United States
Environmental Protection
Agency

Water Engineering
Research Laboratory
Cincinnati OH 45268

Research and Development

EPA/600/S2-85/058 Aug. 1985

EPA

Project Summary

Solidification/Stabilization of Sludge and Ash from Wastewater Treatment Plants

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Tests were performed to determine the physical properties and chemical leaching characteristics of the residuals and the stabilized/solidified products from two publicly-owned wastewater treatment works (POTW). The two POTW waste products included in this study were an anaerobic digester sludge from an Imhoff digester and an ash from a rotating hearth incinerator used to destroy primary settler and digester sludges.

Three different solidification/stabilization systems were used. One of the systems was based on the addition of cement and soluble silicates in various proportions and formed soil-like solids that were soft and easily broken. A second system used lime and flyash to form a pozzolanic material that produced a hard, concrete-like solid. The third system was based on the formation of gypsum in the waste after acidification; these products remained wet and did not harden. The Imhoff sludge (anaerobic digester waste) was treated only with the cement-soluble silicate process; the incinerator ash was treated using all three processes.

None of the treated products were very durable, as none survived the full sequence of 12 cycles and the wet-dry or freeze-thaw testing. The concrete-like, lime and flyash solid had the highest durability, surviving 11 freeze-thaw and 5 wet-dry cycles. The soil-like products survived two or fewer cycles of both durability tests. The gypsum-based product remained moist and putty-like and could not be tested.

The pozzolanic, flyash-lime product reduced the loss of constituents to the leaching medium to the greatest extent. It also produced by far the smallest increase in the weight of the waste to be disposed for any of the processes—170% of the dry sludge solids. These facts coupled with the low cost of the solidification agents make this process the most cost-effective of those tested in this study.

This Project Summary was developed by EPA's Water Engineering Research Laboratory, Cincinnati, OH, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

The effects of contaminating groundwater and surface water with residues from publicly owned sewage treatment works (POTW) are not completely known. POTW wastes are sludges with high-organic content that can be disposed of directly or further treated by incineration, pyrolysis, or composting to lower the water and organic matter content before disposal.

The potential for unacceptable levels of groundwater contamination has been documented in several cases where POTW wastes were disposed of in lagoons, landfills, or land farms. Elevated levels of certain metals (cadmium, copper, iron, lead, magnesium, manganese, silver, and zinc) and the nutrients ammonia and nitrate have all been reported in at least one of several studies of pollutant losses from POTW waste disposal sites. In one study, 85 cases of groundwater and/or surface water contamination were associated with leaky sewage sludge impoundments in 29 States. Several responses to this problem are being actively pursued under government and private auspices. Special land treatment or land cultivation techniques are also under active investigation for sewage sludges. Systems for isolating lagoons and landfills with impervious liners and covers or other containment strategies are also under development.

This study is concerned with another alternative—that of chemically stabilizing, fixing, or structurally isolating the POTW wastes in a suitable matrix so that the resulting treated product can be safely handled, transported, and disposed of using established methods of landfilling or burial. Chemical fixation and solidification/stabilization are general terms used to designate processes that can be used to immobilize, isolate, or otherwise contain wastes. Solidification suggests the production of a solid, monolithic mass with sufficient structural integrity to be handled, transported, and disposed of in some conveniently sized pieces without requiring any secondary containers. Stabilization implies the immobilization of toxic substances by inducing chemical reactions to form insoluble compounds, or by entrapping the toxic element or compound in a watertight, inert polymer or stable crystal lattice. In stabilization, much of the emphasis has been placed on preventing the waste from coming into contact with leaching water, or on creating pH and/or oxidation-reduction conditions that minimize the solubility of the toxic constituents. Many fixation systems combine these two concepts by producing an impermeable mass that isolates the waste (or reduces its leachable surface area) and also maintains minimum solubility of the toxic components.

The three solidification/stabilization processes included in this study are those marketed by companies that volunteered to treat the POTW wastes; they do not necessarily represent a cross-section of those processes available commercially or being developed at this time. One is a pozzolan-based process using flyash and lime as the major treatment reagents; another uses cement and soluble-silicate; and the third is a process that attempts to produce solid gypsum in the waste after acidification.

The POTW residues selected for use included an anaerobic digester (Imhoff) sludge of relatively high organic content and an ash from an open-hearth incinerator.

