0.1

TABLE 7. METAL SOLUBILIZATION

C11			Metals			
Sludge Identification	As	Ве	Ca	Cđ	Cr	Cu
100R	0.76ª	NC <sup>b</sup>	7.8	4.6	0.08	0.38
100A	NC	0.35	4.1	NC	0.30	1.2
100B	0.75	1.4	5.6	0.75	0.83	1.2
100E	0.36	0.40	0.73	0.13	0.05	0.11
100F	2.9	1.3	2.4	0.19	0.03	1.5
200R	NC	0.02	3.6	0.02	<0.01	0.02
200A	0.28	0.04	4.1	0.12	0.02	0.04
200B	0.06	0.02	5.8	0.08	0.07	0.02
200C	2.3	7.2	3.2	19.2	0.11	12.2
300R	NC	NC	0.20	<0.01	0.11	0.05
300A	0.03	0.09	4.6	0.20	0.24	0.05
300B	4.5	NC	0.06	<0.01	0.12	0.03
400R	0.26	NC	6.0	0.08	0.15	0.17
400A	0.05	0.06	3.5	0.50	0.63	0.25
400B	0.31	NC	1.2	0.04	1.2	0.22
400E	0.17	0.11	1.1	0.68	0.38	0.06
500R	0.02	NC	7.0	0.04	0.20	0.14
500A	0.26	0.07	3.1	NC	0.27	0.08
500B	0.02	NC	1.0	0.02	1.2	0.46
500E	0.79	0.12	0.48	0.04	0.55	0.07
500G	0.82	0.82	2.1	NC	0.06	0.04

(continued)

TABLE 7. (CONTINUED)

a			Me-	tals		
Sludge Identification	As	Be	Са	Cd	Cr	Cu
600R	0.41	NC	9.5	0.33	0.31	0.02
600A	0.14	0.21	2.4	NC	0.88	0.10
600В	0.21	0.47	4.1	5.9	0.98	0.31
600E	0.11	0.53	1.1	0.14	0.39	0.04
600F	1.8	0.24	7.2	0.06	0.31	0.07
700R	NC	NC	1.5	0.02	<0.01	<0.01
700C	NC	NC	28.2	12.7	0.01	0.67
800R	NC	NC	4.1	1.9	5.2	0.13
800A	0.21	0.17	2.6	NC	0.61	0.21
800В	2.5	NC	0.78	1.8	>100	0.10
900R	NC	NC	2.8	0.07	0.02	<0.01
900A	0.50	0.06	2.8	NC	0.34	0.64
900B	2.7	NC	0.94	0.41	1.0	5.4
1000R	0.23	NC	0.66	0.87	0.12	0.26
1000A	0.28	0.13	3.5	NC	0.78	0.20
1000B	0.73	NC	0.18	0.59	4.7	2.0
1000E	0.50	0.14	0.49	0.05	0.95	1.0

(continued)

TABLE 7. (CONTINUED)

011				Metals			·
Sludge Identification	Mg	Mn	Мо	Na.	Ni	Pb	Zn
100R	1.3	0.46	NC	NC	1.6	0.05	0.12
100A	0.57	0.60	2.2	7.5	0.28	0.11	0.45
100B	3.4	0.11	3.6	10.5	0.34	0.08	NC
100E	2.2	0.03	1.0	2,3	0.09	0.06	NC
100F	1.4	1.1	2.3	2.4	0.86	0.12	NC
200R	11.1	1.1	NC	NC	0.07	<0.01	0.04
200A	0.47	0.66	2.1	4.6	0.05	0.06	0.24
200B	0.25	0.22	5.7	36.8	0.02	0.08	0.05
200C	42.5	NC	2.5	54.7	17.9	NC	16.5
300R	0.03	NC	NC	NC	<0.01	0.01	0.01
300A	0.34	0.24	1.2	3.9	0.03	NC	0.15
300B	0.06	0.02	NC	26.	<0.01	0.01	NC
400R	0.54	0.22	NC	NC	0.32	0.15	2.2
400A	0.14	0.30	4.0	1.2	0.61	0.06	NC
400B	0.02	0.06	NC	32.5	0.44	0.07	NC
400E	0.26	0.06	1.7	2.2	0.19	0.13	NC
500R	0.01	0.02	NC	NC	0.15	0.01	NC
500A	0.13	0.47	3.2	8.7	0.35	0.03	0.31
500B	<0.01	0.01	7.0	>100	0.10	0.04	NC
500E	0.32	0.01	NC	28.6	0.05	0.14	NC
500G	10.0	6.1	5.2	11.5	1.6	0.03	NC

(continued)

TABLE 7. (CONTINUED)

Cludes				Metals			
Sludge Identification	Mg	Mn	Мо	Na.	Ni	Ръ	Zn
600R	2.4	3.2	NC	NC	1.2	NC	0.41
600A	0.26	0.21	1.8	0.73	0.47	0.03	NC
600В	1.1	0.18	2.2	10.7	0.32	0.24	NC
600E	2.6	0.05	NC	2.4	0.13	0.11	0.64
600F	1.3	0.81	2.1	1.8	0.28	NC	NC
700R	0.70	0.16	NC	NC	0.02	<0.01	NC
700C	40.6	48.4	NC	>100	5.2	20.0	3.9
800R	1.9	0.45	NC	NC	0.17	0.01	NC
800A	0.41	0.13	2.5	10.0	12.0	0.02	NC
800B	1.9	0.10	NC	>100	0.91	0.10	NC
900R	1.7	0.30	NC	NC	0.07	0.01	0.02
900A	0.55	0.42	3.2	9.1	0.19	NC	0.21
900B	0.03	0.06	NC	3.5	1.1	0.03	0.09
1000R	1.7	NC	NC	NC	0.14	0.03	0.21
1000A	0.35	0.43	<0.01	7.2	0.48	10.8	NC
1000B	0.01	0.01	NC	>100	1.2	0.45	NC
1000E	0.32	0.03	NC	23.8	0.33	0.07	0.62

a All values expressed in percent

b Not calculated

and nitrite can result in artifically high values.

The data from the elutriate test indicated high COD values for fixed sludges 200C and 700C. Fixation process C involves an organic resin which probably resulted in the release of organic carbon to the elutriate. These high COD values for process C indicated that material was moving out of the fixed sludges to the elutriate.

High COD values for sludges 300R and 800R and fixed sludge 800B probably resulted from chloride ion interference. Sludge 800R was a brine sludge from a chlorine production process and had a high concentration of chloride ions.

Analysis of the elutriate from sludge 300R also indicated a large nitrite concentration which could interfere with the COD determination.

COD values for the elutriate from the raw and fixed sludges indicated that fixation generally reduced the migration of materials that could be oxidized by a strong chemical oxidant.

## Sulfate and Sulfite --

Sulfate and sulfite were the predominant anionic species in the elutriate from the sludges except for sludge 800 (chlorine brine sludge). The generation of sulfates and sulfites can be expected in FGD sludge production. Many of the industrial processes also involve the use of or generation of some form of sulfur compound. Sulfate and sulfite ions were also introduced into the fixed sludges from certain fixation additives which contain high concentrations of sulfate.

The predominance of sulfate ions in the elutriate was probably due to air oxidation of sulfite ions in sludge holding ponds and in the elutriate sample. Elutriate samples from sludges 500R and 1000R had much higher sulfite concentrations than the other samples. The high sodium concentrations in these double alkali FGD sludges probably resulted in soluble sodium sulfite being leached from the sludges.

