
Thermal Stability Testing of Low-Level Waste Forms

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Thermal Stability Testing of Low-Level Waste Forms

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ABSTRACT

The NRC Technical Position (TP) on Waste Form specifies that waste forms should be resistant to thermal degradation. The thermal cycle testing procedure outlined in the TP on Waste Form was carried out and is believed adequate for demonstrating the thermal stability of solidified waste forms. The inclusion of control samples and the monitoring of sample temperature are recommended additions to the test. An outline for reporting thermal cycling test results is given.

To produce a data base on the applicability of the thermal cycling test, the following simulated laboratory-scale waste forms were prepared and tested: boric acid and sodium sulfate evaporator bottoms, mixed bed bead resins, and powdered resins each solidified in asphalt, cement and vinyl ester-styrene. Thermal cycling does not significantly affect the compressive strength of the solidified wastes, except powdered resins solidified in cement which disintegrated during the test and bead resins in cement which showed a loss of compressive strength. After temperature cycling, cement solidified bead resins showed areas of spalling and solidified sodium sulfate forms had surface deterioration. Asphalt solidified wastes, except powdered resins, deformed by slumping on temperature cycling. Free liquid was released from vinyl ester-styrene solidified waste forms as a result of thermal cycling. Dewatered bead and powdered resins were also tested and no free liquid was released on temperature cycling.

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THERMAL STABILITY TESTING OF LOW-LEVEL WASTE FORMS

1. INTRODUCTION

In the regulation, 10 CFR Part 61, "Licensing Requirements for Land Disposal of Radioactive Waste," the U.S. Nuclear Regulatory Commission (NRC) establishes licensing procedures, performance objectives, and technical criteria for the shallow land burial of low level radioactive wastes. To ensure the long-term safe performance of a burial site, Class B and Class C wastes should be in a form that will maintain its physical integrity for at least 300 yrs. The NRC has issued a Technical Position (TP) on Waste Form to provide guidance to waste generators on test methods and criteria for acceptable waste forms.

The TP on Waste Form specifies that wastes should be resistant to thermal degradation. After being subjected to thermal cycling, waste specimens should maintain a compressive strength greater than 50 psi and should liberate less than 0.5 percent by volume of free liquids. Outlined in the TP is a procedure for the thermal stability testing of waste forms. The purpose of this work is to determine whether the criteria and the test method are reasonable and can be achieved.

Davis and Gause⁽¹⁾ considered the testing for thermal stability of waste specimens and recommended the ASTM-B553-79, "Standard Test Method for Thermal Cycling of Electroplated Plastics,"⁽²⁾ with modifications, thus, making the procedure suitable for testing solid waste specimens. Subsequently, MacKenzie et al.⁽³⁾ reviewed the test procedure and concluded that the thermal cycling test is a reasonable means to identify thermal degradation of a waste form which might occur during interim storage, handling and transport of wastes. Moreover, it was stressed "that the importance of the test hinges on the measurements made after completion of the cycling (compressive strength, weight loss, and amount of free liquid formed) which must demonstrate that the waste form satisfies the stability requirements of Section 56(b) of 10 CFR Part 61."⁽³⁾ The procedure proposed by MacKenzie et al. and the method outlined in the TP on Waste Form are given in Appendix A.

The results presented here are not intended to verify the thermal stability (or the lack of it) of the specific waste forms tested. The aim of this study is to evaluate the test procedure in the TP on Waste Form and determine if it is sufficient to test the thermal stability of low-level waste forms. Simulated laboratory scale waste forms of the following fuel-cycle waste streams are considered: boric acid and sodium sulfate evaporator bottoms, mixed-bed bead resins, and powdered resins. These wastes are solidified in asphalt, cement, and vinyl ester-styrene. The specimens may not be representative of actual industrial waste forms but are acceptable for evaluating the test procedure. Control samples were added to the tests to distinguish changes that result primarily from temperature cycling. After thermal

cycling, the test and control specimens were examined for deformation, release of liquid and they were tested for compressive strength. Also considered in this work is the effect of thermal cycling on the release of liquid from dewatered bead and powdered ion-exchange resins.

2. EXPERIMENTAL PROCEDURES

2.1 Specimen Size

The specimen size is determined by the requirements of the compressive strength test procedure used in this study (see Section 2.3). A right cylindrical specimen with a length approximately twice the diameter is required. Therefore, the samples used have a nominal diameter of 2 in. and a nominal length of 4 in. This form will be referred to as a 2 x 4 throughout this report.

Test specimens were prepared in Nalgene 160-mL sample vials (SYBRON Nalge Cat. No. 6250-0160). The containers have a slight taper (diameter at the top is approximately 1.87 in. and the diameter at the bottom is approximately 1.82 in.) and are about 3.8-in. long. The actual diameter at the top, the bottom, and in the middle of each waste form and the length of the sample were measured before and after thermal cycling.

2.2 Thermal Cycling Procedure

The procedure used for the thermal cycle testing of waste forms is based on ASTM B553-79, "Standard Test Method for Thermal Cycling of Electroplated Plastics." The basis for using this test was discussed previously and modifications were suggested to make it suitable for testing solid waste specimens.⁽¹⁻³⁾ Details of the procedure used are outlined in this section.

2.2.1 Temperature Cycle

Four replicates of each waste stream/ solidification agent mixture were temperature cycled, and four specimens were held under controlled conditions. The number of samples tested was recommended by MacKenzie⁽³⁾ (Appendix A) and in an early draft of the Technical Position on Waste Form. The authors believe that at least three replicate samples be tested thereby providing some evidence of the reproducibility of the specimen performance.

Each 2 x 4 specimen was removed from the plastic mold and sealed in a metal can*. The cans used were 2.9-in. diameter and 4.4-in. high. It was intended that the cans be tightly sealed to prevent the possible evaporative loss of free liquid and provide no support to the forms during the temperature cycling. The canned specimens (test samples and controls) were placed in a temperature controlled chamber (maintained at $22 \pm 2^{\circ}\text{C}$) at least 16 h before the start of temperature cycling. Control samples remained in this chamber throughout the period of the test.

A single temperature cycle consisted of keeping thermally equilibrated waste forms at $22 \pm 2^{\circ}\text{C}$ for a minimum of 1 h. Tests specimens were then placed in an $60 \pm 2^{\circ}\text{C}$ oven where they remained for 1 h. Next, the test

*300 x 407 steel can with a tinned surface for food packaging (1/4-lb. electroplate) obtained from May Co., Brooklyn, NY.

specimens were placed in the $22 \pm 2^{\circ}\text{C}$ chamber. The test forms were equilibrated to $22 \pm 2^{\circ}\text{C}$ and were held at that temperature for a minimum of 1 h. Finally, the test samples were placed in a freezer set at $-40 \pm 3^{\circ}\text{C}$ for 1 h. All test samples were subjected to 30 complete cycles and then allowed to equilibrate at $22 \pm 2^{\circ}\text{C}$ before the containers were opened.

A Forma Scientific Environmental Chamber (Model 69737-1) and a standard laboratory incubator (General Electric) were used for the 22°C temperature chamber. Throughout the period of the test the temperature of these chambers fluctuated by $\pm 2^{\circ}\text{C}$. A Tenney Environmental Chamber (Model T-5-110240) was used for the high and the low temperatures. The chamber was preheated to 60°C before inserting the specimens; there was a $\pm 2^{\circ}\text{C}$ temperature fluctuation. After opening and closing the chamber, the air temperature inside the chamber would decrease and it would take several minutes for the temperature to return to 60°C . The 1-h exposure to 60°C was measured from the time the samples were placed in the oven. In similar fashion, the chamber was cooled to -40°C prior to inserting the test samples. There was a $\pm 3^{\circ}\text{C}$ fluctuation at this temperature over the period of the test. The air temperature inside the chamber would increase when the samples were introduced and it would take several minutes for the air temperature to return to -40°C . Again, the 1-h exposure to -40°C was measured from the time the samples were placed in the chamber. Throughout the test, the position of the samples during a particular cycle were randomly changed within a chamber in an attempt to minimize any possible effects from temperature variations in the chamber.

2.2.2 Waste Form Temperature

The centerline temperature of the waste forms were estimated using 2 x 4 forms of asphalt, cement, and vinyl ester-styrene with an iron-constantan thermocouple situated approximately at the center of the forms. Each of these forms was sealed in a metal can which was thermal cycled with the appropriate test samples. The temperatures measured in the three forms were considered to be representative temperatures of all the forms of the specific type.

