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Demonstration of Macroencapsulation of  
Mixed Waste Debris Using  
Sulfur Polymer Cement

C. H. Mattus

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Chemical Technology Division

DEMONSTRATION OF MACROENCAPSULATION OF MIXED WASTE DEBRIS  
USING SULFUR POLYMER CEMENT

C. H. Mattus

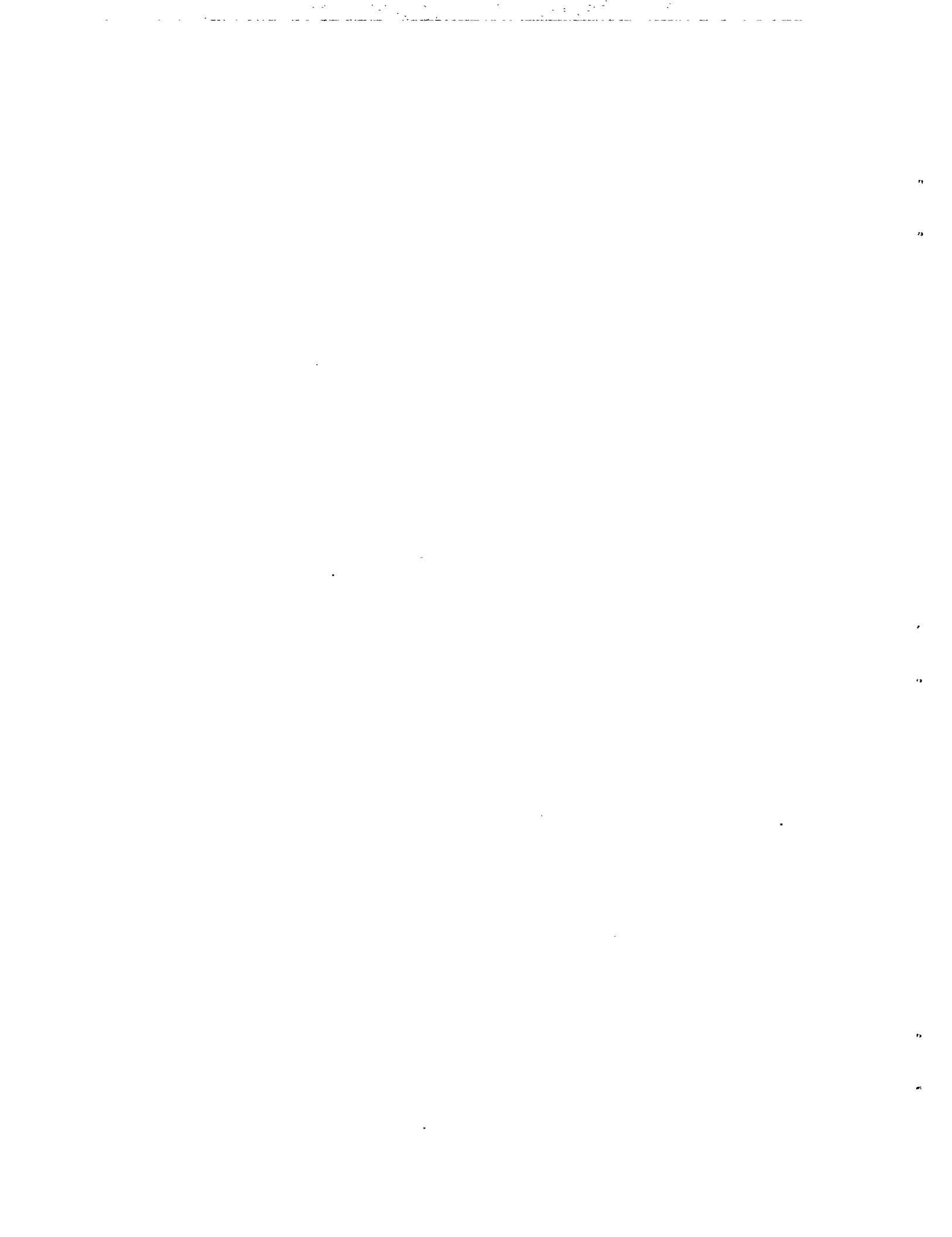
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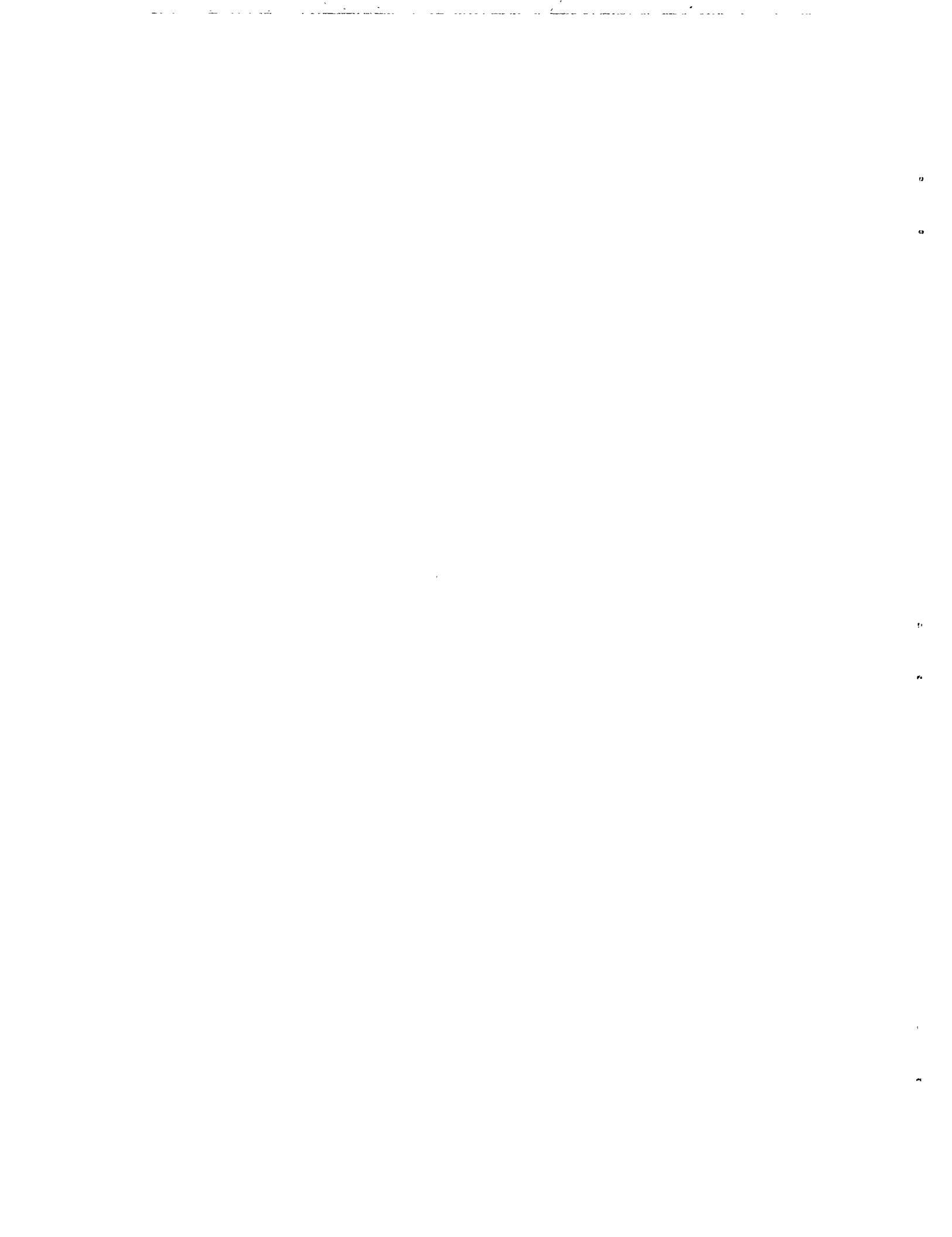
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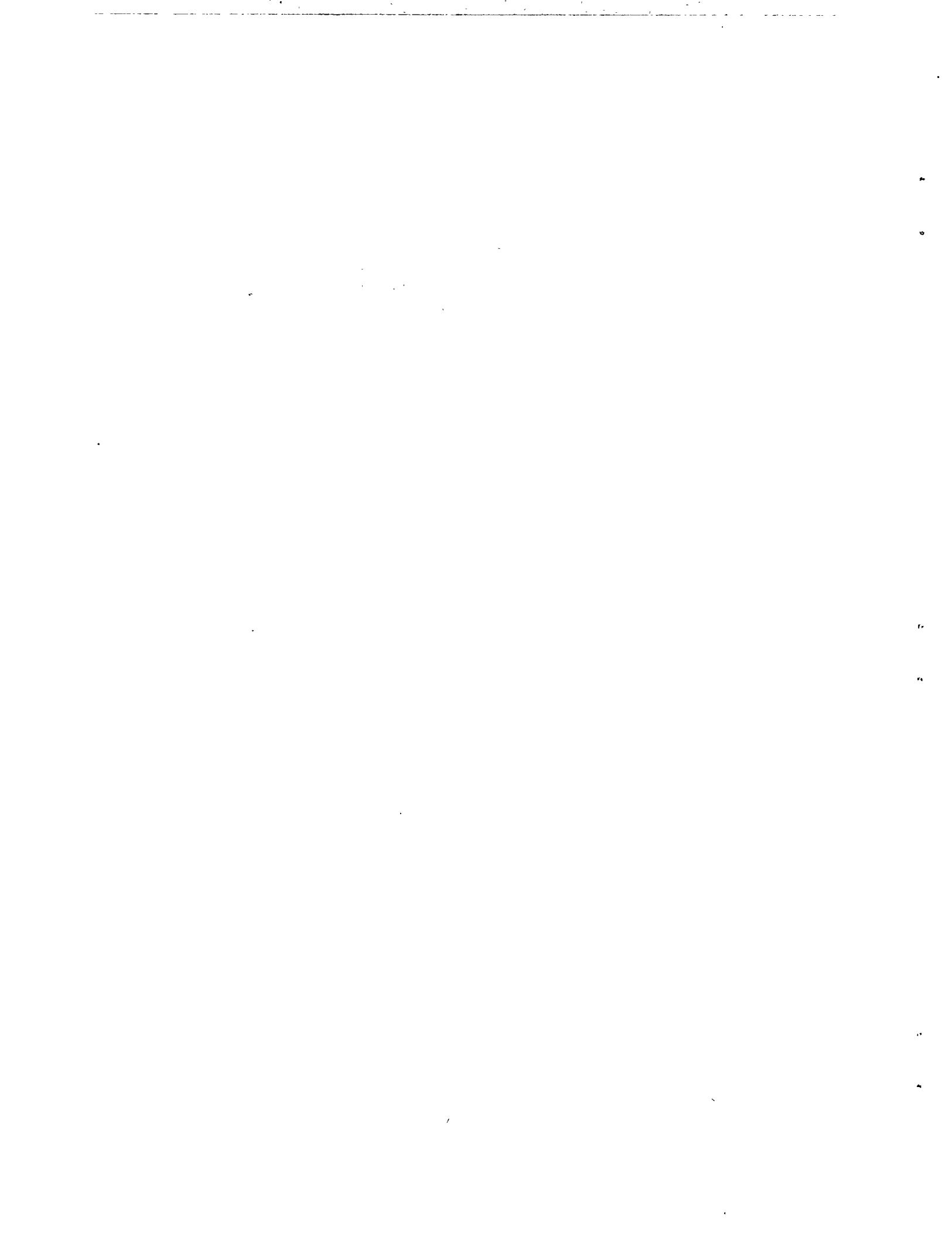
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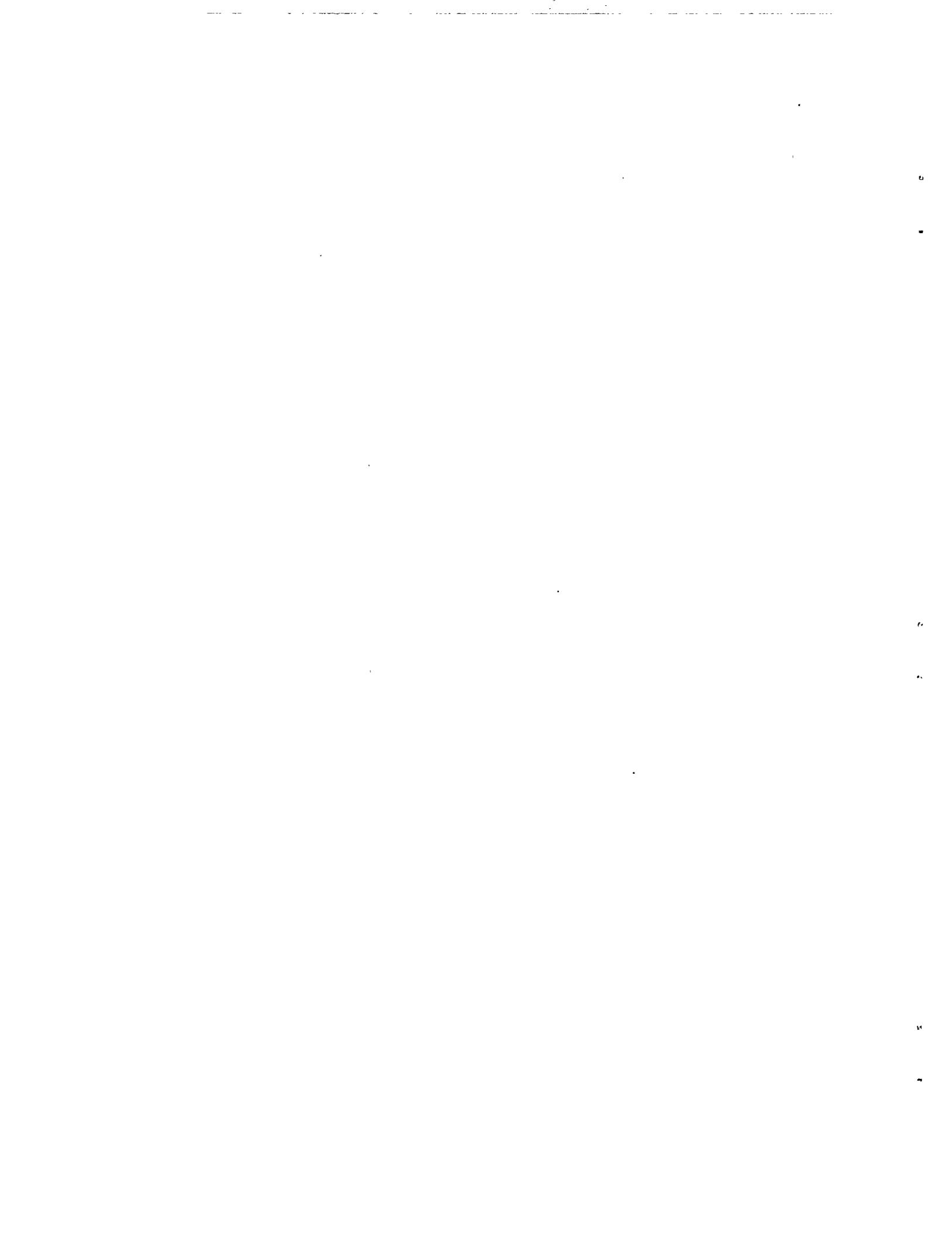
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## ACRONYMS

DOE	U.S. Department of Energy
DOT	U.S. Department of Transportation
EPA	U.S. Environmental Protection Agency
INEEL	Idaho National Engineering and Environmental Laboratory
LDR	Land Disposal Restrictions
MWFA	Mixed Waste Focus Area
MWIR	Mixed Waste Inventory Report
NRC	U.S. Nuclear Regulatory Commission
ORNL	Oak Ridge National Laboratory
ORR	Oak Ridge Reservation
RCRA	Resource Conservation and Recovery Act
SPC	sulfur polymer cement
WAC	waste acceptance criteria



## EXECUTIVE SUMMARY

This report covers work performed during FY 1997 as part of the Evaluation of Sulfur Polymer Cement Fast-Track System Project. The project is in support of the "Mercury Working Group/Mercury Treatment Demonstrations-Oak Ridge" and is described in technical task plan (TTP) OR-16MW-61.

Macroencapsulation is the treatment technology required for debris by the U.S. Environmental Protection Agency Land Disposal Restrictions (LDR) under the Resource Conservation and Recovery Act. Based upon the results of previous work performed at Oak Ridge, the concept of using sulfur polymer cement (SPC) for this purpose was submitted to the Mixed Waste Focus Area (MWFA). Because of the promising properties of the material, the MWFA accepted this Quick Win project, which was to demonstrate the feasibility of macroencapsulation of actual mixed waste debris stored on the Oak Ridge Reservation.

The waste acceptance criteria from Envirocare, Utah, were chosen as a standard for the determination of the final waste form produced. During this demonstration, it was shown that SPC was a good candidate for macroencapsulation of mixed waste debris, especially when the debris pieces were dry. The matrix was found to be quite easy to use and, once the optimum operating conditions were identified, very straightforward to replicate for batch treatment. The demonstration was able to render LDR compliant more than 400 kg of mixed wastes stored at the Oak Ridge National Laboratory.

## 1. INTRODUCTION

The Mixed Waste Focus Area (MWFA) strives to sponsor and develop treatment technologies for mixed wastes that clearly demonstrate that a specific technology meets the requirements of the end users. Among the inventory of mixed wastes, one class—mixed waste debris—presents a challenge because of its diversity.

