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EPICOR-II Resin Waste Form Testing

Prepared by R. M. Neilson, Jr., J. W. McConnell, Jr.

EG&G Idaho, Inc.

Prepared for
U.S. Nuclear Regulatory Commission

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ABSTRACT

One goal of the EPICOR-II Resin/Liner Investigation Program is to confirm the adequacy of the test procedures specified in the U.S. Nuclear Regulatory Commission "Technical Position on Waste Form" (TP) relative to compliance with stability requirements for solidified Class B and C wastes. In previous work, sponsored by the Department of Energy under Contract No. DE-AC07-76IDO1570, formulations were developed to solidify radioactive wastes from EPICOR prefilters PF-7 (organic ion exchange resins) and PF-24 (organic ion exchange resins with zeolite) from Three Mile Island Unit-2 using Portland Type I-II cement and vinyl ester-styrene. Those waste forms were fabricated and then subjected to the specified stability test procedures. This report describes later work funded by the U.S. Nuclear Regulatory Commission. That work consisted of performing the comprehensive waste form testing specified in the TP. Test methodologies used to verify compliance with test criteria for free standing water, compressive strength, thermal stability, leachability, and radiation stability are described. The waste form performance data are presented and evaluated in this report.

SUMMARY

Portland Type I-II cement and vinyl ester-styrene waste forms incorporating ion exchange resin waste from EPICOR-II prefilters were subjected to the tests specified in the "Technical Position on Waste Form" (TP) issued by the U.S. Nuclear Regulatory Commission (NRC). These waste forms incorporated ion exchange resin waste from EPICOR-II prefilters PF-7 and PF-24, which were expended in the cleanup of Three Mile Island Unit 2 liquid waste. The test procedures addressed: (a) free liquids, (b) homogeneity, (c) compressive strength, (d) resistance to thermal degradation, (e) leachability, (f) immersion, (g) radiation stability, and (h) biodegradability. The purpose of this work was to evaluate the adequacy of those procedures relative to assuring compliance with the stability requirements for solidified Class B and C low-level waste in 10 CFR 61, "Licensing Requirements for Land Disposal of Radioactive Waste." Waste form performance data were also obtained as a result of this work. Since the formulations utilized for solidification of the EPICOR-II wastes have low waste loadings relative to commercial practice in order to assure integrity during testing, these performance data may overestimate the conservatism of commercial products.

The Portland cement and vinyl ester-styrene waste forms tested were found to meet the waste form stability requirements in 10 CFR 61. (Biodegradation testing is continuing and will be the subject of a later report.) This work demonstrated that appropriate administrative procedures and controls can be implemented to minimize contamination and personnel exposure while utilizing the procedures specified in the TP.

While the procedures specified in the TP were generally satisfactory for demonstrating compliance with the stability requirements of 10 CFR 61, recommendations were developed to improve the guidance presented in the TP. These recommendations address both clarification of specific items and modification of the designated procedures in order to better satisfy the intent of the regulation.

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INTRODUCTION

EPICOR-II prefilters expended in the cleanup of Three Mile Island Unit 2 (TMI-2) liquid wastes were transported to the Idaho National Engineering Laboratory (INEL) as part of the EPICOR-II Research and Disposition Program. In one aspect of this program, funded by the U.S. Department of Energy (DOE), the solidification of EPICOR-II wastes from prefilters PF-7 and -24 using Portland Type I-II cement and vinyl ester-styrene was investigated by EG&G Idaho, Inc. (EG&G Idaho). A total of 267 radioactive waste form specimens was prepared by hot cell solidification operations in this activity. Development of formulations for the solidification of these EPICOR-II wastes has been described previously.[1]

The formulations used for the immobilization of EPICOR-II waste forms in this previous work were developed to produce waste forms meeting the regulatory requirements of 10 CFR 61, "Licensing Requirements for Land Disposal of Radioactive Waste."[2] This regulation classifies low-level waste on the basis of overall disposed hazards as Class A, Class B, or Class C. While certain minimal requirements must be met by all waste, Class B and Class C waste must also meet stability requirements.