2.3 Compressive Strength Tests

The compressive strength of the test and the control 2 x 4 waste forms was measured at the end of thermal cycling. The ANSI/ASTM C39-72 "Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens"⁽⁴⁾ was used as prescribed for testing the cement forms. However, since asphalt and vinyl ester-styrene are non-rigid materials, the stress at 10% deformation of the axial length was determined. This value serves as a measure of compressive strength of these forms. Cement and vinyl ester-styrene forms were capped with CT-55 concrete capping compound (Soiltest, Inc.) prior to compression testing. The ends of the asphalt forms were machined to flat parallel surfaces. This machining process resulted in specimens of varying lengths.

2.4 Free Liquid

2.4.1 Amount of Free Liquid

The percent free liquid, relative to the mass of the 2 x 4 form after thermal cycling, was determined according to the following equation:

$$\% \text{ free liquid} = 100 (M_T - M_f - M_c)/M_f$$

where

M_T = total mass of the can, 2 x 4 form and any free liquid after thermal cycling

M_f = mass of 2 x 4 wiped free of liquid

M_c = mass of can wiped free of liquid

2.4.2 pH of Free Liquid

The pH of droplets of free liquid was estimated using narrow range pH paper calibrated in divisions of 0.5 pH units.

2.5 Mass Change

The percent mass change reported is based on the mass of a sample (sealed container plus 2 x 4 waste form) measured, after the sample was equilibrated at 22°C, prior to the 30 temperature cycles and at the end of the cycling. The mass change is expressed as a percent of the sample mass at the start of the test.

2.6 Preparation of Waste Forms

Formulations for all of the test samples used in this study were either taken from the literature or supplied by a vendor of the solidification process used. No effort was made to optimize the formulations since the objective of this study is to determine the applicability of the thermal cycle testing procedure.

Definitions of terms and sources of the materials used in these preparations are given in Appendix B.

2.6.1 Wastes Solidified in Asphalt

The method used to prepare laboratory-scale simulated asphalt waste forms was suggested by the Werner and Pfleiderer Corporation.* Their asphalt solidification process, available to the nuclear industry, mixes a

*Telephone conversation between W. Klein, Werner and Pfleiderer Corporation, and P. L. Piciulo, August 2 and 19, 1982.

water-based waste stream with hot asphalt. Water is driven off by the heat and the final waste material is moisture free. To simulate this on a laboratory scale, it was suggested that all waste be dried prior to mixing with the asphalt. Since the waste was predried it was not expected that free liquid would be observed after thermal cycle testing. A vendor should use samples prepared according to their actual solidification processor when this testing is carried out.

Asphalt (Pioneer 221) was first melted in a stainless steel beaker, which was partially immersed in a mineral oil bath, maintained at 180°C. The pre-dried solid waste was then slowly added to the beaker while the mixture was stirred. A Premire Series 2000 Dispersator equipped with a high viscosity mixing head was used for stirring each batch of samples for 15 minutes after the materials were combined. The mixture was then poured into the sample vials and allowed to set for a minimum of 24 h before testing. The composition of the simulated waste streams and the ratio of waste-to-asphalt are given in the following sections.

2.6.1.1 Boric Acid Evaporator Bottoms

<u>Material</u>	<u>Weight Percent</u>
Borax	99.2
Crud	0.8

Dry Borax (sodium tetraborate decahydrate) was used to simulate the form of processed, neutralized boric acid waste in asphalt. The ratio of dry waste: asphalt used in the laboratory preparation was 0.43.

2.6.1.2 Sodium Sulfate Evaporator Bottoms

<u>Material</u>	<u>Weight Percent</u>
Sodium Sulfate, anhydrous	92.2
Sodium Chloride	7.4
Crud	0.4

The ratio of dry waste:asphalt used in the laboratory preparation was 0.67.

2.6.1.3 Bead Resins

<u>Material</u>	<u>Weight Percent</u>
IRN-150 (H ⁺ , -OH form)	100

The resins were dried at 110°C for 2 days and partially crushed in a blender for a minute before mixing with asphalt. The ratio of dry waste: asphalt used in the laboratory preparation was 0.67.

2.6.1.4 Powdered Resins

<u>Material</u>	<u>Weight Percent</u>
Powdex (PAO) (^{-}OH Form)	40
Powdex (PCH) (H^{+} Form)	40
Crud	10
Sodium Chloride	10

The resins were dried in an oven at 100°C for 2 days before use. The ratio of dry waste : asphalt used in the laboratory preparation was 0.67.

2.6.2 Wastes Solidified in Cement

All samples were mixed in batches and poured into forms. The specimens were capped to prevent water evaporation during the 28-day curing period. The composition of the simulated waste stream, the type of cement, and the conditions for solidification are given in the following sections.

2.6.2.1 Boric Acid Evaporation Bottoms^(5,6)

<u>Material</u>	<u>Weight Percent</u>
Boric Acid	12.0
Crud	0.1
Water	87.9

Prior to solidification the waste stream was heated to 77°C and the pH was adjusted to 12 using sodium hydroxide. Portland type III cement used for solidification was stirred into the simulated waste. The ratio of waste stream : cement was 0.5.

2.6.2.2 Sodium Sulfate Evaporator Bottoms⁽⁷⁾

<u>Material</u>	<u>Weight Percent</u>
Sodium Sulfate	14.9
Ammonium Sulfate	9.6
Sodium Chloride	2.0
Crud	0.1
Water	73.4

Prior to solidification the waste stream was heated to 77°C and the pH was adjusted to between 2.5 and 4.0 using sulfuric acid. Portland Type II cement used for solidification was stirred into the simulated waste. The ratio of waste : cement was 0.6.

2.6.2.3 Bead Resins⁽⁷⁾

<u>Material</u>	<u>Weight Percent</u>
IRN-150 (NH_4^+ , ^-OH form)	33
Water	67

Amberlite IRN-150 is a 50:50 mix of cation and anion exchange resins supplied in the H^+ and ^-OH forms, respectively. The counter ion of the cation-exchange resin was converted from the H^+ form to the NH_4^+ form prior to solidification by washing with ammonium hydroxide (NH_4OH). The resins were subsequently washed with water to $\text{pH} = 7$. The simulated waste stream was solidified in Portland Type I cement using a waste:cement ratio of 0.6.

2.6.2.4 Powdered Resins⁽⁷⁾

<u>Material</u>	<u>Weight Percent</u>
Powdex (PAO) (^-OH Form)	15.3
Powdex (PCH) (NH_4^+ Form)	15.3
Sodium Chloride	3.8
Crud	3.8
Water	61.8

Prior to solidification the H^+ form cation resins were converted to the NH_4^+ form using ammonium hydroxide. The simulated waste was solidified in Portland Type I cement using a waste:cement ratio of 0.65.

2.6.3 Wastes Solidified in Vinyl Ester-Styrene

The solidification system used in this work was supplied by Dow Chemical Company. It consists of a vinyl ester-styrene copolymer binder, a catalyst, and a promoter. Details of the procedures are considered proprietary by Dow Chemical Company. The waste streams outlined in the following four sections were solidified on a laboratory scale using the methods prescribed by Dow.

2.6.3.1 Boric Acid Evaporator Bottoms

<u>Material</u>	<u>Weight Percent</u>
Boric Acid	9.0
Sodium Borate	1.5
Sodium Sulfate	1.5
Crud	0.1
Water	87.9

The mixture was heated to 54°C and stirred to dissolve all solids. The pH of the solution was adjusted to 7 with sodium hydroxide before solidification. The ratio of waste stream : binder was 1.5. Waste and binder were mixed at room temperature.

2.6.3.2 Sodium Sulfate Evaporator Bottoms

<u>Material</u>	<u>Weight Percent</u>
Sodium Sulfate	11.0
Sodium Chloride	0.5
Sodium Phosphate	0.5
Crud	0.1
Water	87.9

The mixture was heated to 54°C and stirred to dissolve all solids. The pH of the solution was adjusted to 10.6 with sodium hydroxide. The ratio of waste stream : binder was 1.5. Mixing was performed at room temperature.