The U.S. Environmental Protection Agency (EPA) gives the following definition of “debris” and “hazardous debris” (*Fed. Regis. 57*, 37194) in the Resource Conservation and Recovery Act (RCRA) Land Disposal Restrictions (LDR): “*Debris* means solid material exceeding a 60 mm particle size that is intended for disposal and that is: a manufactured object; or plant or animal matter; or natural geologic material.”

The regulatory options for treating such waste are to use one of the following technology groups: extraction, destruction, or immobilization. Macroencapsulation is a subpart of the immobilization category and is defined in the *RCRA Land Disposal Restrictions: A Guide to Compliance*, 1996 edition, as follows: “Application of surface coating materials, such as polymeric organics (e.g. resins and plastics), or use of a jacket of inert inorganic materials to substantially reduce surface exposure to potential leaching media.”

In the same document (Table 8.1—Alternative Treatment Standards for Hazardous Debris, p. 8.10), the performance standard for this technology is as follows: “Encapsulating material must completely encapsulate debris and be resistant to degradation by the debris, its contaminants, and materials into which it may come into contact after placement.” In other words, the use of macroencapsulation technology is sufficient for treating hazardous debris and rendering it LDR compliant. However, EPA does not provide guidance about the physical characteristics that could reduce the surface exposure to leaching media. Consequently, the waste acceptance criteria (WAC) from Envirocare, Utah, were used as a guide for the design of the experimental setup to be used during this demonstration.

The MWFA initiated the Quick Win Program to bridge the gap between technology development and end users of the technology. Macroencapsulation of mixed waste debris was tested at other U.S. Department of Energy (DOE) sites with the use of various matrices such as epoxy [1] or polymer [2, 3]. This project meets the needs of the MWFA by demonstrating macroencapsulation of debris at the bench scale using sulfur polymer

cement (SPC) and doing so on actual inventories of such debris presently located in the Mixed Waste Inventory Report (MWIR) of the Oak Ridge Reservation (ORR).

SPC is a relatively new material in the waste immobilization field, although it was developed in the late seventies by the Bureau of Mines [4-10]. Its physical and chemical properties are interesting (e.g., development of high mechanical strength in a short period of time and high resistance to many corrosive environments). Because of its very low permeability and porosity, SPC is especially impervious to water, which, in turn, has led to its consideration for immobilization of hazardous or radioactive waste [11-14]. The ability of this material to form very insoluble products with many metals renders the macroencapsulation matrix even more attractive than others, because a chemical reaction actually occurs during the encapsulation [15].

The Y-12 Development Division of the ORR has investigated several stabilization technologies in support of the conceptual design for the Mixed Waste Treatment Facility and the Oak Ridge Applied Technology Program [16, 17]. Recent efforts have focused primarily on SPC and have involved sludge from the West End Treatment Facility, K-1232 B&C Pond, and Central Pollution Control Facility. Thermodynamic modeling of toxic metal reactions with SPC has been conducted and over 200 laboratory-scale experiments have been completed to assess the extent of waste stabilization and the effect of additives. Although numerous tests at both the laboratory and bench scale [at sister facilities such as Idaho National Engineering and Environmental Laboratory (INEEL)] have established the potential for this technology [18-21], it has not been demonstrated to meet end user needs of acceptance for disposal at Envirocare. Based upon these results, the idea of using SPC for this purpose was submitted to the MWFA. The SPC enhanced bench-scale or "fast-track" system has been designed for macroencapsulation of D004-D011 contaminated debris. The process generates minimal secondary wastes. Secondary solid wastes such as clothing, wipes, etc., can be encapsulated with primary debris wastes if deemed necessary.

Experiments were conducted initially to determine the conditions required to meet the selected WAC, particularly the absence of void spaces and discernible interfaces between the waste and the SPC matrix, based primarily upon visual inspection. Verification was performed by cross-cutting completed drums and recording the absence of boundaries by photographs. Once optimum conditions had been established, actual mixed waste material was similarly treated using debris from MWIR number OR-W063.

## 2. PROPERTIES OF THE MATERIAL

### 2.1 Origin and Properties of SPC

Sulfur has a large number of allotropic forms. The more common form,  $\alpha$ -sulfur, is rhombic and at 95.5°C undergoes transition to  $\beta$ -sulfur, which is monoclinic. This transformation is associated with a volume change and residual stresses in the final product due to the higher density of the  $\alpha$ -form [9]. The physical properties and final shrinkage of the sulfur-based materials were found to be incompatible with commercial objectives, because tests such as thermal cycling could result in final form disintegration when the sulfur is not used with stabilizing admixtures.

The Bureau of Mines initially studied this material. By adding 5% admixtures (i.e., dicyclopentadiene and oligomers of cyclopentadiene in equal amounts) to the sulfur, they created a modified sulfur cement that is now commercially available as SPC (or Chement) [4, 5]. This material possesses several interesting properties, the most important of which are its rapidly achieved high mechanical strength and its high resistance to corrosive environments [7, 8]. SPC has a viscosity at 135°C of  $50 \pm 25$  cp and a specific gravity at 25°C of  $1.90 \pm 0.02$ . Table 1 provides data on the chemical composition and physical characteristics of SPC.

**Table 1. Composition and properties of sulfur polymer cement**

Species or property	Value
Sulfur, %	$95 \pm 1$
Carbon, %	$4.5 \pm 0.2$
Hydrogen, %	$0.5 \pm 0.05$
Viscosity at 135°C, cp	$50 \pm 25$
Specific gravity at 25°C	$1.90 \pm 0.02$

*Source:* McBee, C. W., and Weber, H. H., "Sulfur Polymer Cement Concrete," in *Proceedings of the Twelfth Annual Department of Energy Low-Level Waste Management Conference*, CONF-9008119, National Low-Level Waste Management Program, Idaho National Engineering Laboratory, Idaho Falls, Idaho, 1990.

SPC is resistant to mineral acids and high-salt environments and also resists corrosive electrolyte attack. Table 2 summarizes test results obtained with sulfur concrete materials. As a result, some researchers predict that SPC could have twice the durability of portland cement; they base their conclusion on the impervious nature of the material.

Table 2. Results of industrial testing of sulfur concrete materials

Environment	Performance <sup>a</sup>
Sulfuric acid	Nonreactive
Copper sulfate-sulfuric acid	Nonreactive
Magnesium chloride	Nonreactive
Hydrochloric acid	Nonreactive
Nitric acid	Nonreactive
Zinc sulfate-sulfuric acid	Nonreactive
Copper slimes	Attacked by organics used in processing
Nickel sulfate	Nonreactive
Vanadium sulfate-sulfuric acid	Nonreactive
Uranium sulfate-sulfuric acid	Nonreactive
Potash brines	Nonreactive
Manganese oxide-sulfuric acid	Nonreactive
Hydrochloric acid-nitric acid	Nonreactive
Mixed nitric-citric acid	Nonreactive
Ferric chloride-sodium chloride-hydrochloric acid	Nonreactive
Boric acid	Nonreactive
Sodium hydroxide	Attacked by > 10% NaOH
Citric acid	Nonreactive
Acidic and biochemical	Nonreactive
Sodium chlorate-hypochlorite	Attacked by solution at 50 to 60°C
Ferric-chlorate ion	Nonreactive
Sewage	Nonreactive
Hydrofluoric acid	Nonreactive with graphite aggregate
Glyoxal-acetic acid formaldehyde	Nonreactive
Chromic acid	Deteriorated at 80°C and 90% concentration; marginal at lower temperature and concentration

<sup>a</sup>Nonreactive means that test results show no sign of corrosion or deterioration for test period of 6 to 9 years.

Source: McBee, C. W., and Weber, H. H., "Sulfur Polymer Cement Concrete," in *Proceedings of the Twelfth Annual Department of Energy Low-Level Waste Management Conference*, CONF-9008119, National Low-Level Waste Management Program, Idaho National Engineering Laboratory, Idaho Falls, Idaho, 1990.

McBee and Weber indicated that the optimum processing temperature range for SPC was between 127 and 138°C, while Darnell suggested a range of 129 to 141°C. Above the range of 150 to 160°C, a sharp rise in viscosity occurs because of additional polymerization within SPC that makes the material "gummy and unpourable" [22]; hydrogen sulfide (H<sub>2</sub>S) gas, which is poisonous and flammable, also forms. Additionally, if the processing temperature is too low, the sulfur will be incompletely or partially melted.

SPC is not recommended for use with strong bases and oxidizing agents, aromatic or chlorinated hydrocarbons, or oxygenated solvents because of the risk of chemical corrosion due to H<sub>2</sub>S formation [15]. Chemical reactions may also occur when SPC is used with nitrate salts due to the risk of combustion [23, 24], especially when carbon is present.

If some admixtures are not perfectly dry, the steam produced during the mixing, and especially during the cooling phase, will generate tiny vents to the surface of the waste form, making it porous [9, 24].

## **2.2 Use of SPC for Immobilization of Hazardous, Radiological, or Mixed Wastes**

The impervious properties of this new matrix drew interest among researchers trying to develop new waste forms to stabilize the mixed waste stored on many DOE sites. Scientists from Brookhaven National Laboratory first used this new matrix [11-14] and tested it with four different waste streams. Their studies were performed only at the bench-scale level; however, they performed all the testing requested by the U.S. Nuclear Regulatory Commission except one: the demonstration of feasibility at full scale. This part was then carried out by INEEL.

The approach of INEEL researchers was to develop a sulfur concrete comparable to cement concrete [19, 22, 23, 25-27]. They performed tests that always included pouring a mixture of 40 wt % incinerator ash (<0.95 cm) with 60 wt % SPC (SPC concrete) over coarse bottom ash and pipes of various sizes stacked in a large container. The filling efficiency of voids was determined by cross-sectioning and observation afterwards of the resulting waste form. INEEL researchers performed various tests with different mixers; however, their results were somewhat disappointing. They observed shrinkage, frothy-like crust on the surface (air venting was not completed when the mixture solidified), and some air pockets in the waste form.

In Europe, Van Dalen and Rijpkema [15, 21] studied borate waste (a simulation of waste from a pressurized-water reactor evaporator), lead iodide, a sludge resulting from water treatment, incinerator ash produced from burning a simulation of low-level wastes, and ion-exchange resins. Their list of preferred waste types for encapsulation in an SPC matrix includes inorganic low-solubility compounds like sludges, precipitates, and incinerator ashes. According to these researchers, organic materials (because they do not adhere to SPC), ion exchangers (because they destroy the matrix by the pressure of swelling), and highly soluble compounds (because they capture water by osmotic pressure and promote cracks of the waste form) are not good candidates. In France, Kertesz et al. [20] also used SPC for the immobilization of incinerator ashes.

Among the studies performed on this material, it is interesting to relate the tests performed in The Netherlands by Van Dalen [15], which provide some insight as to the sulfide formation between some metals and SPC. Strips of lead, stainless steel, aluminum, copper, and brass were dipped in the molten SPC, and the formation of a dark layer of metal sulfide was observed for those metals that have a higher affinity for sulfur than oxygen (i.e., lead, copper, and brass). This renders the matrix more attractive than others (e.g., polyethylene, resins), because it not only macroencapsulates the waste but also forms, to some extent, an insoluble form of the hazardous chemical.

### **3. DESIGN OF THE EXPERIMENTAL SETUP**

#### **3.1 Selection of the Casting Container Size**

The treatment standard for debris is macroencapsulation; however, EPA does not provide criteria for the design of such technology. Therefore, the WAC of Envirocare, Utah, which could be the location for the final disposal of the treated waste, were followed. Before selecting the equipment for the experimental work, it was necessary to select the size and shape of the final waste form that would be cast. Three main parameters were taken into consideration for the selection.

3. There was the possibility that the final waste forms would have to be shipped to Envirocare, Utah, and therefore, should follow the U.S. Department of Transportation (DOT) requirements for shipment of hazardous material (49 CFR 173.12).
4. The requirements of Envirocare for the macroencapsulated waste form were obtained.

Three conditions had to be met:

- a. A sample of the actual waste to be macroencapsulated should be provided, as well as a statement indicating that it represents the waste being encapsulated.
- b. A barrier of at least 2 in. made of the encapsulating material should surround the waste. Intimate contact with the waste inside is required. The waste should be encapsulated during a continuous pour.
- c. A copy of the Treatment Plan, showing that the methodology meets the definition of macroencapsulation, must be provided.

3. The demonstration being performed in a laboratory without special handling tools (e.g., a crane) could not generate a waste form too heavy for manual handling. Also, because of the short time frame available for the project, commercial equipment was needed and had to be readily available.

After considering all the issues involved, a DOT-approved, 5-gal metallic pail (1A2/Y1.8/100) was selected as the container to cast the waste form in.

### **3.2 Practical Information Gathered for Using SPC**

Useful data for the demonstration were found in the literature or by contacting SPC vendors. It was determined that it was necessary to maintain the heat of the material at a temperature of  $135^{\circ}\text{C} \pm 6^{\circ}\text{C}$ . At temperatures below  $128^{\circ}\text{C}$ , hard solidified blocks of material are formed, while at temperatures above  $150$  to  $160^{\circ}\text{C}$ , SPC starts emitting hydrogen sulfide gas. When this happens, the properties of the materials are altered such that pouring becomes impossible.

The container receiving the molten SPC mixture, as well as its contents, should be heated prior to pouring and during operations at  $135^{\circ}\text{C}$ . If not, voids occur in the waste form and cause it to lose its impervious properties. Such voids also induce thermal stress cracks in the final form.

Because the SPC matrix is not well suited for wet waste, all admixtures added to SPC must be dried prior to addition. McBee, who actually developed this process when working at the Bureau of Mines, recommended that aggregates to be incorporated with the sulfur cement have a moisture content less than 1% [9]. In the case of waste form production, Darnell et al. [19] reported a 0.2% moisture content for ash introduced into the SPC matrix during their full-scale test and recommended that the goal be 0% water to obtain a good waste form.

#### 4. EXPERIMENTAL SETUP

Based upon the various parameters described in Sect. 3, a design for the experimental setup was elaborated. The necessary pieces of equipment were purchased and modified, if necessary, to meet the needs of the experiment: a 5-gal pail heater equipped with a temperature thermostat, a melting pot equipped with thermostatic controls, heating tapes, and controllers. Figures 1-3 illustrate the arrangement and setup chosen.

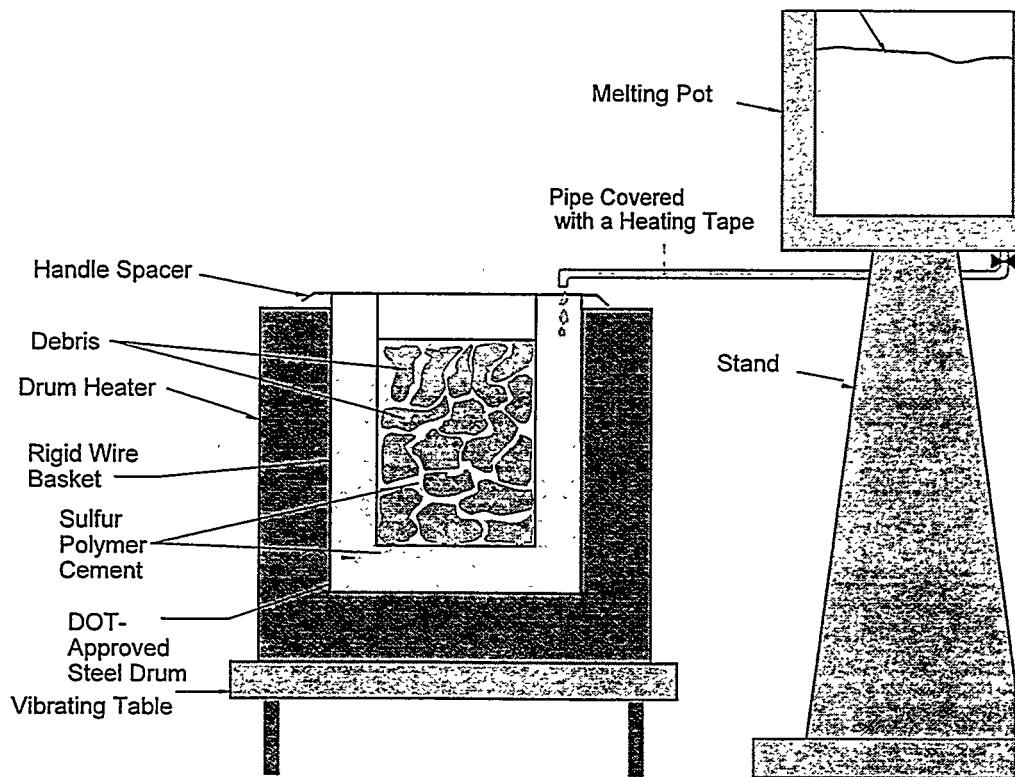


Fig. 1. Experimental setup for the demonstration.

The debris to be macroencapsulated was introduced into a wire basket that was sitting in the 5-gal pail by using a device that supported the weight, centered the basket, and maintained the basket securely to create a 2-in. surrounding layer of SPC. The space between the drum and the wire was filled with SPC to ensure that no pathway existed between the debris and the outside of the waste form. Molten SPC was poured into the drum to provide the outer layer of SPC and to fill the voids between the debris pieces. The pour of sulfur was

continuous until it reached about 2 in. from the top of the drum, at which level it was stopped. After hardening of the bottom part, a cap layer of molten SPC was added to the drum as a final barrier after removal of the holding device. Upon cooling and hardening of the SPC, the drum was sealed and could then be shipped for land disposal.

