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**Volume Reduction System for
Solid and Liquid TRU Waste from
the Nuclear Fuel Cycle:
January-March 1978**

**Barbara M. Alexander, Dana A.
Batchelder, Jay W. Doty and Edward L. Lewis**

May 5, 1978

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Foreword

Under the sponsorship of the DOE Division of Waste Management, Production and Reprocessing, Mound is responsible for the development of design criteria for a volume reduction system to treat combustible solid and liquid waste generated in the nuclear industry.

This report is submitted by W. T. Cave, Director, Nuclear Operations, and B. R. Kokenge, Manager, Nuclear Technology, from contributions prepared by members of the Nuclear Waste Technology Section: K. V. Gilbert, Manager, The Volume Reduction Technology group: W. H. Bond, Leader, and the Waste Immobilization Group: J. W. Doty, Leader.

Previous reports of this and the related project, TRU Waste Cyclone Drum Incinerator and Treatment System, are listed below:

Volume Reduction System for Solid and Liquid TRU Waste From the Nuclear Fuel Cycle:

October-December 1977	MLM-2509
July-September 1977	MLM-2498
April-June 1977	MLM-2463
January-March 1977	MLM-2436

TRU Waste Cyclone Drum Incinerator and Treatment System:

October-December 1977	MLM-2510
July-September 1977	MLM-2496
April-June 1977	MLM-2482
January-March 1977	MLM-MU-77-64-0004

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Abstract

The laboratory cyclone incinerator, which started operation in January, 1978, has been developed to study the effects of burning feed containing radionuclides other than plutonium-238. These isotopes, which will be primarily products of fission, must be efficiently removed from the off-gas, if combustible waste containing such species is to be incinerated.

In the first two months of operation, a number of burns were made and efforts were made to identify and remedy problem areas. Preliminary data from early runs show that the combustion efficiency of the incinerator can still be improved upon and indicate the changes necessary to improve combustion performance.

Controlled experiments were conducted on the pressed ash-cement pellet matrix to define compressive strength, mechanical stability, density, and effect of curing environment (wet cure and dry cure). Leachability studies were initiated on pressed sludge/cement matrix in distilled water at ambient temperature. Compressive strengths of sludge/cement pressed matrix samples were investigated. Physical and chemical attributes of incinerated ash were evaluated in relationship to the ash/cement matrix.

Effects of burning beta and gamma contaminated waste

The identification and removal of radioactive gases and particles from the incinerator off-gas, different from those encountered in burning plutonium-contaminated wastes, have been major concerns of the commercial incinerator project since its inception. This problem is being studied through a laboratory-scale, experimental incinerator to develop new technology which will expand the horizons for application of cyclone incinerator technology in the commercial nuclear industry. This incinerator system is approximately 1/20 the size of the Cyclone Incinerator.

The objectives of the selection of an ash-immobilization technique and product characterization activities are to demon-

strate a chosen process, characterize the product of the process, and compare characterization criteria of the product to the criteria developed in Task II of the TRU Waste Cyclone Drum Incinerator and Treatment System Project.

Bench-scale incinerator

B. M. Alexander

The laboratory cyclone incinerator project was developed to study the effects of burning wastes contaminated with isotopes other than plutonium-238 in a cyclone system. The original design (Figures 1 and 2) of the laboratory incinerator included an off-gas cleanup system for the removal of Cl^- and particulate matter. This consists of a deluge tank (Figure 3) using a caustic scrubbing solution and several stages of filtration (Figures 1 and 4). The initial stages of the experimental program are

FIGURE 1 - Laboratory-scale incinerator.

FIGURE 2 - Overall view of laboratory-scale cyclone incinerator.

FIGURE 3 - Deluge (spray) tank for laboratory-scale cyclone incinerator.

directed toward optimizing the combustion efficiency and particle removal capability of this system. After this is accomplished, the feed will be doped with various isotopes of interest, such as fission products which are found in wastes from reactors and reprocessing facilities. The removal efficiency of the system for each isotope will be determined, and necessary modifications will be made to improve the decontamination factor across the system for each isotope.

Construction of the laboratory cyclone incinerator was completed in January, 1978. Early problems were experienced with the deluge tank in that the caustic scrubber solution drained from the tank so slowly that very low gas and liquid flow rates [on the order of $20 \text{ ft}^3/\text{min}$

FIGURE 4 - First stage filtration unit.

(at a velocity of 1630 SFPM^*) for the off-gas and 1 gal/min for the scrubber solution] were necessary to avoid flooding the deluge tank.