Both sulfate and sulfite concentrations in the elutriate were highly variable. From the available data, fixation process E proved to be the most successful at retarding the migration of sulfate and sulfite to the elutriate. Process A also provided some attenuation of sulfate and sulfite while other processes included in the study proved only partly successful.

#### Nitrite and Nitrate --

Only small amounts of nitrite and nitrate were found in the elutriate from the sludges except for sludge 300. Elutriate from sludge 300R and fixed sludge 300B contained high levels of both nitrite and nitrate. Fixed sludge 300A produced a high concentration of nitrate in the elutriate.

Sludge 300 was generated in a nickel/cadmium battery process where nitrate salts of nickel and cadmium are used as raw materials (7). Any metallic cadmium remaining in the waste stream could reduce nitrate to nitrite (8). This reaction was probably the source of the nitrite found in the sludges.

Fixed sludge 400A resulted in a high nitrate concentration in the elutriate, but from the available data this value appears to be an anomaly. Overall, the fixation processes were generally successful in reducing the migration of nitrite and nitrate to the elutriate.

## Cyanide--

The elutriate from the FGD sludges (100, 400, 500, 600, and 1000) contained very little cyanide. Cyanide was formed in appreciable amounts in the elutriate from the fixed samples of sludge 200, sludge 300R, and fixed sludge 100F. Sludge 200 was generated in an electroplating process where cyanide baths are used to hold plating ions in solution. As a result, the low value for cyanide in the elutriate of sludge 200R may be considered questionable.

Cyanide is also used in the nickel/cadmium battery production process which probably resulted in residual cyanide in sludge 300R. The concentration of cyanide in fixed sludge 100F along with other fixed sludges may have been the result of addition of cyanide-containing fixation additives. The data indicated no significant trends in the attenuation of cyanide by the various fixation processes.

#### Chloride--

Chloride concentrations in the elutriate of the sludges were highly variable. The elutriate from sludge 800R had a high concentration of chloride. This was as expected since the sludge was generated in a chlorine production process using a brine. Fixed sludge 800A resulted in a low concentration of chloride indicating that fixation process A was successful in limiting chloride migration. The elutriate from fixed sludge 800B had a higher concentration of chloride than did the elutriate from sludge 800R. The higher concentration probably resulted from the addition of chloride by a fixation additive or an addition of a contaminant in the fixation additives which helped to solubilize the chloride. For certain other sludges, fixation process B was able to limit chloride migration. Other fixation processes were generally successful in reducing chloride concentrations in the elutriates.

### Carbonate and Bicarbonate--

The carbonate/bicarbonate determination was essentially a measure of the alkalinity of the elutriate. The particular form (carbonate or bicarbonate) found in the elutriate was pH dependent. At lower pH values, the bicarbonate form was predominant and at higher pH values the carbonate form was predominant.

Bicarbonate salts of metals are generally more soluble than are carbonate salts. For example, calcium carbonate can be leached from limestone in the form of calcium bicarbonate by the action of water rich in dissolved carbon dioxide. Other metals can react similarly. In terms of metal migration from sludges, it is therefore important to maintain a sufficiently high pH for predominance of the less soluble carbonate salt.

Carbonate/bicarbonate concentrations in the elutriates of the fixed sludges were generally high. Since carbonate/bicarbonate analyses were not conducted on the raw sludges, no comparison can be made with the fixed sludges.

--Hq

pH is an important parameter in determining the solubility of metals. Metals are generally more soluble at low pH values and less soluble at high pH values. Metal hydroxides exhibiting an amphoteric character (e.g., Be, Cu, Cr, Al, Zn, etc) do not follow this general rule (9). They form complexes at high pH values which result in increased solubility.

The pH values of the elutriate varied from the acidic to the strongly basic range. The lowest pH values were associated with fixation process C. A low pH must be maintained in the process C fixation procedure to force the fixation reaction to occur. The highest pH values were associated with sludges produced in processes that involved the use of sodium hydroxide (sludges 300, 500, 800, and 1000).

The fixation processes varied greatly in their ability to maintain or increase the pH of the elutriate. A decrease in pH was in general considered undesirable due to the resulting mobilization of metals. The metal analyses indicated a correlation between low pH values and metal mobilization but very little correlation between high pH values and metal immobilization.

## Conductivity--

Specific conductivity is a measure of the total ionic concentration in a sample. The optimum pH range for conductivity determinations is between six and nine. The conductivities due to hydrogen ions and hydroxyl ions are much greater than those due to other ions and therefore will produce invalid results when the pH of a sample is outside the optimum range (8). Therefore, elutriate samples with high pH values (300R, 300B, 400B, etc) could not be compared on the basis of conductivities.

The conductivities of the elutriates of the sludges with sample pH in the optimum range are highly variable. The data indicated that fixation processes were capable of limiting the migration of contaminants only for certain sludges. All of the fixation processes on sludge 600 were successful in reducing specific conductivity. For other sludges, success was dependent on the fixation process. The specific conductivity provided only a limited overall view of the success or failure of the fixation processes used on the various sludges.

## Metal Analysis

As mentioned previously, a large number of metal analyses were conducted on the elutriate from the sludges (Table 5). Many of the same metal analyses were conducted on the samples generated in the total digest tests (Table 6). Data from the two tests was used to determine percent solubilization for certain metals (Table 7).

### Arsenic--

High arsenic levels were indicated in the total digest test for sludges 100R, 500R, and 700R and for fixed sludge 500B. Sludges 100 and 500 are FGD sludges from power plants burning eastern coal. This coal is probably the source of the arsenic. Sludge 700 is an inorganic pigment production sludge. Some form of arsenic is probably used in or found as a contaminant in the pigment production process.

The elutriate from sludge 100R had a high arsenic concentration corresponding to the bulk analysis, but sludges 500R and 700R did not. The solubilization of arsenic in the elutriate test was generally low with most values being under one percent.

### Boron--

Boron analyses were conducted on the elutriate from the fixed sludge samples. Elutriate samples from sludges fixed by process A generally contained higher boron concentrations than samples from sludges fixed by the other processes. Elutriate from certain sludges fixed by processes C, F, and G had high boron concentration, but not as high as those from process A. It is possible that certain additives for processes A, F, and G contain boron as a contaminant.

## Beryllium--

Sludge 200R and fixed sludges 200A, 200B, and 200C were shown to contain high levels of beryllium in the total digest test. Sludge 200 is produced in an electroplating process. Some mobilization of beryllium was indicated in the elutriate test for sludge 200R. The elutriate test for fixed sludge 200C indicated a fairly high mobilization of beryllium which probably resulted from the low pH associated with fixation process C. It should be noted that the differences in beryllium concentrations for the raw and fixed sludges were a result of lowering the detection limit for beryllium after the analysis of elutriate samples from the raw sludges was conducted.

The solubilization of beryllium in the elutriate test was generally low except for fixed sludge 200C.

#### Calcium--

High calcium concentrations were indicated in each sludge by the total digest test. Lime, lime derivatives, and other compounds of calcium are used in most of the processes in which the sludges are generated as either a treatment step or as an integral part of an industrial process. Some of the additives used in the fixation processes also contain calcium compounds. Calcium concentrations in the elutriates were generally high. These values are probably strongly dependent on the amount of excess lime or other calcium compounds used in a particular process and the form of calcium contained in the sludge (i.e., sulfate, sulfite, carbonate, etc). Calcium solubilization in the elutriate test was generally in the range of 1 to 10 percent.