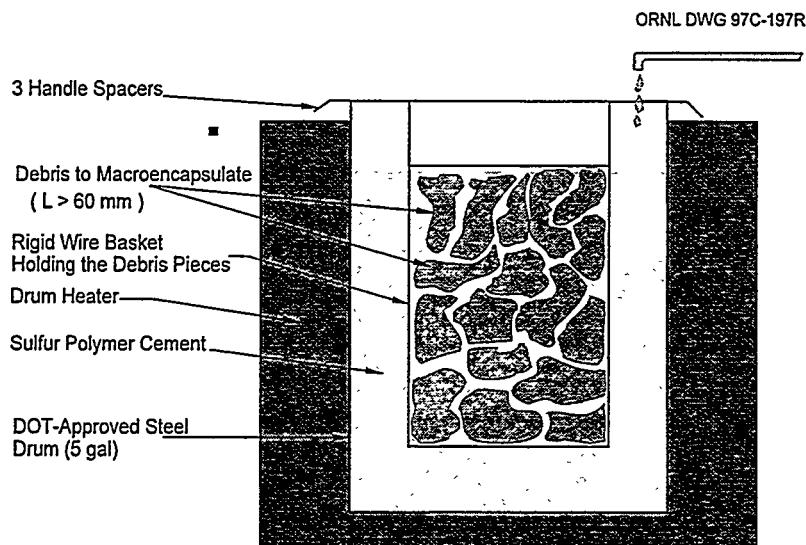


Fig. 2. Transverse view of the setup.

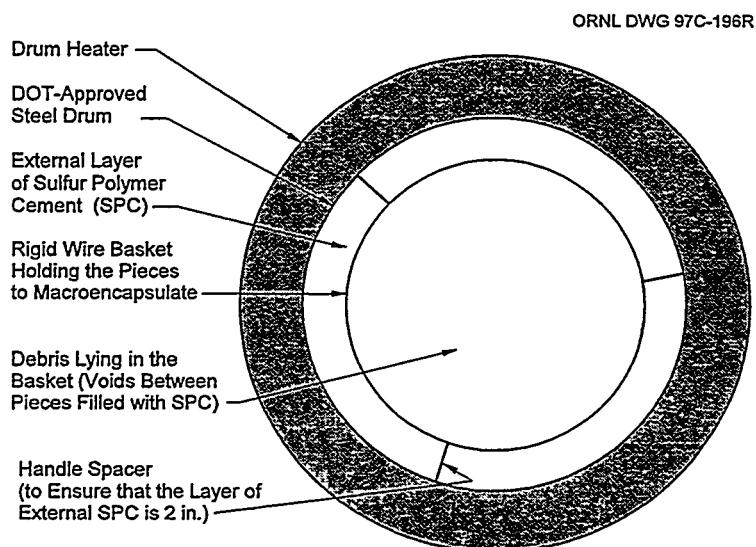


Fig. 3. Top view of the setup.

## 5. BENCH-SCALE TESTING

It is not possible to cut a pail containing macroencapsulated mixed waste; therefore, the demonstration of the quality of the product had to be done with "surrogates," pieces of scrap metal found in some shops around the building. The bench-scale tests were performed using metallic 1-gal pails. Some scrap metal pieces were used and placed in a wire mesh basket used for holding the pieces. Various conditions were tested: (1) heating/nonheating of the debris, (2) slow/rapid cooling of the waste form, (3) vibration/nonvibration during the pour, (4) continuous/noncontinuous pour, and (5) heating of the debris in an oven or in a heating mantel.

### 5.1 First Test Using a 1-gal Pail

In this test, the pieces of scrap metal were sitting in a wire mesh basket hanging in the 1-gal pail (Fig. A.1). The pail was placed in an oven at a temperature of about 130°C. The pieces were allowed to remain at this temperature for 2 h to ensure that complete dryness was achieved. Some chunks of SPC were added to the pail and allowed to melt. More SPC was added until the molten material covered the metal pieces. The molten SPC was mixed as well as possible. The oven was then turned off, and the pail was allowed to cool slowly inside the oven without opening the door. The transverse cut of the sample showed that void pockets were present at about 1 to 2 cm from the top (Fig. A.2).

### 5.2 Second Test Using a 1-gal Pail

In this test, the metallic pieces in the wire basket were heated with a heating tape placed around the metal pail. A thermocouple was placed inside the pail, with another one between the heating tape and the pail wall. The temperature inside the pail was 130°C, and that for the heating tape was 136°C for 4 h before pouring the molten SPC, which was performed in an oven regulated at 134°C. Only one-half of the pail was filled with SPC. The heating tape was turned off after pouring, but some insulating material was placed around the pail to prevent a too-rapid cooling rate. After cooling, a large crack was noticed at the surface of the hardened SPC. The whole pail was then reheated to correct this problem. More molten SPC was then added, and the pail was cooled down by gradually reducing the heat of the tape. After cooling, the pail was cut, and as in the previous test, the presence of air voids was observed at about 1 to 2 cm from the top of the hardened material (Fig. A.3). The hypothesis was made that the top layer of the material,

which was cooling faster, prevented the bubbles generated by the molten material underneath from escaping and that the next test would remedy this problem.

### 5.3 Third Test Using a 1-gal Pail

The pail containing the debris was heated in the oven at 130–135°C. The SPC was also melted while the debris was heated. After 9 h at that temperature, the pail was removed from the oven and placed in a metallic pan on a vibrating table. The molten SPC was then poured on top of the debris while the table was vibrating. Some insulating material was put on top and around the pail to reduce the cooling rate and prevent crack formation. After cooling, some large pores were observed at the surface of the hardened material. A heating tape was placed around the top portion of the pail in an attempt to remelt this portion only to remove the pores. After 5 h, only 2 cm of material was melted; therefore, the entire pail was put back in the oven for remelting. After one night in the oven at 130°C, the can of debris was removed, it was put on the vibrating table, and a heating tape was placed at the top of the pail to allow the top portion of the SPC to remain molten longer (Fig. A.4). After 10 min of vibration, the heating tape was set up at 140°C and an alumina cover was put on top of the pail to minimize the heat loss. Meanwhile, some SPC was melted in the oven to completely recover the debris pieces. The molten SPC was added to the pail and was vibrated for a brief period (~1 min). The alumina cap was then put on, and the heating tapes were left on for 5 h. After the SPC had cooled, the pail was cut transversely. Very few air voids were observed on top of the material (Fig. A.5).

### 5.4 Fourth Test Using a 1-gal Pail

The pail containing the debris was left in the oven overnight at 130°C for removal of any possible moisture and to heat the pieces. At the same time, some SPC was melted in the oven. The debris was removed from the oven and put on the vibrating table, and the molten SPC was introduced on top. An alumina cover was put on top of the pail. A heating tape was placed around the top portion of the pail to maintain the SPC in a molten state. The thermocouple placed between the pail and the tape indicated 139°C. The vibrating table was turned on for 20 min and then stopped. The heating tape allowed the top part to remain melted overnight. After cooling, the pail was cut and no voids were observed (Fig. A.6).

## 5.5 First Test Using a 5-gal Pail

It was then decided to make a couple of tests in a 5-gal pail to verify that the effect of scaling up would not modify the conditions found with the 1-gal size. These tests were also necessary to evaluate the equipment that would be used for the demonstration with mixed wastes. Cutting a pail with mixed waste debris would not be possible, so a surrogate was used to confirm that the process was set up satisfactorily.

The first test did not employ the standard basket designed for the demonstration (Fig. A.7), but rather a commercial size that was larger than needed. The basket was held in place by wire, not by the device described earlier. However, except for these two differences, the equipment was set up the same way throughout the demonstration (Fig. A.8).

The pail and its contents were heated in the drum heater at 130–140°C for at least 6 h. A thermocouple measuring the temperature inside the pail was located about 4 cm from the side and about 3 cm from the bottom of the pail. An insulated cover was put on top of the pail. Two 5-cm-wide heating tapes were placed at the top of the pail to provide another source of heat close to the top and to maintain the SPC in a molten state while the bottom part was cooling. Thermocouples placed on each tape, between the tape and the wall of the pail, were set to regulate the temperature at 130°C. The tapes were turned on at the same time as the drum heater.

While the debris was heating up, the SPC was melted in the temperature-regulated melting pot (Figs. A.9 and A.10). The temperature was set at 130–135°C and was checked with a thermocouple located about 2 to 3 cm from the bottom of the pot. Because of the larger volume of the chunks, many additions of SPC were necessary to fill the pot almost completely with molten material. The SPC was stirred from time to time to determine if more unmelted chunks were present.

The valve and pipe permitting the pour of the molten SPC inside the pail were also covered with a heating tape to prevent the molten SPC from freezing during the pour (Fig. A.11). After the first test was performed, it was found that it was best to turn the tape on at least 30 min before the pour and keep it at a temperature (with the thermocouple placed between the pipe and the tape) of 150°C.

After all the various pieces had reached the desired temperatures, the SPC was continuously poured to the top of the pail by opening the valve. The vibrating table was turned on

during the pour and for about 3 min after. It took about 8 to 10 min to fill the first layer. The two heating tapes were left on for 6 to 8 h while the drum heater was turned off. After cutting the pail of cooled SPC, as shown in Fig. A.12, some air bubbles were observed at about 2 to 3 cm from the top surface.

### 5.6 Second Test Using a 5-gal Pail

A second test was performed utilizing all the equipment intended for use with the actual mixed waste. The same operating conditions and setup as those in the first test were used, with one exception: the operating temperature for the heating tape was raised because it was suspected that this was the reason for the formation of bubbles in the previous test. The heating tapes were thus maintained at 150°C before and for 12 h after the pour. The molten SPC was poured to a level just below the basket holding device (Figs. A.13–A.16). The drum heater was stopped after the pour was completed. The next morning, the SPC had hardened (Fig. A.17), and the second layer was poured after removal of the basket holding device (Figs. A.18 and A.19). The surface of the hardened SPC appeared to have no visible defects (Figs. A.20 and A.21). After cooling, the pail was cut transversely (Figs. A.22 and A.23); no air bubbles were observed (Figs. A.24–A.26). Intimate contact between the SPC and the debris pieces was also observed as shown in Figs. A.25 and A.26. It was then decided to continue the demonstration using actual mixed wastes stored on-site at Oak Ridge National Laboratory (ORNL).

### 5.7 Summary of the Bench-Scale Testings

- As recommended in the literature, heating the debris to a temperature of 130–150°C is better for ensuring that no fast cooling of SPC occurs on the cold surfaces; this reduces the formation of air pockets.
- Vibrating the container holding the basket of debris proved to give better results in general. The vibration time was found to be sufficient when vibration was applied throughout the time of pouring and was maintained for a couple of minutes after the end of the pour.
- Samples obtained from a continuous pour were compared with samples made in three successive additions of SPC. After transverse cut of the samples, no obvious discontinuity was observed for multiple pours if the proper cooling procedure was followed.
- Without a cooling treatment step, the formation of air bubbles was observed at the top surface of the SPC. This was noticed whenever the cooling was slow (sample cooling

in the closed oven after it was turned off) or more rapid (the sample removed from the oven and allowed to cool down on the bench top). It was decided that the cause might be the faster cooling and hardening of the top layer, which prevented the bubbles generated by the still-molten SPC underneath from escaping. To remedy this problem, heating the top portion of the container while the rest was cooling down was tested. The heating was maintained for about 8 h. After cutting the sample, it was confirmed that no air pockets were present.

## 6. SELECTION OF MIXED WASTES

When this proposal was written, the goal was not only to demonstrate this technology for the treatment of mixed wastes but also to take advantage of this demonstration to eliminate one MWIR stream—OR-W063—from the national inventory.

ORNL Waste Management personnel were contacted to obtain a copy of their MWIR, indicating waste present on-site, as well as all information available to aid in selecting the waste candidates. The majority of the items present in this MWIR were only suspected to be radiologically contaminated; therefore, the known mixed wastes were targeted for this demonstration. Among these, some were not compatible for use with SPC (e.g., oxidizers). Some others did not meet the EPA definition of "debris." A copy of the Waste Management database, at the time of the demonstration, is provided in Appendix B.

Finally, only two waste streams were available for use in the demonstration:

- 208 kg (457 lb) of cadmium sheets, EPA waste code D006, waste item X10IH008284-001 (TS22), and
- 204 kg (448 lb) of lead pipes, EPA codes D008 and D009, waste item X10IH006100-001 (TS24).

Both of these wastes were perfect candidates because they were dry and therefore did not require an extensive and expensive drying process.

## 7. TREATABILITY STUDY USING ORR MIXED WASTES

ORNL Waste Management personnel delivered the first drum of cadmium sheets, which was opened under health physics supervision. The sheets of cadmium were individually

folded to fit inside the 55-gal drum. The size of the debris ranged from 10 cm to more than 1 m in one dimension; different thicknesses of metal were present (Fig. A.27). Size reduction was necessary (Fig. A.28) and was performed using sheet metal scissors in a radiological fume hood under a radiological work permit. Some of the thickest pieces could not be rendered flat enough to allow maximum compaction in the basket (Fig. A.29) in an effort to increase the waste loading. The data gathered during this treatability study are compiled in Table 3. Twelve 5-gal pails of macroencapsulated waste were generated.

Upon treatment of all the cadmium sheets, the second drum of waste was delivered and underwent the same operations. In this drum, pieces of lead pipes and seals were found (Figs. A.30 and A.31). Size reduction was necessary only for two pieces of 4-in. pipe, which were too long to fit in the basket and needed to be cut in half. A handsaw was used for this purpose. All the other pieces of debris could be folded or bent to the desired size (Fig. A.32). The data gathered during this treatability study are compiled in Table 4.

Because of Envirocare's very stringent WAC requiring a 5-cm layer of material surrounding the macroencapsulated debris, the waste loadings obtained were not very satisfactory. For the cadmium sheets, the waste loading volume obtained in the basket ranged from 19.4 to 42.5% (average 29.8%), depending on the possibility of flattening the pieces sufficiently to increase the quantity added in one basket. However, the waste loading volume obtained in the pail ranged from 5.7 to 12.5% (average 8.7%) only. For the lead pipes, which were easier to fold and break, the results are a little better, with a volume loading in the basket ranging from 27.5 to 52.3% (average 38.8%), which corresponds to a volume loading in the pail ranging from 8.1 to 15.4% (average 11.4%). It should be noted that the waste loading volume obtained in the basket is the one that reflects the waste loading as it is usually calculated. The value obtained for the pail includes another variable, the size of the pail itself, as shown in Table 5.

To conform to the criteria from Envirocare, a calculation was done to determine the available volume for the waste inside drums of various sizes. Having a 5-cm barrier of inert material in a 55-gal container makes more sense than having such a barrier in a 5-gal container, considering the volume available for waste: almost 60% of the 55-gal drum is available for waste but only 30% of the 5-gal drum.

Another criterion that is not taken into account with this 5-cm barrier is the nature and porosity of the matrix used for macroencapsulation. SPC and similar materials are very impervious when compared with cement-based matrices; therefore, consideration should be

Table 3. Data collected during the treatability study TS22 (cadmium sheet waste)

	TS22 A	TS22 B	TS22 C	TS22 D	TS22 E	TS22 F	TS22 G	TS22 H	TS22 I	TS22 J	TS22 K	TS22 L
Wt empty basket (kg)	0.30	0.30	0.35	0.35	0.40	0.35	0.35	0.35	0.35	0.35	0.40	0.35
Wt empty pail (kg)	1.65	1.65	1.65	1.65	1.80	1.80	1.70	1.75	1.70	1.75	1.75	1.65
Wt basket + debris (kg)	10.75	9.80	13.00	7.75	10.40	14.80	14.70	11.55	14.80	12.95	15.85	10.80
Wt debris (kg)	10.45	9.50	12.65	7.40	10.00	14.45	14.35	11.20	14.45	12.60	15.45	10.45
Gross wt (kg)	49.03	48.71	50.08	46.85	48.58	51.35	52.07	48.90	52.85	50.53	53.94	49.58
Wt SPC total (kg)	36.63	37.26	35.43	37.45	36.38	34.75	35.67	35.60	36.35	35.83	36.34	37.13
Wt SPC in basket (kg)	8.64	9.27	7.44	9.46	8.39	6.76	7.68	7.60	8.35	7.84	8.34	9.14
Vol SPC in basket (L)	4.55	4.88	3.91	4.98	4.41	3.56	4.04	4.00	4.40	4.13	4.39	4.81
Volume debris (L)	1.63	1.30	2.27	1.20	1.77	2.62	2.14	2.18	1.78	2.05	1.79	1.37
Vol loading in basket (%)	26.40	21.02	36.67	19.41	28.56	42.45	34.56	35.23	28.84	33.23	28.93	22.19
Vol loading in pail (%)	7.75	6.17	10.77	5.70	8.39	12.47	10.15	10.34	8.47	9.76	8.50	6.52
Wt loading in pail (%)	21.31	19.50	25.26	15.79	20.59	28.14	27.56	22.91	27.34	24.94	28.65	21.08
Density waste (g/cm <sup>3</sup> )	6.41	7.32	5.58	6.17	5.67	5.51	6.72	5.15	8.11	6.14	8.64	7.62

Table 4. Data collected during the treatability study TS24 (lead pipes)

	TS24 M	TS24 N	TS24 O	TS24 P	TS24 Q	TS24 R	TS24 S	TS24 T
Wt empty basket (kg)	0.40	0.35	0.40	0.30	0.35	0.35	0.30	0.35
Wt empty pail (kg)	1.75	1.70	1.70	1.75	1.70	1.65	1.60	1.65
Wt basket + debris (kg)	18.00	18.60	23.00	20.35	20.05	21.40	18.60	21.50
Wt debris (kg)	17.60	18.25	22.60	20.05	19.70	21.05	18.30	21.15
Gross wt (kg)	56.25	55.84	58.29	56.43	57.57	58.07	55.93	58.02
Wt SPC total (kg)	36.50	35.54	33.59	34.33	35.82	35.02	35.73	34.87
Wt SPC in basket (kg)	8.51	7.55	5.60	6.34	7.83	7.03	7.74	6.88
Vol SPC in basket (L)	4.48	3.97	2.95	3.34	4.12	3.70	4.07	3.62
Volume debris (L)	1.70	2.21	3.23	2.84	2.06	2.48	2.10	2.56
Vol loading in basket (%)	27.52	35.68	52.28	45.99	33.34	40.16	34.06	41.40
Vol loading in pail (%)	8.08	10.48	15.35	13.51	9.79	11.79	10.00	12.16
Wt loading in pail (%)	31.29	32.68	38.77	35.53	34.22	36.25	32.72	36.45
Density of the waste (g/cm <sup>3</sup> )	10.35	8.28	7.00	7.06	9.56	8.48	8.70	8.27

**Table 5. Calculation of the volume of the drum available for the waste when following Envirocare's WAC**

Vol (gal)	Vol (L)	Diam (cm)	Ht (cm)	Inner ht (cm)	Inner diam (cm)	Inner vol (L)	Vol, 2-in. layer (L)	Total vol (L)	% Inner vol in drum
5	18.90	28.58	33.02	23.02	18.58	6.23	14.93	21.17	29.5
10	37.80	35.40	39.37	29.37	25.40	14.88	23.86	38.73	38.4
15	56.70	35.40	59.13	49.13	25.40	24.89	33.29	58.18	42.8
20	75.60	40.64	58.74	48.74	30.64	35.92	40.24	76.15	47.2
30	113.40	46.36	69.37	59.37	36.36	61.60	55.42	117.02	52.6
55	207.90	57.15	83.03	73.03	47.15	127.44	85.43	212.87	59.9

given to the porosity of the material when determining the annular space required as a barrier.