2.6.3.3 Mixed Bed Bead Resins

<u>Material</u>	<u>Weight Percent</u>
IRN-150 (NH ₄ ⁺ , OH ⁻ Form)	90
Water	10

Prior to solidification, IRN-150 resins were converted from the H⁺ and ⁻OH form to the NH₄OH form by washing with ammonium hydroxide. The resins were rinsed with water until they reached pH = 7. The waste stream was mixed at room temperature with binder in a ratio of waste:binder of 2.0.

2.6.3.4 Powdered Resin Filter Sludge

<u>Material</u>	<u>Weight Percent</u>
Powdex (PAO) (Cl ⁻ Form)	15
Powdex (PCH) (Na ⁺ Form)	15
Water	70

The cation and anion exchange resins were combined and converted from the H⁺ and ⁻OH forms with sodium chloride. The waste stream was mixed at room temperature with binder in a ratio of waste:binder of 1.5. An extender (the type and quantity are proprietary) was added to the binder prior to mixing.

2.7 Sample Identification

An explanation of the sample identification numbers is included here because the numbers appear in the figures in this report. Each sample identifier consisted of seven alphanumeric characters. Starting at the left character a T indicates that the sample belongs to the thermal cycle testing program. The second character, a letter, specifies the solidification agent (i.e., A = asphalt, C = cement, and D = vinyl ester-styrene). The next three digits indicate the date (month, day) the sample was prepared. The sixth character is a letter representing the waste stream (i.e., B = boric acid, S = sodium sulfate, R = bead resin, P = powdex resins). Finally, the last digit

is the sequential number assigned to replicate samples. Specimens numbered one through four were temperature-cycled, whereas, numbers five through eight were controls.

2.8 Dewatered Resin

Unsolidified bead and powdered organic ion-exchange resins were subjected to the temperature cycling as described in Section 2.2.1. Bead resins, IRN-150 were used as supplied and after converting to the Na^+ and Cl^- form. Powdered resin samples consisted of a mixture of equal amounts of Powdex (PAO) (^-OH form) and Powdex (PCH) (H^+ form) used as supplied and after conversion with NaCl . The resins were washed with water and initially dewatered in large batches by vacuum filtration. Cans were loosely packed with the resins (200 g) to about 80% capacity and sealed. Eight samples of each resin type were prepared. Four samples were thermal cycled and four served as controls.

2.8.1 Free Liquid

After temperature cycling, the containers were opened. Free liquid in each sample was determined by dewatering the resin using vacuum filtration for 5 minutes.

2.8.2 Resin Moisture Content

After dewatering, the percent moisture in the resins was determined by drying the resins for ~2 days at 110°C . The weight of the sample was measured before and after heating. The percent moisture content was computed as follows:

$$\% \text{ M.C.} = 100 (M_R - M_R')/M_R$$

where

M_R = mass of wet resins (before heating)

M_R' = mass of dry resins (after heating)

3. EFFECTS OF THERMAL CYCLING ON ASPHALT WASTE FORMS

3.1 Temperature Changes

The temperature of the asphalt form containing the thermocouple was recorded after each 1-h period that the waste forms were in the 60°C oven and -40°C freezer. The average centerline temperature reached a maximum of $43 \pm 2^\circ\text{C}$ and a minimum of $-11 \pm 2^\circ\text{C}$.

It is evident from these measurements that the temperature of the interior of the test forms did not reach the temperature limits of the test. One hour is an insufficient period of time for the samples as tested here to reach either 60°C or -40°C.

3.2 Mass Changes

The mass of the containerized test samples and control samples was recorded before and after thermal cycling. In every case, there was a small (<0.07%) apparent increase in the mass of samples. The averaged percent change of the sample mass is listed in Table 3.1. The difference between the increase in mass of the test samples and that of the control samples for any of the four waste streams was not significant. Therefore, the mass increase cannot be attributed to thermal cycling.

Table 3.1

Results of Thermal Cycling of Asphalt Waste Forms^a

Waste Stream		% Mass Change	Stress at 10% Deformation (MPa)
Boric Acid	Test	0.02 ± 0.01	0.28 ± 0.07
	Control	0.031 ± 0.007	0.28 ± 0.04
Sodium Sulfate	Test	0.018 ± 0.004	0.20 $\pm 0.05^{b,c}$
	Control	0.011 ± 0.002	0.26 ± 0.05
Bead Resin	Test	0.025 ± 0.002	0.27 $\pm 0.01^c$
	Control	0.042 $\pm 0.007^b$	0.31 ± 0.03
Powdered Resin	Test	0.032 ± 0.008	0.68 ± 0.05
	Control	0.043 ± 0.008	0.72 $\pm 0.06^b$

^aThe standard deviations are based on the average results of four samples.

^bAverage based on three samples.

^cSamples were machined before compression test.

3.3 Free Liquid

No free liquid was observed on the forms or in the containers after thermal cycling. This is not surprising since in the preparation of these forms, all simulated wastes were pre-dried.

3.4 Deformation of the Forms

Slumping of the asphalt forms was observed at the end of thermal cycling. Examples of typical waste form deformation for asphalt samples containing boric acid, sodium sulfate, bead resin and powdered resin are pictured in Figures 3.1 through 3.4, respectively. Each thermally-cycled form together with a control specimen was photographed at the end of the test. The simulated boric acid/asphalt composite showed extensive surface deformation and slumping (see Figure 3.1). Asphalt composites with sodium sulfate (Figure 3.2) and bead resin (Figure 3.3) simulated wastes also showed significant slumping. All powdered resin/asphalt forms had very little deformation resulting from temperature cycling. No control specimens showed any deformation. Therefore, it is concluded that the deformation of the asphalt forms is the result of temperature cycling.

The observed slumping of the asphalt specimens appeared more pronounced at the bottom of the forms and at other points where the form made contact with the metal container. This suggests that the point of contact of the form with the can is a favored point for heat transfer and that there is a temperature gradient through the form.

3.5 Compressive Strength

The average values of the stress measured at 10% axial deformation of the asphalt forms are listed in Table 3.1. Only the asphalt forms containing powdered resin have an estimated compressive strength greater than the 0.34 MPa (50 psi) which is a minimum compressive strength for a waste form recommended in the TP on Waste Form. However, the specimens used in this test were prepared in the laboratory and may not adequately represent actual asphalt waste forms produced by industry. Comparison of the average compressive strengths of samples that were temperature cycled with the average compressive strengths of control samples reveals no differences that are significant. Thus, it is concluded that thermal cycling of the asphalt waste forms has no effect on the compressive strength.

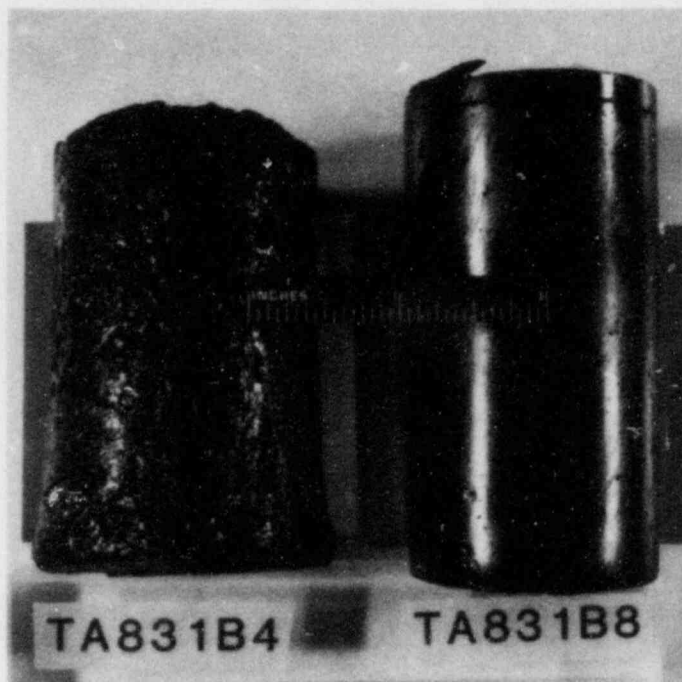


Figure 3.1 Examples of asphalt/boric acid waste forms photographed at the end of thermal cycling. Test specimen (left): TA831B4. Control specimen (right): TA831B8.

