

MARINE SCIENCES RESEARCH CENTER
STATE UNIVERSITY OF NEW YORK
STONY BROOK, NEW YORK 11794

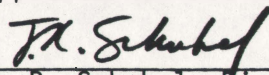
CHARACTERIZATION AND STABILIZATION OF
OGDEN-CHICAGO RESIDUE

Peter M. J. Woodhead
Frank J. Roethel

Working Paper #14

Reference 85-5

Approved for Distribution



J. R. Schubel, Director

TABLE OF CONTENTS

	<u>Page</u>
SUMMARY AND CONCLUSIONS	1
INTRODUCTION	2
CHARACTERIZATION	4
Mineralogy and Chemical Composition	4
X-ray Diffraction Analysis-Scanning Electron Microscopy	4
Results	5
Energy Dispersion X-ray Analysis	8
Elemental Analysis	12
Radiochemistry	17
Bulk Properties	18
Particle-size Analysis	18
Density	18
Moisture Content	21
Loss on Ignition	21
Acid-base Reaction pH	21
BLOCK STABILIZATION	24
Introduction	24
Cement-based Processes	24
Pozzolanic Reaction	25
Gypsum-enhanced Pozzolanic Cementation	25
Process Selection Considerations	26
Moisture-Density Relations	27
Block Mix Design	27

BBP1564

32491187

5/5/95 RL

TABLE OF CONTENTS (Continued)

	<u>Page</u>
Block Characteristics	29
Appearance	29
Density	29
Compressive Strength	33
Discussion of Block Stabilization	33

LIST OF TABLES

<u>Table</u>		<u>Page</u>
1	Elemental concentrations of NBS fly ash standard	13
2	Approximate concentration ratio: Peak height intensity (PHI) of element in incineration ash divided by PHI of the same element in NBS standards	14
3	Elemental composition of Ogden-Chicago residue	16
4	Size fraction of residue	20
5	Moisture content of residue	22
6	Loss on ignition at 550°C and at 900°C	23
7	Block mix designs for solidification	30
8	Physical properties of blocks	32

ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
1	Scanning electron micrographs of Ogden-Chicago incineration waste	6
2	EDXA of Ogden-Chicago incineration waste (Sample 1)	9
3	EDXA of Ogden-Chicago incineration waste (Sample 2)	10
4	Comparison of EXDA's of NBS coal fly ash and Ogden-Chicago incineration waste	11
5	Particle size fracations	19
6	Moisture density curve of Ogden-Chicago incineration waste	28
7	Solidified proctors of Ogden-Chicago incineration waste	31

SUMMARY AND CONCLUSIONS

A sample of urban-waste incinerator residues from a Chicago Resource Recovery Plant was characterized for physico-chemical and mineralogical properties. The potentials of the waste material for stabilization and solid block fabrication were tested using cement-based mix designs. Feasibility of the technique was demonstrated, blocks of satisfactory properties were made from the material, using relatively small additions of cement. Possibilities for using other stabilization techniques were considered, but they were discounted as inappropriate for this specific material on the basis of amount of material available, and the properties and components which we had measured.

Further investigations are warranted if the full potential for stabilization-solidification of this waste is to be realized.

INTRODUCTION

A sample of fresh (wet) ash residues from an urban incinerator in the Chicago area was given to the Marine Sciences Research Center of SUNY for investigation and to consider the suitability of the waste material for processing into solid, hard blocks. The incinerator residue came from a conventionally operated Ogden Company resource recovery plant.

Complete details of the waste feedstocks and operational details of the plant are not available and for the purposes of this initial investigation of the waste product we will call it the Ogden-Chicago material.

Development of technology for solidification and stabilization of waste materials is a response to concerns for environmental protection and is designed to accomplish:

- improved handling and physical characteristics
- compaction and volume reduction
- decrease surface area across which loss of waste components can occur
- limit the solution or to toxify potentially hazardous constituents in the wastes

Solidification produces blocks of treated waste with good structural integrity. Stabilization is primarily meant to limit the solubility and detoxify the potential contaminants in waste although the physical characteristics of the waste may not be changed; stabilization involves the use of additives to reduce dissolution of contaminants. Solidification usually also achieves the effects of stabilization and our investigations of the Ogden-Chicago residues had the objective of both solidification and stabilization, as block-stabilization of this waste.

The first step toward stabilizing a waste is to measure the chemical and physical properties of the material and its constituents. With this detailed information selection may be made of the best candidate treatment systems for that specific

waste stream. A fairly complete knowledge of all the components of the waste is of great importance in order to best guide the selection of the stabilization-solidification technique to be tried--this was particularly the case with the Ogden-Chicago residue provided which was small in size and so allowed only 2 or 3 experiments to be attempted with nothing to spare. We have measured several properties of this material and the first section of the report described those properties. In the second section we describe the method of stabilization employed and the characteristics of the blocks which were made.

CHARACTERIZATION

Mineralogy and Chemical Composition

The mineralogy and composition of the Ogden-Chicago waste material was examined using a variety of techniques including x-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive x-ray analysis (EDXA). XRD was used to identify mineral phases present in the waste while SEM was used to determine particle size and morphology. Photomicroscopy was helpful in the identification of microcrystalline phases normally not observed by standard XRD techniques. Scanning electron photomicrographs and accompanying elemental distribution of x-ray specific elements using EDXA techniques were made of the incineration ash.

X-ray Diffraction (XRD) Analysis - Scanning Electron Microscopy (SEM)

The mineralogical composition of the incineration waste was determined by XRD analysis of unoriented mounts of powdered samples. The powdered samples were prepared by grinding a freeze-dried sample and passing it through a No. 200 sieve (mesh-size $75 \mu\text{m}$). A portion of the sample was mixed with an organic binder and pressed at $2.95 \times 10^3 \text{ Pa}$ for two minutes to form a disc. The disc was then analyzed on a Picker (New Hyde Park, New York) x-ray diffractometer using $\text{Cu-K}\alpha$ radiation at 40 kv and 17 mA and a 5° to 65° 2θ scan.

The diffractograms were examined for the presence of minerals, using for peak identification the alphabetical index for inorganic materials compiled by the Joint Committee on Powder Diffraction Standards.

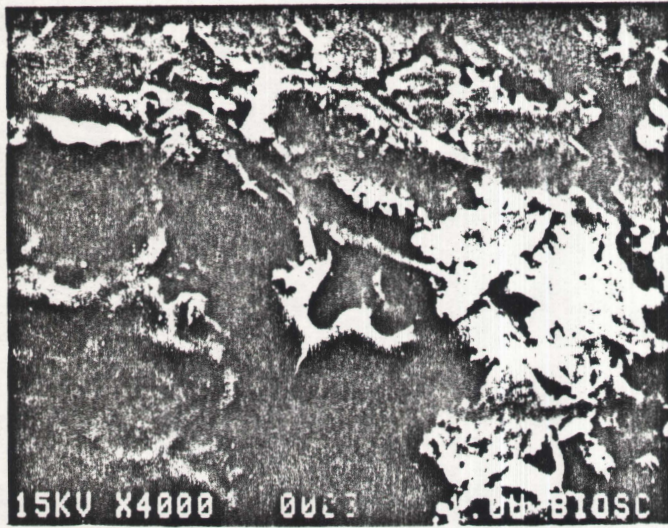
Classification of mineral phase presence as major, minor or trace was based upon the number of diffraction peaks identified and the peak intensities. Since the intensity of x-ray diffraction by a given mineral phase is a function of the degree of mineral crystallinity as well as crystal size, this classification is qualitative. An authigenically precipitated phase may yield a weak diffraction pattern even though present in large quantity since it will be poorly crystallized in incineration waste materials.