The leaching procedure used in this study incorporated several unique aspects compared with those found in the literature. The sample specimens were of a size and shape (a right cylinder 5 cm high × 5 cm diam) suggested for use in the universal leaching procedure (ULP), which is a proposed standard test for solidified wastes. The sample specimens had a surface-to-volume ratio of 1.20/cm. The leaching samples were placed in a cellulose thimble to prevent particulates from being lost to the leaching medium. The thimble also allowed the leach testing of untreated and treated specimens using identical protocols. The volume of leaching medium (1 L) was 10 times that of the waste and was changed daily over the 90-day leaching test.

Methods and Materials

Description of POTW Wastes

The wastes used in this study represent typical residuals produced in POTW operations: ash from sludge incineration and an anaerobic digester sludge of relatively high organic content. The two wastes were obtained from two wastewater treatment plants—the ash directly from a rotating-hearth incinerator and the sludge from the drying bed associated with the operation of an Imhoff digester tank. Both treatment plants received waste from industrial areas that contain heavy metals, so both residues cause some disposal problems.

The Imhoff tank sludge is an air-dried, felt-like material containing recognizable debris such as hair, plastic scraps, and rubber bands; it has little odor and has a greenish to grey-black color. This sludge has been marketed as a soil additive. The incinerator ash is a dry, light powder, brown to orange in color, with a few large (5- to 10-cm diameter, black, slag-like agglomerates. The ash was collected directly beneath the incinerator gratings.

The constituents determined in the chemical analyses (Table 1) were chosen based on previous studies that indicated they were important indicators of leaching activity. The Imhoff sludge had a much higher percent water (42.3% w/w compared with 22.8% w/w for the ash and total organic carbon than the incinerator ash. The sludge also generally had higher levels of most heavy metals (especially chromium, selenium, copper, iron, and zinc) and volatile constituents such as sulfate and cadmium. Of the toxic metals, only manganese was present in higher concentrations in the ash.

Stabilization Techniques

After thorough mixing, each sludge was sampled using standard procedures, and samples were stored in sealed plastic containers maintained at

Table 1. *Concentration of Selected Constituents in POTW Wastes (mg/kg dry sludge solids)**

Parameter	*Imhoff Sludge*	*Incinerator Ash*
Arsenic	*17.4*	*13.4*
Cadmium	*213*	*2.26*
Chromium	*3,060*	*566*
Copper	*1,370*	*750*
Iron	*22,600*	*16,800*
Lead	*1,130*	*1,160*
Manganese	*283*	*783*
Selenium	*2.65*	*0.20*
Zinc	*4,140*	*705*
Sulfate	*1,880*	*6,200*
Chloride	*333*	*455*
Total organic carbon	*127,000*	*1,700*

**All values in mg/kg dry sludge solids as digested in hot, concentrated nitric acid.*

room temperature until use. The relative amounts of dry sludge solids, dry reagents, and water in the treatment processes for each sludge processed are listed in Table 2. Specific information on the additives used by each processor is not given, as the formulations are proprietary; only the general category of each additive is given when known.

Process X

Process X processed both sludge types as shown in Table 2. The waste and water were slurried in a cement mixer before the proprietary dry reagents (cement and other materials) were added. A liquid reagent was then added, and the mixture was pumped into the molds. The molds were covered with plastic and set aside to cure.

Process Y

Mixes were prepared in a container equipped with a turbine mixer. After the ash was adjusted to optimum moisture, 5% additive (on a dry weight basis) was added. The final mixture was placed in the molds, compacted with a hand tamper, and covered with plastic to cure.

Process Z

The ash, water, and acid reagents were mixed using a propeller mixer. More than an hour was required to dissipate the considerable heat produced. Then a lime and water slurry was added while mixing continued for another 30 min. The residue was placed in containers to settle. The treated waste had to be filtered to remove excess moisture before it could be compacted in molds.

Leaching Apparatus

The leaching apparatus (Figure 1) allows the manipulation of such parameters as contact time, surface area, and leachate quantity and renewal frequency. For this study, leaching fluid was renewed daily, and samples of leachate for chemical analysis were taken weekly for the first month, biweekly for the next month, and monthly thereafter for a total of 90 days of leaching (pH and conductivities were taken daily). All leaching tests were run and sampled in triplicate so that a total of 192 leachate samples were generated for each test. The leaching vessel was constructed of materials that were inert with respect to the waste and leaching fluid. Blanks were carried through each set of tests.