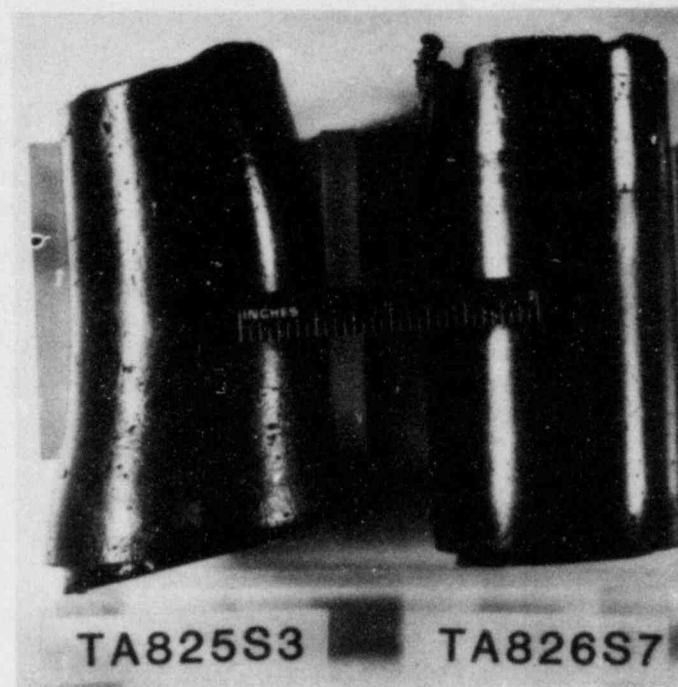


Figure 3.2 Examples of asphalt/sodium sulfate waste forms photographed at the end of thermal cycling. Test specimen (left): TA825S3. Control specimen (right): TA826S7.

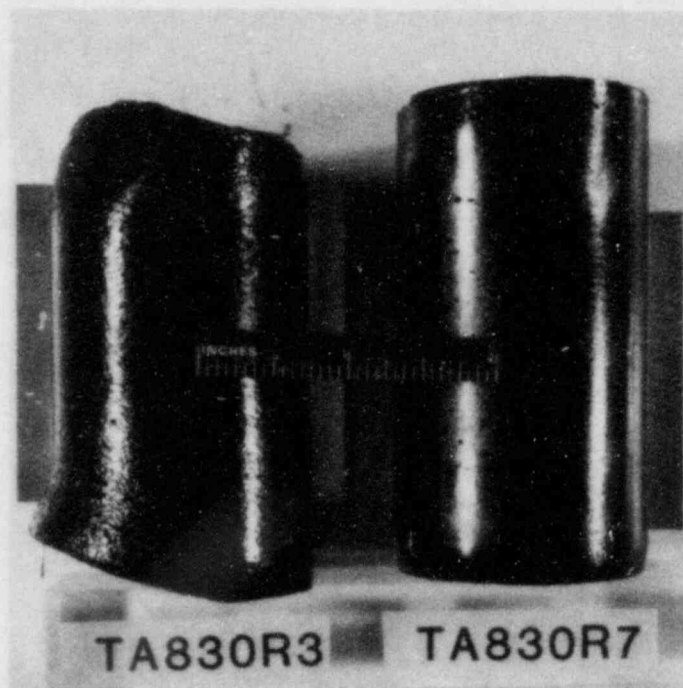


Figure 3.3 Examples of asphalt/bead resin waste forms photographed at the end of thermal cycling. Test specimen (left): TA830R3. Control specimen (right) TA830R7.



Figure 3.4 Examples of asphalt/powdered resin waste forms photographed at the end of thermal cycling. Test specimen (left): TA827P4. Control specimen (right): TA830P8.

4. EFFECTS OF THERMAL CYCLING ON CEMENT WASTE FORMS

4.1 Temperature Changes

The average temperature of the interior of the cement waste forms after 1 h in the 60°C oven was $47 \pm 3^\circ\text{C}$. After 1 h in the -40°C freezer, the temperature of the cement forms was $-14 \pm 3^\circ\text{C}$. Apparently 1 h is an insufficient period of time for heating the cement forms to 60°C or cooling them to -40°C under the conditions of the test.

4.2 Mass Changes

All containerized test samples and control samples had some decrease in mass as a result of thermal cycling. The average values of mass change listed in Table 4.1 indicate a greater decrease in mass for the thermally-cycled specimens as compared to the control specimens for all waste streams except boric acid. The observation suggests a change in the waste form caused by thermal cycling. Since the mass comparison is based on the mass of sealed sample containers, volatile material(s) must have leaked from the cans. The net loss of mass as a result of temperature cycling is greatest for the sodium sulfate waste forms. The bead and powdered resin samples show about equal mass change but somewhat lower than that of the sodium sulfate samples. Boric acid waste solidified in cement shows no apparent loss of mass resulting from temperature cycling. Possible sources of these relatively small weight losses are given in the following sections.

Table 4.1

Results of Thermal Cycling of Cement Waste Forms^a

Waste Stream		% Mass Change	Compressive Strength (MPa)
Boric Acid	Test	-0.02 \pm 0.01	30 \pm 11
	Control	-0.025 \pm 0.006	30 \pm 11
Sodium Sulfate	Test	-0.5 \pm 0.3	42 \pm 15
	Control	-0.07 \pm 0.02	43 \pm 16
Bead Resins	Test	-0.2 \pm 0.1	16 \pm 10
	Control	-0.04 \pm 0.02	22 \pm 2
Powdered Resins	Test	-0.16 \pm 0.08	b
	Control	-0.08 \pm 0.02	28 \pm 8

^aThe standard deviations are based on the average results of four samples.

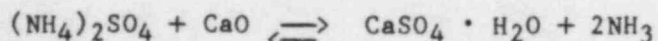
^bNo compressive strength could be determined because the forms disintegrated during thermal cycling (Section 4.4 and Figure 4.4).

4.3 Free Liquid, Container Corrosion and Gas Release

Free liquid was not observed in cans with cement forms containing boric acid, bead resin, or powdered resin wastes when they were opened after thermal cycling.

A small amount of brown oily liquid was present on the inside covers of cans with control specimens of cement solidified sodium sulfate waste. The coating initially present in the surface of the cover was gone from all of the control and test specimens containing sodium sulfate waste and from the four test specimens containing bead resins. It is suspected that the coating reacted with some component (i.e., liquid or gas) released from the forms. A liquid may be initially produced which then vaporizes.

An odor, apparently that of ammonia, was observed on opening the test and control samples containing sodium sulfate and to a lesser degree in the specimens containing bead resins. The source of ammonia is the reaction of components in the waste forms. The simulated sodium sulfate waste stream contains approximately 10% ammonium sulfate (see Section 2.6) which can react with cement. Ammonia (NH_3) can be produced according to the following reaction:⁽⁸⁾



Since the solidified cation-exchange resins have a sulfonic functional group (i.e., R-OSO_3^-) and an ammonium (i.e., NH_4^+) counter ion, a similar reaction may account for the ammonia in the cement solidified resin samples.

It is uncertain whether ammonia gas released during temperature cycling reacted with the coating on the container cover since the coating on a similar cover was unaffected after submerging in concentrated ammonium hydroxide.

4.4 Deformation of the Forms

Figures 4.1 through 4.4 are photographs taken at the end of thermal cycling showing typical physical changes in the cement solidified wastes. Only small surface cracks were observed on some thermally-cycled samples of cement/boric acid waste forms (see Figure 4.1). The cement/sodium sulfate test specimens in Figure 4.2 shows the extent of surface deterioration observed after the test. This deterioration may be attributed to the reaction of ammonium sulfate with cement as discussed in Section 4.3. Ammonium sulfate (present in the waste stream) has been regarded as possibly "the most destructive of all the sulfate salts to Portland cement concrete."⁽⁹⁾ Also apparent in Figure 4.2 is the white powdered material on the surface of the control specimen. This may be a product of the reaction accounting for the release of ammonia from the forms. A test and a control specimen of bead resin solidified in cement are depicted in Figure 4.3. The test sample showed extensive spalling at the bottom of the form and on the sides, whereas, the control sample remained intact. The predominant crumbling of the form at the bottom,

where the form contacted the can, may be directly related to inhomogeneous heating and cooling of the form (i.e., the form was warmer (or cooler) at the bottom than at the top). It is seen in Figure 4.4 that the cement/powdered resin waste forms that were temperature cycled almost completely desintegrated, whereas the control samples remained intact.

