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STATUS REPORT
WASTE INCINERATION AND IMMOBILIZATION FOR NUCLEAR FACILITIES
(OCTOBER 1977 - MARCH 1978)

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Chemistry Research and Development
PILOT PLANT DEVELOPMENT

MASTER

Rockwell International

Energy Systems Group
Rocky Flats Plant
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U. S. DEPARTMENT OF ENERGY
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SUBJECT DESCRIPTORS

Fluidized Bed
Incinerators
Waste Management
Residue Fixation
Vitrification

ROCKWELL INTERNATIONAL
ENERGY SYSTEMS GROUP
ROCKY FLATS PLANT
P.O. BOX 464
GOLDEN, COLORADO 80401

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CONTENTS

Abstract	1
Introduction	1
Summary	1
Discussion	2
Fluidized Bed Incineration	2
Incinerator Modifications	2
Solid Waste Handling	2
Solid Waste Incineration	3
Offgas Sampling System	4
Residue Immobilization	4
Process Equipment	4
Furnace And Reactor	4
Product Characteristics	5
Offgas Cleaning	7
Product Physical Integrity	7
Joule Reactor Development	10
Conclusions	10

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Abstract. Fluidized bed incineration and processes for immobilization of wastes generated at nuclear facilities are undergoing development. After minor piping modifications to eliminate dust collecting points, a pilot plant fluidized bed incinerator run of 225 continuous hours was successfully completed in a demonstration of component reliability. Vitrification of incinerator ash and other wastes is now being accomplished using a pilot scale unit developed as a continuous flow process.

INTRODUCTION

This project is a part of the ongoing programs for the safe handling and disposal of wastes generated at nuclear facilities. Volume and weight reduction of combustible materials are accomplished utilizing a 9 kg/hr pilot plant fluidized bed incinerator designed and constructed at Rocky Flats. Solid combustible wastes, generated in production operations, are burned in a primary fluidized bed of sodium carbonate granules which provides *in situ* neutralization of hydrochloric acid from thermal decomposition of the large amount of polyvinylchloride (PVC) plastics contained in the waste. Liquid wastes, including chlorinated hydrocarbons, are burned in a similar manner. The low temperature process (below 600 °C in both the primary bed and fluidized catalyst afterburner) eliminates the need for refractory lining in the system. The *in situ* neutralization of acid gases and sintered metal filters in the offgas line negates the necessity of an aqueous scrubber. Low operating temperatures should result in a low fired plutonium oxide in the residue, a form much more easily solubilized in the plant's aqueous recovery process than the refractory oxide produced in conventional open flame incinerators.

An immobilization method is also under development for converting the incinerator residue into a product that meets the acceptance criteria at a permanent repository. Residues from incineration are fine powders that would be unacceptable under the ban on respirable or dispersible powders. After laboratory-scale tests on several immobilization techniques, vitrification was selected as the method best suited for waste handling because of process simplicity, safety, and product desirability. A major objective is that the immobilization method be relatively inexpensive for processing the large volume of waste in the non-high level category. Another objective is that the amount of additives required to produce an acceptable glass product be kept low as possible.

SUMMARY

Minor modifications to the pilot plant fluidized bed incinerator involved piping changes to eliminate right angles and tees that were found to be dust collection points. Modification of the incineration process was investigated; the second-stage, fine-bladed shredder was removed from the waste preparation system for a 100-hour solid waste run. The resultant once-shredded waste was difficult to feed at a uniform rate, and it was concluded the extra mechanical step in the two-shredder system was justified. Using the regular two-shredder system, a successful 225-hour continuous run was made to test the reliability of system components. Vitrification of incinerator ash and other low-level waste has been extensively tested using bench-scale equipment. A pilot plant is now operational, producing glass buttons in a continuous flow operation. The combined wastes are mixed with boric acid and sodium carbonate, screw fed into a metal reactor vessel, heated to 1050 °C, and the

molten glass allowed to drip at a controlled rate onto a rotating forming plate. Some problems have been encountered in the offgas system and various solutions are being implemented. The annealing and strain points of the product glass have been determined and an annealing system is in operation.