Table 2. *Formulation of Treated and Untreated Waste Samples Used in Physical and Leach Testing**

	Formulation				
Sludge and Process Code	*Dry Sludge Solids (%)*	*Treatment Reagents (%)*	*Water (%)*	*Core Weight (g)*	*Dry Solids in Core (g)*
Imhoff Sludge					
Untreated	*57.6*	-	*42.4*	*121.2*	*69.9*
XA	*5.5*	*26.0*	*68.5*	*104.8*	*66.4*
XB	*15.8*	*17.7*	*66.6*	*105.2*	*65.6*
Incinerator Ash					
Untreated	*77.2*	-	*21.8*	*67.4*	*52.0*
X	*20.6*	*11.3*	*68.1*	*106.0*	*62.2*
Y	*(59)†*	*(3)*	*(39)*	*134.3*	*87.4*
Z	*20.0*	*15.6*	*64.4*	*132.7*	*67.9*

**All data are averages of three cores used in leaching experiment. Variations in weight were less than 5%.*
†Formulation for Process Y is not known because of unspecified water additions. Figures given are estimates derived from chemical analyses and vendor information.

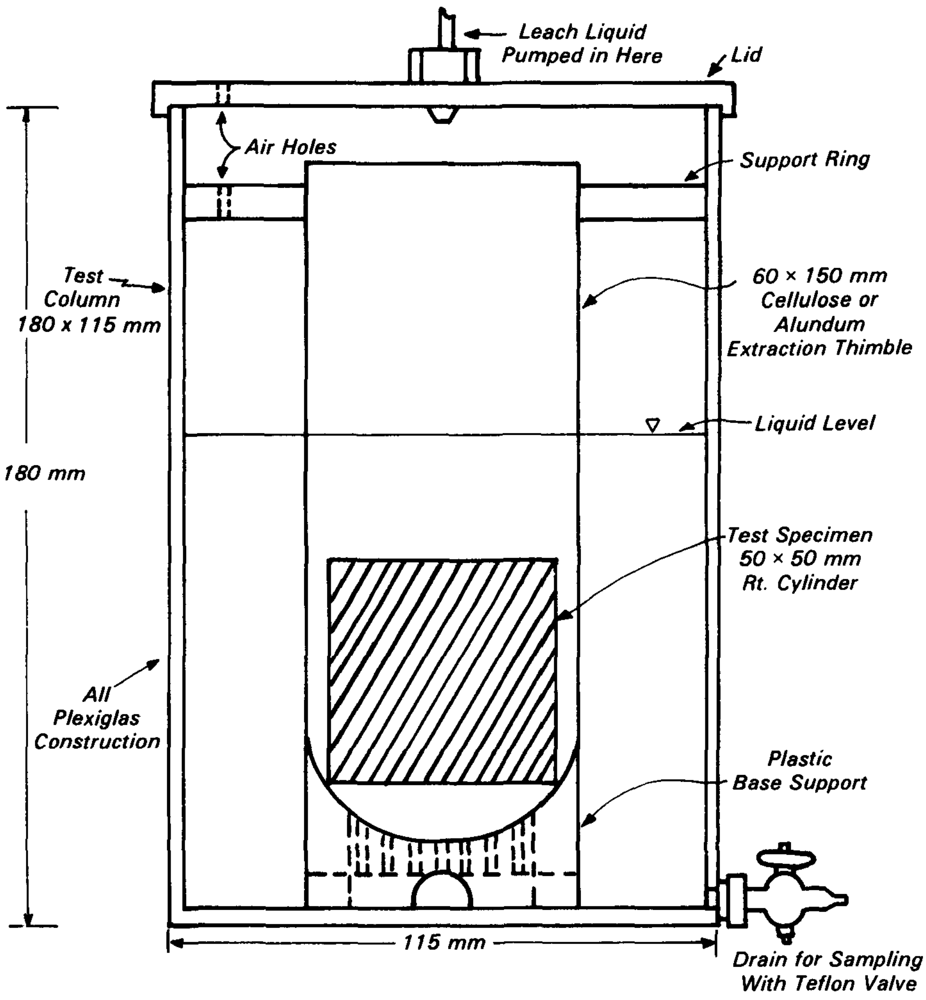

Figure 1. *Design of leaching apparatus.*

The test specimen (a 50- × 50-mm right cylinder) was supported in the leaching medium (carbon-dioxide-saturated, deionized water at pH 4.0 to 4.2) in a 60- × 150-mm cellulose extraction thimble that was supported both at the top and the base. The cylinders of untreated samples were formed in a small pellet press. All treated samples were cored from the solid or semisolid products cured in large molds.