In summary, temperature cycling caused noticeable deterioration of the cement forms containing sodium sulfate, bead resin, and powdered resin simulated wastes. However, thermal cycling had little effect on the forms of cement solidified boric acid waste.

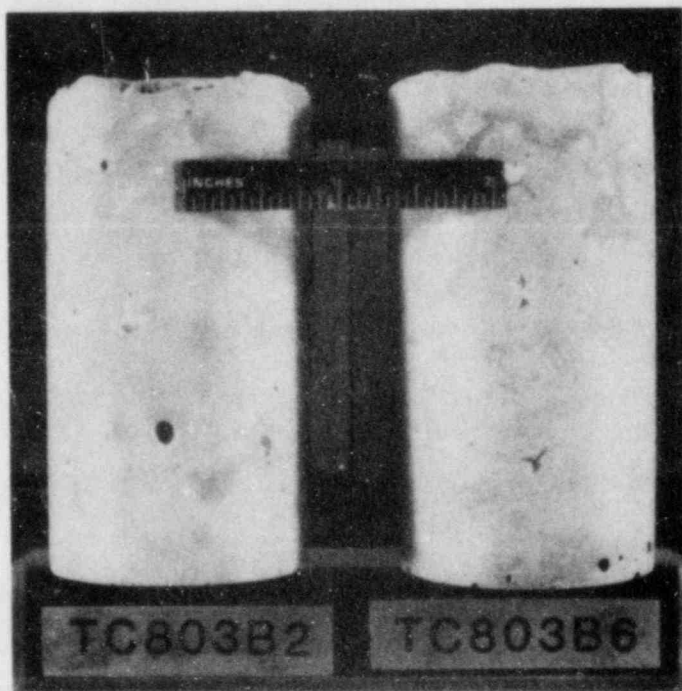


Figure 4.1 Examples of cement/boric acid waste forms photographed at the end of thermal cycling. Test specimen (left): TC803B2. Control specimen (right): TC803B6. Note: specimens were photographed top side down.

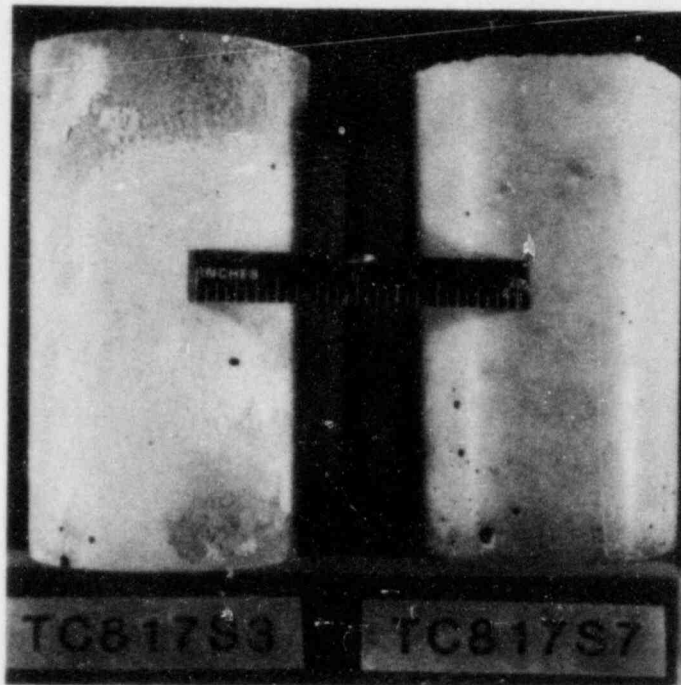


Figure 4.2 Examples of cement/sodium sulfate waste forms photographed at the end of thermal cycling. Test specimen (left): TC817S3. Control specimen (right): TC817S7. Note: specimens were photographed top side down.



Figure 4.3 Examples of cement/bead resin waste forms photographed at the end of thermal cycling. Test specimen (left): TC723R1. Control specimen (right): TC723R5. Note: specimens were photographed top side down.

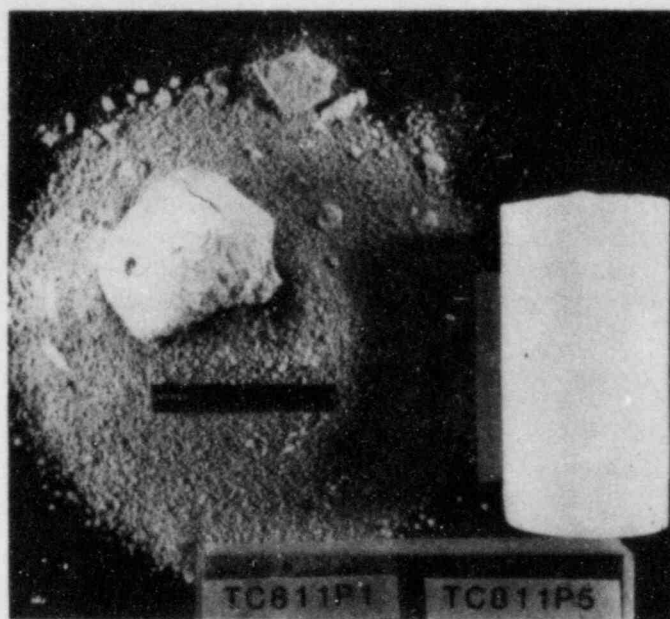


Figure 4.4 Examples of cement/powdered resin waste forms photographed at the end of thermal cycling. Test specimen (left): TC811P1. Control specimen (right): TC811P5.

4.5 Compressive Strength

Averages and standard deviations of the compressive strengths of the cement forms are listed in Table 4.1. In each case where compressive strength could be measured, the value was greater than 50 psi (0.34 MPa), which is the recommended minimum compressive strength given in the TP on Waste Form (see Appendix A). No compressive strengths could be measured for the powdered resin/cement test forms because they disintegrated during the thermal cycling (see Figure 4.4). All powdered resin/cement samples were prepared in the same manner and separated into the test group and control group by random selection. Thus, it is expected that the compressive strength given for the controls is representative of all the forms and that the disintegration is a direct result of temperature cycling. There is no difference between the compressive strengths of test specimens and control specimens of the boric acid/cement forms and the sodium sulfate/cement forms. Bead resin/cement test forms have an average compressive strength more than 25% lower than that measured for the control forms. The significance of this difference is questionable since the standard deviation is quite large. The performance of the cement forms, however, appears to present three cases. First, thermal cycling does not affect the compressive strength of the cement forms of boric acid and sodium sulfate. Next, forms of powdered resin in cement were disintegrated by thermal cycling.

Finally, the compressive strength of the cement/bead resin forms is affected by temperature cycling although, after cycling, it does exceed the 50 psi lower limit recommended in the TP on Waste Form. However, it is only by including control samples with test samples in the procedure that a loss of mechanical strength of a waste form can be observed. It is possible that thermal cycling can cause a very large decrease (e.g., a factor of 10) in the compressive strength of a waste form and yet the final value will be greater than 50 psi. This hypothetical form would comply with the TP, but the comparison of the results of the tests and control samples would reveal the deleterious effects of temperature cycling. An observation such as this may indicate a potential problem with an actual waste form.

5. EFFECTS OF THERMAL CYCLING ON VINYL ESTER-STYRENE WASTE FORMS

5.1 Temperature Changes

After the 1-h period in the 60°C oven the interior of the vinyl ester-styrene waste forms reached an average temperature of $46 \pm 2^\circ\text{C}$. When exposed to -40°C , the average temperature of the forms was $-15 \pm 3^\circ\text{C}$. Clearly 1-h exposure to the temperature extremes under the conditions of this test does not result in heating or cooling the forms to the temperature limits of the test.

5.2 Mass Changes

There was a small decrease in the mass of the containerized test samples and control samples of vinyl ester-styrene waste forms. The average values of the percent mass change for the different streams are listed in Table 5.1. There is no statistical significance for the decrease in mass of the test samples and that of the control samples for any of the four waste streams. This loss of mass, therefore, is not attributed to the thermal cycling of the waste forms.