## 8. FINAL DISPOSAL OF THE TREATED WASTES

The treatment technology required by the EPA to render the waste LDR compliant is not only a function of the codes it carries; it also depends upon the description of the waste made by the generator on some occasions. This is especially true for wastes in the "debris" category. To be considered "debris" by EPA, a waste must have been declared as such by the generator.

In the case of the lead pipes contaminated with mercury, both treatment standards were met when using SPC. For radiologically contaminated lead, the treatment standard is macroencapsulation; this is the process used in this demonstration. For mercury, amalgamation is the treatment standard required, and sulfur is cited by EPA as one of the chemicals that can be used. Even if not considered "debris," the macroencapsulation could render this waste LDR compliant.

Some discussions were held with Waste Management personnel to discuss the Environmental Compliance explanation about the meaning of the word "debris" in the waste

description. When paperwork is filled out by the generator at the site, this individual provides a description of the waste he or she wishes to dispose of. However, this person is often not aware of the various treatment options for the waste and may not provide in the description the type of wording that would permit some kinds of treatment (e.g., macroencapsulation for debris). The Waste Management Organization, which is in charge of disposing of the waste, can provide an extension of the waste description to allow such types of treatment.

The Waste Management database was modified to reflect the fact that the two waste streams used in the demonstration actually meet the EPA requirements for debris. The database now includes the word "debris" in the waste description. With this change, the debris cadmium sheets were rendered LDR compliant by their macroencapsulation in SPC. Both treated wastes will be disposed of as low-level radioactive waste on-site.

## 9. CONCLUSIONS

The use of SPC as a macroencapsulation matrix for mixed waste debris has been demonstrated successfully during this project. One waste stream from the national MWIR has been treated for the known mixed wastes it contained (more than 400 kg) when these were suitable for this technology. The waste has been rendered LDR compliant and is therefore ready for final disposal.

This treatment process was generally found to be simple to use. It was very well suited for the solid, dry wastes treated. Debris preheating was performed only to bring the pieces to a temperature slightly above the melting point of SPC, not to evaporate water contained in the waste. One of the advantages of using SPC is that there is no need for characterization of the waste to be encapsulated, thus reducing the cost associated with the use of this technology. Also, SPC has a great advantage over some other macroencapsulating matrices (resins, polyethylene, etc.) because it actually forms very insoluble sulfide compounds with

many metals such as mercury, lead, or cadmium, providing at least some minimal chemical stabilization during the encapsulation process.

## 10. REFERENCES

1. Zirker, L. R., Thiesen, T. J., Tyson, D. R., Beitel, G. A., "Macroencapsulation of Lead and Steel Swarf," in *Mixed Waste—Proceedings of the Third Biennial Symposium*, Baltimore, Md., August 7–10, 1997, Idaho National Engineering Laboratory, Idaho Falls, Idaho.
2. Getty, R. H., Riendeau, M. P., *Polymer Macroencapsulation of Low Level Radioactive Lead Wastes*, Interim report TI95-018, Rocky Flats Environmental Technology, Golden, Colo., 1995.
3. Getty, R. H., Riendeau, M. P., *Thermoset Macroencapsulation of Beryllium Contaminated Low Level Radioactive Mixed Debris Waste*, TI95-019, Kaiser Hill, LLC, Golden, Colo., 1996.
4. Sullivan, T. A., McBee, W.C., *Development and Testing of Superior Sulfur Concretes*, RI 8160, Bureau of Mines, Boulder City Metallurgy Research Laboratory, Boulder City, Nev., 1976.
5. McBee, W. C., Sullivan, T. A., *Development of Specialized Sulfur Concretes*, RI 8346, Bureau of Mines, Pittsburgh, 1979.
6. McBee, W. C., Sullivan, T. A., Jong, B. W., *Modified-Sulfur Cements for Use in Concretes, Flexible Pavings, Coatings, and Grouts*, RI 8545, Bureau of Mines, Pittsburgh, 1981.
7. McBee, W.C., Sullivan, T.A., Jong, B.W., *Corrosion-Resistant Sulfur Concretes*, RI 8758, Bureau of Mines, Pittsburgh, 1983.

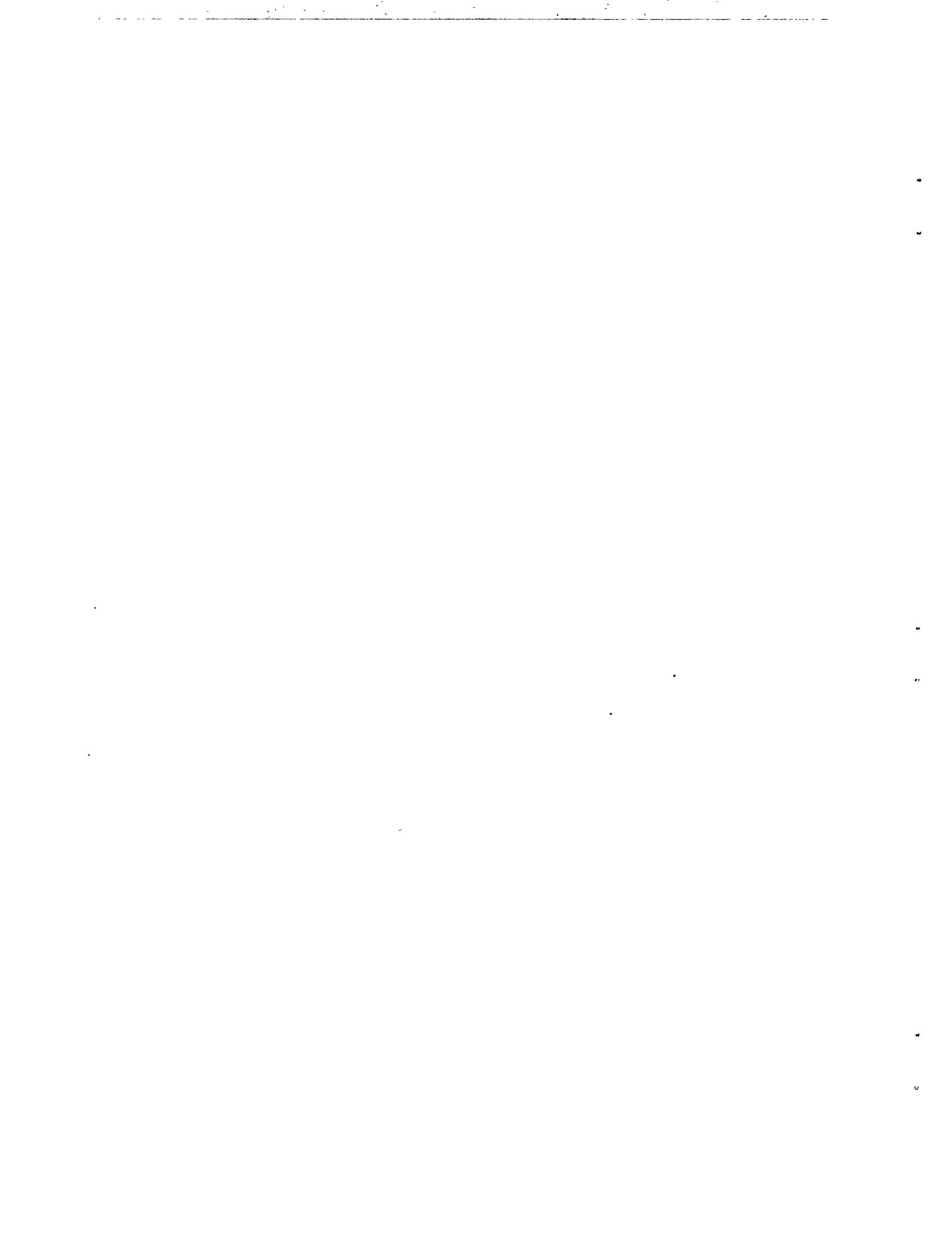
8. McBee, W. C., Sullivan, T. A., Jong, B. W., *Industrial Evaluation of Sulfur Concrete in Corrosive Environments*, RI 8786, Bureau of Mines, Pittsburgh, 1983.
9. McBee, W. C., Weber, H. H., "Sulfur Polymer Cement Concrete," in *Proceedings of the Twelfth Annual Department of Energy Low-Level Waste Management Conference*, CONF-9008119, National Low-Level Waste Management Program, Idaho National Engineering Laboratory, Idaho Falls, Idaho, 1990.
10. Mattus, C. H., Mattus, A. J., *Evaluation of Sulfur Polymer Cement as a Waste Form for the Immobilization of Low-Level Radioactive or Mixed Waste*, ORNL/TM-12657, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1994.
11. Arnold, G., et al., *Modified Sulfur Cement Solidification of Low-Level Wastes*, BNL-51923, Brookhaven National Laboratory, Upton, N.Y., 1985.
12. Kalb, P. D, Heiser, J. H., III, Colombo, P., "Comparison of Modified Sulfur Cement and Hydraulic Cement for Encapsulation of Radioactive and Mixed Wastes," in *Proceedings of the Twelfth Annual Department of Energy Low-Level Waste Management Conference*, CONF-900819-2, National Low-Level Waste Management Program, Idaho National Engineering Laboratory, Idaho Falls, Idaho, 1990.
13. Kalb, P. D, Heiser, J. H., III, Colombo, P., *Encapsulation of Mixed Radioactive and Hazardous Waste Contaminated Incinerator Ash in Modified Sulfur Cement*, BNL-43691, Brookhaven National Laboratory, Upton, N.Y., 1990.
14. Kalb, P. D., et al. *Durability of Incinerator Ash Waste Encapsulated in Modified Sulfur Cement*, BNL-45292, Brookhaven National Laboratory, Upton, N.Y., 1991.

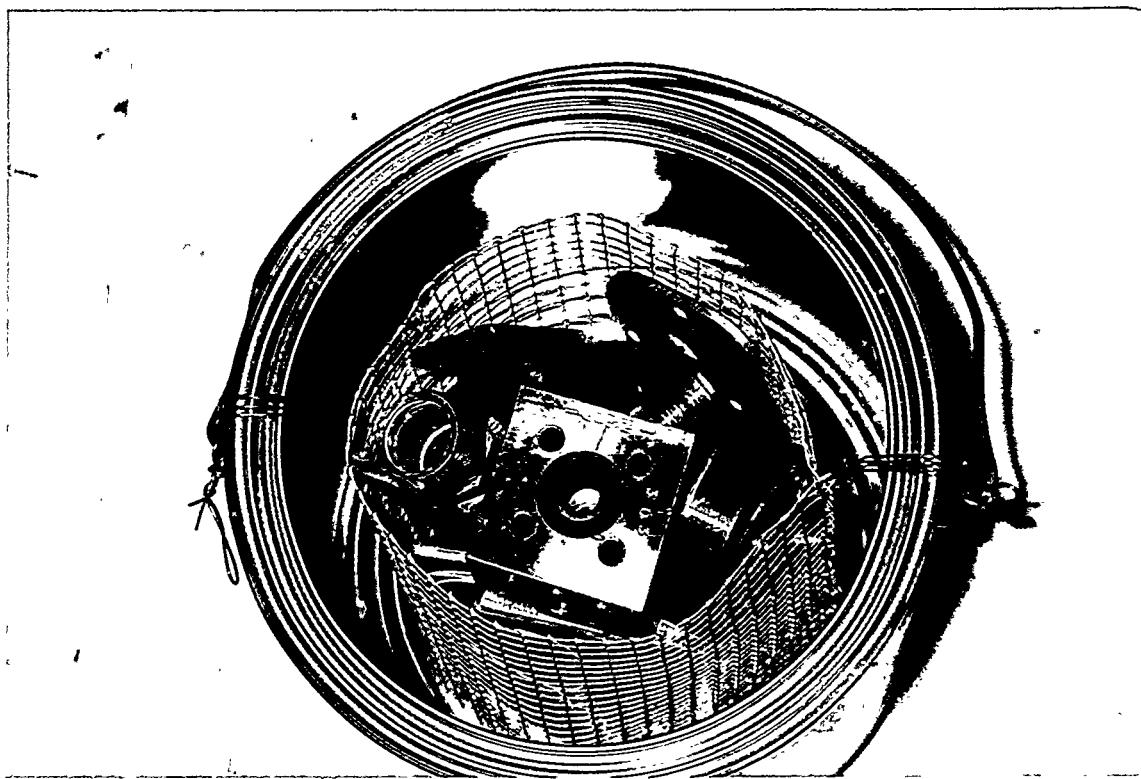
15. Van Dalen, A., Rijkema, J. E., *Modified Sulphur Cement: A Low Porosity Encapsulation Material for Low, Medium and Alpha Waste*, EUR 12303 EN, Commission of the European Communities, Luxembourg, Belgium, 1989.
16. Calhoun, C. L., Jr., Nulf, L. E., Gorin, A. H., *Sulfur Polymer Cement Encapsulation of RCRA Toxic Metals and Metals Oxides*, Y/DZ-1145, Oak Ridge Y-12 Plant, Oak Ridge, Tenn., 1995.
17. Calhoun, C. L., Jr., Nulf, L. E., Fedorov, V. V., *Sulfur Polymer Cement Encapsulation of Oily Matrix Mixed Low-Level Sludge*, Y/DZ-2010, Oak Ridge Y-12 Plant, Oak Ridge, Tenn., 1996.
18. Gering, K. L., "Solidification Results from a Treatability Study of Nonincinerable Low-Level Mixed Wastes," pp. 1.4.1-1.4.28 in *Proceedings of the Second International Symposium on Mixed Waste*, Baltimore, Md., August 17-20, 1993, A. A. Moghissi, R. K. Blauvelt, G. A. Benda, and N. E. Rothermich, eds., Temple University, Philadelphia, 1993.
19. Darnell, G. R., "Intense Volume Reduction of Mixed and Low-Level Waste, Solidification in Sulphur Polymer Concrete and Excellent Disposal at Minimum Cost," in *Proceedings of the Twelfth Department of Energy Low-Level Waste Management Conference*, CONF-900819, National Low-Level Waste Management Program, Idaho National Engineering Laboratory, Idaho Falls, Idaho, 1990.
20. Kertesz, C. J., Chevanas, P. R., Aufret, L., *Conditionnement de cendres d'incinérateur alpha et beta gamma, obtenues par incinération des déchets radioactifs, par enrobage, dans différentes matrices*, EUR 14365 FR, Commissariat à l'Energie Atomique, Centre de Cadarache, Commission des Communautés Européennes, Luxembourg, Belgium, 1993.