#### Cadmium--

Bulk analysis data indicated high cadmium concentrations in raw and fixed samples of sludges 200, 300, and 700. Sludge 200 (electroplating), sludge 300 (nickel/cadmium battery), and sludge 700 (inorganic pigment) are produced in processes that use cadmium. Cadmium was not mobilized in the elutriate test except for fixed sludges 200C, 300A, 700C, and 800A. The high concentrations of cadmium in the elutriate samples for sludges fixed by process C were probably a result of the low pH associated with process C. Since the concentration of cadmium in the elutriate of fixed sludge 800A is higher than the total digest concentration, it may be assumed that the samples used in the two tests were not homogeneous. The high cadmium value in the elutriate of fixed sludge 300A appears too high when compared with sludges 300R and 300B. This same situation

occurred for certain other metal analyses for fixed sludge 300A.

Solubilization of cadmium in the elutriate test was generally low. Only sludges 100R and 800R and fixed sludges 200C, 600B, 700C, and 900B had solubilization rates greater than one percent.

### Cobalt--

Cobalt analyses were conducted on samples from the total digest test. Varying amounts of cobalt were found in the sludges with the highest concentrations being in sludges 200R (electroplating) and 300R (nickel/cadmium battery) and fixed sludge 300B. The higher concentrations in these sludges is probably the result of cobalt contamination of metals used in the two electroplating processes.

### Chromium--

High chromium concentrations were indicated in both raw and fixed sludges for sludge series 200 (electroplating) and 700 (inorganic pigment). Significant mobilization of chromium was indicated by the elutriate test results for fixed sludges 200B and 200C. Most of the fixation processes were not successful in limiting the migration of chromium in the elutriate test. High pH associated with some of the fixation processes may help mobilize chromium since the hydroxide of chromium is amphoteric.

The solubilization of chromium in the elutriate test was generally less than one percent. The extremely high value for 800B may indicate an anomalous value for the elutriate test or the total digest test. This difference could also have resulted from nonhomogeneous samples of sludge being used in the tests.

### Copper--

Copper was found in all the sludges with the highest concentrations being found in sludge series 200 (electroplating) and 700 (inorganic pigment). Mobilization of copper in the elutriate test was high for all the sludges in sludge series 200 and for fixed sludges 700C and 900B. Copper solubilization was less than two percent except for fixed sludges 200C and 900B.

## Mercury--

Mercury concentrations were generally low in the total digest test except for sludges in sludge series 300 (nickel/cadmium battery), 700 (inorganic pigment), and 800 (chlorine production). The highest concentrations of mercury were found in sludges in sludge series 800 where a mercury cell is used in the production process.

Mercury mobilization in the elutriate test was very low with most of the concentrations being below the detectable limit of the analytical equipment used.

### Potassium--

Potassium analyses were conducted on elutriate from the fixed sludge samples. Varying concentrations of potassium were found in the elutriate from the fixed sludges but in most cases, the elutriate from sludges fixed by process B had the highest potassium concentrations. These high concentrations

in the elutriate samples were probably the result of a high potassium concentration in one of the fixation additives for process B. The elutriate from the one sludge fixed by process G also had a very high potassium concentration.

## Magnesium--

High concentrations of magnesium were indicated in both raw and fixed sludges by the total digest test. Magnesium is a common contaminant in limestone and other naturally occurring calcium compounds. As mentioned previously, most of the sludges are produced in processes where some calcium compound is used and therefore magnesium is introduced into the process as a contaminant.

Magnesium mobilization in the elutriate test was significant for sludges 200R, 600R, and 700R and for fixed sludges 200C, 500G, and 700C. Solubilization of magnesium was generally low except for fixed sludges 200C and 700C. The low pH associated with process C produced a solubilization of approximately 40 percent for both sludges.

## Manganese--

Manganese concentrations in the samples from the total digest test were variable with the highest concentration coming from sludge 700R (inorganic pigment). Mobilization of manganese in the elutriate test was generally low, less than one percent. Sludges fixed by process C showed significant mobilization of manganese. Solubilization of manganese was generally low with the exception being fixed sludge 700C which produced a 48 percent solubilization.

## Molybdenum--

The total digest test indicated that varying amounts of molybdenum were contained in the sludges. The highest concentration of molybdenum was found in sludge 700R (inorganic pigment). Molybdenum analyses were conducted on elutriate from the fixed sludges. The results indicated little mobilization of molybdenum in the elutriate test. The solubilization of molybdenum was generally below five percent.

## Sodium--

High sodium concentrations were indicated in all sludges by the total digest test. Sodium compounds are used in some of the sludge generating processes either as part of the industrial process or as a treatment step. Sodium, as a contaminant, is found in many materials used in the processes that generate the sludges. Sodium is also a contaminant in some of the fixation additives.

Sodium migration along with solubilization in the elutriate test was generally high. This was to be expected since many sodium compounds are fairly soluble. Several fixed sludges resulted in solubilizations of over 100 percent. These high solubilization values are probably due to nonhomogeneous samples being used in the elutriate test and the total digest test.

#### Nickel--

Nickel concentrations in samples from the total digest test were variable with the highest concentrations in the raw and fixed sludges from sludge series 200 (electroplating) and 300 (nickel/cadmium battery). Nickel migration

in the elutriate test was generally low except for sludge 200C. It should be noted that even with the high concentrations of nickel associated with sludge 300, very little nickel was released in the elutriate test indicating that nickel is fairly immobile under these conditions. Solubilization of nickel was generally low, less than one percent, with the exception of fixed sludges 200C and 800A.

### Lead--

The total digest test data indicated high lead concentrations for sludge series 200 (electroplating) and 700 (inorganic pigment). Sludge 300R (nickel/cadmium battery) also had a high lead concentration. Lead migration in the elutriate test was generally low except for fixed sludges 700C and 1000A. Solubilization of lead was generally low, less than 0.1 percent, with fixed sludge 700C being the exception. Overall, the data indicated that lead was fairly immobile in the elutriate test.

#### Selenium--

Selenium analyses were conducted on elutriate from raw and fixed sludges. Selenium concentrations were variable but no sludge produced a significantly high concentration in the elutriate. Selenium analyses were not conducted on samples from the total digest test and therefore no comparisons could be made.

#### Tin--

Tin analyses were conducted on samples from the total digest test. High tin concentrations were indicated for raw and fixed sludges from sludge series 200 (electroplating) and 700 (inorganic pigment).

### Vanadium--

Vanadium analyses were conducted on samples from the total digest test. Concentrations of vanadium were varible with the highest concentration being indicated from sludge 600R (FGD, lime, western coal).

#### Zinc--

High zinc concentrations were indicated in the total digest test for raw and fixed sludges in sludge series 200 (electroplating), 300 (nickel/cadmium battery), and 700 (inorganic pigment). Zinc mobility as indicated in the elutriate test was low except for sludges fixed by process C. Solubilization of zinc was generally low with sludges fixed by process C resulting in the only significant solubilization.