All chemical analyses and sample preservation were done using EPA-approved methods. The leaching fluid was monitored throughout the testing, and a triplicate set of vessels without waste samples was run as a control. In all cases, blank corrections were insignificantly small.

Physical Testing

Physical testing of the treated samples was performed to assess their strength, durability, and trafficability. Cores of the treated wastes 10 cm in diameter were submitted for testing for moisture content, unconfined compressive strength, cone penetration, and freeze-thaw and wet-dry durability.

Results

Leach Testing

The leaching test was designed to define quickly and accurately the potential loss of constituents from treated and untreated sludges. The waste samples frequently broke apart or sloughed (except those of process Y) in the cellulose extraction thimbles, increasing their surface area and accelerating constituent losses. But suspended materials were confined to the extraction thimble and were not collected in the leachate samples, so not all constituents in the leachates were in solution. Chemical factors such as solubility, diffusion rates, and adsorption/desorption were probably of major importance to the leaching behavior of the materials in the vessels because of the small sample size, its complete submersion, and the daily replacement of the leaching fluid.

Three patterns of leaching were apparent for the constituents of the sludges studied here. For highly soluble constituents such as chloride and sulfate, most leaching took place in the first few changes of leaching fluid, their concentration quickly dropping to a constant, low value. The more insoluble constituents (lead, manganese, and chromium, for example) showed relatively constant, low concentrations throughout the test period. The third pattern consisted of low initial concentrations followed by higher levels later in the leaching period resulting from pH changes, common ion effects, or ion exchange phenomena. The pH of the leachate became lower (more acidic) in all vessels throughout the leaching period, especially in the untreated wastes.

The results of the leaching tests are summarized as the total mass of each constituent leached averaged for the triplicate samples. These data are shown in Tables 3 and 4. Other presentations and derivations of the data are given in the complete report along with a thorough statistical analysis.

The three treatment processes produced very different treated waste products. Only those of process Y were truely solidified into a monolithic block with good structural integrity. This solidified product also contained the sludge constituents best of all the treatment processes, releasing smaller amounts of most metals and anions than the untreated incinerator ash. Leachates from process Y-treated ash also had the lowest conductivities and caused less change in leaching fluid pH (indicating little interaction) than those of the other treated specimens.

Process X produced a cured material that looked liked a hardened, clay-soil material that crumbled easily into small chunks. The samples produced from the Imhoff sludge by this process were much softer, perhaps because of the higher organic content (Table 1). Overall, the specimens produced by process X from both wastes lost smaller amounts of many constituents to the leaching medium than the respective untreated wastes. Leachates from these samples were more alkaline than those of any other treated or untreated samples, indicating the strongly alkaline character of the treated material. After 2 or 3 weeks of leaching, the conductivities of the leachate from these samples were generally higher than those of the untreated wastes after a comparable period of leaching, meaning that the long-term levels of dissolved materials in their leachate exceeded those of the untreated sludges. This fact should be considered in any future evaluation procedures or leaching test designs.

Process X was the only formulation used to treat both waste types. Since treatments XB of the Imhoff sludge (Table 3) and X of the incinerator ash (Table 4) were most similar in the amounts of dry solids and treatment reagents, they gave the best basis of comparison of the treatability of the two

Table 3. *Total Mass of Constituents Leached from Treated and Untreated Imhoff Digester Sludges (mg)**

Parameter	*Untreated Imhoff Sludge*	*Process X, Treatment A (XA)*	*Process X, Treatment B (XB)*
Arsenic	*0.742*	*0.049*	*0.020*
Cadmium	*3.30*	*0.126*	*0.341*
Chromium	*0.566*	*1.15*	*3.29*
Copper	*3.86*	*3.20*	*12.38*
Iron	*48.4*	*1.57*	*11.86*
Lead	*0.238*	*0.019*	*0.040*
Manganese	*11.5*	*1.48*	*2.48*
Selenium	*BDL†*	*0.255*	*0.005*
Zinc	*120*	*3.02*	*12.22*
Sulfate	*2680*	*621*	*1440*
Chloride	*130*	*28.3*	*65.0*
Total organic carbon	*199*	*190*	*654*

**All data are the means of three replicates.*
†Below detection limits in all leachate samples.

wastes. The treated products of both sludges had significantly lower masses leached than their respective untreated sludges for arsenic, cadmium, manganese, zinc, sulfate and chloride; but both leached higher masses of chromium, copper, and total organic carbon (TOC) than did their untreated counterparts. Both of these treated products had similar physical properties. Both the Imhoff sludge products contained high levels of TOC and heavy metals, and the relative containment properties of the two treated specimens were quite similar.