The first burn on the incinerator was made under these conditions, and the results were unsatisfactory. The amount of turbulence in the combustion chamber was low, and the feed did not burn completely. Because of the low caustic flow rate, the off-gases were cooled only to approximately 61°C in the deluge tank, and particle removal was poor. Part of the flooding problem was resolved with a change in the venting of the caustic storage tank.

*SFPM-feed/minute at standard temperature and pressure.

A second burn was made with a higher off-gas flow rate. This time a hotter, more complete combustion was achieved, but, about 10 min into the burn, the deluge tank flooded and backed up into the off-gas lines. It was obvious that the 3/8 in. stainless steel tubing which drained from the deluge tank to the storage tank did not have sufficient capacity. This was then replaced with 5/8 in. tubing, at which time the rest of the caustic line was also replaced with 1/2 in. tubing to reduce line resistance. After this replacement was completed, the caustic flow rate increased to 2.3 gal/min, and no further flooding problems have been experienced.

The feed material used in the laboratory cyclone usually consists of waste similar to that produced in the Waste Processing Building (WD) (shoe covers, paper towels, work gloves, plastic bags, cardboard, etc.) or a shredded material consisting mainly of paper and polyethylene, mixed with a small amount of styrofoam, or a combination of both.

The earliest laboratory incinerator runs, which were made with shredded feed, used one section of 1/2 in. stainless steel tubing for an air inlet. Under these conditions, efficient combustion could not be achieved because of the low air flow rate into the chamber. The incomplete combustion of plastic produced a great deal of tar in the off-gas, which then clogged the fine borosilicate glass filters in the off-gas line.

To increase airflow to the combustion chamber, a second cyclone air inlet was added opposite the first (Figure 5). Greatly improved combustion was noted, but the pressure drop across the combustion

FIGURE 5 - Upper part of combustion chamber valved lines are the two air inlets.

chamber was still quite high, as the configuration of the burn chamber had made it necessary to put an 80° bend in each air inlet. The simulated WD waste burns much more completely than the shredded material. (See Table 1, Weight Reduction Results.) It is believed that the plastic in the shredded material will burn much more completely if still more air flow and a higher combustion temperature can be achieved.

Sludge removed from the filter in the caustic line after runs four through six was analyzed, along with a sample of the caustic liquid, to determine their major constituents. The results are tabulated in Table 2. The high fluoride level in the samples is caused by burning Teflon in the incinerator. The Cl^- is produced by burning polyvinyl chloride and other chlorinated materials. Trace metals

Table 1 - WEIGHT REDUCTION RESULTS

Run No.	Date	Feed Material	Off-Gas Flow Rate (SFPM) ^a	Weight Reduction
4	1/25/78	shredded	2100	8.6:1
5	2/2/78	WD	2100	19.0:1
6	2/6/78	WD	2200	19.0:1
7	2/7/78	WD/shredded	2200	11.4:1
8	2/8/78	WD(mostly paper take)	2200	8.7:1
9-10	2/23/78	shredded	1950	11.4:1

^aSFPM - ft/min - standard temperature and pressure

Table 2 - CHEMICAL COMPOSITION OF FILTER SLUDGE

Caustic Solution ^a	Dry Sludge
Major Components:	Cl ⁻ - 225 ppm
Water	F ⁻ - 155 ppm
NaOH	
Trace Components:	C - 49.2%
TOC ^b - 7.2x10 ⁴ mg/liter	Al - >1% (~5%)
Cl ⁻ - 2512 mg/liter	Si - >1% (~3%)
F ⁻ - 1700 mg/liter	Ca - >1% (~3%)
	Ti - >1% (~2%)
	Fe - >1% (~1.5%)
Si - 24 mg/liter	Mg - 0.9%
Ti - 12 mg/liter	Zn - 0.8%
Ca - 12 mg/liter	Na - 0.75%
Al - 5 mg/liter	Pb - 0.3%
Va - 4 mg/liter	Cr - 0.15%
B - 3 mg/liter	Mn - 0.1%
Fe - 3 mg/liter	Ni - 0.08%
Pb - 2 mg/liter	Sb - 0.05%
Cr - 1 mg/liter	Ag - <0.05%
Cu - 1 mg/liter	Sn - <0.05%
Mg - <1 mg/liter	Cu - <0.05%
	B - <0.05%
	Co - <0.05%

^apH - 11.83

^bTOC - total organic carbon

which show up in the analysis may originate as constituents of the incinerator feed (e.g., titanium is used as a filler in paper), or they may be picked up from the Haynes 25 alloy used in the combustion chamber.