### COMPARISON OF FIXATION TECHNIQUES

The elutriate test was developed to provide a fast, easy method for predicting the migration of contaminants from a sludge to the environment. By comparing the migration of contaminants from sludges fixed by different processes, it is possible to determine the relative success of each fixation process. This comparison has been simplified through use of several comparative matrices. These matrices are presented in Tables 8-11. The metals included in the matrices are those for which analyses were conducted on the elutriate from both raw and fixed sludge samples. The first three matrices illustrate metal attenuation (or containment) in each sludge by each fixation

TABLE 8. METAL ATTENUATION BY FIXATION (O PERCENT)

		Fix	ation Ide	entifica	tion		
Sludge Identification	A	В	С	E	F	G	Avg
100	42 <sup>a</sup>	67	$^{ m N}^{ m p}$	83	67	N	65
200	67	58	25	N	N	N	50
300	33	67	N	N	N	N	50
400	75	83	N	75	N	N	78
500	25	55	N	64	N	45	47
600	75	58	N	83	73	N	72
700	N	N	0	N	N	N	0
800	27	36	N	N	N	N	32
900	33	17	N	N	N	N	25
1000	42	33	N	33	N	N	36
Avg	47	53	13	68	70	45	

a Expressed as percent.

b Sludge not fixed by processor or sample not evaluated.

TABLE 9. METAL ATTENUATION BY FIXATION (50 PERCENT)

		F:	1				
Sludge Identification	A	В	C	E	F	G	Avg
100	33 <sup>a</sup>	42	$^{ m N}^{ m b}$	83	58	N	54
200	42	33	17	N	N	N	31
300	25	42	N	N	N	N	34
400	50	58	N	67	N	N	58
500	8	27	N	45	N	9	22
600	50	58	N	83	64	N	64
700	N	N	0	N	N	N	0
800	27	9	N	N	N	N	18
900	17	17	N	N	N	N	17
1000	17	17	N	25	N	N	20
Avg	30	34	9	61	61	9	

a Expressed as percent.

b Sludge not fixed by processor or sample not evaluated.

TABLE 10. METAL ATTENUATION BY FIXATION (90 PERCENT)

		Fi	xation	Identi	fication	1	
Sludge Identification	A	В	C	E	F	G	Avg
100	17 <sup>a</sup>	8	$N_{\mathcal{P}}$	42	42	N	27
200	8	8	8	N	N	N	8
300	17	8	N	N	N	N	13
400	17	25	N	25	N	N	22
500	0	0	N	9	N	9	5
600	50	33	N	42	33	N	40
700	N	N	0	N	N	N	0
800	0	9	N	N	N	N	5
900	8	0	N	N	N	N	4
1000	8	17	N	8	N	N	11
Avg	14	12	4	25	38	9	

a Expressed as percent.

b Sludge not fixed by processor or sample not evaluated.

TABLE 11. ATTENUATION OF VARIOUS METALS

		Fix	ation Ide	entifica <sup>.</sup>	tion	
Metal	A	В	С	E	F	G
As	5/9	6/9	0/1	3/5	1/2	0/1
Ca	5/9	8/9	1/2	5/5	2/2	1/1
Cd	2/9	3/9	0/2	4/5	2/2	0/1
Cr	2/9	1/9	0/2	2/5	2/2	1/1
Cu	6/9	4/9	0/2	4/5	1/2	1/1
Mg	7/9	9/9	1/2	4/5	2/2	0/1
Mn	6/9	7/9	0/2	4/5	1/2	0/1
Ni	5/9	5/9	0/2	4/5	2/2	0/1
Pb	3/9	3/9	1/2	4/5	1/1	1/1
Se	4/9	6/7	0/2	3/4	2/2	0/0
Zn	4/8	6/7	0/2	3/4	2/2	0/0

process. The first matrix (Table 8) is based on a greater than zero percent attenuation of a metal; the second matrix (Table 9) is based on a greater than fifty percent attenuation of a metal; and the third matrix (Table 10) is based on a greater than 90 percent attenuation of a metal. The number of metals successfully contained attenuated is expressed as percent of the total number of metals for which analyses were conducted. For example, in Table 8 (greater than zero percent attenuation) under sludge 100 and fixation process A, the 42 indicates that out of 12 metals, the concentration of five of these was the same or lower in the elutriate of fixed sludge 100A than in the elutriate of sludge 100R. Achieving a 50 percent attenuation indicates that the concentration of a metal in the elutriate of the fixed sludge sample is less than or equal to one-half the concentration of the same metal in the elutriate of the raw sample. Similarly, a successful 90 percent attenuation indicates that the concentration of a metal in the elutriate of a fixed sludge sample is less than or equal to one-tenth the concentration of the same metal in the elutriate of the raw sample. In the case where both concentrations were below the detection limit of the analytical equipment, no comparison was made. The matrices can be used to compare the fixation processes on an individual sludge basis or on an overall basis. It should be noted that each fixation processor did not work with the same number of sludges and therefore average values may represent work conducted on only one or two sludges.

The highest success rate in Table 8 (greater than zero percent attenuation) was 83 percent for sludges 100E, 400B, and 600E. This indicates that no fixation process was able to limit the migration of all the metals to a level below the concentration of the metal in the elutriate of the raw sludge. The lowest success rate was zero percent for sludge 700C. As indicated by the average values, process F had the greatest overall success rate and process C had the lowest overall success rate. In terms of average values for the sludges, sludge 400 was the easiest sludge to fix and sludge 700 was the hardest. In general, the data indicated that the fixation processors had greater success in fixing the FGD sludges than in fixing the industrial sludges.

Table 9 contains percentage values for metal attenuation at the 50 percent level. These values indicate the percent of the total number of metals in the elutriate of a fixed sludge sample that are attenuated to a concentration level at least one-half that of the concentration level in the elutriate of the raw sludge. The highest success rate in Table 9 was 83 percent for sludges 100E and 500E and the lowest success rate was zero percent for sludge 700C. The highest average value for attenuation by a fixation process was 61 percent for fixation processes E and F. The average values indicate that the fixation processors had the highest success rate with sludge 600.

Table 10 contains percentage values for metal attenuation at the 90 percent level. These values indicate how successful the fixation processes were at achieving at least 90 percent attenuation of the metals.

The highest success rate in Table 10 was 50 percent which occurred for sludge 600A. The lowest success rate was zero percent which occurred for sludges 500A, 800A, 500B, 900B, and 700C. Considering average values for

fixation processes, process F was most successful with a success rate of 38 percent. Sludge 600 appeared to be the best sludge for the fixation processors with a success rate of 40 percent.

The data from Tables 8-10 indicate that sludge 600 was the easiest to fix and that excluding sludge 700 (only one fixation processor), the processors had the lowest success rate with sludge 900. It should be noted that the matrices consider all metals to be equal for purposes of calculating attenuation. Obviously, certain metals present a greater hazard to the environment than do others. This problem must be considered in any evaluation of a fixation technique. It should also be noted that different fixers chose to work with only certain sludges and therefore strict comparison of fixers is difficult. Such a comparison is outside the scope of this study.

A fourth matrix (Table 11) was developed to provide information on the success of each fixation process on various metals. This matrix is based on a better than zero percent attenuation. For example, under fixation process A and arsenic, the ratio 5:9 indicates that out of nine sludges fixed by process A, the concentrations of arsenic in five of the fixed sludge elutriate samples were less than or equal to the concentrations of arsenic in the corresponding raw sludge elutriate samples. If the concentrations of the metal in the two elutriate samples were both below detectable limits, then no comparison was made. The information in Table 11 indicates that cadmium and chromium were the most difficult metals to attenuate. The fixation processes appeared to have the best success with the attenuation of calcium, magnesium, and manganese.