DISCUSSION

Fluidized Bed Incineration

Incinerator Modifications

The pilot plant fluidized bed incinerator (FBI) was described fully in RFP-2655,¹ and only minor modifications to the interconnecting piping have been made to improve performance. These changes are shown as shaded areas on Figure 1. Past run inspection of the pilot plant fluidized bed incinerator have shown that small build-ups of fine particulates were restricting flow in some sections of the offgas stream. This condition resulted in increased pressure drops and necessitated periodic cleanout. The problem was caused by several sharp bends in the offgas lines forming pockets for particle entrapment. These sections of the offgas piping were modified to eliminate the right angle tees and elbows; the major vessels were not altered. Flow calculations were made to determine the optimum pipe sizing and radii of bends, and to achieve equal distribution of gas flow to both sides of the afterburner gas distributor. The calculations showed a 5.0 cm (2 in) diameter offgas line with all radius bends at 33 cm (13 in) would be the most efficient.

Previously, unheated nitrogen purges were used in the afterburner gas distributor to maintain a uniform gas velocity. The temperature drop caused by the purge would allow heavy hydrocarbons to condense in the distributor and cause minor plugging. The purge lines have subsequently been heated, providing a flow of 30 SCFH at 120 °C, and no further plugging problems have been encountered.

An air-operated, silent vibrator was installed on the primary cyclone discharge to test its ability to eliminate ash bridging. Design calculations were used to locate the correct position for vibrator installation and to determine the appropriate operating pressure. This silent vibrator has proven to be as effective as the standard vibrator currently in use, while being less audibly irritating.

After the completion of the offgas piping modifications, the vessels and connecting pipework were insulated with a dense ceramic fiber insulation. The previous insulation was a mineral wool blanket with wire mesh covering. The change was effected to eliminate a possible health and safety hazard and to decrease thermal radiation losses.

Solid Waste Handling

A 100-hour solid waste run was completed with the fine-toothed primary shredder removed from the feed preparation stream. Under normal operating conditions, all solid waste entering the incinerator system is hand-sorted to remove large pieces of metal before feeding to the coarse-tooth shredder. This shredder reduces the feed stream to chunks and strips of approximately 14-mm width. The shredded waste then is air-classified for tramp metal removal and pneumatically conveyed to the fine-toothed shredder for final grinding. The feed stream leaving the fine-toothed shredder will be pieces approximately 7-mm in width. This size of materials has been found to be very conducive to good screw operation. It was hoped that the removal of the fine-toothed shredder would eliminate one item of equipment, while not adversely affecting the feed screw operation. Cloth and plastic sheets were allowed to pass into the feed screw hopper as long strips rather than small bits. This created a problem for the feed screw hopper bridge-breaking apparatus. A constant feed rate was difficult to achieve, as strips of material would entangle around the bridge-breaker shaft, blocking the screw feeder flights. It was determined from this run that the operating advantages associated with the fine-toothed shredder far outweigh the periodic maintenance disadvantages.

¹ D. L. Ziegler, J. W. White, A. J. Johnson, L. Q. Fong, A. R. Teter, and S. F. Chung. *Waste Incineration and Fixation for WMFR*. U. S. DOE RFP-2655. Rocky Flats Plant, Rockwell International, Golden, Colorado. November 1977.