These stability requirements, which are applicable to EPICOR-II waste, necessitate that (a) the waste forms have structural stability under the expected disposal conditions and (b) the waste forms contain as little free-standing noncorrosive liquid as is reasonably achievable (no more than 0.5% of the volume of the waste for waste processed to a stable form). [For non-stable waste, the structural stability requirement can be met by processing the waste to a stable form or placing the waste in a disposal container or structure that provides stability after disposal.] The formulations developed for the solidification of EPICOR-II prefilter waste with Portland cement are particularly significant, since published information concerning the immobilization of ion exchange resin wastes in Portland cement indicates instances of low product integrity, waste form disintegration, and free-standing water.[3-5] Thus, the formulations utilized for solidification of actual EPICOR-II wastes have low waste loadings compared to commercial practice in order to assure waste form integrity during performance testing.

The U.S. Nuclear Regulatory Commission (NRC), in its "Technical Position on Waste Form" (TP),[6] provides guidance to waste generators on waste form test methods and acceptable results for compliance with the waste form requirements of 10 CFR 61. The stability performance requirements and test methods specified for Class B and C waste address the following: (a) free liquids, (b) homogeneity, (c) compressive strength, (d) resistance to thermal degradation, (e) leachability, (f) immersion, (g) radiation stability, and (h) biodegradation. In this study, EPICOR-II waste forms were subjected to the specified test procedures to confirm the adequacy of the procedures to assure compliance with stability requirements. The validity of the performance requirements with respect to the intent of 10 CFR 61 was not assessed. The testing described in this report also resulted in the development of performance data for EPICOR-II waste forms. Biodegradation testing is continuing and is not included in this report.

MATERIALS AND PROCEDURES

EPICOR-II Waste Forms

EPICOR-II prefilter waste was obtained from prefilters PF-7 and PF-24. Waste from prefilter PF-7 contained a mixture of synthetic organic ion exchange resin types (phenolic cation, strong acid cation, and strong base anion resins), while prefilter PF-24 contained a mixture of synthetic organic ion exchange resins (strong acid cation and strong base anion resins) with an inorganic zeolite. The specific ion exchange resins employed in this process and their relative quantities are proprietary.

EPICOR-II prefilter waste was solidified with Portland cement and vinyl ester-styrene in August 1983. Solidification operations were conducted remotely in a hot cell, as shown in Figure 1. Portland cement waste forms were prepared using Portland Type I-II cement (Oregon Portland Cement Co., Inkom, ID). Vinyl ester-styrene is a proprietary thermosetting polymer solidification agent developed and supplied by the Dow Chemical Company (Midland, MI).

EPICOR-II waste was homogenized and water was added to convert the waste to decanted form prior to solidification; i.e., ion exchange resins have absorbed water to saturation and the interstitial void space between resin beads has been filled with water. For solidification with Portland cement, additional water was added to the EPICOR-II waste for cement hydration and mix fluidity. A detailed description of the solidification of EPICOR-II waste can be found in Reference 1.

The formulations of Portland cement and vinyl ester-styrene waste form batches incorporating EPICOR-II prefilter waste are found in Tables 1 and 2, respectively. The designation scheme for waste form batches is a letter (C or D) indicating the solidification agent (C = Portland Type I-II cement, D = Dow vinyl ester-styrene), followed by a number signifying the waste type (1 = organic resin from prefilter PF-7, 2 = organic resin with zeolite from prefilter PF-24). A letter may follow, indicating successive batches of a given type (same solidification agent and waste type). Individual waste form specimens are identified with the batch designation followed by a hyphen and a successive specimen number (1 to 36). Waste batches typically contained 36 specimens; D1-1 is the first specimen prepared in batch D1 (vinyl ester-styrene with prefilter PF-7 waste). Tables 1 and 2 indicate that the vinyl ester-styrene formulations contain more than twice the amount of decanted resin waste on a weight basis than the Portland cement formulations. However, waste loading in terms of packaging efficiency (volume of decanted waste incorporated per unit volume of the solidified waste form) is a more meaningful measure of comparison. Average waste form packaging efficiency and density data are provided in Table 3.