Incineration waste was prepared for SEM by freeze drying prior to placement upon a stainless steel stub and gold plated. The analysis was carried out on a JOEL Model 35C (Tokyo, Japan) scanning electron microscope with resolution capability of 100Å.

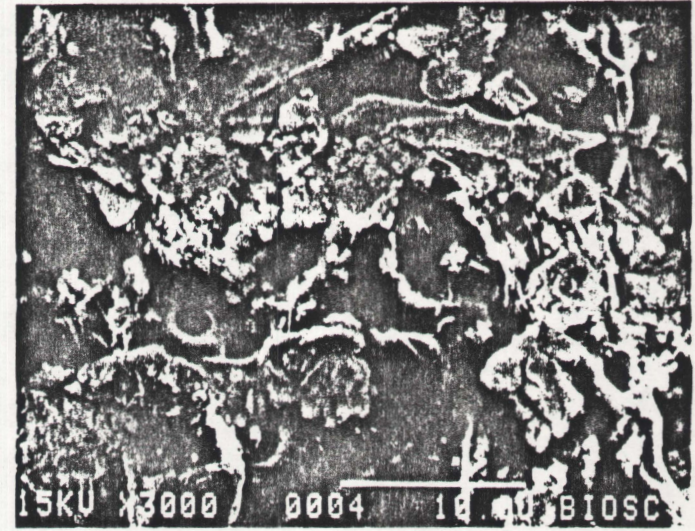
Results

The x-ray diffraction results indicate that the mineralogical composition of the incineration residue consists in part of quartz (SiO_2), gypsum $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ and calcite (CaCO_3). The diffractograms also reveal that SiO_2 is the dominant mineral phase with minor amounts of the other constituents. Amorphous compounds can not be detected using x-ray diffraction techniques due to their non-crystalline nature. Additionally, the presence of large quantities of amorphous material may have some shielding effect for any crystalline material present in the powdered mount. The x-ray diffractograms of incineration ash examined exhibit a low signal to noise ratio and that is attributed to the presence of large amounts of amorphous material.

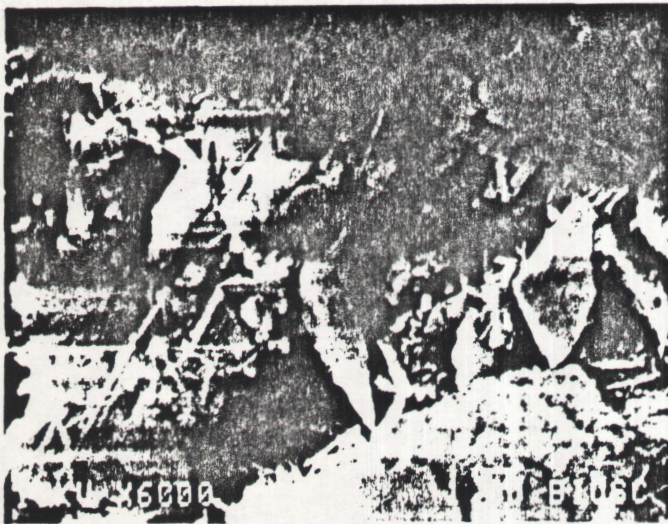
Scanning electron micrographs (Figure 1) of the sample of incineration residue reveal large variation in particle size and morphology. Particles consisting of fly ash spheres, flat platelets, well defined crystals and amorphous material were commonly observed. The fly ash nodules generally ranged in size from 1 to 25 μm ; the average size being about 8 μm . Additional mineral phases were noted while examining the photomicrographs. Hematite (Fe_2O_3) was observed at many locations throughout the sample. Classically appearing like 'swiss cheese' in Figure 1a,b, the material was observed to range in size from 25 μm to greater than 100 μm . Adhering to hematite are crystals and amorphous material. Throughout the series of photomicrographs well defined gypsum crystals were found. They appear elongated and vary in size from 1 to 50 μm in lateral dimension and $\frac{1}{2}$ to 5 μm in thickness. Figure 1f shows apparent mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) crystals, which long and slender, adhere to and almost surround a fly ash nodule. Other crystals can be seen radiating from much of the amorphous material (Figures 1d,e,h). From the energy dispersive x-ray analysis these crystals are known to be extremely high in calcium. Such crystals, ranging in size from $\frac{1}{2}$ to 1 μm , have a very different structure than gypsum and they may be calcite.



(a)



(b)

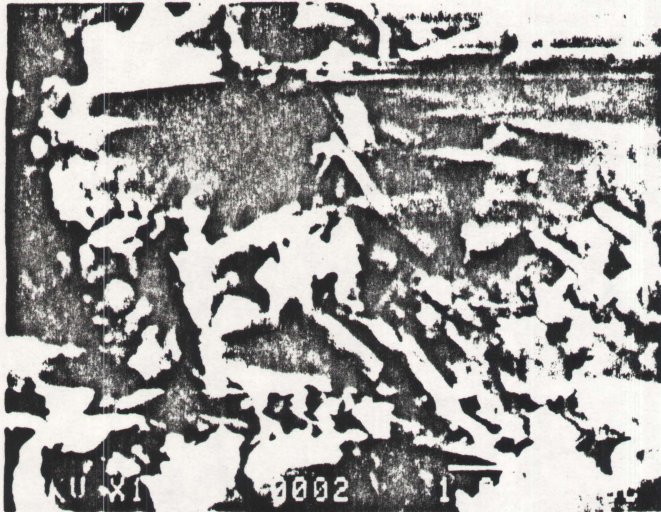


(c)



(d)

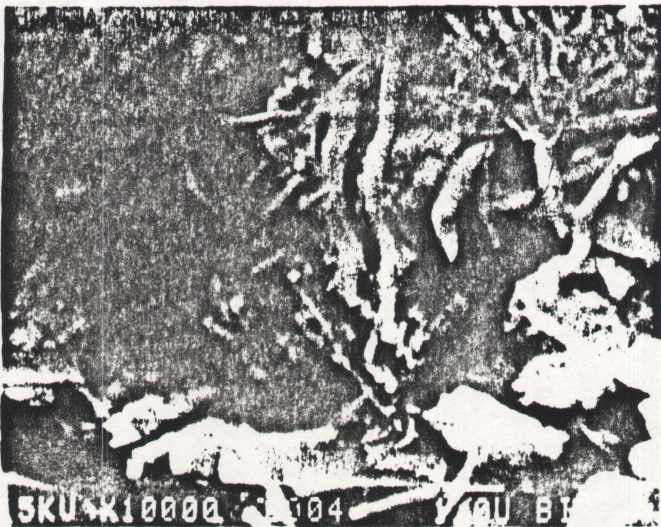
Figure 1. Scanning electron micrographs of Ogden-Chicago incineration waste.



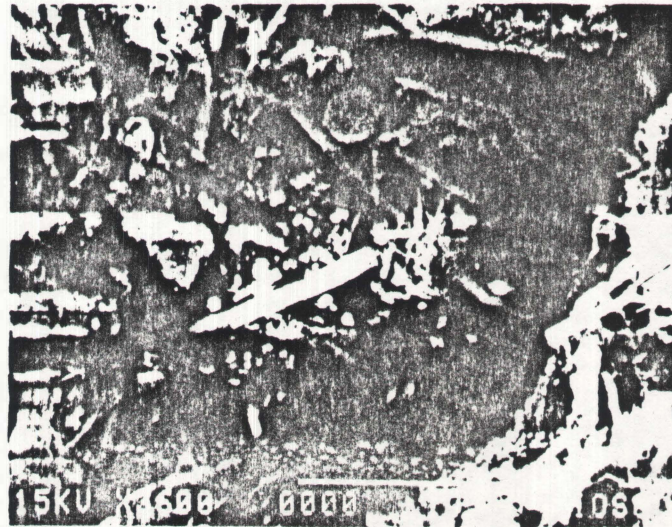
(e)



(f)



(g)



(h)

Figure 1. Continued.