21. Van Dalen, A., Rijkema, J. E., "Matrix Properties of Modified Sulfur Cement for Radioactive Incinerator Ashes," in *Treatment and Conditioning of Radioactive Incinerator Ashes*, L. Cecille and C. Kertesz, eds., Elsevier, New York, 1991.
22. Darnell, G. R., Aldrich, W. C., Logan, J. A., *Full-Scale Tests of Sulfur Polymer Cement and Non-Radioactive Waste in Heated and Unheated Prototypical Containers*, EGG-WM-10109, Idaho National Engineering Laboratory, Idaho Falls, Idaho, 1992.
23. Darnell, G. R., "Sulphur Polymer Cement, a New Stabilization Agent for Mixed and Low-Level Radioactive Waste," pp. 7.1.1-7.1.11 in *Proceedings of the First International Symposium on Mixed Waste*, Baltimore, Md., August 26-29, 1991, A. A. Moghissi and G. A. Benda, eds., University of Maryland, Baltimore, 1991.
24. *Mixed and Low-Level Waste Treatment Facility Project, Vol. 3: Waste Treatment Technologies*, EGG-WMO-10244, Idaho National Engineering Laboratory, Idaho Falls, Idaho, April 1992.
25. Darnell, G. R., *Progress Report: Full-Scale Tests with Sulfur Polymer Cement Since the Previous Report, February 1992*, GRD-73-93, Idaho National Engineering Laboratory, EG&G Idaho, Idaho Falls, Idaho, August 1993.
26. Darnell, G. R., *Sulfur Polymer Cement, a New Stabilization Agent for Mixed and Low-Level Radioactive Waste*, EGG-M-91419, Idaho National Engineering Laboratory, Idaho Falls, Idaho, 1992.
27. Darnell, G. R., *Sulfur Polymer Cement, a New Final Waste Form for Radioactive and Hazardous Wastes*, GRD-29-92, Idaho National Engineering Laboratory, EG&G Idaho, Idaho Falls, Idaho, Nov. 19, 1992.

## **APPENDIX A**

### **PHOTOGRAPHS ILLUSTRATING THE DEMONSTRATION**



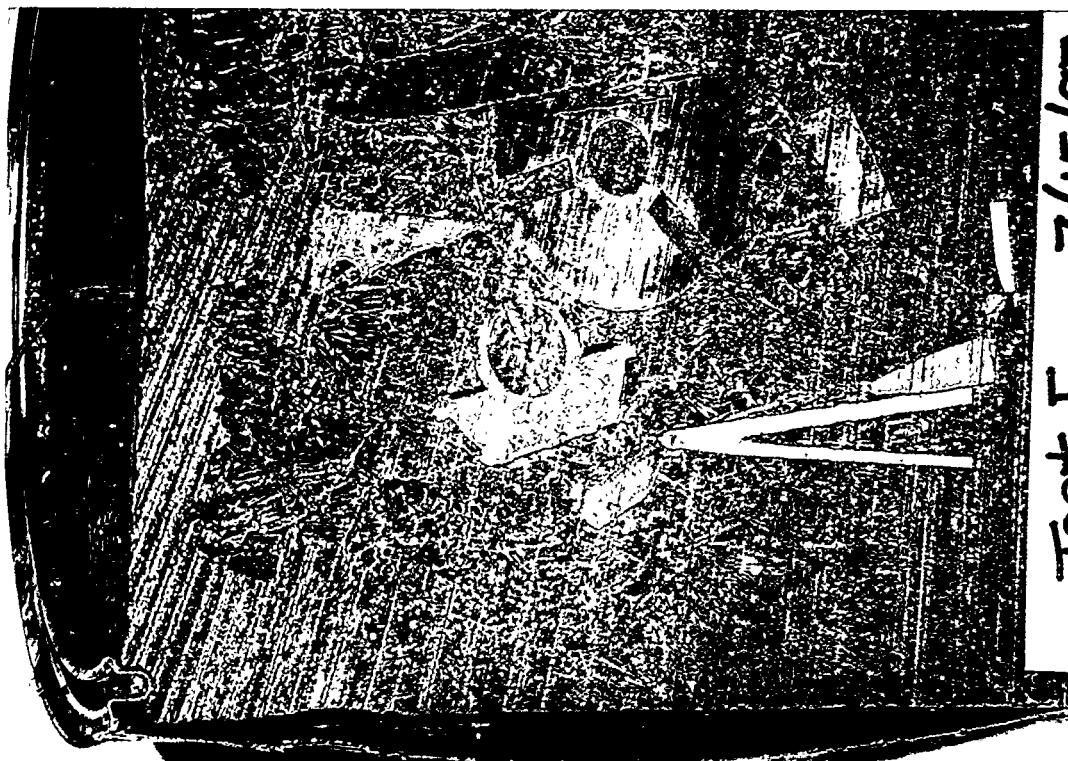


**Fig. A.1. Bench-scale test using scrap metals and 1-gal pail.**



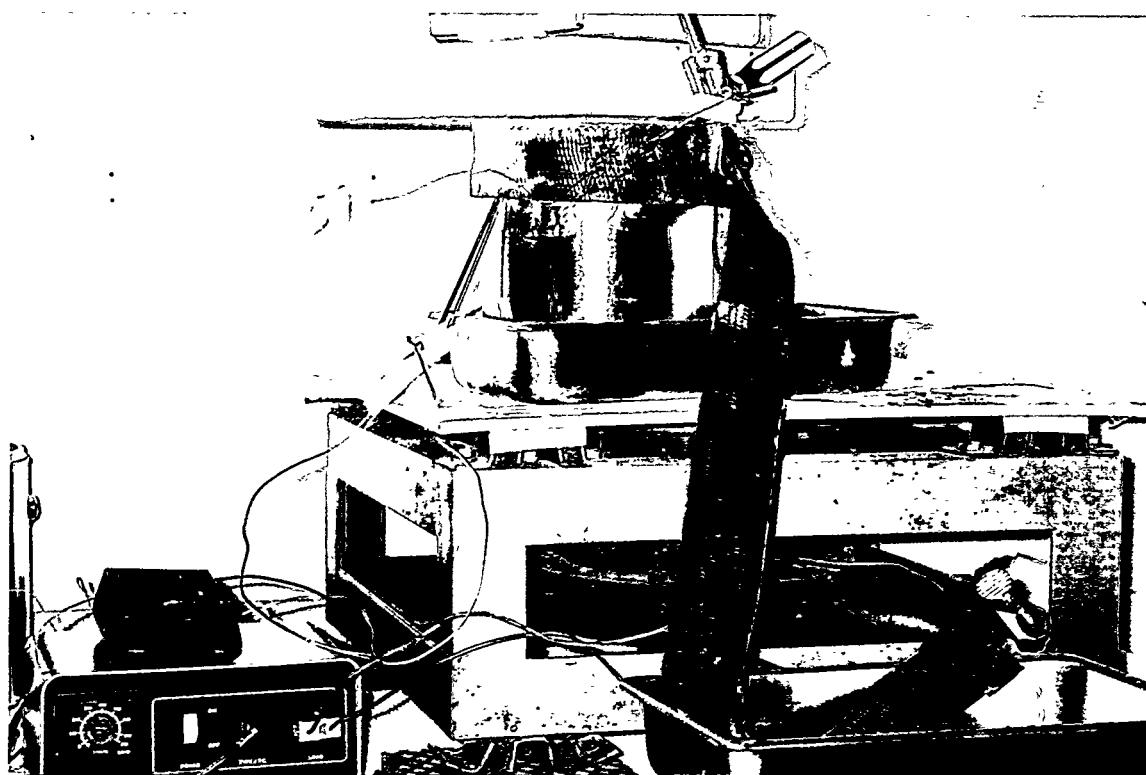
Test G 7/17/97

Fig. A.3. Transverse cut after introducing melted SPC into the heated pot.

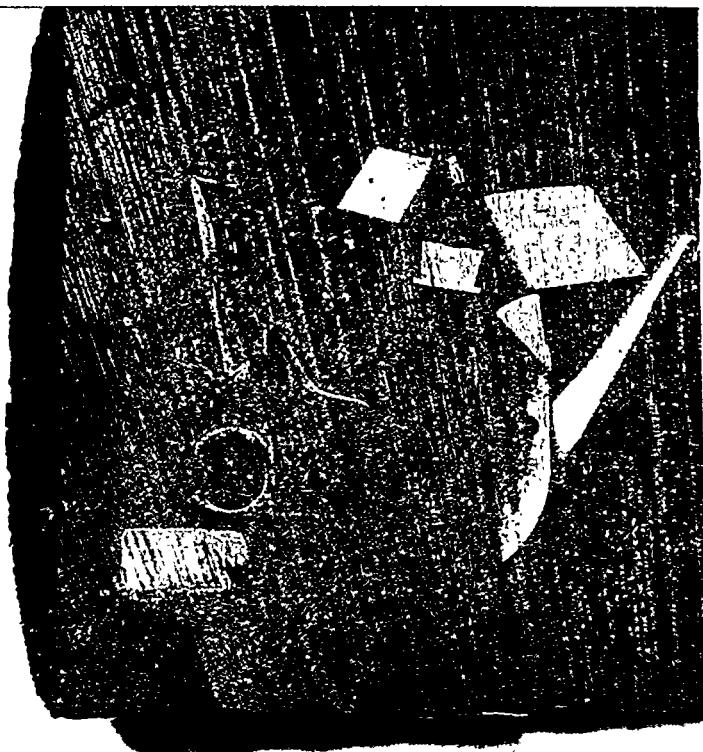


Test F 7/15/97

Fig. A.2. Transverse cut after direct melting of SPC in a pail in the oven. The presence of air voids is noted at the top.

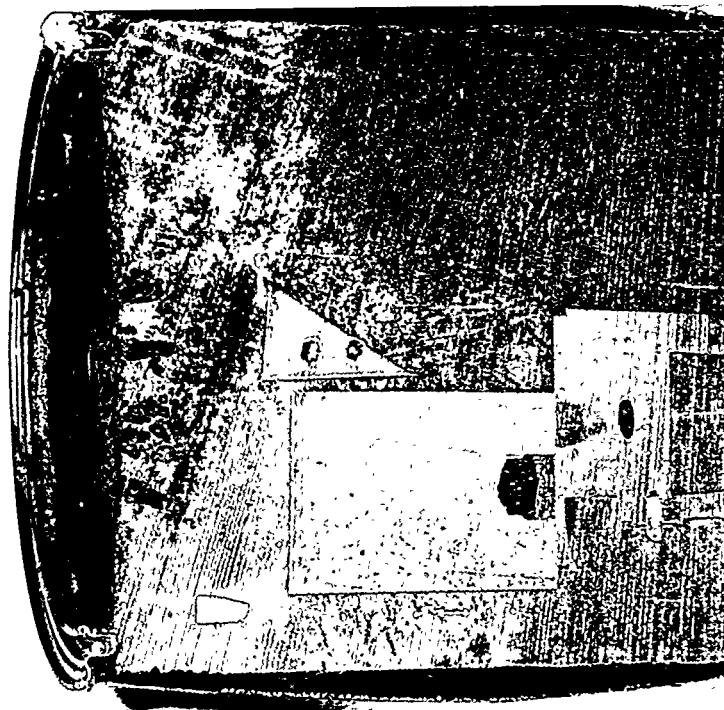


**Fig. A.4.** Operational setup for the bench-scale test.



7/21/97 Test T

Fig. A.5. Transverse cut after pouring molten SPC over the heated debris with accompanying vibration.



7/21/97 Test H

Fig. A.6. Transverse cut after pouring molten SPC over the heated debris with accompanying vibration and extended melting time for the top portion.

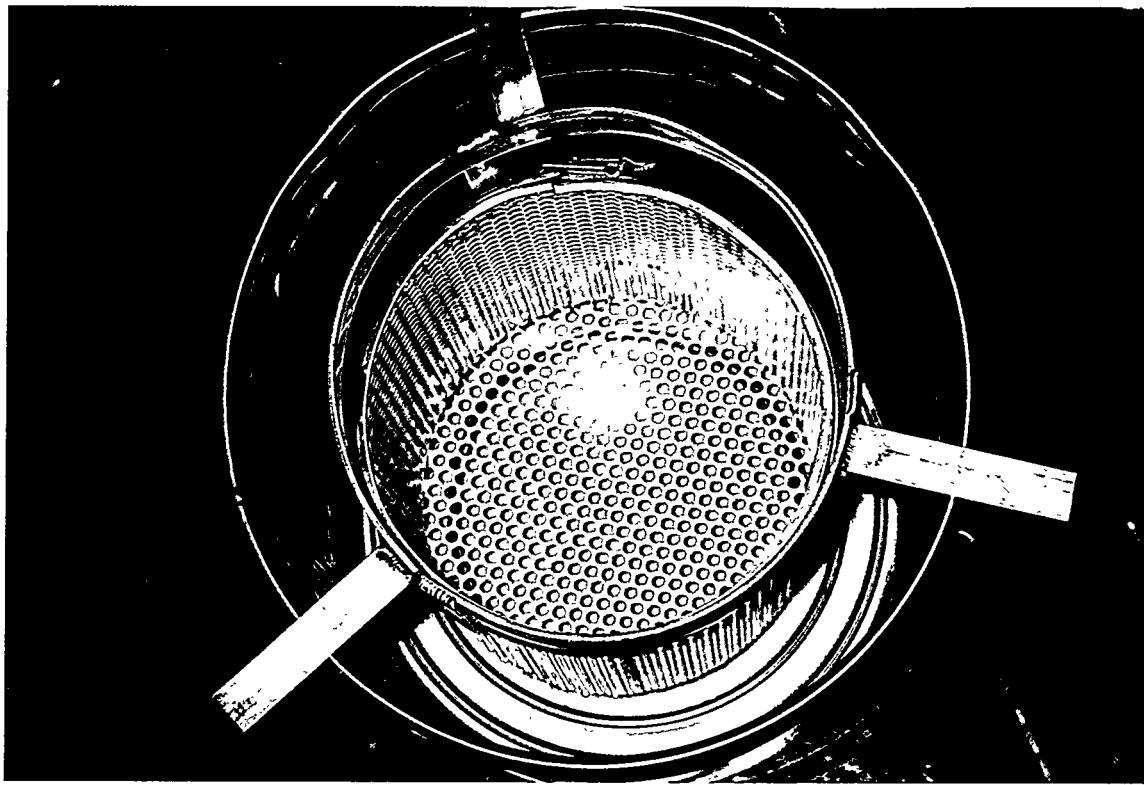


Fig. A.7. Holding device, which supports and centers the basket during pouring of the SPC.

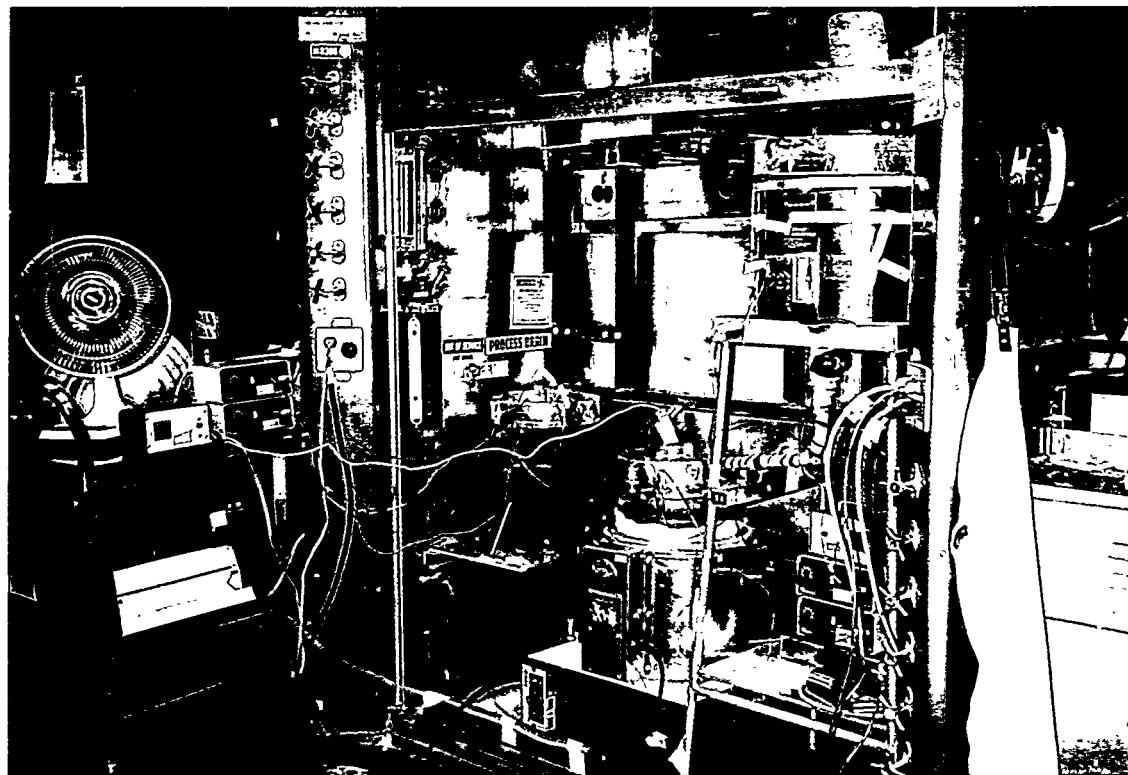


Fig. A.8. Experimental setup.



Fig. A.9. Detail of the SPC chunks.



Fig. A.10. Melting of SPC performed in the warming pot.