Table 5.1

Results of Thermal Cycling of Vinyl Ester-Styrene Waste Forms^a

Waste Stream		% Mass Change	Free Liquid % pH ^b		Stress at 10% Deformation (MPa)
Boric Acid	Test	-0.08 \pm 0.03	0.82 \pm 0.08	5.5 \pm 0.5	17 \pm 1
	Control	-0.04 \pm 0.02	0.10 \pm 0.04	ND	18.6 \pm 0.3
Sodium Sulfate	Test	-0.08 \pm 0.02	0.7 \pm 0.1	5.3 \pm 0.5	11.3 \pm 0.4
	Control	-0.05 \pm 0.02	0.13 \pm 0.02	ND	10.8 \pm 0.5
Bead Resin	Test	-0.07 \pm 0.02	0.8 \pm 0.1	4.5 \pm 0.5	18 \pm 1
	Control	-0.05 \pm 0.02	0.18 \pm 0.02	4.5 \pm 0.5	18.7 \pm 0.3
Powdered Resin	Test	-0.05 \pm 0.04	1.24 \pm 0.07	4.5 \pm 0.5	13.0 \pm 0.1
	Control	-0.05 \pm 0.04	0.42 \pm 0.09	4.5 \pm 0.5	13.2 \pm 0.5

^aThe standard deviations are based on the average results of four samples

^bND means not determined.

5.3 Free Liquid and Container Corrosion

Droplets of free liquid were present in the test samples and the controls of the boric acid, sodium sulfate, bead resin and powdered resin waste streams solidified in vinyl ester-styrene. Figures 5.1 through 5.4 show examples of typical test and control specimens of the four waste streams solidified in vinyl ester-styrene. Droplets of liquid are visible on each of the test specimens. Additional liquid condensed on the inside surface of the cans. Liquid found in the control samples was largely on the container interior and thus none is visible on the control specimens in the photographs. The average amounts of liquid, as a percentage of the waste form mass, are listed in Table 5.1. The values given are similar to the volume percent free liquid since the average density of the waste forms is $1.04 \pm .04$. For each waste stream, the amount of free liquid released from the test specimens is significantly greater than that found in the control samples.*

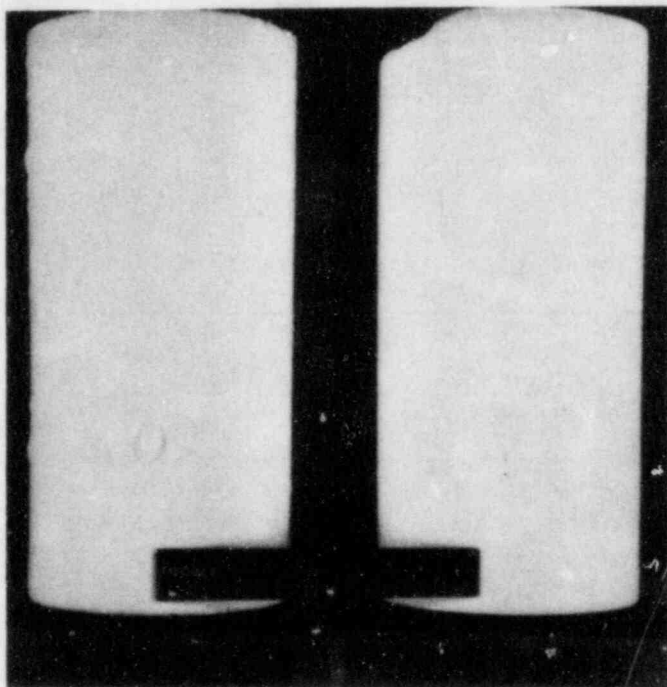


Figure 5.1 Examples of vinyl ester-styrene/boric acid waste forms photographed at the end of thermal cycling. Test specimen (left): TD812B4. Control specimen (right): TD812B8.

*The percent mass change in Table 5.1 does not directly correlate with the loss of free liquid because the mass change is based on measurements of containerized samples. It is recommended that the mass of each sample component (e.g., form, can, liquid) be measured before and after cycling to better evaluate any physical changes.

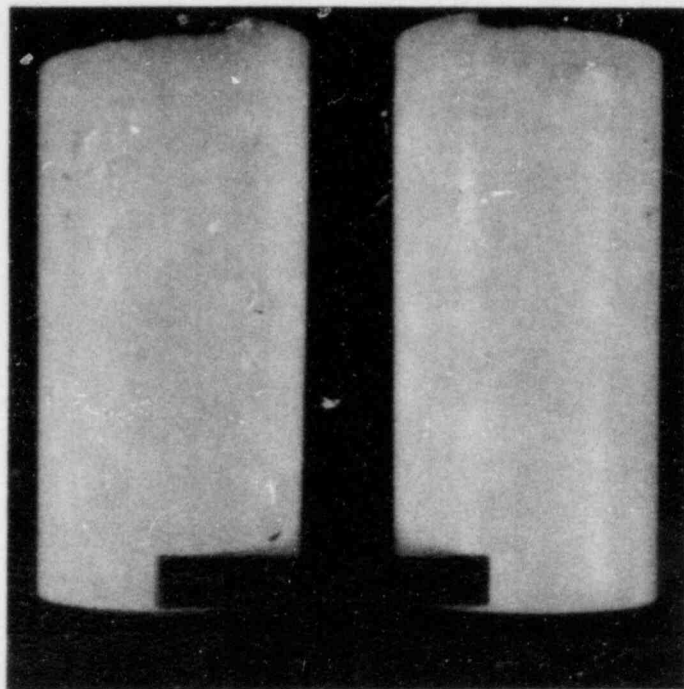


Figure 5.2 Examples of vinyl ester-styrene/sodium sulfate waste forms photographed at the end of thermal cycling. Test specimen (left): TD811S3. Control specimen (right): TD811S7.

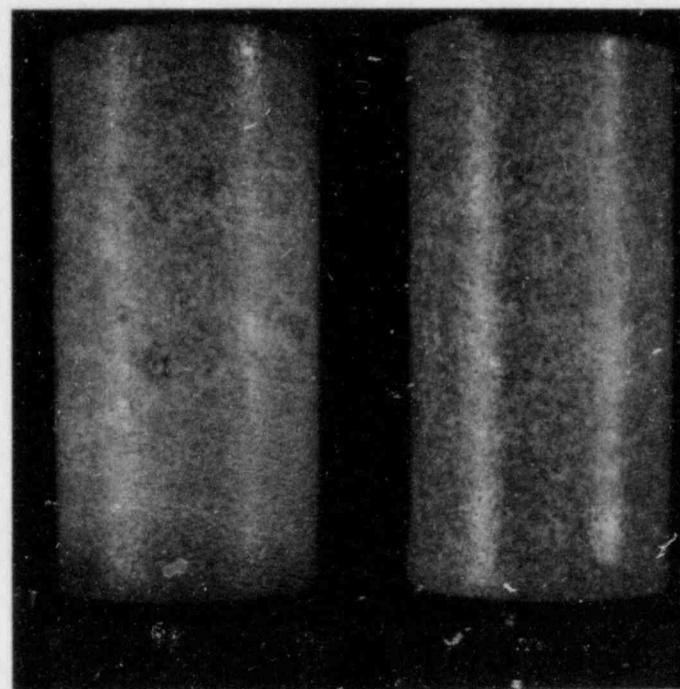


Figure 5.3 Examples of vinyl ester-styrene/bead resin waste forms photographed at the end of thermal cycling. Test specimen (left): TD813R4. Control specimen (right): TD816R8.

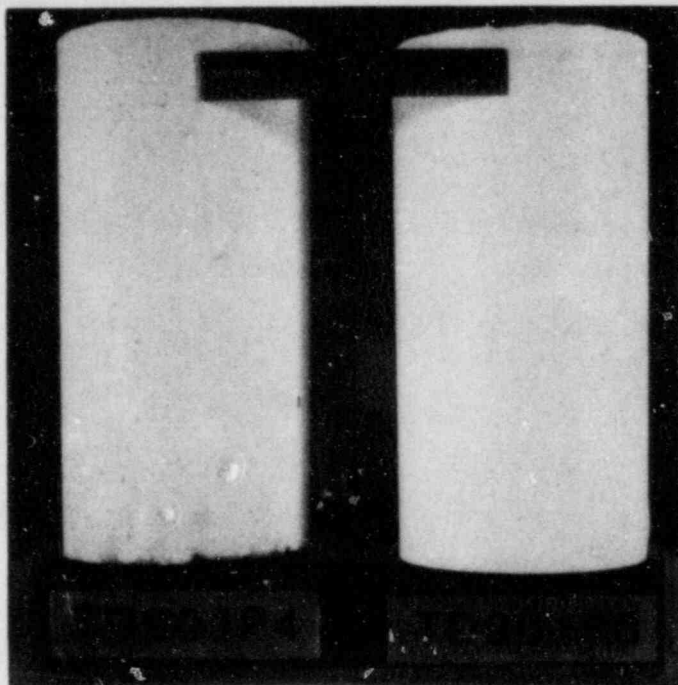


Figure 5.4 Examples of vinyl ester-styrene/powdered resin waste forms photographed at the end of thermal cycling. Test specimen (left): TD901P4. Control specimen (right): TD901P8.