FIGURE 1. Pilot Plant Fluidized Bed Incinerator

Solid Waste Incineration

Upon completion of the various equipment modifications, an extensive run was accomplished to test the long term capabilities of the fluidized bed incinerator. The run covered 225-hours of continuous operation, and has been the longest continuous effort for the pilot plant unit. The waste stream consisted of polyethylene sheet, PVC plastics, paper, cloth, and various materials generated in the plant production operations. The feed preparation system, which includes the shredders, and air-classifier (metal removal), a feed hopper bridge-breaking apparatus, and the feed screw,

functioned exceptionally well. No down time was credited to this portion of the total system, even though it could be the greatest potential trouble spot. The primary reactor vessel was filled with sodium carbonate granules to a depth of 50 cm. These granules are approximately 500 to 1000 micrometers in diameter and offer a very good gas-solid contact area. This sodium carbonate bed material provides *in situ* neutralization of the acid gases normally associated with incinerator operation.

During the 10-day period, more than one metric ton of plant production waste was processed by the

incinerator. Of that total, 34 kg of metal was either hand sorted or air-classified out of the incinerator system. The majority of this metal came from snaps and buttons attached to the coveralls. These pieces of metal are light and small enough to be pneumatically conveyed into the feed screw hopper with the scraps of cloth. They present no problem for the system, as they can be drained from the primary vessel without interfering with normal operation.

Offgas Sampling System

The pilot plant unit currently has the capacity to continuously monitor the offgas stream for unburned total hydrocarbons (THC), carbon monoxide (CO), carbon dioxide (CO₂), oxygen (O₂) and hydrogen chloride (HCl). The sampling system has undergone several changes to improve accuracy and reliability. Previously, the sample was removed from the offgas stream by a single diaphragm pump, transported through a stainless steel tube to the analyzer bank, then pumped through the analyzers. Plugging of the sample piping and moisture collection in the analyzers would periodically occur. The sample lines have now been heated from the sample take-out point to a water-chilled moisture trap. This change has served to remove the moisture and particulates while allowing the CO, CO₂, and HC gases to remain intact. The sample line was also split so that diaphragm pumps could pull the sample through each analyzer separately. The modifications have led to increased reliability for each of the instruments and made it considerably easier to control the varying flows that each analyzer requires.

An oxygen analyzer was recently installed to measure the amount of unreacted oxygen remaining in the incinerator flue gas. The in-stack analyzer does not require a sampling system and is sensitive to low levels of oxygen with a fast response time. This instrument enables the operator to regulate the amount of excess oxygen in the offgas stream by controlling the combustion air added to the catalytic afterburner. It was found that by operating at an excess oxygen level of from 5 to 7%, offgas levels of CO and THC's are minimized. Currently, the O₂ analyzer is monitored and appropriate operating changes made manually, but the instrument has the

capability to provide an automatic control function. As a total unit, the sampling system appears to give accurate values with a minimum of operating care.

Residue Immobilization

A process has been developed to immobilize various types of transuranic wastes resulting from typical operations at nuclear material production facilities. Several methods of immobilization were initially tested and evaluated; vitrification, the conversion of residues into glass, was selected for further development because of the inherent product advantages of non-flammability, chemical stability, and mechanical durability. Glass has been produced containing, by weight, approximately 67% residue comprised of: ash from fluidized bed incineration of combustible solid and liquid wastes, diatomaceous earth filter aid, and inorganic ion exchange resin. The balance of the product consists of glass forming additives, boric acid, and sodium carbonate, necessary for vitrification.

Process Equipment

A bench-scale vitrification facility has been designed, constructed, and operated during the development of the process. The facility consists of an externally resistance-heated metal vitrification reactor, a rotating table product former, a vertical tube annealing furnace, and transport mechanisms to handle input and output.

Furnace and Reactor

First generation equipment consisted of a reactor fabricated from a length of pipe heated by an encircling arrangement of resistance wires. Reactor corrosion was a problem which was anticipated and did occur. Previous laboratory work indicated that, of relatively common materials, Inconel® 600 and Inconel® 690 were least corroded by glass melts. The first pipe reactor was constructed of Hastelloy® C pipe, a material of lower corrosive resistance that was on hand. Test runs were made to determine the desired design for the Inconel reactor. At the end of approximately 40-hours of operation, the

Hasteloy had corroded through in the gas space above the glass level. The second reactor, fabricated from a length of Inconel® 690 pipe, has exhibited no serious corrosion during the four-month period of operation. Another problem, however, forced a change in the furnace design. The early reactors were heated by closely encircling ceramic half-cylinders containing embedded resistance wires. Continuous operation at production temperature of 1050 °C resulted in rapid destructive deterioration of the wires. Many failures and much maintenance time demonstrated that such a method of heating was unacceptable for continuous remote operation.