#### SECTION 7

### INTERPRETATION OF ELUTRIATE TEST RESULTS

The data obtained from the elutriate test was used to compare the relative success of each fixation process in limiting the migration of pollutants from the sludges. The success rates of the various fixation processes were discussed in the previous section of this report. It should be noted that most of the fixation processes were only partially successful in limiting the migration of certain toxic metals (especially cadmium, chromium, and selenium). The success rates for metal attenuation were extremely low for the industrial wastes. Tables 8 and 11 indicate that the fixed sludges produced higher concentrations of certain metals in the elutriate than did the raw sludges.

A review of the fixation processes used in the study was conducted to identify factors that could contribute to the poor success of the fixation processes in limiting metal migration. Several problem areas associated with the fixation processes were identified during the review. Since all of the processes are commercial, detailed process descriptions are proprietary and cannot be included in this report. Therefore, only a general discussion of problems associated with certain fixation processes has been included. These problems can generally be associated with changes in sludge permeability, pH control, and the composition of the process additives.

The fixation processes under study reduce pollutant mobility primarily by altering the physical properties of the sludges. These properties include the surface area-to-volume ratio and the permeability. During the fixation process, if the pollutants can be trapped in a mass with a very low permeability, the leaching potential for the pollutants will be small. Any reaction taking place between the sludge and the fixation additives during or after the fixation procedure that would effectively increase the permeability of the material would probably result in an increased leaching of contaminants. Results from studies involving the corrosion of concrete due to sulfate ions have indicated two such reactions that could occur during or after certain fixation processes. (10,11) The first reaction involves the formation of gypsum from sulfate ions and calcium hydroxide by crystallization with the adsorption of two molecules of crystal water. (10) This reaction is accompanied by an increase in volume which can induce expansion of the material in which the gypsum is being formed. The resulting expansion and fracturing of the material increases the permeability.

The second reaction can occur in fixation processes where tricalcium aluminate is available. The tricalcium aluminate reacts with gypsum to produce a complex calcium sulfoaluminate hydrate.(10,11) This compound

crystallizes with the adsorption of 31 molecules of crystal water which results in a considerable increase in volume. This reaction is especially detrimental during the later stages of curing. The resulting fracturing of the fixed sludge mass will greatly increase the permeability. (The resulting swelling of the fixed sludge mass has actually been observed in a column leaching test).

The fracturing of any fixed sludge samples by these or other reactions could cause an accelerated physical deterioration of the sample during the elutriate test. The resulting increase in exposed surface area would increase the pollutant migration rate to the elutriate. In a disposal site, an increase in the permeability of the sludge mass would accelerate the migration of the pollutants to the surrounding environment.

The next major problem associated with the fixation processes is that of pH control. The pH of the sludge-additive mixture is particularly important in several of the fixation processes. For example, in one process, the pH must be maintained at a fairly low value for the fixation reaction to occur. The metals become more soluble as the pH decreases. During this fixation process, the metals in solution are trapped in interstitial water and are later released during the elutriate test. This can result in higher concentrations of certain metals being found in the elutriate of a fixed sludge.

Another problem resulting from pH control involves the increased solubility of amphoteric metal hydroxides at high pH values. Many of the fixation processes use lime or lime derivatives which tend to increase the pH of the sludge. An increase in the pH of the elutriate can result in increased concentrations of these amphoteric metals in the elutriate.

The final problem to be discussed concerns the composition of the process additives. The additives used in the fixation processes can contain high levels of contaminants. An example of such an additive is fly ash. Table 12 presents trace element compositions for several fly ash samples from different coal-fired generating stations. As illustrated in this data, the concentrations of certain trace elements are as high or higher than the concentrations in the sludges being fixed. The use of this type of additive can result in a higher concentration of certain pollutants being available for leaching from the fixed sludges. It should be noted that these higher available pollutant concentrations in the sludges will not necessarily result in higher concentrations in the elutriate because of permeability and surface area-to-volume changes resulting from fixation reactions. The effect of different additives on the performance of a fixation process will be dependent on the proportion of additives used and on other factors as discussed above.

TABLE 12. FLY ASH TRACE ELEMENT ANALYSIS (12)

Sample Number									
Element	1	2	3	4					
Sb	1.0ª	1.5	2.5	4.4					
As	3.2	74.	56.6	61.					
Ba	3600	700	750	15000					
Ве	5.2	7.5	13.1	5.2					
В	179	274	392	1040					
Cd	.39	1.4	5.3	4.2					
Cr	5.6	3.6	28.0	8.9					
F	377	83.9	210.	2880.					
Ge	1.2	12.1	25.1	9.2					
Hg	.126	<.010	.146	<.010					
Pb	6.4	17.	27.	4.0					
Mn	157.	242.	273.	374.					
Мо	7.0	6.4	5.9	12.					
Ni	34.2	75.1	108.	92.9					
Se	1.7	3.27	14.7	16.4					
Λ	<50.	<100.	<25.	<100.					
Zn	92.	102.	854.	386.					
Cu	43.	113.	59.	238.					

a All results are reported as the average of duplicate analyses. Analyses are in ppm on a dry sample basis.

## SECTION 8

## ELUTRIATE TEST AND LONG-TERM LEACHING TEST COMPARISON

#### BACKGROUND

The applicability of the elutriate test in comparing relative pollutant migration from raw and/or fixed sludges has been discussed in previous sections of this report. In order to further evaluate the applicability of the elutriate test, a comparison was made between elutriate test results and long-term leaching column results. The main objective of this comparison was to determine if the elutriate test results could be used to predict the total pollutant migration from a sludge.

Data on total pollutant migration was obtained from analysis conducted on leachate from a series of columns containing both raw and fixed sludges. The leaching of the sludges was conducted over a two-year period with samples being collected periodically. A discussion of this study can be found in an earlier project report (5). The analysis of the leachate samples taken late in the study has not been completed and therefore this report includes column leaching data for only the first 39 days of the study. This is sufficient data to provide a trend in the comparison of the leachate test results and the column test results.

After examining the vast amount of data available from the ten different sludges, the decision was made to examine only one sludge in detail with regard to a comparison between the two tests. Sludge 500 was chosen for close examination due to the amount of analytical data available on various metal species at the time the comparison work was done. Data for the comparison work included results from tests on raw Sludge 500 and fixed Sludge 500A, 500B, 500E, and 500G.