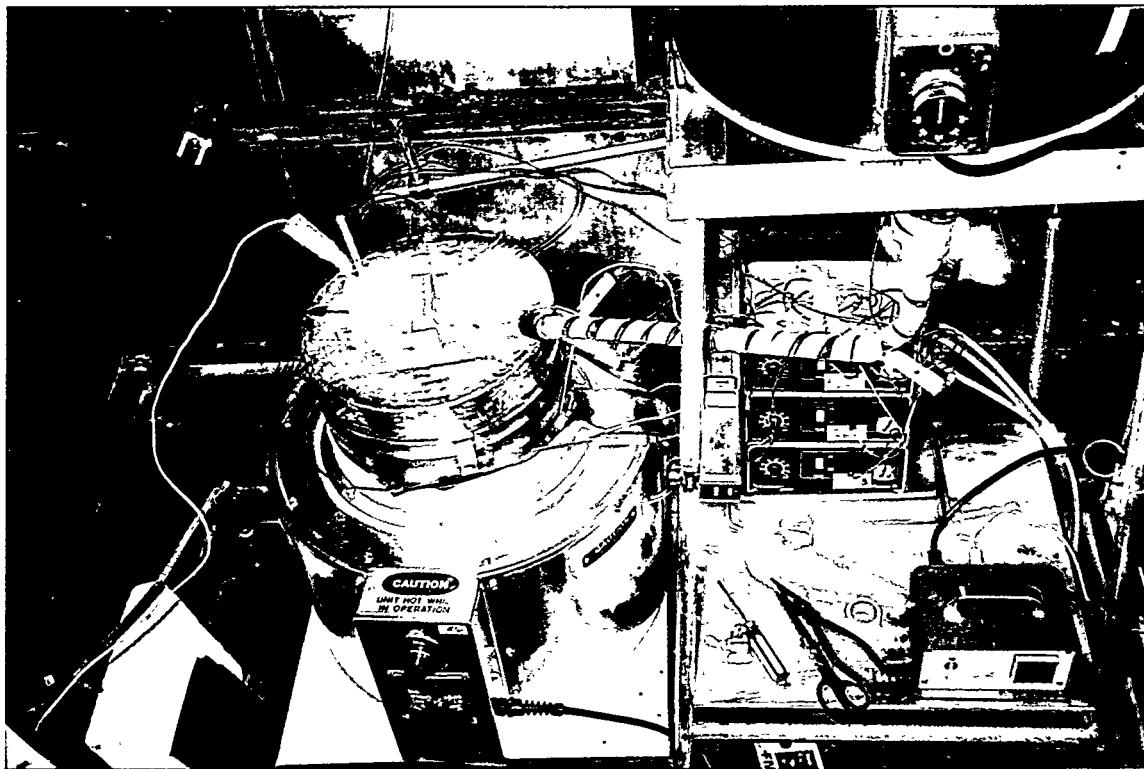


Fig. A.11.. Use of an insulated cover to minimize the heat loss.



**Fig. A.12. Transverse cut of the first test, made by using a 5-gal pail.**

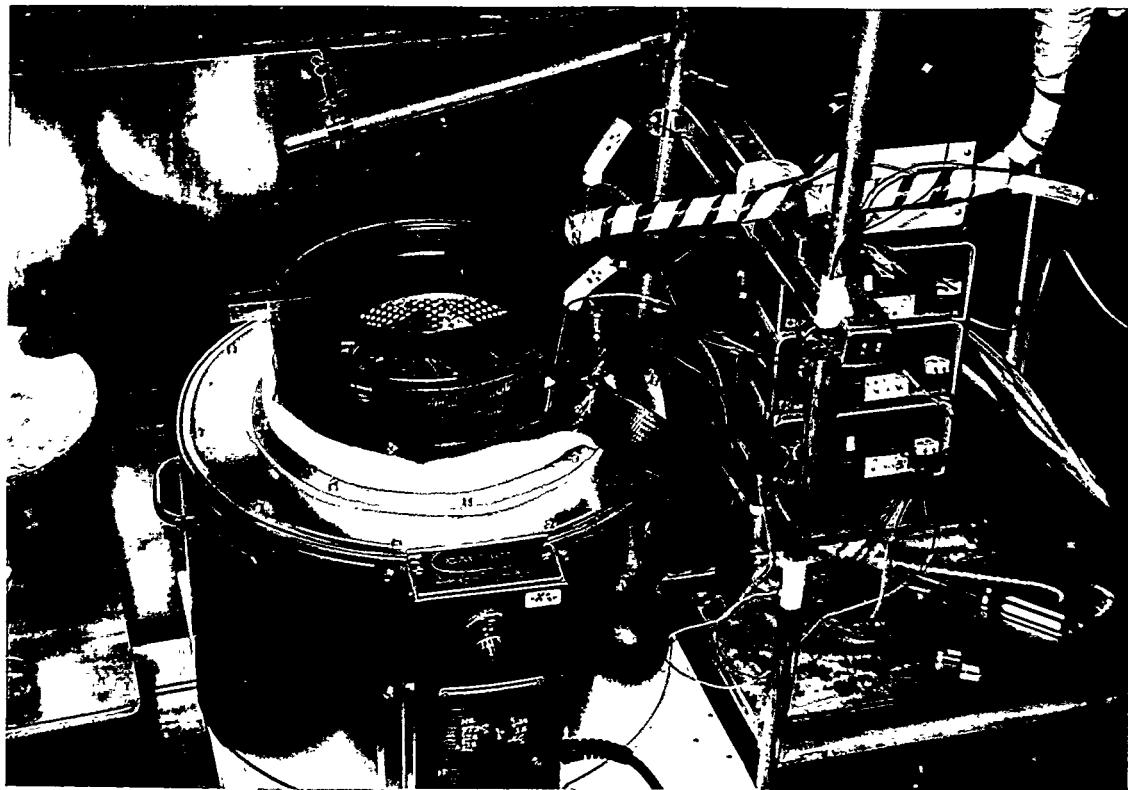


Fig. A.13. View of the basket during the drying stage.

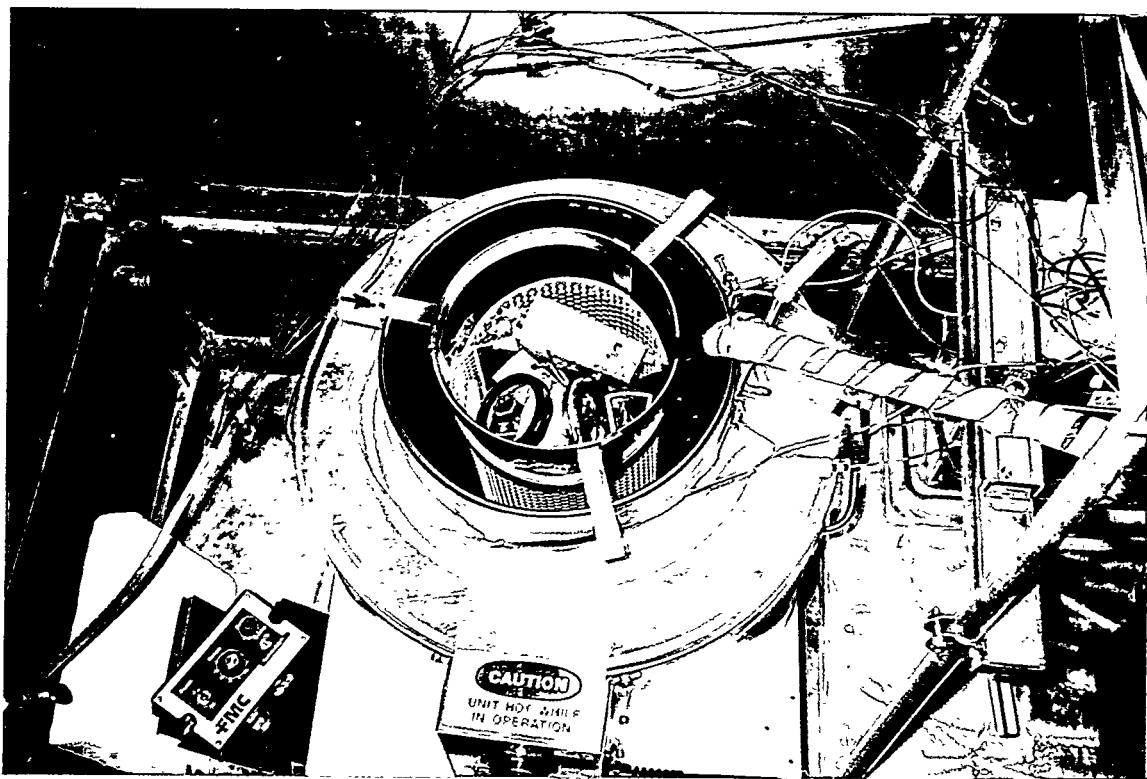


Fig. A.14. Pouring of the molten SPC while the vibrating table is operating.

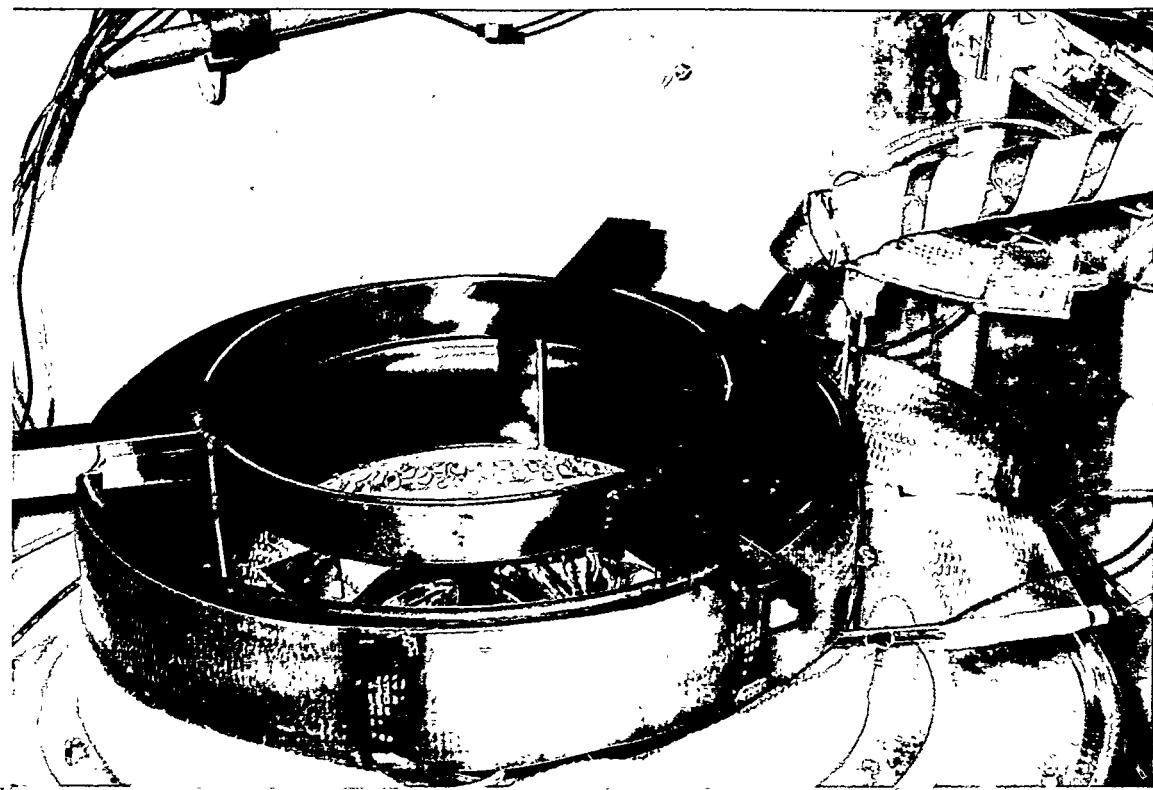


Fig. A.15. Fluidity of the molten SPC, which permits even small pores to be filled.

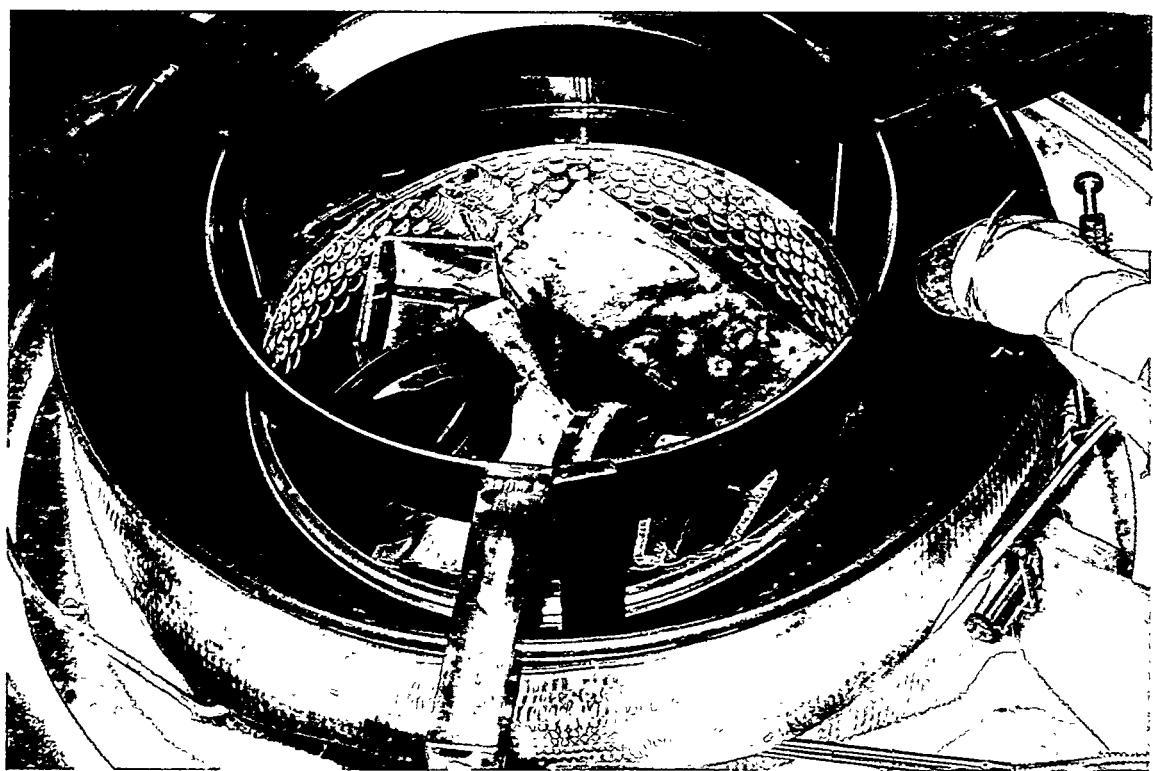


Fig. A.16. Preventing the molten SPC from reaching the holding device.

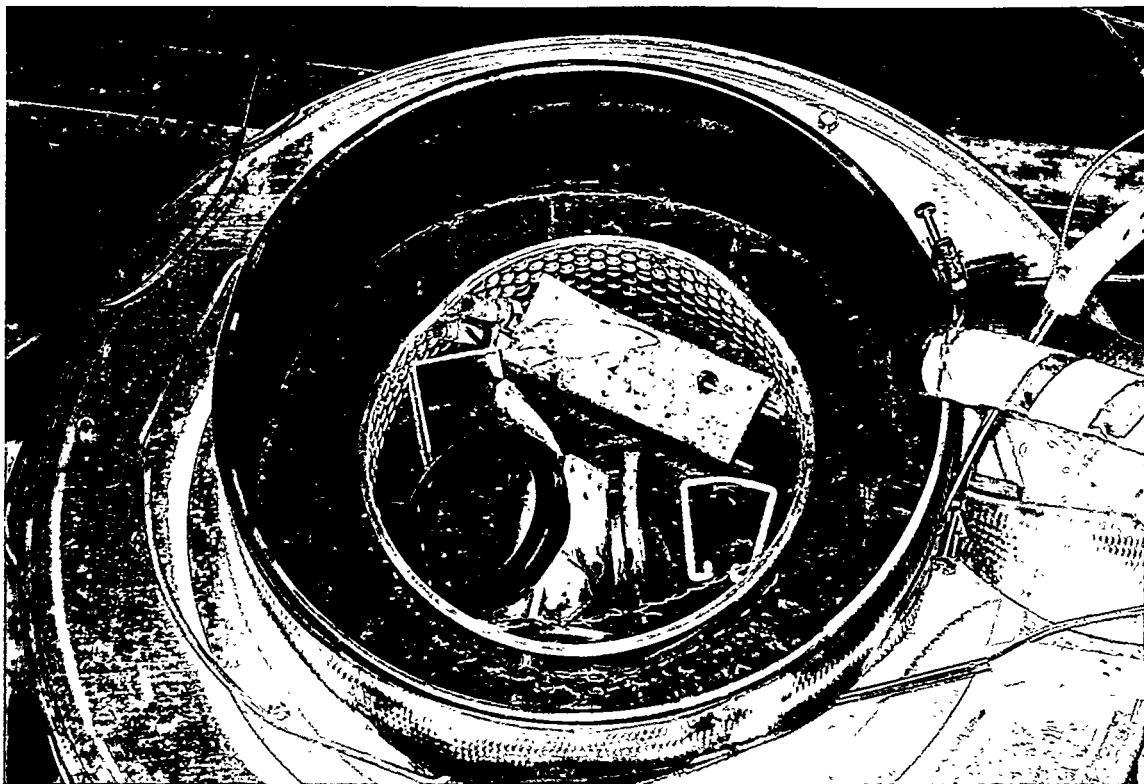


Fig. A.17. Removal of the holding device after cooling of the SPC.