Another type of resistance heating device, the silicon carbide glow bar, is generally considered more appropriate for high temperature service than resistance wires. A furnace heated by glow bars was therefore installed in January 1978. The exterior is approximately a meter cube with an interior heater chamber approximately 60 cm long, 38 cm deep, and 38 cm wide, containing 14 glow bars, 2.5 cm in diameter. Power is provided by 220 volt, 3 phase, SCR supply capable of delivering 8 kilowatts to each of three independently controlled zones of the furnace. A digital proportional temperature controller, integral with the power supply, provides overall temperature regulation up to 1500 °C. The furnace is a standard item of its manufacturer and therefore required no on-site design or construction effort aside from minor modifications to allow installation of the reactor.

The greatly increased capacity of the heated chamber of the new furnace allowed use of a larger and somewhat differently constructed reactor, shown in Figure 2. Fabricated primarily of Inconel 690 plate 0.64 cm thick, the reactor is approximately square in cross-section (the corners having been rounded to require minimum welding during fabrication) measuring 23 cm wide and deep, and 50 cm long. Penetrating the top near one end is an Inconel 600 pipe measuring 5.25 cm inside diameter, which serves as the feed port or inlet to the reactor. Another Inconel 600 pipe, 2.66 cm inside diameter, penetrates the top near the other end and serves as control rod guide and offgas outlet. Directly below this pipe, is the glass outlet, an Inconel 600 pipe cap which

measures 2.66 cm inside diameter with 0.64 cm diameter hole through the bottom. The glass flow control rod, a 1.9 cm diameter Inconel 690 rod threaded at one end and machined to a point at the other, is installed through a threaded portion of the offgas outlet in such a way that rotation of the rod causes it to move vertically. The conical end of the rod can be withdrawn from sealing the hole in the glass outlet allowing glass flow to be initiated and regulated. As the glass outlet is totally within the heated chamber of the furnace, a hole has been provided through the refractory and furnace bottom directly below the outlet to allow the glass to drop onto the forming table. The feed port and control rod/offgas outlet are of a sufficient length to extend through the refractory and furnace top so as to be accessible from outside.

Product Characteristics

The product of the process is a pellet of glass with a flat bottom and domed top, round in plane, measuring approximately 10-mm in diameter and 6-mm in height. The glass is dark green to black in color and highly opaque. Each pellet weighs about 0.75 gram and occupies a volume of 0.3 cm³. Simulated packaging and shipping tests indicate that the packing density of the product is 1.5 g/cm³ and that a standard 0.2 m³ drum filled with pellets will weigh approximately 320 kg. Physical tests of the glass have been performed to determine optimum operating parameters of the process. As it is expected that annealing the glass is necessary to ensure mechanical stability, the temperatures of the annealing point and strain point are two parameters which must be known. The annealing and strain points have been determined, through application of the "Standard Method of Test for Annealing Point and Strain Point of Glass by Fiber Elongation" (ASTM C336), to be 505 and 475 °C, respectively. Further tests were performed in which the duration of exposure of the glass to the annealing temperature was varied in order to determine the proper annealing time. Results from these tests indicate that pellets annealed for 15 minutes exhibit the best characteristics.

FIGURE 2. Glowbar Heated Iconel Reactor

Such pellets require forces in excess of 2300 N (517 lbs) to cause fracture.