#### CORRELATION DEVELOPMENT

The amount of pollutant migration was calculated using the volume of leachate collected and the concentration of a particular metal in each sample of leachate as follows:

$$W = \sum_{i=1}^{n} VC$$

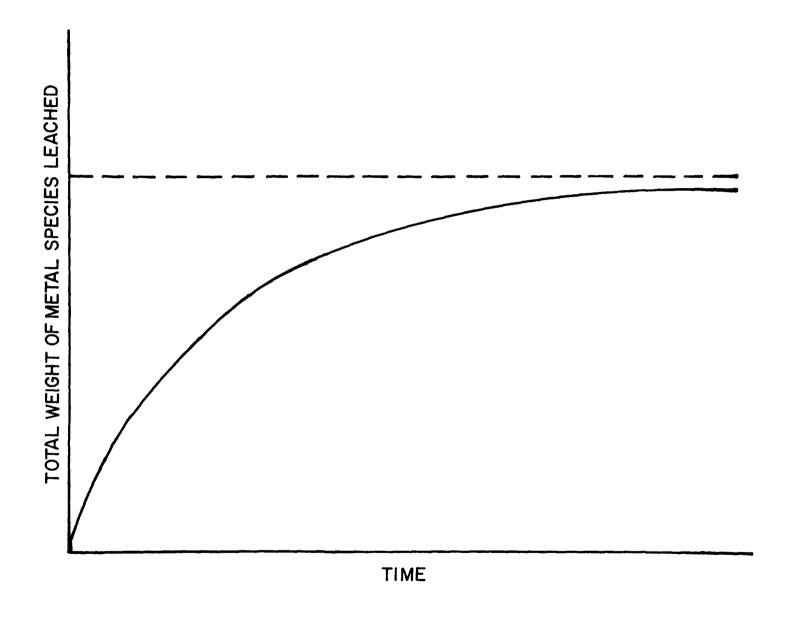


Figure 1. Total weight of metal species leached with respect to time.