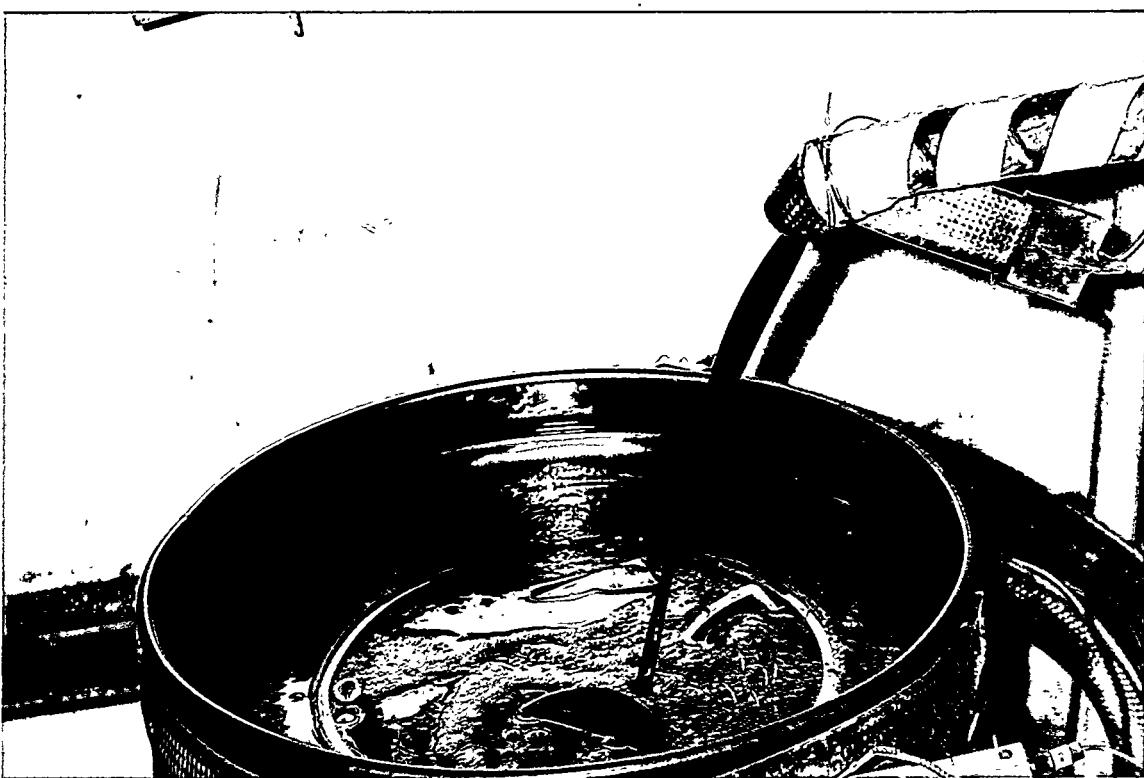


Fig. A.18. Heating the top portion of the drum prior to pouring the second layer of SPC.

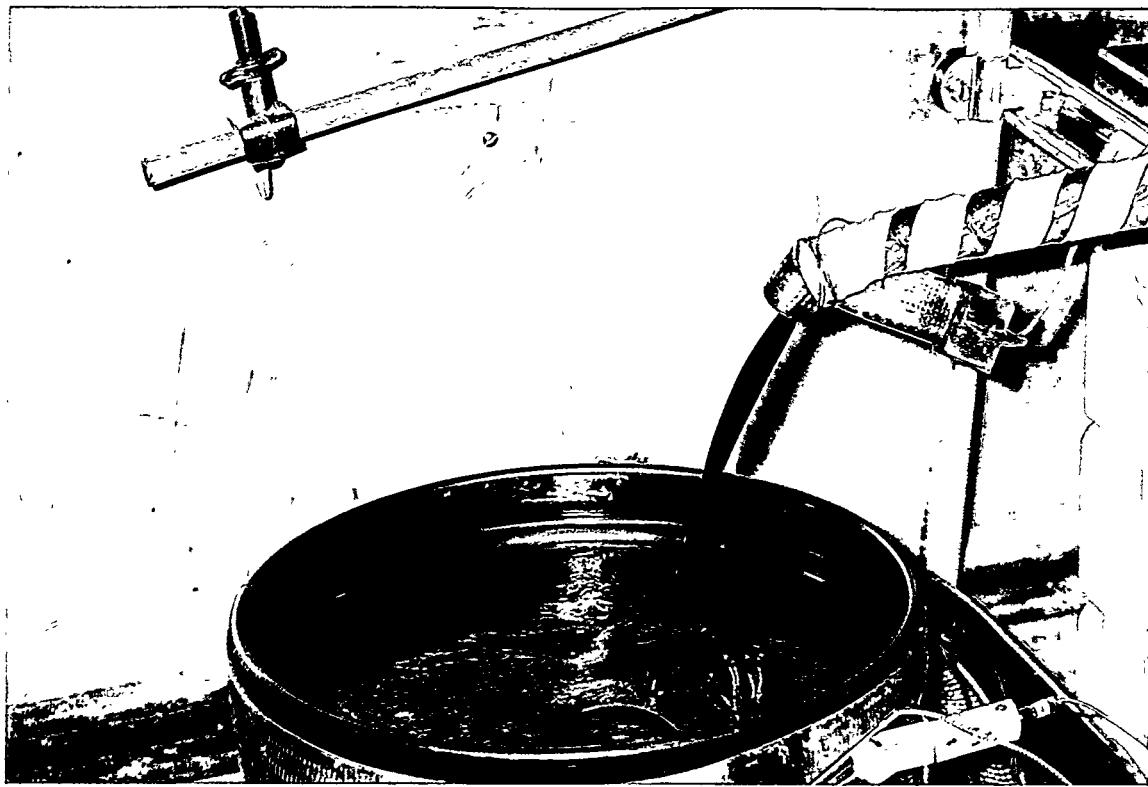


Fig. A.19. Filling the remainder of the pail with SPC.

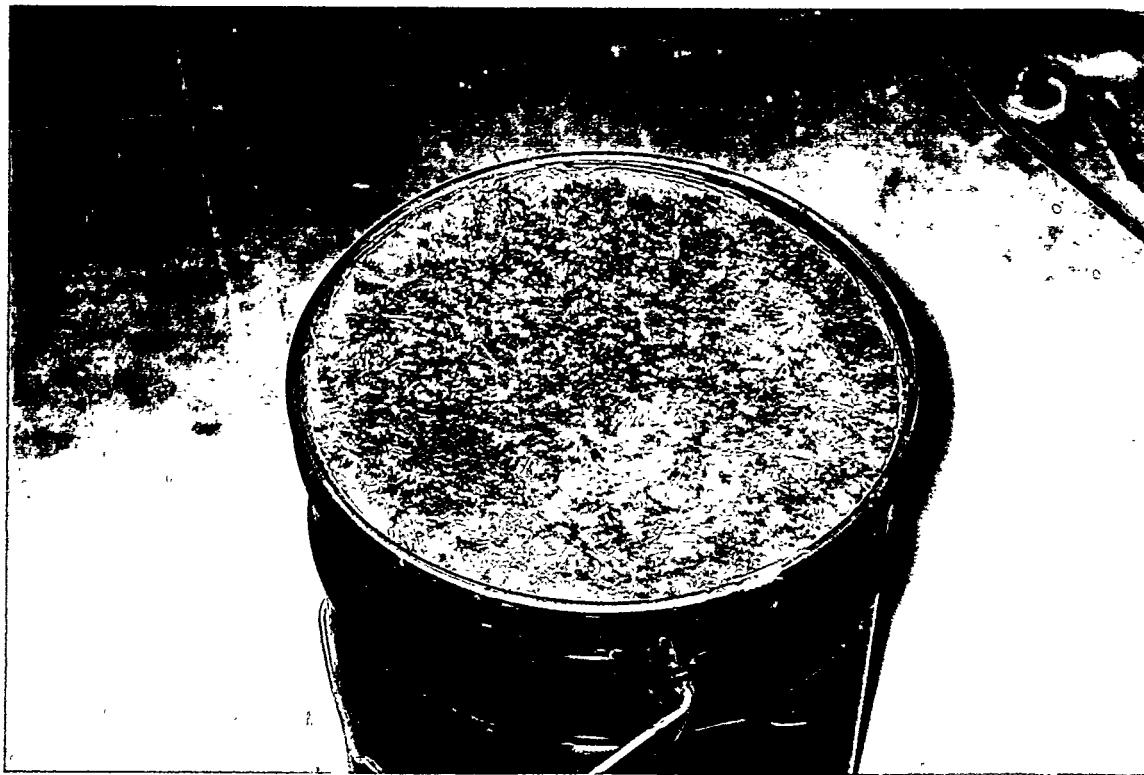


Fig. A.20. Completion of the macroencapsulation—after cooling of the second layer.



**Fig. A.21.** Surface of the SPC, revealing no apparent defect.



Fig. A.22. Transverse cut of the second test, made by using a 5-gal pail.

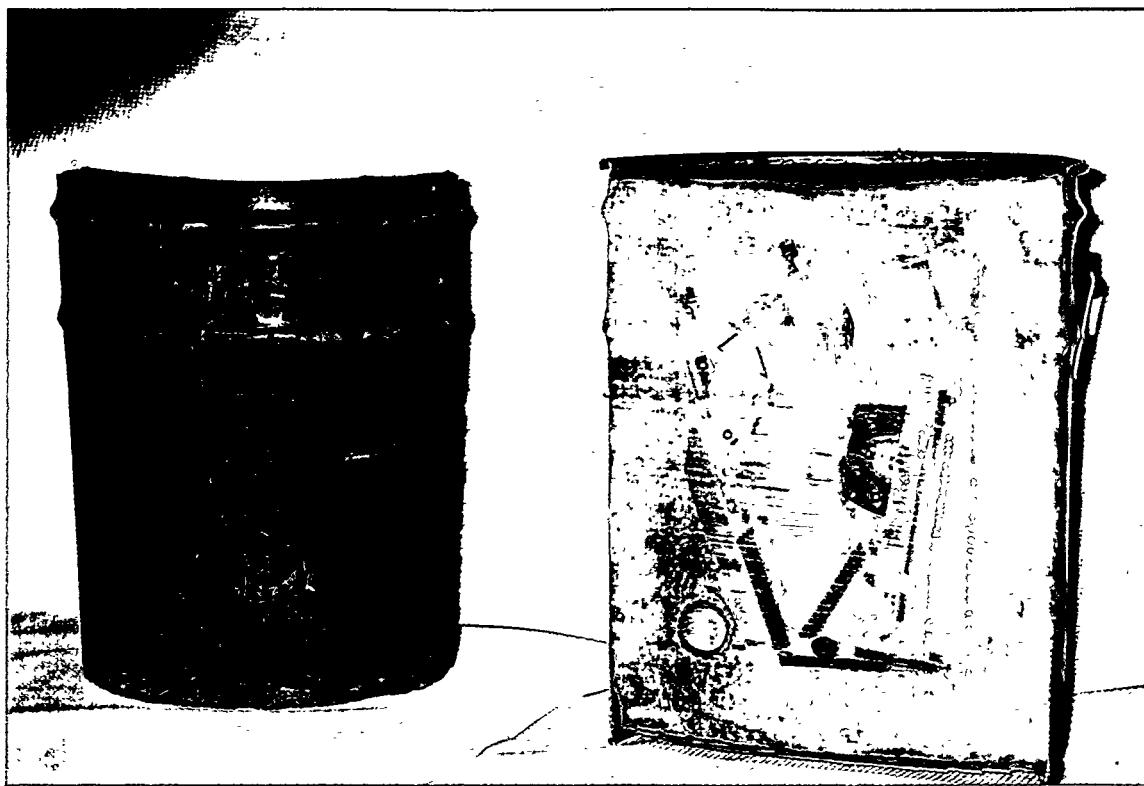


Fig. A.23. Outside and inside views of the waste form.



Fig. A.24. Detail of the peripheral surfaces.



Fig. A.26. Transverse cut, revealing moderate porosity on the top that does not appear to connect to the outside.

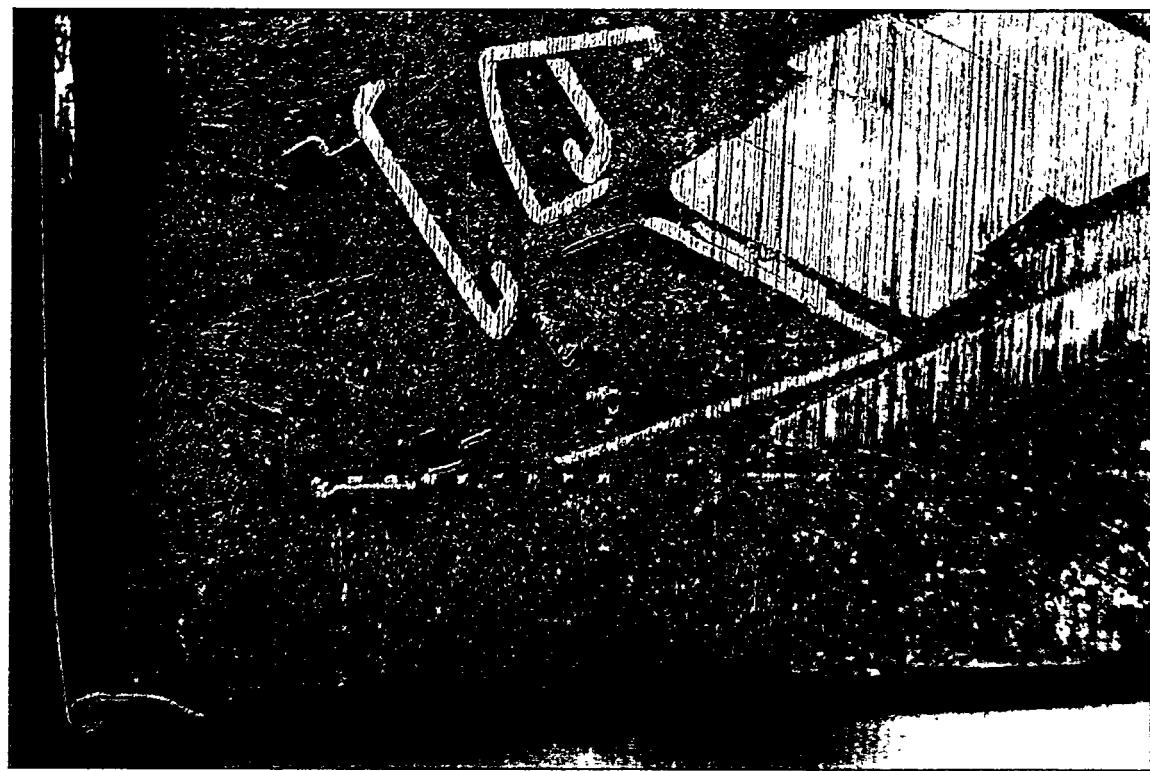


Fig. A.25. Transverse cut, revealing no apparent porosity on the side.



Fig. A.27. Bags of cadmium sheets (TS22) before size reduction.

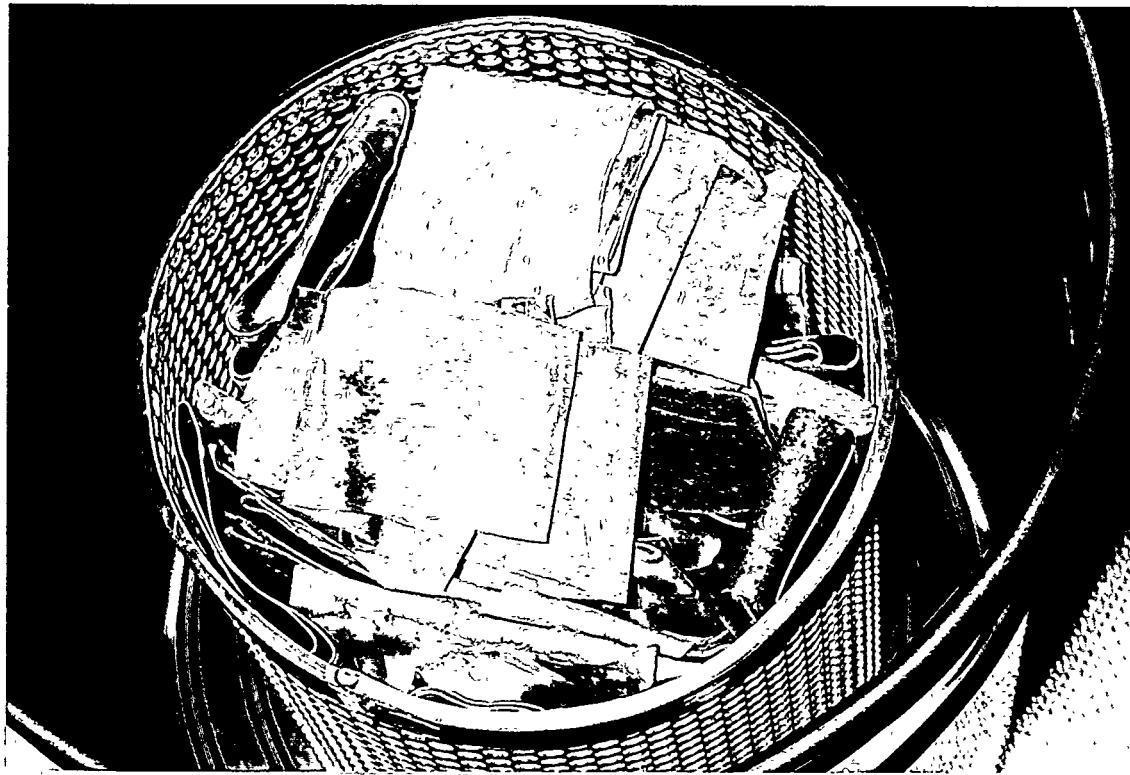


Fig. A.28. Using size reduction to achieve maximum waste loading.