Two different levels of solids and treatment additives were used by process X on the Imhoff sludge (see Table 2). Treatment XA was designed to give optimum containment, and treatment XB was typical of commercial treatment proportions. Although both treated specimens yielded leachates significantly lower than the untreated sludge leachates in arsenic, cadmium, manganese, zinc, sulfate and chloride, treatment XA lost significantly lower levels of chromium, copper, manganese, sulfate and TOC than treatment XB specimens (see Table 3). The much higher relative additive level in XA specimens did increase the containment efficiency for some constituents and did increase the strength to a small degree (Table 5). Selenium, however, appears to be added in the treatment process, since none was detected in the untreated sludge leachate, and since process XA leached significantly more selenium than process XB (the reverse of that for the constituents listed above).

The treated product from process Z remained wet and did not harden. At the suggestion of the vendor, the material was filtered and air-dried before being tested. Even after this processing, the material remained wet and would not hold its shape. Process Z specimens lost the largest proportion of their constituents in the leaching test and had the most acidic leachates of all samples tested. The addition of acidic reagents to the ash apparently brought many metals into solution; also, the level of sulfate in these leachates was extremely high and persistent.

Table 4. *Total Mass of Constituents Leached from Treated and Untreated Incinerator Ash (mg)**

Parameter	*Untreated Incinerator Ash*	*Process X*	*Process Y*	*Process Z*
Arsenic	*0.472**	*BDL†*	*0.511*	*2.19*
Cadmium	*0.046*	*0.044*	*0.0185*	*0.083*
Chromium	*0.336*	*0.637*	*0.0627*	*0.038*
Copper	*2.77*	*0.389*	*0.156*	*1.20*
Iron	*BDL*	*BDL*	*BDL*	*8.00*
Lead	*0.325*	*0.042*	*0.074*	*0.625*
Manganese	*5.61*	*3.56*	*0.10*	*11.7*
Selenium	*BDL*	*BDL*	*BDL*	*BDL*
Zinc	*4.48*	*BDL*	*BDL*	*1.38*
Sulfate	*1050*	*656*	*111.5*	*8970*
Chloride	*102*	*55.0*	*BDL*	*BDL*
Total organic carbon	*70.6*	*30.8*	*30.2*	*42.2*

**All data are means of three replicates.*
†Below detection limits in all leachate samples.

Table 5. *Summary of Physical Properties of the Treated Wastes*

	Imhoff Sludge		*Incinerator Ash*		
Physical Property	*XA*	*XB*	*X*	*Y*	*Z*
Free moisture content (%)	*36.6*	*37.7*	*41.3*	*34.9*	*49.8*
Cone penetrometer					
Center (cm)	*0.8*	*0.8*	*0.4*	*ND**	*ND*
Edge (cm)	*1.4*	*Fractured*	*1.4*	*ND*	*ND*
Unconfined compressive strength					
Load (N)	*100.8*	*114.3*	*118.1*	*169.9*	*ND*
N/sq cm	*24.3*	*27.6*	*25.5*	*41.6*	*ND*
Durability tests (cycles to failure)					
Freeze/thaw	*2*	*2*	*2*	*11*	*ND*
Wet/dry	*2*	*2*	*2*	*5*	*ND*

**Not done on this sample.*

Physical Testing

A comparison of the physical properties of the treated sludges permits judgments about the relative ability of the treatment processes to improve the handling characteristics of the wastes and to provide durable materials suitable for landfilling. Results of the physical properties testing are summarized in Table 5.

All of the treated products made using process X had physical properties similar to a crumbly soil-cement with low strength durability. All contained between 37% and 42% free moisture (not bound in hydration reactions). Process-Y-treated ash was the hardest and most durable material and had the lowest free moisture. The treatment products from process Z did not harden and were not tested.