Energy dispersion x-ray analysis (EDXA)

Incineration waste and National Bureau of Standards (NBS) coal fly ash 1633a were analyzed through the use of energy dispersive x-ray analysis (EDXA). - Ash samples were compacted in plastic SEM containers and then examined using an International Scientific Inc. Model SX-30 scanning electron microscope equipped with a Princeton Gamma-Tech System 4 x-ray dispersion capabilities. Computer enhancement and data manipulation was made possible with this system.

Through the use of energy dispersive x-ray (EDXA) capability of the scanning electron microscope (SEM) system, replicate elemental analysis was performed on the incineration waste (Figures 2 and 3). For comparison purposes EDXA was also used to examine National Bureau of Standards coal fly ash (NBS SRM, 1633a), having known elemental concentration. The x-ray spectrum of the Ogden-Chicago incineration ash shows the presence of silicon (Si), calcium (Ca), aluminum (Al), lead (Pb), potassium (K), magnesium (Mg), copper (Cu), iron (Fe) and zinc (Zn). An examination of the energy spectrum shows high reproducibility in the relative elemental concentration of the replicate samples of waste. Elements found in high concentrations are calcium, silicon and aluminum. The other elements are minor constituents with magnesium and copper having the lowest concentration. Trace elements having concentrations $\frac{1}{100}$ μ g/g are difficult to detect and this technique is therefore not appropriate to trace element analysis.

Two peaks observed in the spectra located at 2.6 Kev and 4.5 Kev remain unidentified. These peaks might represent elements but could be the artifacts due to secondary electron reflections.

Comparison of the NBS coal ash spectra with the incineration ash data (Figure 4) reveals higher elemental concentrations with the exception of iron and aluminum in the Ogden-Chicago waste material. While silicon and potassium are only slightly enriched, most noticeable is the very high concentration of calcium observed in the incineration ash samples. In addition, magnesium, copper and zinc have a relatively low concentration in the incineration ash spectra are not present in the coal ash spectra.

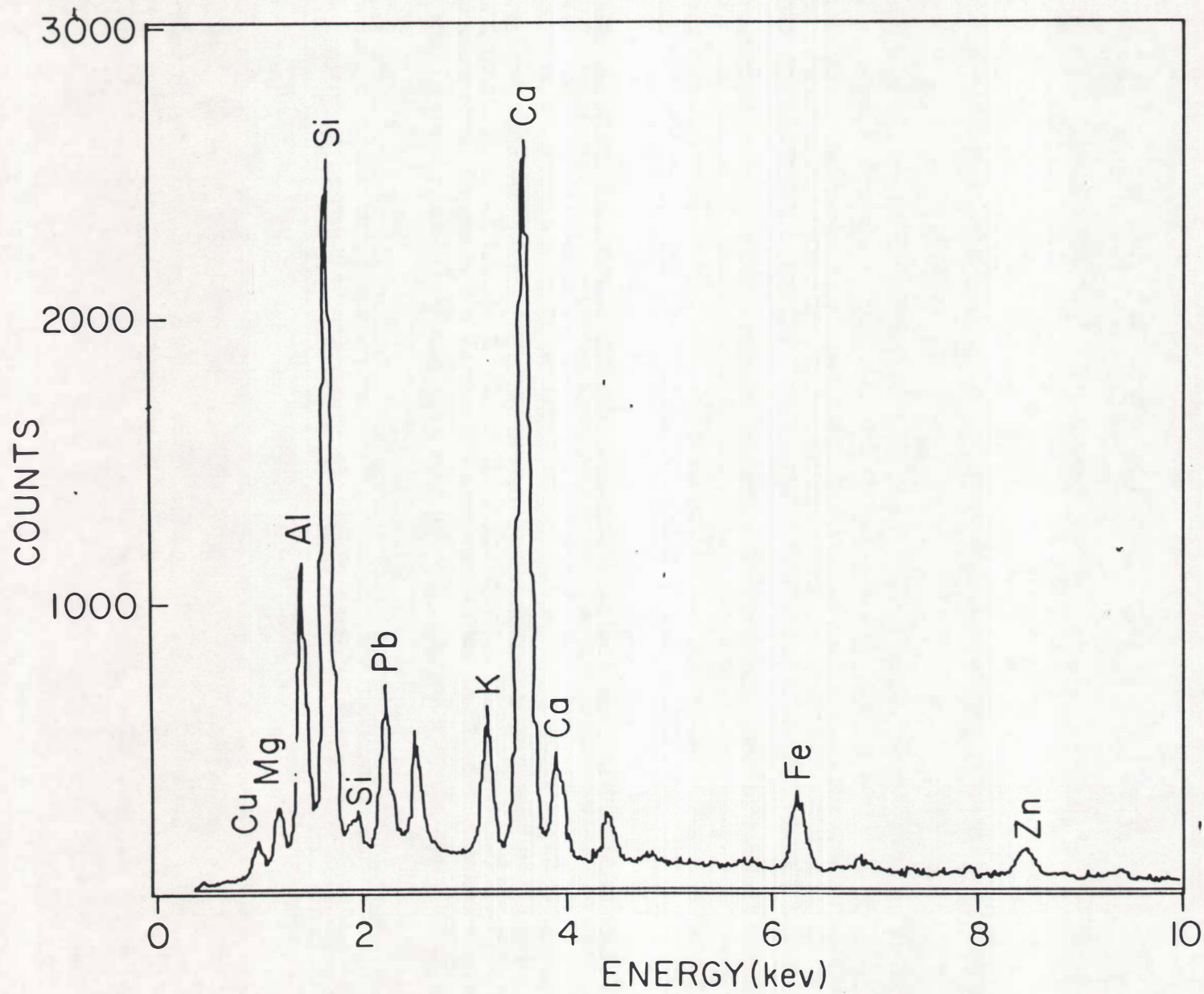


Figure 2. EDXA of Ogden-Chicago incineration waste (Sample 1).