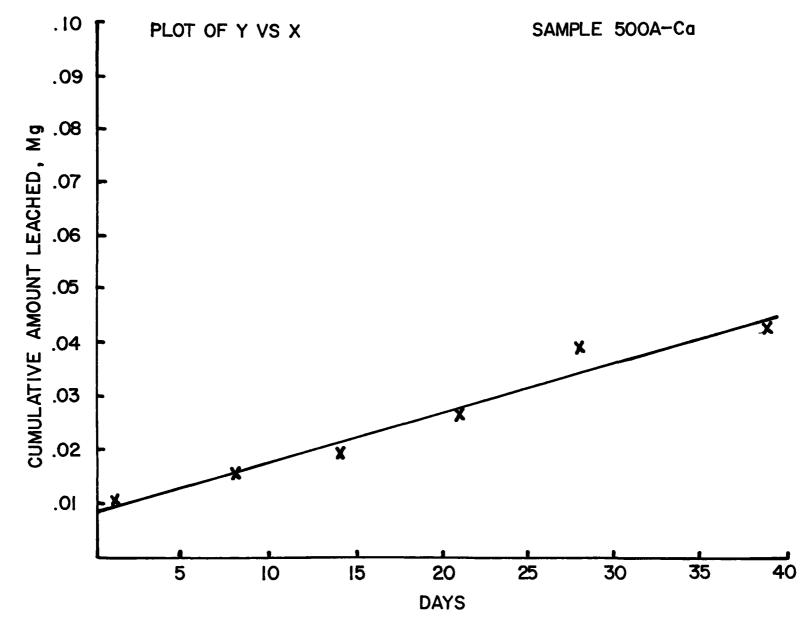


Figure 2. Sample 500A - Ca linear plot

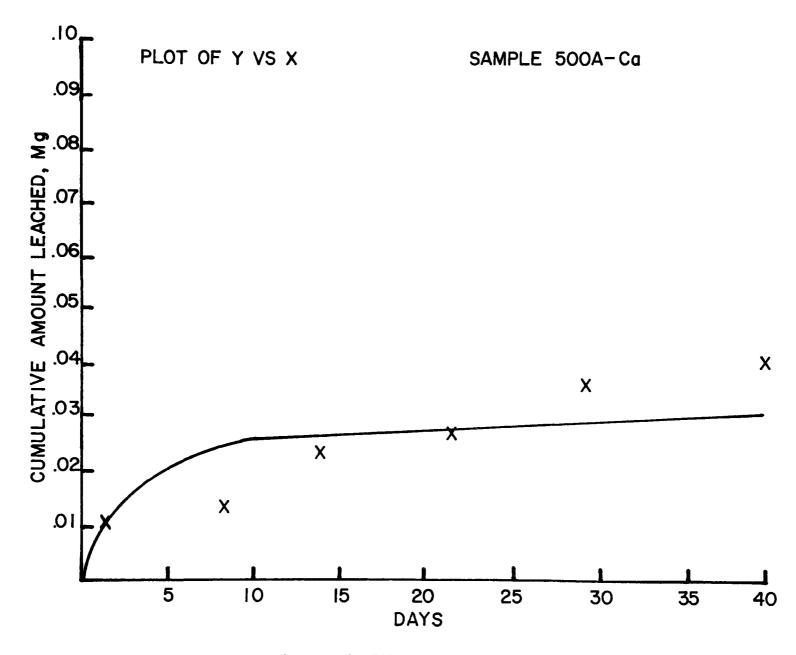


Figure 3. Sample 500A - Ca exponential plot

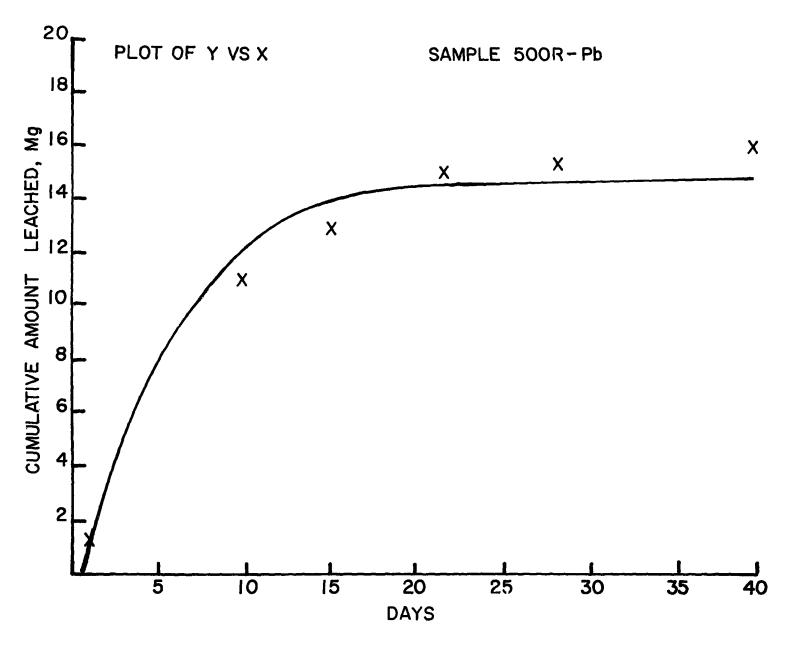


Figure 4. Sample 500R - Pb

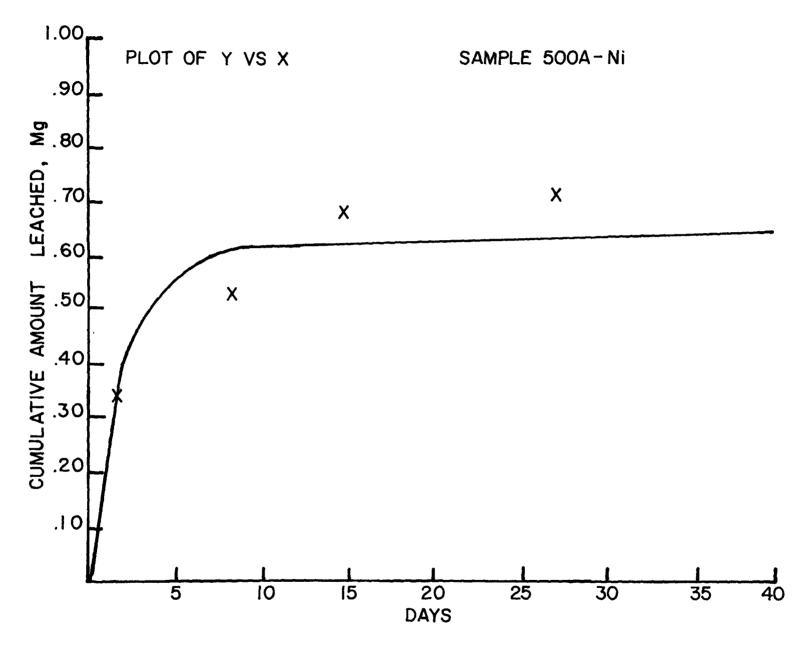


Figure 5. Sample 500A - Ni

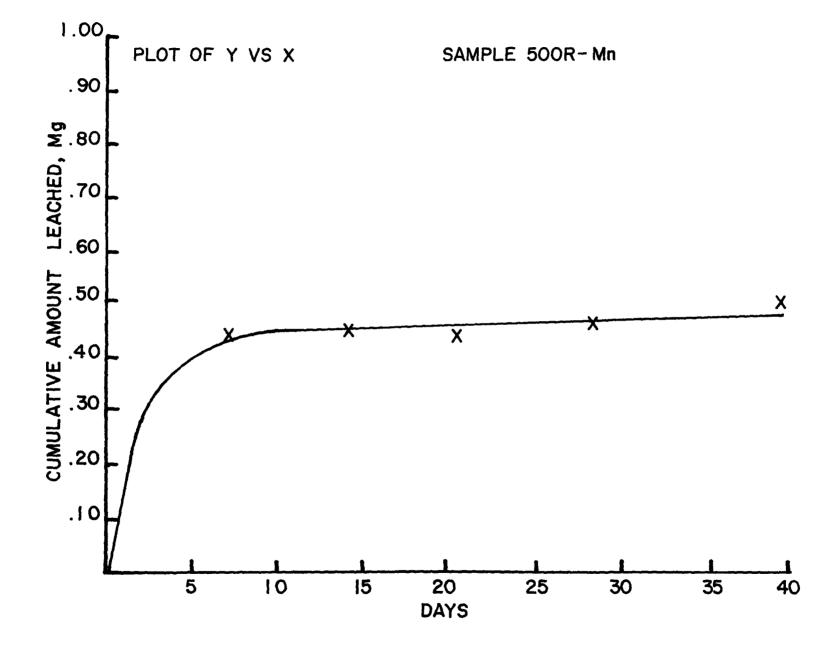


Figure 6. Sample 500R - Mn

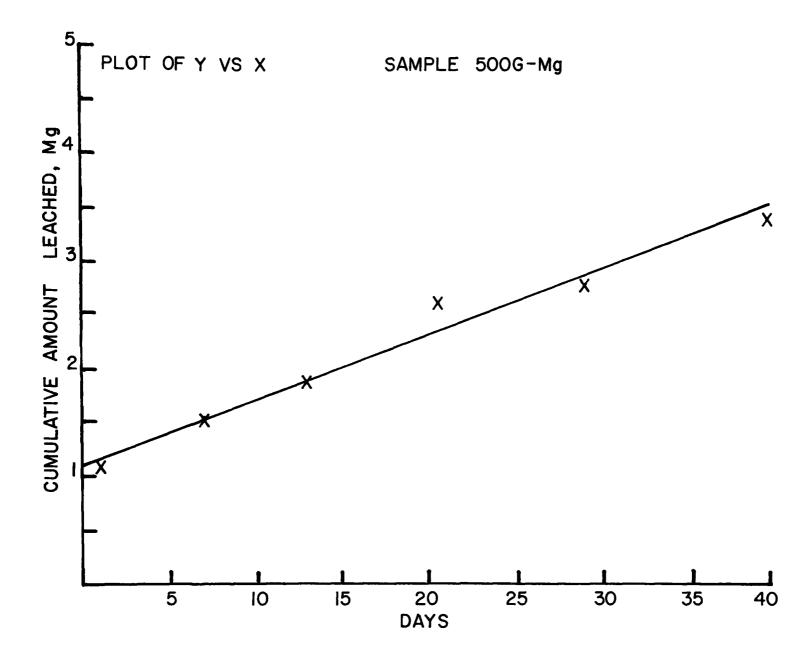


Figure 7. Sample 500G - Mg

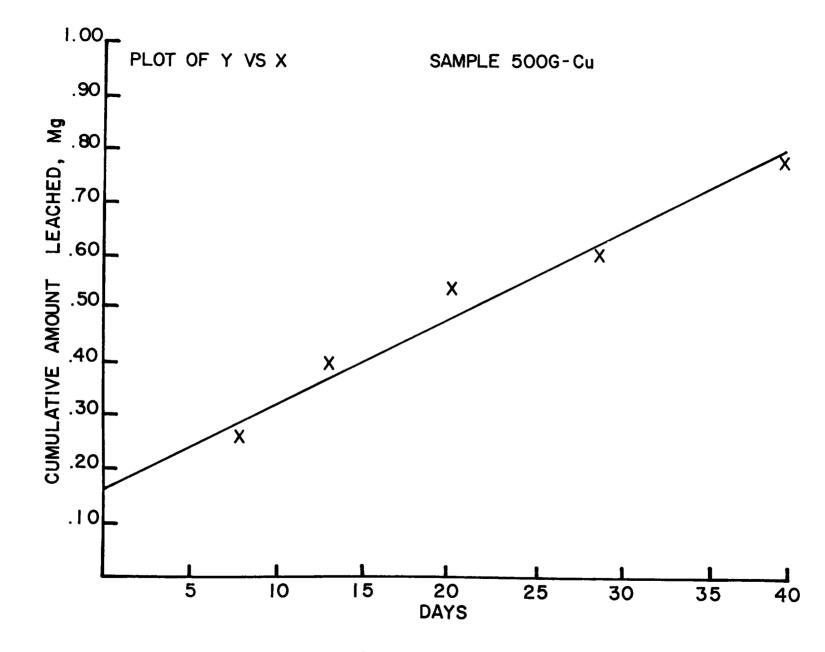


Figure 8. Sample 500G - Cu

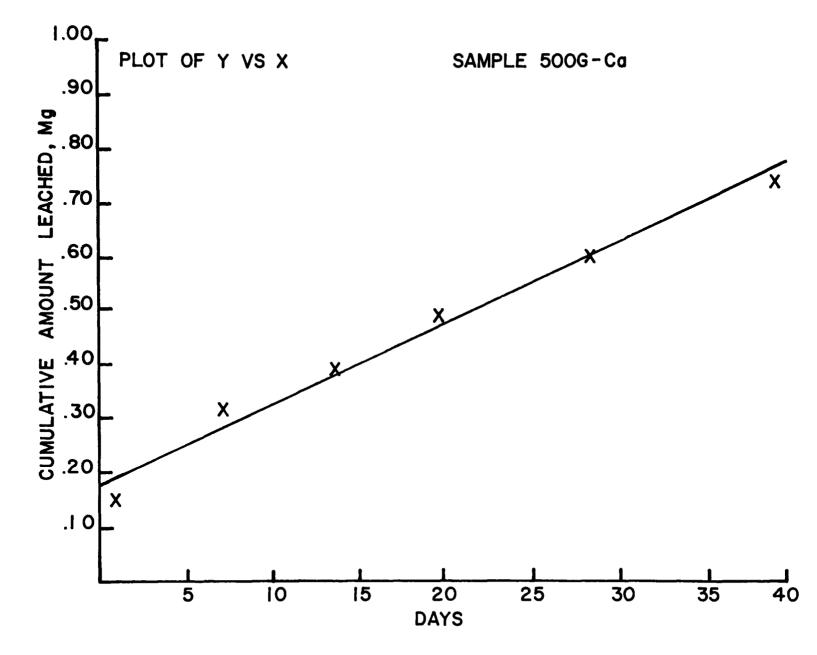


Figure 9. Sample 500G - Ca

where: W = Total weight of metal species leached in the column test (mg)