The carbon content of both samples is at least double the minimum attainable because of inefficient combustion and high particle carryover from the combustion chamber. Many small particles are carried over from the combustion chamber before they can be burned.

With the addition of the second cyclone air inlet, the maximum temperature in the combustion chamber, as recorded by a platinum-rhodium thermocouple, was approximately 1300°F. It is hoped that, by reducing the pressure drop through the incinerator system, an even higher air flow rate can be achieved, which may produce combustion temperatures around 2000°F. Equipment modifications will be necessary to reduce particle carryover from the combustion chamber.

Waste immobilization

D. A. Batchelder, E. L. Lewis and J. W. Doty

Introduction

The objectives of this task are to evaluate various TRU waste immobilization methods and to evaluate performance of the immobilized waste forms against existing and newly developing criteria for accepting immobilized waste product forms for terminal isolation. In addition, this program has as its objective the development of processes to effectively immobilize various waste residues such as sludges, absorbents, and incinerator ash.

Pressed ash-cement

pellet experiments

Controlled experiments were conducted this quarter on the selected pressed-pellet matrix chosen for the immobilization process of incinerator ash residue. The experiments were designed to provide data for evaluation and selection of the best pellet matrix for incinerator ash and Portland Type 1A cement.

Certain process parameters previously determined and evaluated during screening studies previously reported with the pressed ash-cement matrix were fixed throughout the entire experimental sequence. The parameters in the process which were held constant are:

1. Pressing pressure of 25,000 psi for 1-min duration.
2. Three-gram matrix weight.
3. One-half inch pressing die, which, with a 3-g charge, produces a relatively standard 1/2-in. diameter by 1/2-in. high pellet (L/D ratio approximately 1).
4. Dry Cure Cycle - two days (48 hr) in open atmosphere.
5. Wet Cure Cycle - four-day (96 hr) cure in distilled water followed by a two-day cure in open atmosphere.

Four groups of waste matrices samples were evaluated during this reporting period. Pellets were pressed with 60%, 70%, 80%, and 0% ash residue (100% Portland 1A cement). The pure cement pellets were pressed for control comparison conditions. The water-to-cement ratio was varied in each sample group to determine

the water-to-cement ratio which would produce acceptable pressed pellets. Normally six water-to-cement ratios were evaluated ranging from a too dry condition (fracturing pellet matrix after pressing) to a wet condition (excess water ejected from die during pressing operation).

The control condition matrix (100% Portland 1A cement) containing 0% incinerator ash at acceptable water-to-cement ratios produced nearly identical pressed pellets. The dry-cure method produced pressed pellets of uniform compressive

strength, density, and final weight. Based on the final weight of the pellets the calculated percentage of water remaining in the matrix is approximately 2% for the dry-cure method. Similarly the wet-cure method produced pressed pellets also of uniform but higher compressive strength, density, and final pellet weight. However, the wet-cure method retained approximately 10% water in the matrix form.

The data shown in Table 3 for the 60%, 70% and 80% ash-cement pressed matrices are given only for the two best water-