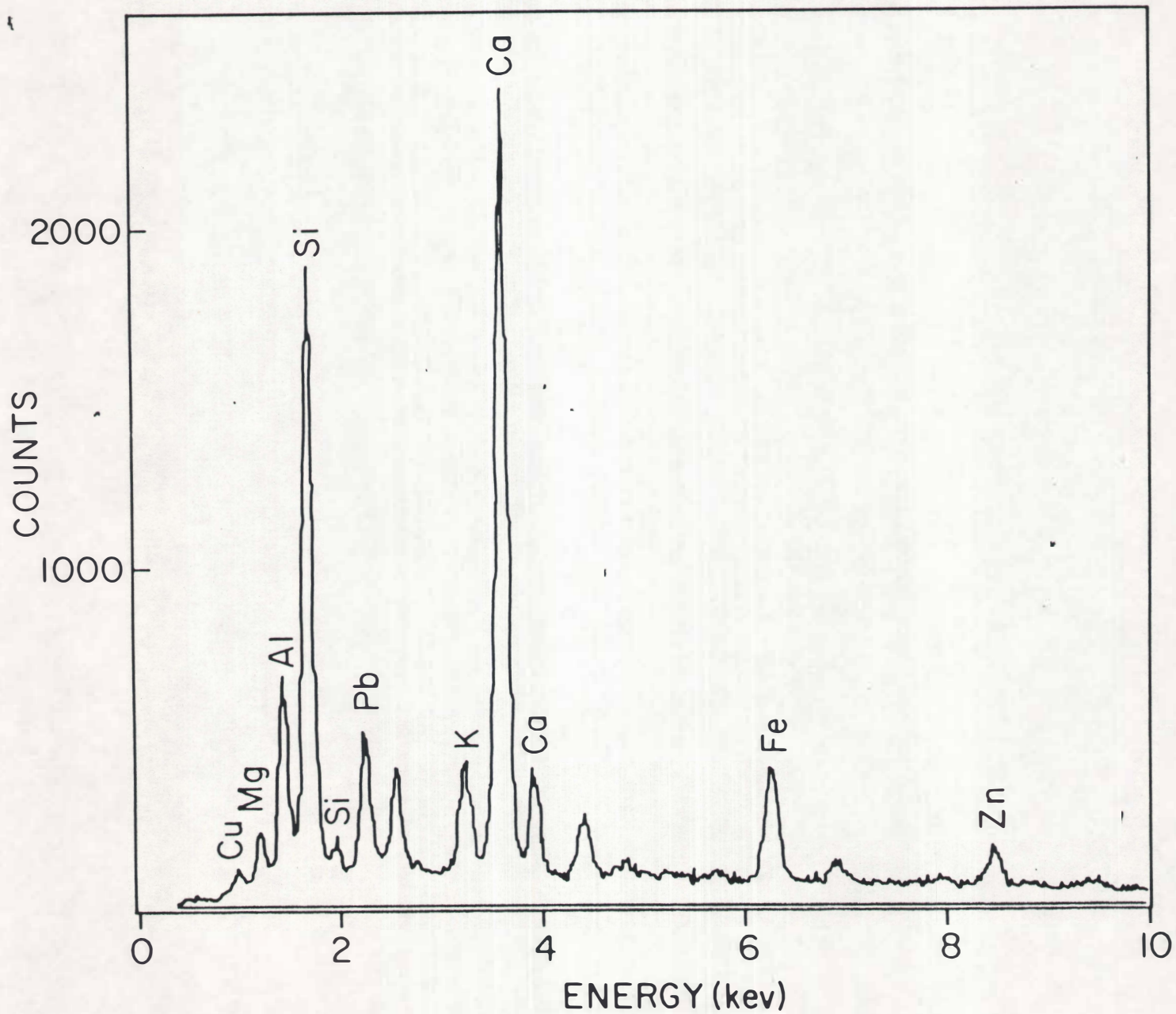


Figure 3. EXDA of Ogden-Chicago incineration waste (Sample 2).

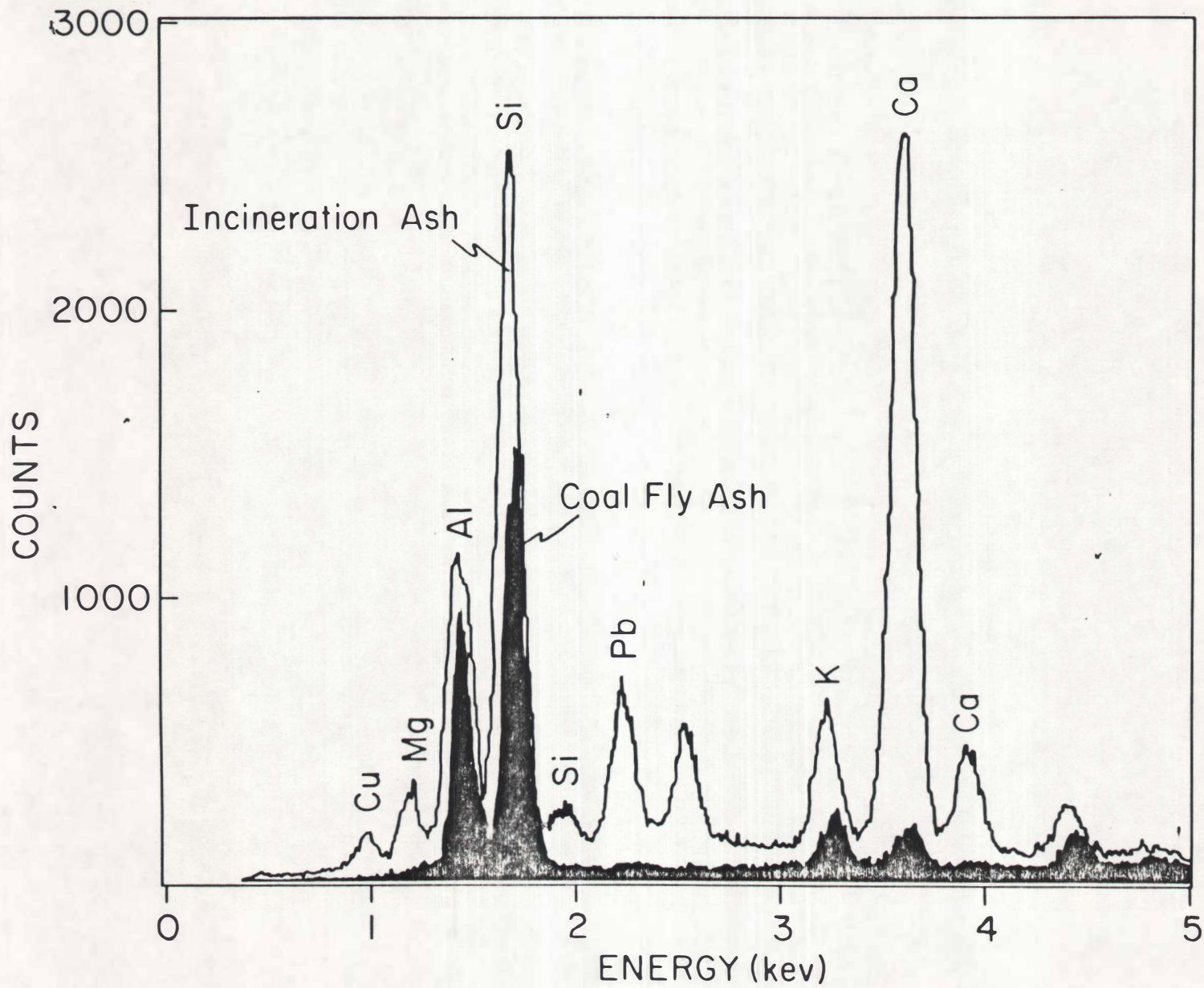


Figure 4. Comparison of EXDA's of NBS coal fly ash and Ogden-Chicago incineration waste.

Table 1 provides the concentration of selected elements present in the NBS fly ash standard. By comparing the peak height intensities of the two different samples and knowledge of the NBS elemental concentration, one can make approximate estimates of the concentrations in the incineration ash samples. Table 2 illustrates the relative ratio of elemental concentration in the Ogden-Chicago ash as compared to the concentration of the same element found in the NBS standard. Calcium concentration in the incineration ash based on this ratio is estimated to be about 10%. In a similar manner the iron concentration is estimated at approximately 2%, silicon 26% and aluminum 9%.

Elemental Analysis

The Ogden-Chicago waste residue is so very heterogeneous that rather large samples would need to be homogenized and several replicate analyses made to make reliable quantitative measures of elemental concentrations in the material under investigation. Since it is also not known how representative this sample is of the waste-stream from which it was taken, quantitative elemental concentration data are not presented here. For our initial purposes, to stabilize the waste and fabricate solid blocks, sufficient information on the composition of the material could be obtained quickly with less precise analytical methods.

Semi-quantitative analysis of the elemental composition of the Ogden-Chicago residue was made by two relatively new techniques, a) proton induced fluorescence x-ray analysis (PIXE) and b) inductive coupled plasma analysis (ICP). Both techniques are relatively rapid and allow elemental composition to be categorized as major, minor, or trace components.

The material for analysis was dried in air oven at 80°C then ground finely in an agate pestel and mortar until it could be passed through a U.S. Standard No. 100 sieve (mesh-size 150 μm). Only a few metallic particulates remained after sieving was completed, they comprised less than 1% of the total weight of the sample. For the inductive coupled plasma analysis, part of the powdered Ogden-Chicago residue was first digested in an acid mixture (hydrofluoric-nitric acids, 1:1 by volume).