Leach resistance of the glass is being measured by immersion of weighed glass pellets in a saturated salt solution at room temperature. The solution is one that could be present in a salt mine repository if water intrusion occurs. Periodically, the pellets have been reweighed to determine weight loss. Preliminary results indicate no statistically significant weight changes have occurred in the initial six months time span.

Offgas Cleaning

Up to 25% by weight of the reactor feed is converted into gases during the vitrification process. The offgas stream contains some vaporized constituents of the feed, notably boron compounds and sodium chloride, but is primarily CO, CO₂, and water vapor. The oxides of carbon are generated by combustion of carbon from the incinerator ash and decomposition of sodium carbonate. Some carbon in the mix is desirable for the reduction of Na₂SO₄ to Na₂S, but a large excess could reduce the metallic oxides in the melt to metals. To decrease the possibility of forming free metals, the offgas system is designed to provide an air sweep across the melt surface for efficient carbon burnout. Typical analytical results show a less than 20 ppm carbon content for the current glass buttons.

Offgas from the reactor is first passed through a sintered metal filter and then a HEPA filter. The reactor end of the piping has been subject to plugging by partially vitrified glass. The glass was formed by fines from the feed short-circuiting the melt and collecting in the hot exit piping. Some problems have also been encountered in obtaining an effective blowback on the sintered metal filters, when the filter cake becomes wetted by the condensed water in the offgas. Equipment modifications are now being implemented which solve both of these problems.

Product Physical Integrity

Physical integrity of the glass product concerns both long and short term physical stability. One of the long term effects on product integrity, studied

Table 1. Comparison of Glass Compositions

Compound	Standard Borosilicate, Glass No. 717	Borosilicate Glass, Rocky Flats Plant
Cr ₂ O ₃	-	4.6
Al ₂ O ₃	3.0	16.3
SiO ₂	70.0	38.8
CaO	-	4.6
Na ₂ O	1.0	13.4
MgO	-	2.3
B ₂ O ₃	17.0	20.0
K ₂ O	8.0	-
Li ₂ O	1.0	-
	100.0	100.0

earlier, is the possible breakage due to internal pressure from helium generated by contained radionuclides.¹ Short term concerns include the ability of the product to maintain its physical integrity after the forming step and through the subsequent handling, shipping, and final storage operations. The commercial glass industry commonly uses an annealing step after product forming to impart physical stability to its product. The annealing serves to relieve the internal stress that is frozen into the glass object during the forming step.

In the present Rocky Flats Plant (RFP) vitrification process, a controlled sized drop of molten glass falls onto a rotating plate. The free-formed button is allowed to cool below its deformation temperature and then raked into an annealer. The annealer is a vertical metal tube jacketed by a resistance furnace for maintaining the buttons at the proper annealing temperature. A laboratory determination of the correct annealing temperature for the waste-glass was necessary, as this glass composition differs from the standard borosilicate commercial glass. The composition of the waste-glass will vary with the composition of the incinerator ash added; however, an approximate composition can be calculated. In Table 1, the composition of the waste-glass is

¹D. L. Ziegler, J. W. White, A. J. Johnson, L. Q. Fong, A. R. Teter, and S. F. Chung. *Waste Incineration and Fixation for WMPR*. U. S. DOE RFP-2655. Rocky Flats Plant, Rockwell International, Golden, Colorado. November 1977.

compared to the standard NBS Borosilicate Glass Number 717 used in the tests.

The fiber elongation rate (ASTM Standard Method)² was used to determine the annealing temperature. This method used two relationships: (1) the viscosity of a glass at its annealing temperature is always $10^{13.4}$ poise, and (2) the viscosity to elongation rate can be correlated. The elongation rate of a glass fiber is related to the viscosity as:

$$\eta = \frac{4Lmg}{3\pi d^2 e}$$

Where:

- η = viscosity (poise)
- L = effective fiber length (cm)
- m = load (g)
- g = acceleration of gravity (980 cm/sec²)
- d = fiber diameter (cm)
- e = elongation rate (cm/sec)

Five thin glass fibers of differing diameter were drawn of the borosilicate standard glass and of the test glass. A standard laboratory fiber elongation apparatus was used to determine elongation rate function of temperature for both compositions of glass.