The free liquid released from the forms was acidic, having a pH ~ 5 (see Table 5.1). However, the pH of the liquid is not consistent with the pH of the various waste streams. Prior to solidification, the pH of the waste streams was adjusted as follows: boric acid, pH ~ 7 ; sodium sulfate, pH ~ 10 ; bead resins, pH ~ 7 ; and powdered resins, pH ~ 7 . A possible reason for the low pH of the free liquid may be the dissolution of CO_2 from the air into pure water released from the form. Pure water might be liberated in a distillation process during thermal cycling.

Corrosion of the tin cans was observed in containers holding forms of three of the four waste streams solidified in vinyl ester-styrene. Neither the test samples nor the control samples of solidified bead resins caused container corrosion. Test specimens of solidified boric acid and sodium sulfate wastes caused a corrosive reaction with the containers primarily originating at the point(s) of contact of the 2 x 4 form with the container. None of the control samples for these two waste streams showed any corrosion of the cans. All forms containing powdered resins corroded the bottoms of the cans, however, the corrosion in the test samples was more severe than in the controls. The corrosion is not only the result of the free liquid, but is also dependent on the waste stream solidification and temperature cycling.

It is concluded that thermal cycling promotes the release of liquid from vinyl ester-styrene solidified waste forms. Moreover, the quantity of free liquid present after thermal cycling is greater than the 0.5% limit for waste processed to a stable form according to the Regulation 10 CFR Part 61.56. However, the observations, based on testing laboratory-scale samples, may not be representative of full size waste forms. For example, the liquid released from a vinyl ester-styrene form may come from a layer of finite thickness near the surface of the form. If the thickness of this layer were independent of sample size, then the percent of liquid released would be related to the sample size. That is, as the sample size increases, the percent liquid released relative to the sample volume would decrease. An effect such as this may alter the ultimate conclusion of the test. If this is the case, the vendor of the solidification matrix should demonstrate whether or not the quantity of liquid released is dependent on the test sample size.

5.4 Deformation of the Forms

None of the vinyl ester-styrene forms showed signs of deformation at the end of thermal cycling.

5.5 Compressive Strength

Average values of the stress measured at 10% axial deformation of the vinyl ester-styrene waste forms are listed in Table 5.1. The estimated compressive strengths are substantially greater than the 0.34 MPa (50 psi) minimum compressive strength for a waste form recommended in the TP on Waste Form. Compressive strengths of specimens that were thermally-cycled show no significant difference from the compressive strengths of corresponding control samples. Therefore, temperature cycling of the vinyl ester-styrene waste forms, which enhanced the release of liquid from the forms, is not detrimental to mechanical properties of the material.

6. EFFECTS OF THERMAL CYCLING ON DEWATERED ORGANIC ION-EXCHANGE RESINS

6.1 Mass Changes

The masses of the samples were compared before and after the 30-cycle tests. Averages of the change in mass are listed in Table 6.1. In general, the samples that were temperature cycled had a larger decrease in mass than the controls. However, the difference in mass change between the test and control samples is not significant for the bead resins or the powdered resins in the H^+ and OH^- form. It is suspected that the loss of mass is the result of moisture leaking through the container seal. A large vapor pressure during the heating cycle may account for the mass loss being larger for the test samples than for the controls which were kept at constant temperature.

Table 6.1

Results of Thermal Cycling Dewatered Organic Ion-Exchange Resins^a

Resin	% Mass Change	Free Liquid ^b (%)	Moisture Content ^c (%)
Bead (H^+ , OH^-)			[58.2]
Test	-0.07 \pm 0.05	0	59.9 \pm 0.5
Control	0.00 \pm 0.02	0	60.3 \pm 0.3
Bead (Na , Cl^-)			[49.6]
Test	-0.3 \pm 0.3	0	49.2 \pm 0.6
Control	-0.03 \pm 0.08	0	49.3 \pm 0.4
Powdered (H^+ , OH^-)			[70.1]
Test	-0.13 \pm 0.03	2.6 \pm 0.4	63.3 \pm 0.4
Control	-0.07 \pm 0.03	3.0 \pm 0.8	63.8 \pm 0.3
Powdered (Na , Cl^-)			[67.2]
Test	-0.12 \pm 0.02	4.6 \pm 0.9	64.3 \pm 0.2
Control	-0.06 \pm 0.02	5 \pm 1	64.0 \pm 0.3

^aThe standard deviations are based on the averaged results of four samples.

^bFree liquid is given as a percent of mass of liquid relative to the mass of resins.

^cValue in brackets are the moisture contents determined before the resins were containerized.

6.2 Free Liquid

No free liquid was recovered from any of the test samples or control samples of bead type mixed-bed resins in either the H^+ and OH^- forms or the Na^+ and Cl^- forms.

Powdered resins (both test samples and control samples) released liquid on redewatering after the thermal cycle test. The average amount recovered is given in Table 6.1 as a percent of the resin mass. However, since the amount of free liquid was similar for the test samples and the controls, the release of liquid cannot be attributed to temperature cycling. This is most likely due to the dewatering procedure used. Apparently, dewatering powdered resins in sample size quantities (~200 g) is more efficient than dewatering in a large batch, as before the test, when the containers were initially filled. Bead resins do not appear to be as sensitive as the powdered resins to the dewatering procedure used.

6.3 Resin Moisture Content

The average percent moisture content for the test and control samples of the various resin types is listed in Table 6.1. Also listed are the percent moisture contents of the resins measured before they were put in cans. There is no difference between the percent moisture content of the test specimens and control specimens for any of the resin types tested. Thus, it is concluded that, under the conditions of this test, temperature cycling does not significantly affect the amount of water contained by the ion-exchange resins.

A comparison of the moisture content of the resins before the thermal cycling test with that after the test supports the hypothesis for free liquid release made in Section 6.2. The resins in the H^+ and OH^- form and in the $NaCl$ have moisture contents similar to those measured before the resins were containerized. However, the moisture content of the powdered resins after redewatering at the end of thermal cycling is smaller than the moisture content measured before the test.

7. CONCLUSIONS

7.1 Test Procedure

The procedure outlined in the draft Branch Technical Position on Waste Form (see Appendix A) is believed to be adequate for testing the thermal stability of solidified low level waste forms based on the limited testing carried out in this program. However, the following additions to the procedure are recommended:

- Control samples should be added to the tests. These specimens should be prepared the same way as the samples for temperature cycling. During the period of the 30 cycles, the controls should be stored at room temperature (i.e., 22°C). At the end of thermal cycling, the controls should be examined and compression tested together with the thermal-cycled samples. Comparison of the results can serve to distinguish the effects (if any) of thermal cycling.
- The centerline temperature of at least the thermally-cycled samples should be monitored. At a minimum, it is necessary to know the temperature of the waste forms as they equilibrate to room temperature and must be maintained at that temperature for at least 1 h. The use of one or two forms fitted with thermocouples is believed sufficient to estimate the temperature of the test samples. Monitoring the temperature of the forms during the high and low temperature exposures revealed that 1 h is not a sufficient period for the samples to equilibrate at the temperature extremes. This can be attributed to a combination of the thermal conductivity of the waste form material and any insulation from the container. (It is noteworthy to consider that under field conditions a full scale waste form is also likely to experience thermal cycling which can result in a temperature gradient within the form. Thus, the temperature gradients found during the laboratory testing are believed somewhat typical of field conditions.) Containers that do not provide excessive insulation such that the test forms are subjected to only a very small portion of the desired temperature change should be used in the laboratory testing. Monitoring and reporting the centerline temperature will help a reviewer judge whether the experimental arrangement has biased the conclusions of the test.