The EPICOR-II waste forms have a diameter of 4.76 cm (1.88 in.) and an average length of 7.6 cm (3.0 in.), with a tolerance of ±0.6 cm (0.3 in.) on length. The waste forms were prepared in low-density polyethylene vials with an inside diameter of 4.76 cm (1.88 in.). and a height of 10.2 cm (4.0 in.). The preparation vials had snap-on lids to prevent water evaporation during curing and storage.

Free Liquids

The Technical Position on Waste Form states that:

> "Waste specimens should have less than 0.5 percent by volume of the waste specimen as free liquids as measured using the method described in ANS 55.1.[7] Free liquids should have a pH between 6 and 11."

The ANS 55.1 method consists of examination of the waste package (waste form and container) and the waste form after sufficient time has been allowed for solidification. This method is directed towards 55-gal drums and larger waste containers and involves the following procedure:

- The waste container is opened and the waste form upper surface examined for free liquid.
- With the waste container in an upright orientation, the low point of the container is breached with a minimum 1-in.^{2} opening and any free liquid flowing from the breach observed.
- The waste form is examined by sectioning, coring, or other means to determine if any free liquid exists within the solidified mass.

The method of ANS 55.1 was modified for free liquid determination of EPICOR-II waste forms in the following manner. After sufficient time was allowed for curing (or before compression testing), the preparation vial lid was removed and the upper waste form surface examined for free liquid. The preparation vial was examined for any free liquid after removal of the waste form before inspection or testing. The fracture surfaces of waste forms were also studied after compression testing. Any free liquid observed was collected or its volume estimated. The pH of any free liquid was measured using wide-range pH paper.