Table 3 - PRESSED ASH-CEMENT PELLET MATRIX
CONTROLLED EXPERIMENTAL PROCESS

Ash Type	Ash Weight (g)	Cement Weight (g)	Water/Cement	Cure Dry	Wet	Initial Pellet Weight (g)	Final Pellet Weight (g)	Calculated Weight Loss (g)	Density (g/cm ³)	Average Compressive Strength (psi)	Weight % Loss
	0	3.0	0.10	x		3.23	3.04	0.19	2.29	3635	
	0	3.0	0.125	x		3.25	3.05	0.20	2.30	3661	
	0	3.0	0.15	x		3.23	3.02	0.21	2.32	3668	0.50
	0	3.0	0.175	x		3.26	3.04	0.22	2.33	3670	
	0	3.0	0.10		x	3.32	3.25	0.07	2.43	4580	
	0	3.0	0.125		x	3.36	3.29	0.07	2.45	4330	
	0	3.0	0.15		x	3.34	3.29	0.05	2.47	4192	0.84
	0	3.0	0.175		x	3.32	3.28	0.04	2.49	4293	
1-3	1.8	1.2	0.45	x		3.22	3.02	0.20	2.00	3470	4.45
1-3	1.8	1.2	0.45		x	3.38	3.52	(0.14)	2.30	3600	3.21
1-3	1.8	1.2	0.50	x		3.34	3.04	0.30	2.00	3470	0.63
1-3	1.8	1.2	0.50		x	3.40	3.52	(0.12)	2.29	3620	4.33
1-3	2.1	0.9	0.70	x		3.43	3.05	0.38	1.96	2480	1.78
1-3	2.1	0.9	0.70		x	3.41	3.24	0.17	2.01	3640	3.70
1-3	2.1	0.9	0.75	x		3.40	3.02	0.38	1.96	3470	2.27
1-3	2.1	0.9	0.75		x	3.42	3.31	0.11	2.11	3625	3.63
1-3	2.4	0.6	1.10	x		3.23	3.00	0.23	1.92	2480	3.72
1-3	2.4	0.6	1.10		x	3.38	3.27	0.11	2.08	3600	3.84
1-3	2.4	0.6	1.15	x		3.26	3.00	0.26	1.92	2480	1.85
1-3	2.4	0.6	1.15		x	3.40	3.30	0.10	2.08	3570	4.45
1-1	2.4	0.6	0.70	x		3.30	3.00	0.30	2.03	~2507	
1-1	2.4	0.6	0.70		x	3.30	3.21	0.09	2.15	~3640	
1-1	2.4	0.6	0.85	x		3.30	3.00	0.30	2.04	~2500	
1-1	2.4	0.6	0.85		x	3.18	3.00	0.18	2.10	~3670	

to-cement ratios even though four acceptable and tested water-to-cement ratios were attained. The data clearly indicate that the wet-cure method produces ash-cement matrix having higher compressive strength than pellets which were dry cured. In general, the compressive strength increased as the percent of ash in the matrix decreased. This result was expected since the cement (binder) contributes significantly to the pellet compressive strength.

As the percent of incinerator ash was increased, the water-to-cement ratio also increased. This was also expected as observed in past screening studies conducted on the ash-cement pressed matrices. Although a small portion of water must be required for cement interaction and blending, the ash requires significant amounts of water to wet the ash because of its hydrophilic characteristic. Although this condition exists, the final pellet weights after curing are close in final weight value as the 0% ash matrix final weight. The 60/40 ash-cement matrix after wet-cure processing, however, gained weight; the reason for this is presently being investigated.

The density of the various ash-cement pressed pellets increased as the percent of ash decreased. The density ranged from 83-88% of the 0% ash-cement pressed matrix; however, the density values calculated for the ash-cement various matrices were taken, and the radiographs clearly show that no void, crack, and/or fissure exist in any of the ash-cement pressed pellet matrices.

Mechanical strength and corresponding percent weight loss of the various pressed pellets were also determined.

The pressed pellets were placed in stainless steel containers which were clamped into a Model BT Wrist Action Shaker. The shaker was set at the maximum 10° shake angle to vibrate the samples in a circular motion inside the fixed container. The shaking action was continued for 50 hr, and the weight loss was determined. The resulting weight losses are also shown in Table 3. All the samples experienced a slight rounding of the edge that contacted the stainless steel container; one slight abrasion occurred on the walls of the samples. Figures 6 and 7 are photographs of the 100% cement pressed pellet before and after the shake test. Radiographs of the pellets taken before and after the shake test showed that the pellets were still homogeneous and contained no voids, cracks, and/or fissures. Although the percent weight loss is higher for the 60%, 70%, and 80% ash-cement pressed matrices as compared to the 100% cement pressed matrices, this result would be expected based on the lower compressive strength values.

Volume reduction efficiency

Prior to the initiation of the controlled experimentation on the ash-cement matrix process, a new batch of incinerator ash was prepared with the cyclone incinerator. The composition of the incinerator feed was Type I, that consisted of 32 wt % paper, 9 wt % PVC, 29 wt % polyethylene, 8 wt % polypropylene, 13 wt % rubber, 3 wt % cloth, and 6 wt % metal. A total of 11.2 kg (24.6 lb) of the uncompacted combustible composition (2.2 ft^3) was burned in a 55-gal drum. The resulting ash, containing approximately 20% carbon, was then sintered for 1 hr at 800°C to reduce the carbon content to approximately 0.04% carbon. After grinding and sizing, the sintered ash weighed 340 g and contained a volume of 570 cm^3 .

FIGURE 6 - Pellet before shake test.

Based on the pelletization process for a pressed ash-cement matrix ratio of 70/30, approximately 162 pressed pellets could be produced. These pellets would weigh approximately 494 g if dry cured and approximately 534 g if wet cured. The resulting volume of the immobilized matrix would be approximately 252 cm^3 for the dry-cure product and approximately 256 cm^3 for the wet-cure method. This analysis is based on the preliminary data obtained during the experimentation controlled processes (Table 3).