V = Volume of leachate in sample (1)

 $C = Concentration of metal species in leachate sample (mg/<math>\ell$ )

n = Total number of samples collected

It was anticipated that the total weight of a metal species leached would asymptotically approach a limiting value if the leaching test was continued for a sufficiently long period of time (Figure 1). This limiting value would indicate that most of the metal species available for leaching had migrated out of the sludge.

A comparison was made for a particular sludge between the total weight of a metal species in the column test and in the elutriate test as follows:

$$R = \frac{W}{E}$$

where: R = Ratio of the two weights

W = Total weight of metal species leached in the column test (mg)

E = Total weight of metal species found in the elutriate from the

elutriate test (mg)

The ratio R should have a limiting value of 1 if the elutriate test strictly predicts the total migration of a pollutant.

#### CORRELATION GRAPHS

A series of graphs were developed plotting the cumulative ratio of the weights of metal species leached from each test. Several of these graphs are presented in this report as examples. In each graph, the data points were used to develop a linear regression line of two types (a) a straight line and (b) an exponential curve (Figures 2 and 3). The exponential curve should have provided a good fit if the data points were asymptotically approaching a constant.

The graphs indicated that the exponential curve provided a fairly good fit for certain metals such as manganese (Figure 4), nickel (Figure 5), and lead (Figure 6). The straight line provided a good fit for most of the other metals including calcium (Figure 7), copper (Figure 8), and magnesium (Figure 9). The graphs also indicated that in general the cumulative ratios exceeded a value of 1.

#### INTERPRETATION

The difference between the theoretical correlation and the actual correlation is probable due to several factors. The first factor concerns the solubility of the metal species in the leachate. The solubility of a metal species limits the amount of that metal that can be transported out of the sludge. Metal solubilities are dependent on elutriate pH and temperature, other species in solution, and the chemical form of the particular metal being taken into solution.

In the elutriate test, if the solubility of a metal species was limiting, the test would result in a low indication of metal available for leaching. This phenomenon could easily occur for metals such as calcium and magnesium or for any other metals which are found in high concentrations in the sludges. In this case, the cumulative ratio of the weights of metals species leached from the two tests would be greater than 1.0 since the amount of metal found in the elutriate was limited.

In the column test, if the solubility of a metal species was limiting, a large number of column volumes of leachate would be required to elute the metal. This would result in a fairly constant concentration of the metal species in the leachate resulting in a linear relationship between the cumulative ratio and time. This linear relationship would deteriorate then enough of the metal species was eluted so that solubility is no longer the limiting factor.

Several other factors resulting from differences in testing procedures could also have resulted in the discrepancy. One factor that must be considered is the difference in weight of the sludge samples in the two tests. The much larger weight of sample in the column test could supply much more of a particular metal species than the sample in the elutriate test. This factor would result in a cumulative ratio much higher than 1.0.

Next, the samples from the elutriate test were filtered as a result of separating the sludge from the elutriate. The samples of leachate from the columns were not filtered which could have permitted particulate matter to pass into the leachate samples. The samples were fixed with acid and the resulting drop in pH probably solubilized any metals associated with the particulate matter. This situation would result in a cumulative ratio higher than 1.0.

As mentioned previously, many of the sludge samples deteriorated during the elutriate test due to the shaking procedure in the test. This deterioration resulted in a higher surface area-to-volume ratio for the sludges in the elutriate test than in the column test where most of the sludges did not deteriorate. This higher surface area-to-volume ratio could produce a higher metal migration rate resulting in a cumulative ratio less than 1.0.

The final factor involves the analytical detection limits for the various metals. The concentrations of some metals in the leachate samples from the columns fell below the detectable limit after a period of leaching. Small quantities of these metals were being leached out continuously but could not be detected. This factor could result in an indication of a ceiling for the cumulative ratio whereas the cumulative ratio was actually increasing in value.

## DISCUSSION OF RESULTS

The correlation study between the elutriate test data and the columns test data indicated some problems with the elutriate test in its present form. The solubility problem associated with the elutriate test could probably be solved by repeating the test on the same sludge sample until the concentration

of the metal species of interest is not solubility limited. The solubility problem associated with the column test could be allevaited by continuing the test until the metal species is sufficiently leached so that solubility is not a limiting factor. This extended leaching test may be prohibitive in terms of the time needed to complete the test.

The problem of difference in weights of sludge used in the two tests can be solved by applying a correction factor to the results of the elutriate test. This factor is the ratio of weights of the two sludges as used in each test.

The other problems are not so easily solved and are due primarily to basic differences in testing procedures. Modifications would be required to both tests such as reducing the shaking action in the elutriate test, filtering the leachate samples from the columns or not filtering the elutriate samples, and changes in analytical techniques.

Although the elutriate test as described in this report has proven useful in comparing relative pollutant migration from raw and/or fixed sludges, several modifications of the elutriate test procedure are necessary before the test can accurately predict total pollutant migration as indicated in the column test study.

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15. SUPPLEMENTARY NOTES

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#### 16. ABSTRACT

A distilled water shake test, the elutriate test, was developed and tested to provide a fast, simple, procedure for predicting the escape of pollutants from treated and untreated sludges. The preliminary test consisted of subjecting various treated and untreated flue gas desulfurization (FGD) and industrial waste sludges to the elutriate test procedure and measuring the levels of a wide variety of constituents in the elutriate and comparing these with analyses of digested sludges. The resulting data are presented as percent attenuation and a comparison is made between treated and untreated wastes.

The shortterm elutriate test results were compared to results of a longterm leaching test using the same treated and untreated sludges. The results suggest that the elutriate test may be useful in predicting the pollutant potential of various treated or untreated wastes. Further research and modifications are suggested to improve the predictive value of the test.

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was conducted during the period of August 19/6 through August 19/7. DESCRIPTORS b. IDENTIFIERS/OPEN ENDED TERMS | c. COSATI Field/Group Leachate, Solid Waste Wastes, Stabilization, Leaching, Sludge, Permeability, Pollution, Sulfates, Management 13B Flue Gas Cleaning Sulfites Chemical Stabilization (Fixation) 18. DISTRIBUTION STATEMENT 19. SECURITY CLASS (This Report) 21, NO, OF PAGES UNCLASSIFIED 71 20. SECURITY CLASS (This page) UNCLASSIFIED 22. PRICE RELEASE TO PUBLIC