TABLE 1
ELEMENTAL CONCENTRATIONS OF NBS FLY ASH STANDARD

Element	Concentration ($\mu\text{g/g}$) ^a
Element	NBS Value
Mg (%)	0.45(\pm .01)
Fe (%)	9.4 (\pm 0.1)
Ca (%)	1.11(\pm 0.01)
Si (%)	23(\pm 1)
Al (%)	12.7(\pm .5)
Zn ($\mu\text{g/g}$)	220(\pm 10)
Cu ($\mu\text{g/g}$)	118(\pm 3)
Ni ($\mu\text{g/g}$)	127(\pm 4)
Cd ($\mu\text{g/g}$)	1.00(\pm 0.15)
As ($\mu\text{g/g}$)	145(\pm 15)
Cr ($\mu\text{g/g}$)	196(\pm 6)
Pb ($\mu\text{g/g}$)	72.4(\pm 4)

a Numbers in parenthesis denote standard deviation.

TABLE 2

APPROXIMATE CONCENTRATION RATIO: PEAK HEIGHT
INTENSITY (PHI) OF ELEMENT IN INCINERATION ASH
DIVIDED BY PHI OF THE SAME ELEMENT IN NBS STANDARD

<u>Element</u>	<u>Concentration Ratio</u>
Al	0.72
Si	1.12
Ca	9.46
Fe	0.23
K	1.57

The results of the elemental analyses by the two techniques are given in Table 3; also included in the Table is a summary of the results of EDXA scans described in the preceding section of this report. Not all the same elements were included in the three techniques, but for those elements analyzed by two or more methods agreement in categorization is good. All major and minor cations are probably represented in Table 3, but trace elements contents are certainly not complete.

The elemental composition data for the residue samples which were analyzed by these different techniques (in different laboratories) are in good agreement (Table 3). Data is available for 23 cations in 17 of these were analyzed by two or more methods. Major components of the residue found by all three methods are aluminum, calcium, and silica; magnesium was also estimated to be major by the acid-digest IPC technique. Eight elements were categorized as being in minor abundance and included barium (by IPC only), sodium (by IPC), lead, strontium (by PIXE) titanium, iron and zinc.

The list of trace elements is not complete, but indicates the presence of some elements of potential environmental concern, these include arsenic, cadmium, chromium, mercury, nickel, selenium and silver. The highly alkaline pH measured for the residue, mean pH 12.2, may have significant effects on the solubilities of some of these trace elements. Many multivalent elements will form relatively insoluble hydroxides in the pH range 9 to 11, but other toxic elements such as arsenic may be more easily leached to solution above pH 10.

TABLE 3

ELEMENTAL COMPOSITION OF OGDEN-CHICAGO RESIDUE
Categorization: M-Major; m-Minor; t-trace concentrations

Element	PIXE	IPC	EDXA
Ag		t	
Al	M	M	M
As	t	t	
Ba		m	
Ca	M	M	M
Cd		t	
Cr	t	t	
Cu	m	m	
Fe	M	M	m
Hg	t		
K	m		
Mg		M	
Mn	t	t	
Na		m	
Ni		t	
Pb	m	t	m
Rb	t		
Se		t	
Si	M	M	M
Sr	m		
Ti	m		
Zn	m	m	
Zr	T		

Radiochemistry

Non-destructive gamma spectrometric analysis of Ogden-Chicago incineration waste

Approximately 90 grams of ash were sealed in a polyethylene lined aluminum can and counted on a planar intrinsic germanium gamma detector for approximately 1.5 days. The detector is capable of measuring low energy gamma rays from both naturally occurring and artificial radionuclides. All the gamma emitters identified in the sample are from the naturally occurring uranium and thorium decay series. The following table gives count rates above background for some of the radionuclides identified:

Radionuclide	Decay Series	Count Rate above background (cpm)
^{210}Pb	^{238}U	.15
^{234}Th	^{238}U	.15
$^{226}\text{Ra} + ^{235}\text{U}$	$^{238}\text{U}, ^{235}\text{U}$.07
$^{212}\text{Pb} + ^{224}\text{Ra}$	^{232}Th	.35
$^{214}\text{Pb} + ^{212}\text{Pb}$	$^{238}\text{U}, ^{232}\text{Th}$.12

The sample is consistent with material that contains ^{238}U , ^{235}U , ^{232}Th and associated decay products all of which are naturally occurring.

Bulk Properties

Particle-size Analysis

The distribution of particle sizes in the residue was determined by sieving a sample of approximately 500 g of the fresh residue which was then dried in an air oven at 80°C. The analysis followed ASTM D 422-63 using a series of U.S. Standard Sieves, Numbers 4, 10, 40 and 200. For the three larger sieves the residues were sieved dry and the weight was taken of the materials retained on each sieve. For the No. 200 sieve, the finest residues were washed on the sieve, this wet procedure following ASTM C 786; the No. 200 sieve fractions were dried to constant weight at 80°C.

The results of the particle-size analysis are illustrated in Figure 5 which shows the composition of the different size fractions. The material was very heterogeneous, as expected. In the large size groups glass was predominant together with fragments of pieces of paper, wood and grass clippings. Steel and copper wires, nails were found together with small fragments of metals, which included iron and lead.

The quantitative contributions of the different fractions to the sample are given in Table 4. The greatest fractions by weight were in the size range 0.1 to 2.0 mm, accounting for about 62% of the dry weight. The fineness of the material was calculated from the results of wet sieving through No 200 sieve (ASTM C 786); the fineness value was 6.9%.

Density

The density of the residue mixture was determined after impact compaction in a standard cylindrical metal proctor mold having an internal diameter of 4 in. Compaction was made with a mechanical rammer of 5.5 lb falling 12 in, and followed ASTM D 558, Method B. The mold was filled in three compacted layers, each layer receiving 25 strikes of the rammer. Following compaction the cylinder of waste was trimmed level with the top of the mold, and weighed. The density of the compacted fresh residue was 116.7 lb/ft³ (1.87 g/cm³).