The data indicate the overall process efficiency starting with uncompact combustible Type I composition through incineration and immobilization is approximately 95% in weight reduction and approximately 99% in volume reduction.

FIGURE 7 - Pellet after shake test.

Pressed sludge-cement pellets

Screening studies continued on development of the pressed sludge-cement pellet matrix. Two groups of 1/2-in. diameter pellets were pressed at 25,000 psi. The amount of sludge in the pressed cement matrix was selected at 15 and 20%. The prepared matrices were subjected to both dry-cure and wet-cure methods of curing. As shown in Table 4, again the wet-cured pellets exhibited higher compressive strength than the dry-cured pellets for both sludge percentages prepared.

Leach experiments were also initiated on the 1/2-in. diameter sludge-cement matrix in distilled water at ambient temperature. The leach rate (grams as solid residue leached per cm^2 per hour) is reported in Table 4 for the wet cured pellets.

Elemental analysis of

Type I incinerator ash

Three batches of Type I incinerator ash were sampled and analyzed by emission spectrography after sintering at 800°C. Elements that are present in the three batches of Type I ash are shown in Table 5. Silicon and aluminum are the major constituents and were determined by wet chemistry techniques. As noted, the percent of each element varied considerably in the three batches of ash even though the starting combustible feed composition was identical. As shown in Table 3, the 80/20 ash-cement pressed pellet matrix for Ash Type 1-1 and Ash Type 1-3 varied in water-to-cement ratio in the pressing operation. Ash Type 1-3 required a higher water-to-cement ratio to produce acceptable pellets than the Ash Type 1-1 material. Ash Type 1-3 contains a larger percentage of CaO (quicklime), a compound employed in mortars and cements, which would require water for shaking, leaving a white powder, $\text{CaO} \cdot \text{H}_2\text{O}$. Additionally, the Ash Type 1-3 was fresh-

ly prepared and sintered, and since the ash is hydrophobic, it had not had sufficient time to absorb moisture. Ash Type 1-1 has a lower percent of CaO and is aged ash (having absorbed moisture). Thus, it is believed that these factors contributed to lower water-to-cement ratios for pressing the Ash Type 1-1.

Particle distribution of sintered incinerator ash

The three batches of Type I incinerator ash were sieved using standard stainless steel sieves after sintering at 800°C. The three batches of ash as shown in Table 6 are very similar in particle size distribution; however, the average particle size of Type 1-1 ash is slightly smaller. Although the Type I combustible feed composition is identical, after incineration, the resulting ash residue will vary in particle distribution and elemental composition which will affect the water-to-cement ratios required for acceptable pelletization.

Table 4 - PRESSED SLUDGE-CEMENT PELLETS SCREENING STUDY DATA

Sludge (%)	Cement (%)	Water/Cement Ratio	Cure Method	Average Crush Strength (psi)	Leach Rate (g/cm ² /hr)
20	80	0.20	Dry	3730	
20	80	0.20	Wet	4600	3.55x10 ⁻⁶
15	85	0.14	Dry	3690	
15	85	0.14	Wet	4510	5.05x10 ⁻⁶

Table 5 - ELEMENTAL CONTENT OF SINTERED INCINERATOR ASH

Ash	Element (wt %) ^a																
	B	Si	P	Al	Fe	Mn	Pb	Mg	Cr	Sn	Ni	Ca	Cu	Ag	Zn	Na	Ti
1-1	<0.05	17.8	0.05	17.8	2.5	0.06	0.04	3.0	0.30	<0.05	0.08	2.0	0.05	<0.05	3.0	0.35	3.0
1-2	<0.05	>5	<0.05	>5	3.0	0.05	-	1.5	0.10	-	0.08	2.5	0.10	-	1.0	0.25	1.5
1-3	<0.05	5.0	0.06	3.0	>5	0.10	-	1.0	0.30	<0.05	<0.05	>5	<0.05	-	0.15	0.30	>5

^aElemental analysis will not approach 100% because of volatile products at combustion, the method of determination, and oxide values.

Table 6 - PARTICLE SIZE DISTRIBUTION OF SINTERED INCINERATOR ASH

Ash Type	Ash (wt %) in Size Range			
	>212μm	>106μm	>53μm	<53μm
1-1	20.6	47.0	79.3	20.7
1-2	28.6	57.2	78.6	21.4
1-3	25.3	54.6	79.1	20.9

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