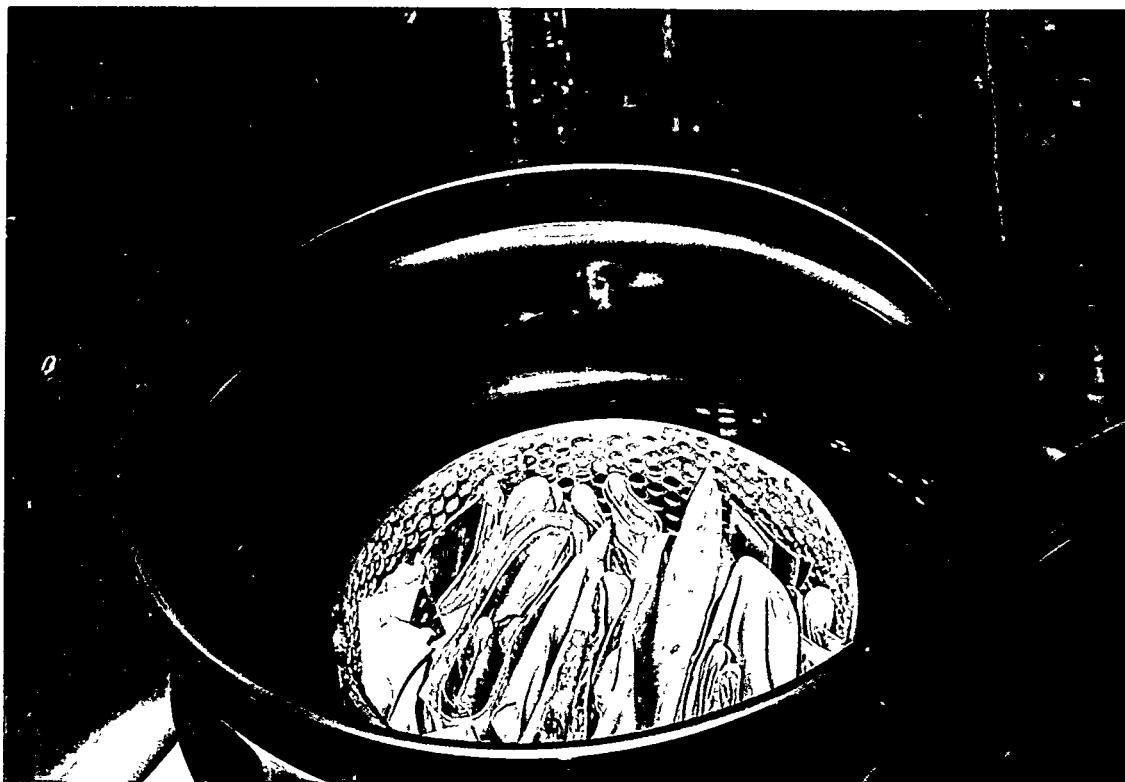


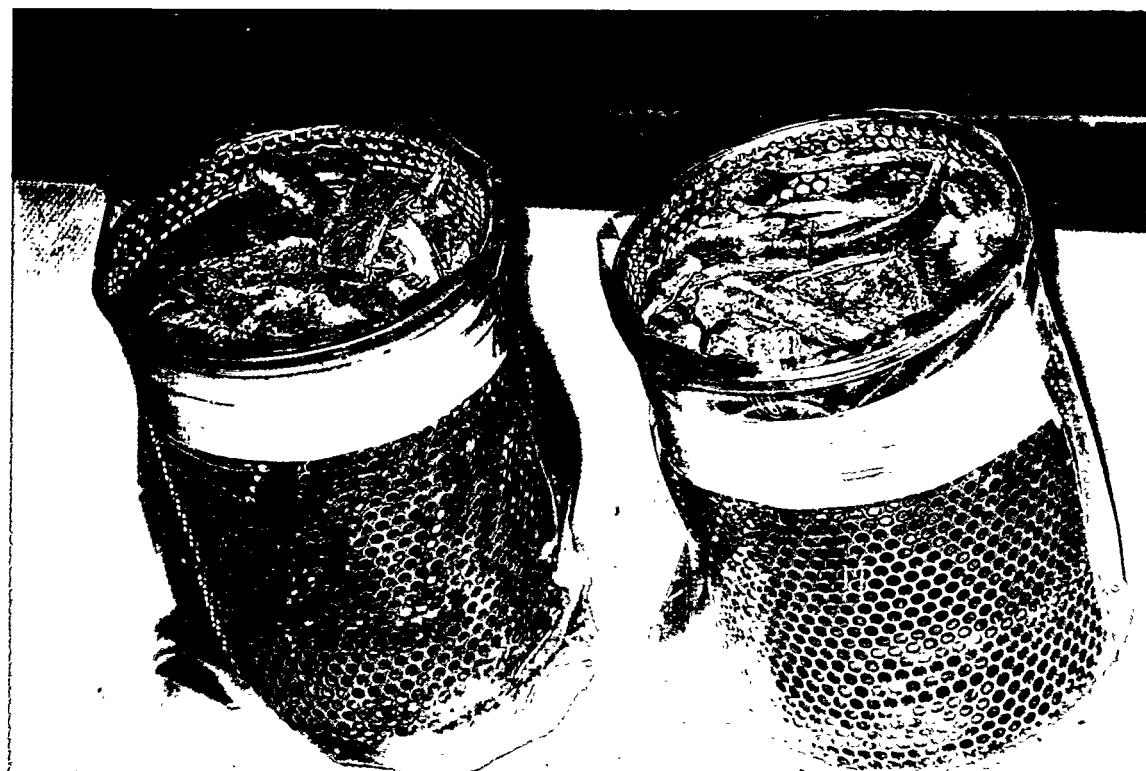
Fig. A.29. Waste pieces that were difficult to reduce to a minimal volume.



Fig. A.30. Detail of the lead joints.



**Fig. A.31. Pliability of the lead joints, which makes them easy to fold.**



**Fig. A.32. Baskets filled with lead joints (TS24)**

## **APPENDIX B**

### **WASTE MANAGEMENT DATABASE FOR OR-W063**



EMW#	Description	Child	X10 Keynum	Storage Area	Plus Both	Waste Description	Gen Facility	Gen Site	Gen Facility	Item EPA	Suspect Rad	Known Rad	Comments
M2721	Elemental Hazardous Metals	NA	X1019511019	7653	SOLID	EXCESS LEAD SEALS (NO LONGER USED)	ORNL	6000	0.218	D008	YES		Not a potential candidate; probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X1019521224	7653	SOLID	LEAD-GRANULAR	ORNL	4500S	0.562	D008	YES		Not a potential candidate; Not a real mixed waste nor a debris
M2721	Elemental Hazardous Metals	NA	X1019521225	7653	SOLID	LEAD GRANULAR	ORNL	4500S	0.444	D008	YES		Not a potential candidate; Not a real mixed waste nor a debris
M2721	Elemental Hazardous Metals	NA	X1019521226	7653	SOLID	LEAD GRANULAR	ORNL	4500S	0.580	D008	YES		Not a potential candidate; Not a real mixed waste nor a debris
M2721	Elemental Hazardous Metals	NA	X1019604000	7653	SOLID	LEAD	ORNL	4500S	0.581	D008	YES		Not a potential candidate; probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X1019313236	7653	SOLID	1 LEAD RING IN PLASTIC BAG	ORNL	2000	0.555	D008	YES		Not a potential candidate; probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X1019604214	7653	SOLID	CADMIUM SHEETS-USED	ORNL	4500N	0.037	D006	YES		Not a potential candidate; probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X1019520753	7653	SOLID	DISCARDED CHEMICAL SELENIUM METAL SOLID. *CNR	ORNL	4500S	0.671	D010	YES		Not a potential candidate; probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X1019514647	7653	SOLID	LEAD DUST	ORNL	7500	0.408	D008	YES		Not a potential candidate; Not a real mixed waste nor a debris (dust)
M2721	Elemental Hazardous Metals	NA	X1019320758	7653	SOLID	DISCARDED CHEMICAL LEAD *CNR	ORNL	4500S	1.179	D008	YES		Not a potential candidate; probably not a real mixed waste

M2721	Elemental Hazardous Metals	NA	X1019320759	7653	SOLID	DISCARDED CHEMICAL CADMIUM	ORNL	4500S	0.399	D006	YES			
M2721	Elemental Hazardous Metals	NA	X1019511480	7653	SOLID	EXCESS CADMIUM METAL IN	ORNL	3047	0.236	D006	YES			
M2721	Elemental Hazardous Metals	NA	X1019511481	7653	SOLID	EXCESS CADMIUM METAL IN	ORNL	3047	0.318	D006	YES			
M2721	Elemental Hazardous Metals	NA	X1019511482	7653	SOLID	EXCESS CADMIUM METAL IN	ORNL	3047	0.218	D006	YES			
M2721	Elemental Hazardous Metals	NA	X1019511491	7653	SOLID	EXCESS CHROMIUM METAL IN	ORNL	3047	0.826	D007	YES			
M2721	Elemental Hazardous Metals	NA	X1019511492	7653	SOLID	EXCESS CHROMIUM METAL IN	ORNL	3047	0.889	D007	YES			
M2721	Elemental Hazardous Metals	NA	X1019511503	7653	SOLID	EXCESS LEAD METAL IN	ORNL	3047	0.599	D008	YES			
M2721	Elemental Hazardous Metals	NA	X1019611217	7653	SOLID	EXCESS CHROMIUM METAL	ORNL	6010	0.018	D007	YES			
M2721	Elemental Hazardous Metals	NA	X1019524703	7653	SOLID	BERYLLIUM FOIL	ORNL	3025M	0.018	P015	YES			
M2721	Elemental Hazardous Metals	NA	X1010H017104-079	7653	SOLID	PURE CADMIUM	ORNL	4500S	0.036	D006	YES			

M2721	Elemental Hazardous Metals	NA	X109611110	1653	SOLID	CHROMIUM NICKEL ROD IN GLASS BOTTLE	ORNL	4500S	0.218	D007	YES		Not a potential candidate: probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X109425971	1653	SOLID	DUTCH BOY WHITE LEAD	ORNL	7055	0.444	D008	YES		Not a potential candidate: Not a real mixed waste nor a debris (paste like material)
M2721	Elemental Hazardous Metals	NA	X10H016255-042	1653	SOLID	LEAD	ORNL	2024	0.032	D008	YES		Not a potential candidate: probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X10H016255-061	1653	SOLID	LEAD	ORNL	2024	0.127	D008	YES		Not a potential candidate: Not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X10H038101-013	1653	SOLID	CADMIUM	ORNL	4500N	0.027	D006	YES		Not a potential candidate: probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X10H016259-001	1653	SOLID	WHITE LEAD	ORNL	3025H	0.454	D008	YES		Not a potential candidate: probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X10H002997-022	1653	SOLID	LEAD	ORNL	7602	0.794	D008	YES		Not a potential candidate: probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X10H013777-019	1653	SOLID	LEAD	ORNL	4500S	0.354	D008	YES		Not a potential candidate: probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X10H036035-011	1653	SOLID	CHROMIUM	ORNL	4515	0.127	D007	YES		Not a potential candidate: probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X109313773	1653	SOLID	USED SELENIUM BRIDGE RECTIFIER(METAL ENCASED SELENIUM) SELENIUM IS POISONOUS WHEN BURNED	ORNL	3001	3.529	D010	YES		Not a potential candidate: probably not a real mixed waste

M2721	Elemental	NA	X1019314774	7653	SOLID	RECTIFIER (SELENIUM METAL)	ORNL	4501	10.127	D010	YES	Not a potential candidate; probably not a real mixed waste	
M2721	Elemental	Hazardous Metals	NA	X1019403263	7653	SOLID	WASTE MERCURY- CONTAMINATED LEAD	ORNL	4500S	0.553	D008	YES	Not a potential candidate; probably not a real mixed waste
M2721	Elemental	Hazardous Metals	NA	X1019036035-009	7653	SOLID	LEAD	ORNL	4515	0.585	D008	YES	Not a potential candidate; probably not a real mixed waste
M2721	Elemental	Hazardous Metals	NA	X1019425964	7653	SOLID	DUTCH BOY WHITE LEAD	ORNL	7055	0.390	D008	YES	Not a potential candidate; probably not a real mixed waste nor a debris (paste like material)
M2721	Elemental	Hazardous Metals	NA	X1019524580	7653	SOLID	LEAD ROD IN METAL SHEATH	ORNL	3010	0.109	D008	YES	Not a real mixed waste nor a debris (paste like material)
M2721	Elemental	Hazardous Metals	NA	X1019426581	HWSF	SOLID	SELENIUM SWITCH FROM OLD CENTRIFUGE	ORNL	4505	0.086	D010	YES	Not a potential candidate; probably not a real mixed waste
M2721	Elemental	Hazardous Metals	NA	X1019503935	7653	LIQUID	LEAD METAL	ORNL	7055	1.352	D001	YES	NRRA item
M2721	Elemental	Hazardous Metals	NA	X1019503971	7653	SOLID	DUTCH BOY WHITE LEAD	ORNL	7055	0.426	D008	YES	Not a potential candidate; probably not a real mixed waste
M2721	Elemental	Hazardous Metals	NA	X1019503972	7653	SOLID	DUTCH BOY WHITE LEAD NATIONAL LEAD COMPANY	ORNL	7055	0.499	D008	YES	Not a real mixed waste nor a debris (paste like material)
M2721	Elemental	Hazardous Metals	NA	X1019505649	7653	SOLID	OUT OF DATE NICKEL METAL	ORNL	4500S	0.145	D001	YES	Not a potential candidate; probably not a real mixed waste
M2721	Elemental	Hazardous Metals	NA	X1019506498	7653	SOLID	ARSENIC LUMPS	ORNL	4500S	0.680	D004	YES	Not a potential candidate; (powder) probably not a real mixed waste

M2721	Elemental Hazardous Metals	NA	X1019521713	7653	SOLID	CHROMIUM, NICKEL POWDER	ORNL	4508	0.004	D001 D007	YES	Not a potential candidate; Not a real mixed waste nor a debris (powder)
M2721	Elemental Hazardous Metals	NA	X1019521714	7653	SOLID	CHROMIUM, NICKEL POWDER	ORNL	4508	0.004	D001 D007	YES	Not a potential candidate; Not a real mixed waste nor a debris (powder)
M2721	Elemental Hazardous Metals	NA	X1019521809	7653	SOLID	BARIUM AND STRONTIUM	ORNL	4500S	0.481	D001 D005	YES	Not a potential candidate; Not a real mixed waste nor a debris (powder)
M2721	Elemental Hazardous Metals	NA	X1019523349	7653	SOLID	EXCESS CHEMICAL IN ORIGINAL CONTAINER (CHROMIUM METAL)	ORNL	4501	0.544	D007	YES	Not a potential candidate; probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X1019523353	7653	SOLID	EXCESS LEAD IN PLASTIC CONTAINER	ORNL	4501	1.633	D008	YES	Not a potential candidate; probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X1019310982	7653	SOLID	LEAD USED FOR SEALING	ORNL	2000	0.014	D008	YES	Not a potential candidate; probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X1019611216	7653	SOLID	EXCESS LEAD METAL PIECES IN PLASTIC BAG	ORNL	6010	0.481	D008	YES	Not a potential candidate; probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X101942412	7652	SOLID	LEAD SLAG FROM LEAD MELTING POTS AND LEAD CONTAMINATED GLOVES AND WIPES.	ORNL	7005	303.90	D008	YES	Not a potential candidate; probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X101942413	7652	SOLID	LEAD SLAG FROM LEAD MELTING POTS AND LEAD CONTAMINATED GLOVES AND WIPES.	ORNL	7005	315.24	D008	YES	Not a potential candidate; probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X1019408688	7652	SOLID	BURNT OUT CIRCUIT BOARD WITH LEAD AND SILVER CONTAMINATED SOLDER *CNR	ORNL	4500S	0.163	D008 D011	YES	Not a potential candidate; probably not a real mixed waste
M2721	Elemental Hazardous Metals	NA	X10194202891	7652	SOLID	LEAD SLAG FROM LEAD MELTING POTS AND LEAD CONTAMINATED GLOVES AND WIPES.	ORNL	7005	161.48	D008	YES	Not a potential candidate; probably not a real mixed waste

M2721	Elemental Hazardous Metals	X10C9402917	X10J9423293	7632	SOLID LEAD SLAG FROM LEAD MELTING POTS	ORNL	7005	200.94	D008	YES		
						ORNL	7500	0.794	D008	YES		
M2721	Elemental Hazardous Metals	X10C9402917	X10J9311274	7632	SOLID LEAD STAND							
						ORNL	7500	1.479	D008	YES		
M2721	Elemental Hazardous Metals	X10C9402917	X10J930930	7632	SOLID LEAD PLATES	ORNL	7500	204.17	D008	YES		
						ORNL	3019H	204.17	D009	Y		
M2721	Elemental Hazardous Metals	X10CH00710R	X10JH006100-001	7507W	SOLID LEAD							
						ORNL	2026	4.537	D005	YES		
M2721	Elemental Hazardous Metals	X10CH01024R	X10JH005691-001	7507W	SOLID BARIUM CHROMATE RESIDUE CONTAMINATED SOLID							
						ORNL	3350	1	D007	YES		
M2721	Elemental Hazardous Metals	X10CH01024R	X10JH013252-043	7507W	SOLID CHROMIUM CARBIDE							
						ORNL	3042	208.45	D006	YES		
M2721	Elemental Hazardous Metals	X10CH01024R	X10JH008284-001	7507W	SOLID CADMIUM							
						ORNL	4500N	0.064	D001	YES		
M2721	Elemental Hazardous Metals	X10CH01898R	X10JH043557-003	7823	SOLID SILVER METAL							
						ORNL	4500N	0.064	D011	Y		