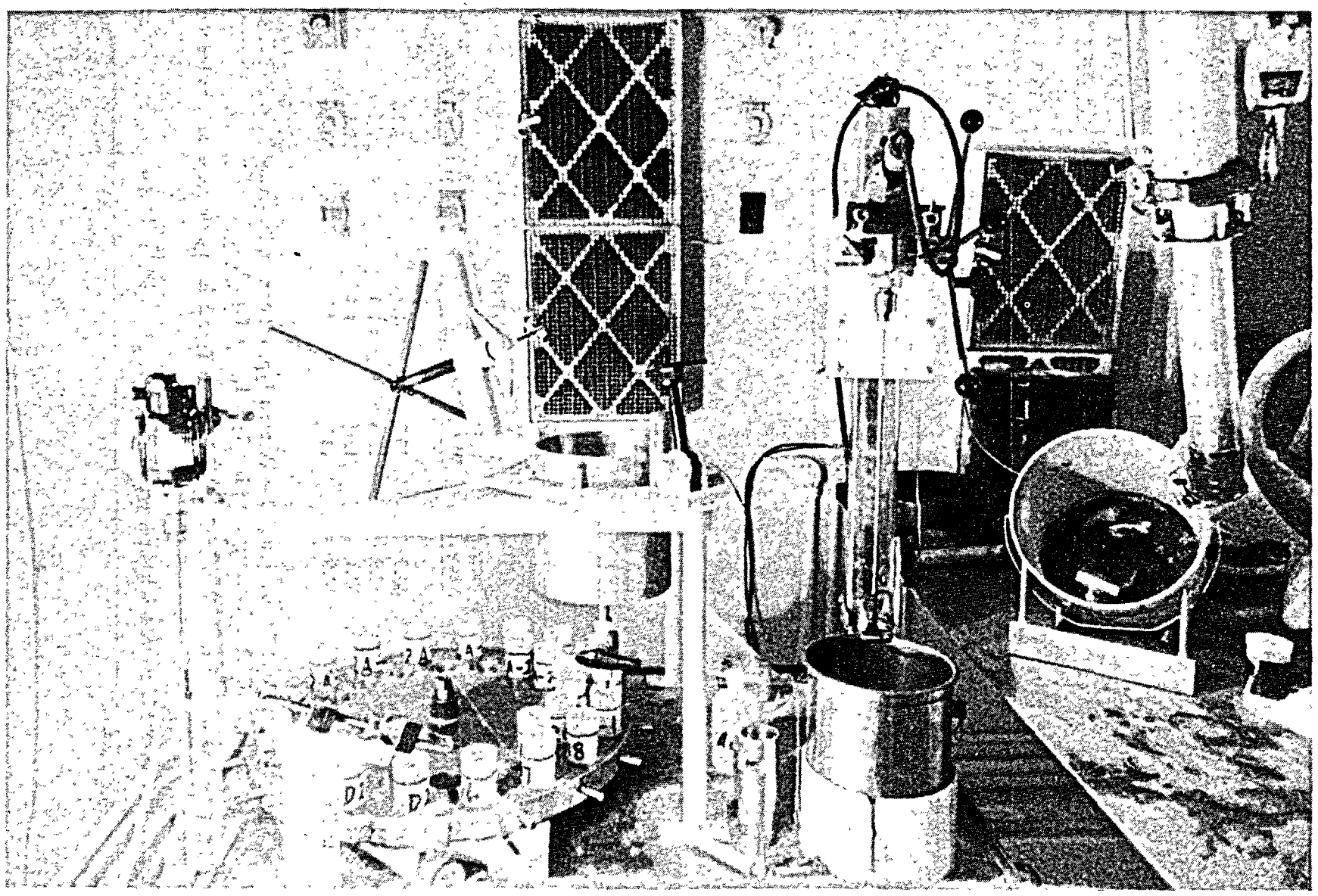


Figure 1. Set-up for hot cell solidification of EPICOR-II resin wastes in Portland cement and vinyl ester-styrene.

Table 1. Formulations for Portland cement waste form batches incorporating EPICOR-II wastes

		Formulation Weight Percentage				
Batch	Waste[a] Type	As-Received Waste	Added Water	Decanted[b] Waste Total	Portland Type I-II Cement	Additional Water
C1	1	15.6	8.5	24.1	62.7	13.2
C1A	1	15.6	8.5	24.1	62.7	13.2
C2A	2	16.8	7.1	24.0	62.5	13.5
C2B	2	16.5	7.1	23.6	61.4	15.1

a. Waste Type 1 is organic resin from prefilter PF-7. Waste Type 2 is organic resin with zeolite from prefilter PF-24.

b. Decanted waste total is the total weight percentage of as-received waste and added water.

Table 2. Formulations for vinyl ester-styrene waste form batches incorporating EPICOR-II wastes

		Formulation Weight Percentage			
Batch[a]	Waste[b] Type	As-Received Waste	Added Water	Decanted[c] Waste Total	Vinyl Ester-Styrene
D1	1	40.9	20.3	61.3	38.7
D1A	1	38.9	22.6	61.5	38.5
D2A	2	43.1	18.3	61.4	38.6
D2B	2	34.9	14.9	49.8	50.2

a. First digit refers to the solidification agent: C = Portland Type I-II cement, D = vinyl ester-styrene. Second digit refers to the EPICOR resin waste type: 1 = Type 1, 2 = Type 2.

b. Waste Type 1 is organic resin from prefilter PF-7. Waste Type 2 is organic resin with zeolite from prefilter PF-24.

c. Decanted waste total is the total weight percentage of as-received waste and added water.