Conclusions

The treatment processes did not always change the physical properties of the wastes in a way that would insure production of a solid. Only one of the treatment processes produced a material that resembled low-strength concrete in character—the pozzolanic flyash-lime process applied to incinerator ash. Other treatment of the incinerator ash produced a soil-like product and a wet, plaster-like product that did not harden even after filtering and air-drying. The two formulations used on the Imhoff sludge both produced soil-like products. The durability of the final products was low; no product survived the full 12 cycles of the wet-dry or freeze-thaw testing (Table 5).

The leaching procedure used in this testing program was designed to control many of the factors that affect leaching rates, such as surface area, leaching time, waste-to-leaching-fluid ratio, and loss of particulates. The concentration of constituents in the leachates from the test samples followed one of three distinct patterns:

1. Highly soluble constituents (e.g., sulfate and chloride) were leached in high concentrations initially, but very quickly reached low, stable concentrations near or below the detection limits.
2. Less soluble constituents (e.g., lead and chromium) were found at relatively constant levels in the leachates over the leaching period. In several cases (cadmium, copper, manganese, and zinc) the initial leachate samples contained elevated amounts before the stable concentration was obtained.
3. Some constituents, like arsenic and iron, were at or below the detection levels initially but increased in concentration in later samples, apparently as the pH of the leaching liquid became more acidic.

The leaching tests indicated that several of the processes significantly slowed the leaching of selected constituents. Overall, both formulations used in treating the Imhoff sludge significantly slowed the leaching of most soluble constituents. The formulations with the higher reagent-to-sludge ratios were more effective, as predicted. With incinerator ash, the gypsum-based process was not effective in solidifying the waste or in limiting the loss to the leaching fluid. The lime-flyash process product was the most effective treatment for both solidification and constituent containment.

The one process used on both types of waste was the cement/soluble silicate system. The results obtained by this process showed no difference in the relative effectiveness of treating the two waste types. The appearance, physical properties, and leachability of the two wastes fixed by the same process were quite similar.

Only process Y produced a stabilized product with more than 20% dry waste solids (by weight). This process contained about 60% dry waste solids, so that the weight after treatment was only about 1.5 times that of the original dry waste. The other processes increased the amount of waste to be landfilled by 4 to 10 times the original dry waste weight. The increase was due largely to the addition of large proportions of water (65% to 70% of the final weight of the treated product). In evaluating the commercial use of solidification/stabilization, the increased mass for disposal must be considered along with the improved pollutant containment afforded by successful treatment.

Recommendations

Solidification/stabilization as used in this preliminary study represents a method of reducing the pollutant potential of POTW wastes when they are disposed of in landfills. The results appear to warrant further testing and evaluation. Specifically, actual field tests using large-scale processing equipment and treated waste samples may show that the material behaves differently from the small samples used in this study. Large-scale, controlled tests using treated waste samples that have surface-to-volume ratios more typical of those actually encountered in landfill situations are needed to give more realistic estimates of treatment benefits. The economics of the process and the increase in waste mass for disposal should also be carefully addressed. Intermittent saturation of the treated samples in contrast to continuous submersion should be considered in future testing.

This study incorporated treatment processes with completely different physical and chemical bases. Tests need to be made using one overall process type to optimize its additive, curing, and mixing requirements. All of these studies should be compared with the results of a standardized solidification technique. Comparing selected physical properties with the leaching characteristics of each process type might reveal those properties of the treated products that can predict their containment efficiencies.

A standardized leaching test must be developed to facilitate valid comparisons and to increase the predictive value of bench-scale leaching tests. An optimum standardized leaching test would be expected to mimic the conditions under which maximum leaching takes place, (i.e., be a worst-case test). Characteristics of such a test would be a short term of leaching, a small and uniform test sample size, an aggressive leaching fluid, and particulate containment for nonsolidified samples. The leaching apparatus developed for this study warrants further use in the development of a standardized leaching test.

The full report was submitted in fulfillment of Interagency Agreement No. EPA-IAG-07-0046 by U.S. Army Engineer Waterways Experiment Station under the sponsorship of the U.S. Environmental Protection Agency.

☆U. S. GOVERNMENT PRINTING OFFICE: 1985/559-111/20628

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The complete report, entitled "Solidification/Stabilization of Sludge and Ash from Wastewater Treatment Plants," (Order No. PB 85-207 504/AS; Cost: $11.50, subject to change) will be available only from:
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