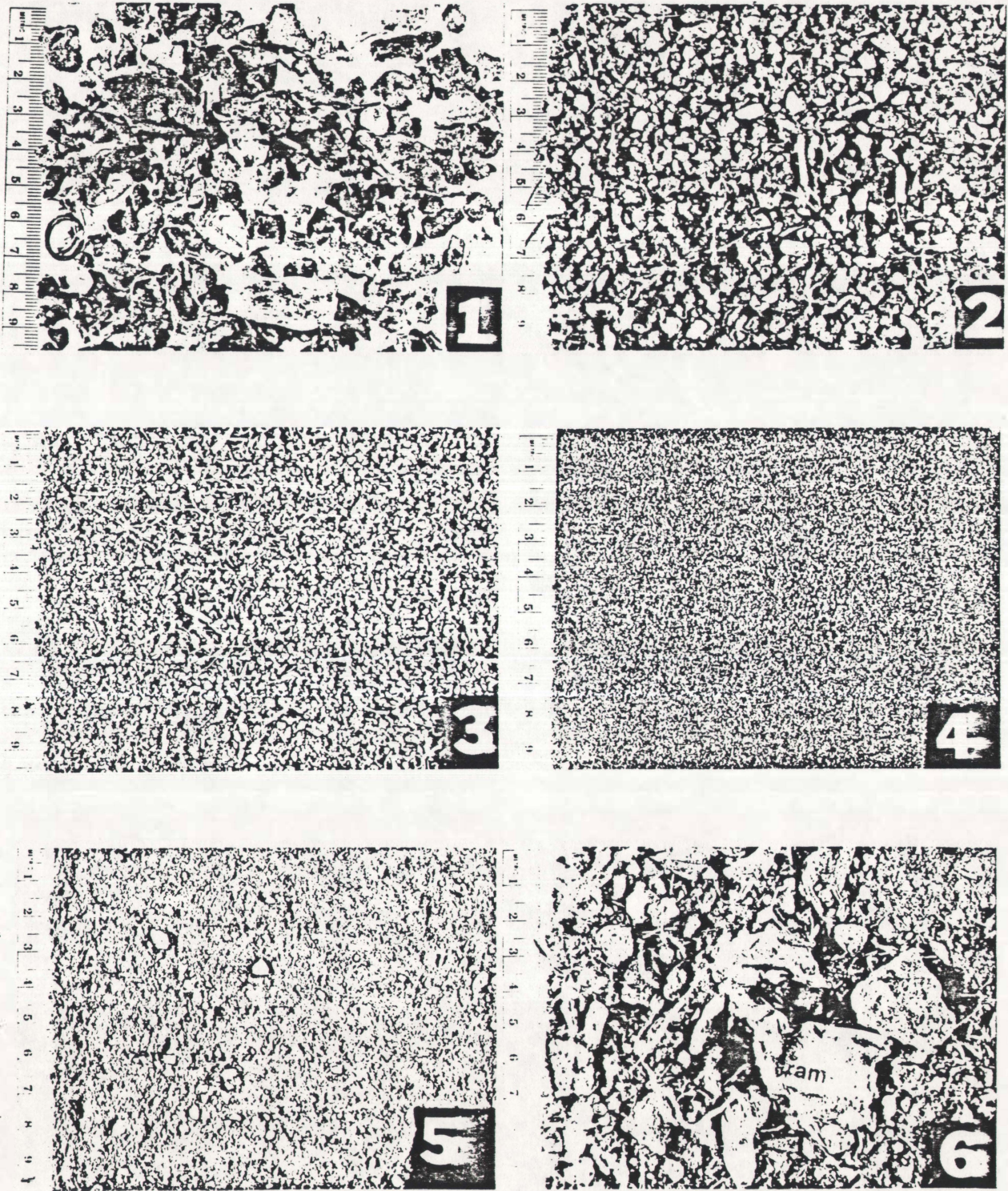


Figure 5. Particle size fractions. 1) retained by #4 sieve; 2) retained by #10 sieve; 3) retained by #40 sieve; 4) retained by #200 sieve; 5) passed through #200 sieve; 6) unsorted.

TABLE 4
SIZE FRACTION OF RESIDUE

Retained by Sieve	Mesh Diameter	Dry Weight, g	Dry Weight %
4	4.75 mm	81.54	21.5
10	2.00 mm	72.05	19.0
40	425 μm	107.27	28.2
200	75 μm	92.58	24.4
Passing 200	(<75 μm)	26.38	6.9

Moisture Content

Moisture content was determined on 30-40 g samples of fresh residue ash which were dried to constant weight in an air oven at about 90°C, Table 5. Moisture contents were fairly uniform, despite the heterogeneity of the material and the large solid inclusions.

Loss on Ignition

The dried samples of residue used for determination of moisture content were used to measure loss on ignition (LOI). In this method the samples were ignited in a covered crucible in a muffle furnace at controlled temperature. Separate determinations were made for LOI at two temperatures, 500±50°C and 900±50°C. LOI is frequently determined at temperatures of 900 to 1,000°C but biogenic organics are burned off at 500°C and this was a materials group of interest for the present characterization.

The combustion loss at 500°C was 3.7% of dry weight (3% of fresh weight); the loss at 900°C was greater, 5.2% of dry weight (4% of fresh weight), Table 6. The higher losses measured at 900°C are probably not only due to the burning of organic compounds, but also include the breakdown of such compounds as calcium carbonate, and the dehydration of others, such as gypsum, or lime.

Acid-base Reaction (pH)

The fresh damp residue was very alkaline, basic, having a mean pH 12.2. This high pH is only slightly below than the U.S. EPA limit of pH 12.5 for categorization as a hazardous waste (Resource Conservation and Recovery Act, Public Law 94-580). At pH 12, the leaching of some elements of environmental concern may be high and will be discussed in the Chemistry section of the report.

Five samples of untreated waste were left in an equal volume of deionized water overnight, decanted and the pH of the water measured. The mean pH of these solutions was 11.4±0.4.

TABLE 5
MOISTURE CONTENT OF RESIDUE

Sample No	Fresh Wt g	Dry Wt g	Weight loss g	Moisture %
1	32.50	25.68	6.82	21.0
2	33.86	26.82	7.04	20.8
3	35.51	28.47	7.03	19.8
4	37.17	29.52	7.65	20.1
Mean				20.4
s.d.				±0.5

TABLE 6

LOSS ON IGNITION AT 550° and at 900°C

Sample No	Dry Wt g	Content Moisture %	LOI5 ¹ g	LOI9 ² g	LOI5 % Dry	LOI9 % Dry ³	LOI5 % Wet ⁴	LOI9 % Wet
1	25.64	21.0	0.95	1.29	3.7	5.0	2.9	4.0
2	26.76	20.8	0.97	1.37	3.6	5.1	2.9	4.1
3	28.49	19.8	0.88	1.49	3.1	5.2	2.5	4.2
4	29.57	20.1	1.35	1.56	4.5	5.3	3.6	4.2
Mean					3.7	5.2	3.0	4.1
± s.d.					0.6	0.1	0.5	0.1

1 LOI5 = Loss on ignition at 500±50°C

2 LOI9 = Loss on ignition at 900±50°C

3 Percent dry weight of sample

4 Percent fresh weight of sample

BLOCK STABILIZATION

Introduction

There are several solidification-stabilization techniques which have the goal of safe disposal of waste materials, either through a productive use or by a variety of disposal or storage methods. Not all methods meet all goals and particular techniques may be appropriate to individual materials, but unsatisfactory for others. We considered three simple, low-cost stabilization and solidification methods for application to the Ogden-Chicago residue:

- a. Cement-based stabilization
- b. Pozzolanic reaction
- c. Gypsum-enhanced pozzolanic cementation

These methods are briefly outlined.

Cement-Based Processes

Most wet or slurried in water waste, can be mixed directly with Portland cement, and the suspended solids will be incorporated into the rigid matrices of the hardened concrete. This process is especially effective for waste with high levels of toxic metals, since at the pH of the cement mixture, many multivalent cations are converted into insoluble hydroxides or carbonates. Metal ions may also be incorporated into the crystal structure of the cement minerals that form. Materials in the waste such as sulfides, asbestos, latex, and solid plastic wastes may increase the strength and stability of waste concrete.

A number of inorganic compounds in the waste can be deleterious to the setting and curing of the waste containing cement. Also, impurities such as organic materials, silt, clay, carbon or lignite may delay the setting and curing of common Portland cement for as long as several days. Insoluble materials passing through a No. 200 mesh sieve are undesirable, as they may be present as dust or may coat the larger particles and weaken the bond between the particles and the cement. Products containing large amounts of sulfate not only retard the setting of concrete, but cause swelling and spalling in the solidified concrete.

Process Selection Considerations

A wide variety of possible techniques exist for processing wastes into solid forms. Our original intention was to utilize at least two of these relatively simple, inexpensive cementation stabilization processes just outlined to make solid blocks of the Ogden-Chicago residue. However before these processes could be adopted, we reviewed our data characterizing this residue with respect to the applicability of each of the three methods being considered for the stabilization/solidification process.