Table 3. Density and packaging efficiency of EPICOR-II waste form batches

Batch[a]	Solidification Agent	Waste Type[b]	Density (g/cm^3)	Packaging Efficiency[c]
C1	Portland Type I-II cement	1	1.82	0.40
C1A	Portland Type I-II cement	1	1.78	0.39
C2A	Portland Type I-II cement	2	1.85	0.39
C2B	Portland Type I-II cement	2	1.77	0.37
D1	Vinyl ester-styrene	1	1.06	0.60
D1A	Vinyl ester-styrene	1	1.03	0.58
D2	Vinyl ester-styrene	2	1.06	0.57
D2A	Vinyl ester-styrene	2	1.03	0.45

a. First digit refers to solidification agent: C = Portland Type I-II cement, D = vinyl ester-styrene. Second digit refers to EPICOR-II resin type 1: 1 = Type 1, 2 = Type 2.

b. Waste Type 1 is organic resin from prefilter PF-7. Waste Type 2 is organic resin with zeolite from prefilter PF-24.

c. Packaging efficiency is the ratio of the decanted waste volume to the waste form volume. This calculation assumes that the density of Type 1 waste = 1.09 g/cm^3 and the density of Type 2 waste = 1.13 g/cm^3 (as determined for simulated EPICOR-II prefilter wastes).

Sixteen waste forms were examined 30 to 49 days after preparation specifically for determination of free liquids and compression testing of as-prepared specimens (described in the following section.) These included eight waste forms with Portland Type I-II cement as the binder and eight waste forms with vinyl ester-styrene as the binder. For both types of binders, four waste form specimens containing Type 1 waste and four specimens containing Type 2 waste were tested. Free liquid examinations were also performed on all EPICOR-II waste forms utilized in subsequent tests.

Compression Testing and Homogeneity

The Technical Position on Waste Form states:

> "Solidified waste specimens should have compressive strengths of at least 50 psi when tested in accordance with ASTM C 39."[8]

In the ASTM C 39 method, a compressive load is applied until the specimen fails and the maximum load to failure is recorded. The compressive strength is calculated by dividing the maximum load by the average cross-sectional area of the specimen. This method also stipulates requirements of the testing apparatus.

Sixteen specimens were selected for compressive strength testing. These included eight waste form specimens with Portland Type I-II cement as the binder and eight waste form specimens with vinyl ester-styrene as the binder. For both types of binders, four waste form specimens containing Type 1 waste and four specimens containing Type 2 waste were tested. Cure times (the time period between waste form specimen preparation and compression testing) were 30 to 49 days.

An Instron Model TTCLM1-4 Tension/Compression machine (Instron Corp., Canton, MA) with a compression cage attachment was used to determine the compressive strength of EPICOR-II waste forms. This machine conforms to the requirements of ASTM C 39 and was operated at a crosshead speed of approximately 0.05 in./min as specified. Waste form specimens were capped before testing using a sulfur base mortar (Cylcap-Humboldt Mfg. Co., Northridge, IL) in accordance with ASTM C 617 to assure that the specimen ends were perpendicular to the specimen axis.[9]

Note that other stability test protocols specified by the TP (thermal stability, immersion testing, radiation

stability, and biodegradation) also include compression testing. ASTM C 39 (with the ASTM C 617 capping method) was also used for compressive strength measurements associated with these other stability tests.

Special procedures were required during specimen handling and compressive strength measurements in order to avoid contamination and personnel exposure due to the high radionuclide content of these waste form specimens. Waste form specimens were removed from their preparation vials using long-handled tongs in a shielded fume hood. The top of each preparation vial was removed and a pointed plunger inserted through the bottom of the vial to push out the waste form. Capping prior to compression testing was also performed in a fume hood with long-handled implements. The specimen removal and capping arrangement is shown in Figure 2.