The results of this study do not provide sufficient data to recommend a single container material to be used. The use of a container material typical of that which would be used in burial is acceptable provided the material is unaffected by the test conditions. (See Appendix A.) Further, the test container should not provide support of the sample and it should close tightly to prevent evaporative loss of any free liquid. Perhaps a metal container would be preferred since metals typically have large thermal conductivities and thus would allow better heating and cooling of the waste form specimen during thermal cycling. Measurements of the waste form centerline

temperature are necessary to provide an indication that as a minimum the temperature of the waste form passes through the freezing point of water.

The procedure for testing the thermal stability of waste forms specifies the use of ASTM C-39 for the compressive strength determinations. This method is designed for testing cement forms but not for testing non-brittle materials such as asphalt and vinyl ester-styrene. Alternative methods are available for testing such materials.^(10,11) Measuring the stress at a given deformation can provide an estimate of mechanical strength of a non-brittle material. In this work, using the apparatus and compression rate prescribed by ASTM C39, the stress at 10% axial deformation was reported as a nominal compressive strength. Other tests may also provide adequate mechanical strength data. The suitability of several methods for determining the mechanical strength of low-level waste forms has been addressed by Davis et al.⁽¹⁾

7.2 Reporting of the Testing Results

A report of the test results should include the following information:

- The compressive strength of the test forms and the controls.
- The amounts of free liquid released from the test forms and control samples. The characteristics of this liquid (at a minimum pH) should be noted.
- The amount of weight loss of the test forms and the control samples.
- The centerline temperatures of the waste forms during the 30 exposures to 60°C and -40°C.
- The visual changes (e.g., slumping, shrinkage, cracking, spalling) in the test forms and the controls. Photographs provide a good documentation of such changes.

A report on the thermal stability testing of solidified low-level waste forms should indicate whether the forms are sufficiently resistant to thermal degradation. To qualify, a waste form should have <0.5% by volume free liquid and have a compressive strength >50 psi after thermal cycling. Additionally, the conditions of the test (i.e., waste form centerline temperature) and observed physical changes (i.e., weight loss and sample deformation) should be addressed. Physical deterioration or changes in the mechanical properties of thermal cycled samples as compared with controls should be included in the evaluation of the waste form stability. If there is doubt that a result from the test is representative of actual industrial waste form behavior, (e.g., as may be the case with the amount of free liquid released from the vinyl ester-styrene forms tested in this program) additional evidence should be provided by the vendor to mitigate any uncertainties.

7.3 Sample Performance

Although the samples tested in this work may not be representative of actual industrial wastes, the following waste-form-specific observations were made:

- Slumping of asphalt solidified boric acid, sodium sulfate and bead resin waste resulted from temperature cycling. This is of little consequence since actual industrial waste will initially be in a container that provides support to the material.
- Temperature cycling of asphalt solidified waste forms does not cause a significant change in the effective compressive strength of the waste forms.
- Thermal cycling between 60°C and -40°C resulted in visible deterioration of the cement forms containing sodium sulfate, bead resin, and powdered resin simulated waste. However, thermal cycling had little effect on the cement solidified boric acid waste forms.
- There was no significant effect on the compressive strength of the cement forms containing boric acid or sodium sulfate which can be attributed to thermal cycling. However, the mechanical strength of the bead resin/cement samples is directly affected by temperature cycling. Cement solidified powdered resin waste was almost completely destroyed by thermal cycling.
- Free liquid was released on thermal cycling vinyl ester-styrene waste forms. The amount of free liquid is greater than the 0.5% limit for stabilized wastes given in 10 CFR Section 61.56. It is suggested that additional work be performed on the part of the vendor to determine the effect of sample size on the release of liquid during temperature cycling.
- Thermal cycling does not significantly effect the compressive strength of vinyl ester-styrene waste forms.
- Temperature cycling (as performed in this work) does not significantly affect the moisture content of bead or powdered organic ion-exchange resins. Moreover, this test indicates that thermal cycling does not contribute to the release of free liquid.

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APPENDIX A

PROCEDURE AND CRITERIA FOR DETERMINING THE RESISTANCE TO THERMAL DEGRADATION OF LOW-LEVEL WASTE FORMS

A.1 Procedure for Demonstrating Stability to Thermal Degradation*

1. The heating and cooling chambers used shall conform to the description given in ASTM-B553-79, Section 3.
2. A minimum of four samples are required. They must be cored samples from simulated full-size waste form (or actual waste form if desired), and must be suitable for use in compressive strength tests carried out according to ANSI/ASTM-C39-72.
3. Each sample is placed in a container which is large enough that it does not provide support to the sample. The container must close tightly so there will be no evaporative loss of any free liquid. Containers can be made of metal or any material unaffected by the test conditions.
4. Marked containers with samples are placed in the test chamber and a series of 30 thermal cycles is carried out following the directions given in Section 5.4.1 through 5.4.4 of ASTM B553. The high temperature limit of the cycles is 60°C and the low temperature limit -40°C. During the cycling, samples are to be rotated randomly or in a pre-determined manner to compensate for any thermal gradients in the temperature-controlled chambers.
5. At the conclusion of the 30 cycles, the samples are removed from their containers and visual changes (e.g. shrinkage, cracking, spalling, deformation) are noted. Weight loss, amount of free liquid and compressive strength are determined and recorded. If statistics are inconclusive for the four samples, the test must be repeated with a larger number.
6. To qualify the waste form, the tested samples must, as a minimum, show <5% change in dimensions (shrinkage), liberate <1% (sic) by volume free liquid, and have a compressive strength >50 psi.

*Reproduced from Reference 3.

APPENDIX A, Continued

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A.2 Guidance for Determining Resistance to Thermal Degradation Given in the Technical Position on Waste Form

Waste specimens should be resistant to thermal degradation. The heating and cooling chambers used for the thermal degradation testing should conform to the description given in ASTM B553, Section 3. Samples suitable for performing compressive strength tests in accordance with ASTM C39 or ASTM D1074 should be used. Samples should be placed in the test chamber and a series of 30 thermal cycles carried out in accordance with Section 5.4.1 through 5.4.4 of ASTM B553. The high temperature limit should be 60°C and the low temperature limit -40°C. Following testing the waste specimens should have compressive strengths greater than 50 psi as tested using ASTM C39 or ASTM D1074.

APPENDIX B

SOURCES OF MATERIALS USED IN WASTE FORM PREPARATIONS

Ammonium Sulfate, $(\text{NH}_4)_2\text{SO}_4$	Analytical reagent grade, Mallinckrodt, Inc.
Borax	See Sodium Borate.
Boric Acid, H_3BO_3	Bio Clinical Laboratories.
Crud	Simulated with fine air cleaner test dust (No. 1543094), AC spark plug division, General Motors Corporation.
IRN-150	Amberlite technical grade mixed cation and anion ion-exchange resin, supplied in the H^+ and OH^- form. Rohm and Haas Company.
Powdex (PAO)	Anion, hydroxide form powdered ion-exchange resin. Ecodyne Grover Water Division.
Powdex (PCH)	Cation, hydrogen form powdered ion-exchange resin. Ecodyne Grover Water Division.
Sodium Borate, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$	Crystals, Fisher Scientific Company.
Sodium Chloride, NaCl	Crystal, analytical reagent, Mallinckrodt, Inc.
Sodium Phosphate, $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$	Reagent grade, Matheson Coleman and Bell, Inc.
Sodium Sulfate, Na_2SO_4	Anhydrous, Reagent grade, J. T. Baker Chemical Company.
Water	Deionized water was used in all cases.

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13. ABSTRACT (200 words or less) <p>The NRC Technical Position (TP) on Waste Form specifies that waste forms should be resistant to thermal degradation. The thermal cycle testing procedure outlined in the TP on Waste Form was carried out and is believed adequate for demonstrating the thermal stability of solidified waste forms. The inclusion of control samples and the monitoring of sample temperature are recommended additions to the test. An outline for reporting thermal cycling test results is given.</p> <p>To produce a data base on the applicability of the thermal cycling test, the following simulated laboratory-scale waste forms were prepared and tested: boric acid and sodium sulfate evaporator bottoms, mixed bed bead resins, and powdered resins each solidified in asphalt, cement and vinyl ester-styrene.</p>					
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