Solidification by compaction and the Portland cement stabilization process seems appropriate for the Ogden-Chicago waste. The fineness of the material is less than 7% and the organic content was relatively low (less than 6% LOI at 500°C). However, soluble salts of copper, lead and zinc may be deleterious to setting of concrete and all three elements are well represented in the waste. The mineralogical analysis had also found calcium sulfate present and this compound may cause breakdown of concretes. However consideration of all of the characterization data suggested that the cement process may be useful in stabilization of these residues and certainly warranted work.

Stabilization with lime addition to form pozzolanic cement and possible enhancement of this reaction by addition of gypsum had been considered attractive candidate methods. However, the chemical and mineralogical characterization data revealed that the residues were already very rich in calcium compounds, with very alkaline reaction so that calcium hydroxide was certainly present, -- indeed it appeared that lime had probably been added to the waste stream at some point. In the same way, the x-ray diffractograms had indicated that the material was also rich in gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), so that further additions were not likely to be effective. Because significant quantities of lime and gypsum were already present in the residues, which had not become stabilized or solidified. We concluded that the untreated material was therefore probably of low pozzolanic activity and that pozzolanic cementation processes would not be effective for block-solidification. On the basis of these data we therefore chose to stabilize the Ogden-Chicago residue using Portland cement processes only.

Cement treatment has a number of advantages which include the production of high load bearing capacities, and low permeability, materials are plentiful and inexpensive and the technology is well known. Extensive filtering or drying are not required; the system is tolerant of much chemical variation; high pH is tolerated and the alkalinity of cement can be used to neutralize acids. Disadvantages include the addition of bulk and weight to the product; particular waste components may be detrimental to the cementation process or may require special cement types or additives.

Pozzolanic Reaction

Waste fixation based on lime usually depends on the reaction of lime with a fine-grained siliceous (pozzolanic) material and water to produce a concrete-like solid (sometimes referred to as a pozzolanic concrete).

Lime-pozzolanic cementation reactions have been used since Roman times and their chemistry is relatively well known. The most common pozzolanic materials used in waste treatment are fly ash, ground blast-furnace slag, and cement-kiln dust; which are themselves waste products with little or no value.

Most of the advantages associated with Portland cement systems apply to pozzolanic systems. These include improved handling and permeability characteristics of the waste, low cost wide availability and ease of processing of lime products. Extensive dewatering is not necessary. Sulfates do not cause spalling of the solid product. Like cement, lime and other additives add bulk and weight.

Gypsum-enhanced Pozzolanic Cementation

We have extensive experience with stabilization of calcium sulfite/sulfate sludges from coal fired power stations. In some instances we have used such related compounds as gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) in the pozzolanic cementation of other materials, the SO_x combines in the reactions to form complex crystalline sulfo-alumina hydrates (ettringites) which enhance cementation. The gypsum-enhanced pozzolanic waste cements are very similar to other pozzolanic cements and have the same advantages and disadvantages.

Moisture-Density Relations

Block fabrication and cementation of soil-like materials is best made and at optimal densities. The density of such materials is closely related to its moisture content. Before blocks were made the moisture-density relationships of the waste residue were determined.

Some of the Ogden-Chicago waste was dried in an air oven and weights of the dried material were mixed with fresh (wet) residue to achieve a range of moisture contents. The mixed materials were compacted in a standard cylindrical metal, proctor, mold having a capacity of $1/30 \text{ ft}^3$ (942.9 cm^3), with an internal diameter of 4.0 in (10.16 cm). The mold was filled in three successive layers, each layer being compacted with a 2 in, 5.5 lb mechanical rammer. Compaction followed ASTM D558-57 Method B, each layer receiving 25 blows from the rammer, with a 12 in fall. The compacted cylinder of waste was trimmed level with the top of the mold, then extruded from the mold and weighed. Densities were calculated from the weight and known volume of the cylinder.

The results for six measurements of compacted density of proctor cylinders with different moisture contents are shown in Figure 6. It is seen that density increases with increasing moisture content, achieving optimum values at about 18% moisture, at higher moisture levels densities decrease again. The fresh (wet) residue has densities of 116 to 117 lb ft^3 , below the optimum of 120 lb ft^3 .

The moisture-density data were used in the calculations of the mix designs calculated for solidification of Ogden-Chicago residue, with the aim of achieving optimum densities for the compacted mixes. However when Portland cement is added to the materials the moisture-density relations are modified and the curve in Figure 6 moves to the left, higher densities are achieved at slightly lower moisture contents; the maximum density achieved also increases somewhat.

Block Mix Design

It was our intention to keep the cement additions low to demonstrate more convincingly the feasibility of this method. There was only sufficient Ogden-Chicago material, together with additives, to make three experimental

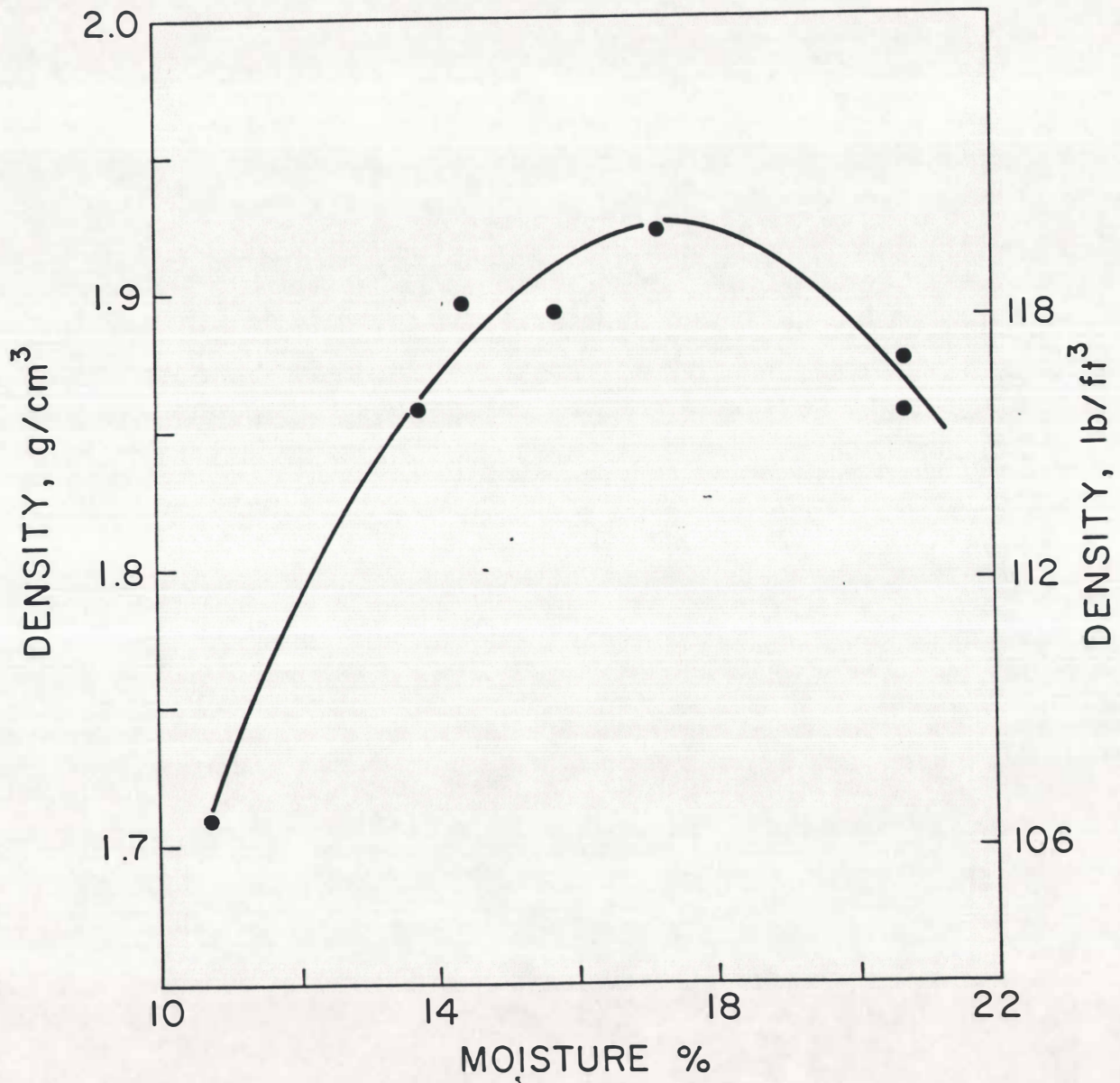


Figure 6. Moisture density curve of Ogden-Chicago incineration waste.

blocks, proctor cylinders. Because new moisture-density relations were produced by addition of Portland cement to the mixtures, the mix designs were modified for each successive proctor fabrication which contributed further information.