The glass fibers were heated to about 25 °C above their estimated annealing temperature, a 200 g weight attached, and the elongation rate measured. The difference between the RFP waste-glass and standard borosilicate glass is seen in Figure 3, plotted on a rectangular scale to exaggerate the difference. Measurements to determine the annealing temperature of RFP glass are shown in Figure 4, with the annealing temperature of 503 to 505 °C indicated. This compares to a 516 °C annealing temperature for the standard borosilicate.

² ASTM Designation 336-71. Standard Method of Test for Annealing Point and Strain Point of Glass by Fiber Elongation. NBS, Washington, D. C. 1977.

- Legend
- 1. Test glass (Rocky Flats) d = 0.0327 cm
 - 2. Standard Borosilicate glass d = 0.0338 cm

FIGURE 3. Elongation Rate Versus Temperature on Rectangular Scale Plot.

Probably a more meaningful characteristic of glass for a waste immobilization project is the working range, which is the temperature span over which the glass can be poured and formed. Experimental work has shown the RFP waste-glass has a wider range and a lower melting point than the standard borosilicate. In Figure 5, a comparison of calculated viscosities for RFP waste-glass and standard borosilicate is shown in the annealing point range. No viscosity data were available at higher temperatures because of the limits of the elongation test.

FIGURE 4. Elongation Rate Versus Temperature for the Glass Produced by Vitrification Process at Rocky Flats Plant. (Dotted lines indicate predicted annealing temperature.)

FIGURE 5. Calculated Viscosities for the Test Glass Compared to the Viscosity Data of the Standard Borosilicate Glass

Viscosity data at temperatures in the working range will become more important when a wider variety of wastes are used as feed. Adjustments in additive amounts and reactor temperature can then be used to adjust the viscosity to the proper range for the product forming step. A 900-mm long spindle has been fabricated for a commercial viscometer that will allow viscometry determinations on the molten glass inside a furnace. The viscometer will be calibrated using a standard borosilicate glass of a known viscosity at different temperatures.

Joule Reactor Development

A bench scale Joule heated refractory vitrification reactor, Figure 6, is in the final stages of construction. This reactor has a melting chamber with a capacity of 2.3 liters and a fining chamber with a capacity of 1.2 liters; operational tests were expected to begin in April 1978. Heat to the reactor is supplied by an electric current passing through the molten glass. Solidified glass and raw feed mix are not conductive enough to allow start-up by simply turning on the power, the glass must be molten to sufficiently conduct. The original start-up design used a sacrificial coil between the electrode to furnish enough heat to melt a thin layer of glass, thus providing a conductive path for the current.

Replacement of the sacrificial coil by remote methods would be difficult in a canyon-type operation. A silicone carbide resistance element placed above the ash mixture will be used in the modified design.

It is anticipated, however, that the corrosive environment of the vitrification chamber will cause a rapid deterioration of the resistance heating element. A method of starting up the Joule reactor using electrically conductive compounds is being investigated. If a compound is found that can be used for starting a cold or frozen melter, the resistance heating element can be eliminated.

CONCLUSIONS

1. Two stages of waste shredding are desirable using the present incinerator feed screw.

Legend

1. Fire Brick
2. Insulator
3. Ceramic Dividers
4. Large Elements
5. Small Elements
6. Drain

FIGURE 6. Joule Heated Vitrification Furnace

2. Components in the FBI system have proven reliable for long (225 hr) continuous runs.
3. The offgas sampling system for the incinerator is now providing more accurate information.
4. Construction of a production scale residue vitrification process with an acceptable long life expectancy, utilizing an externally-heated metal reactor, can be accomplished as demonstrated by the Inconel 690 reactor and glow bar furnace operating success.