The three final mixes that were developed are given in Table 7. Two additions of Portland cement were used, 8% and 10%.

The compacted molded blocks were extruded from the molding cylinder and placed in a high humidity cabinet at a temperature of 50°C. They were held in the cabinet under these conditions for 36 hours to cure and develop structural strength. Following the cabinet curing period blocks were allowed to slowly cool to room temperature for 3 hours and then were tested for their integrity and strength.

Block Characteristics

Appearance

Three satisfactory solid blocks were made from the Ogden-Chicago residue. All were well formed, had firm edges and were fairly smooth-sided. From external appearance the material was densely compacted and of relatively low porosity. The first block formed became somewhat thixotropic during compaction, the rammer blows serving to express water and the texture of the material became clay-like. This block was soft to the touch when newly formed and slumped slightly before developing strength, so that the vertical sides were slightly bulging. The second and third blocks contained less moisture and formed cylinders which were firm to the touch when first extracted from the mold. All three blocks are shown in Figure 7.

Density

The (wet) densities of the blocks were satisfactory. For the freshly formed blocks wet densities of 120 lbs/ft³ were achieved (Table 8). During curing some water loss occurred and density was reduced by about 2 lbs/ft³ in each block, to range 117 to 121 lbs/ft³.

TABLE 7
BLOCK MIX DESIGNS FOR SOLIDIFICATION

Block No.	Ogden Residue %	Portland Cement %	Water %
1	77.5	10.0	16.3
2	76.0	8.0	16.0
3	76.0	10.0	14.0

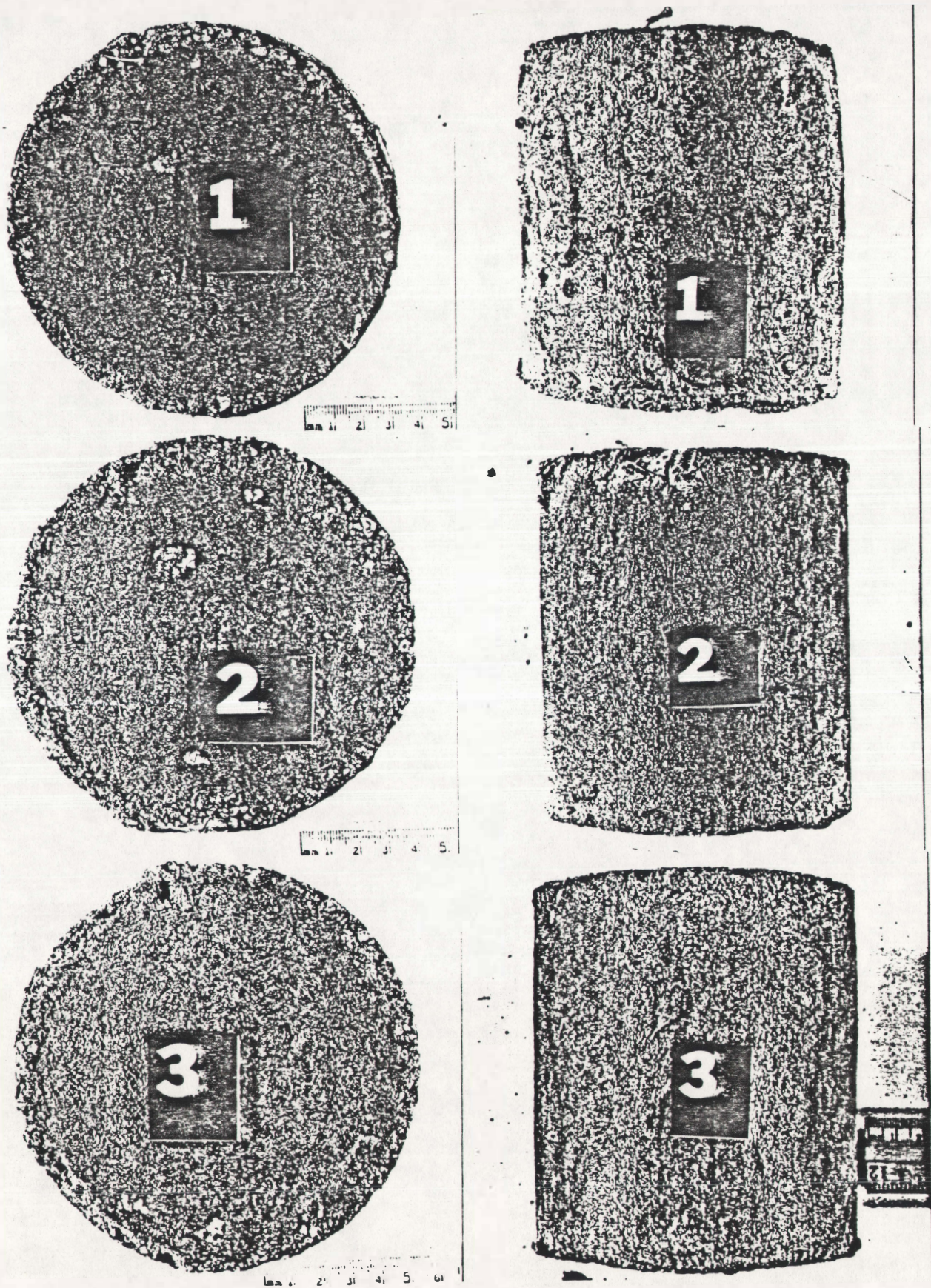


Figure 7. Solidified proctors of Ogden-Chicago incineration waste.

TABLE 8
PHYSICAL PROPERTIES OF BLOCKS

Block No.	Wet Wt. lb	Wet Density lb/ft ³	Dry Density lb/ft ³	Compressive Strength lb/in ²
1	4.0	120.2	117.4	50
2	4.0	120.2	118.0	80
3	4.1	123.2	121.7	100

Compressive Strength

Unconfined compressive strengths were measured by applying a steadily increasing load to the block until the block fails. The load at failure provides a quantitative measure of the integral strength of the block. The test was made following the procedures of ASTM C39-72. We have applied it to a wide range of materials and found it to be a valuable indicator of block integrity and longevity in aquatic systems.

The three blocks which were made had strength of 50, 80 and 100 lbs/in². These strengths were acceptable; they demonstrated the development of integral structure in the material. Curing times were abbreviated and because secondary curing is slow, a more protracted cure at room temperature would certainly have produced further gains in strength.

Discussion of Block Stabilization

The strengths achieved in the short-term were about half of the values that we desire for aquatic disposal, but they very clearly demonstrated the feasibility of block stabilization for this residue. There is no doubt that with further investigation of the materials and development of a range of mix designs to explore the possibilities for stabilization, higher strengths could be developed for solid blocks of the Ogden-Chicago incineration waste.

With a strictly limited amount of material in the sample made available to us, we used the information we achieved when making each block to modify the design of the next block mixture. It is satisfying that we were able to approximately double the strength developed by the three successive blocks we made.