The compression tester was enclosed during use to minimize area contamination resulting from waste form specimen failure. Three methods of enclosure were used at different times in this work in order to demonstrate the level necessary for safe operations. One method used a plastic hood with an access port that enclosed the compression cage attachment only. This hood was ducted to a HEPA-filtered fan to minimize contamination during insertion of test specimens into the compression cage. After fracture, specimen fragments were brushed off the test platen and fell by gravity down the hood duct and into a shielded pail. The second enclosure method was similar in principal, but placed the entire compression tester into a plastic containment room (Figure 3). This arrangement required the technician inserting specimens for testing to enter the containment wearing anti-contamination clothing and a respirator. Figure 4 shows a capped vinyl ester-styrene waste form being placed into the

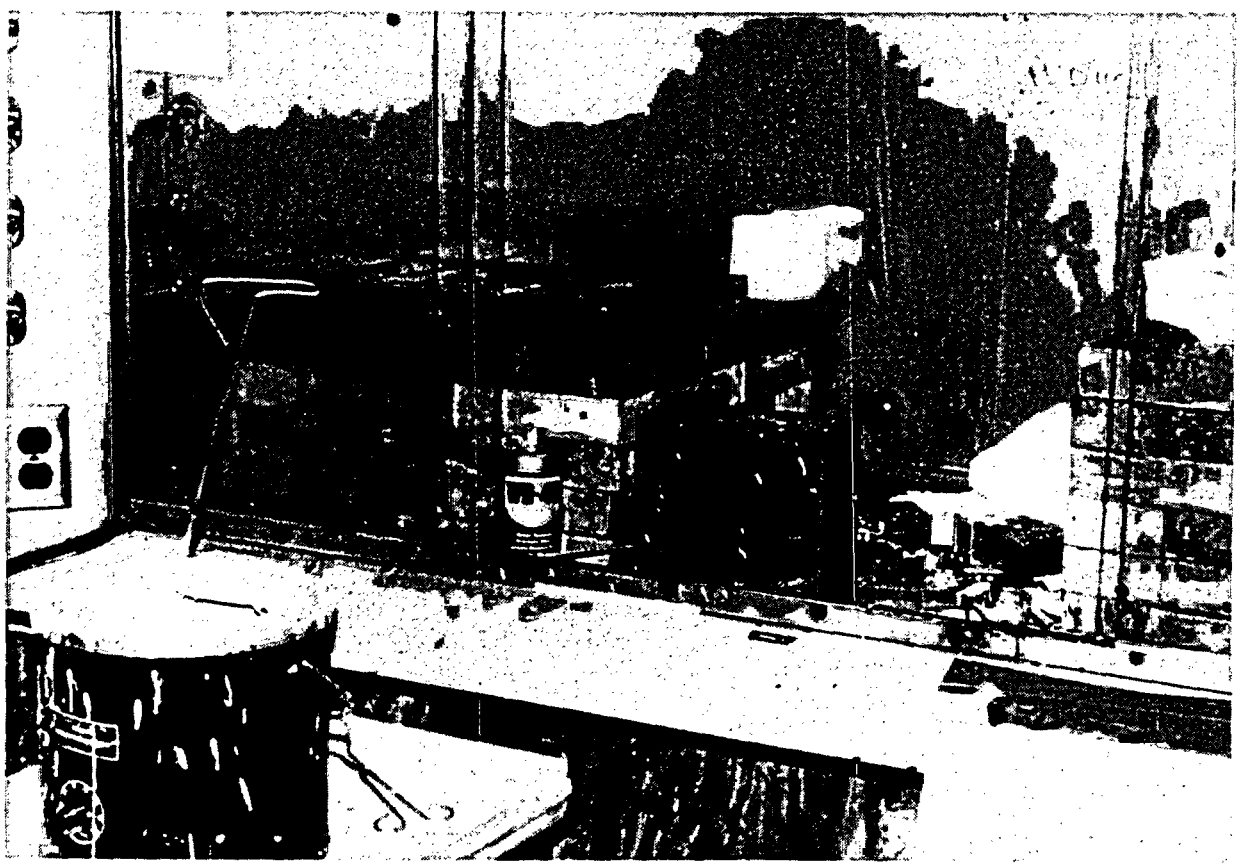


Figure 2. Shielded fume hood facility used for removal of EPICOR-II waste forms from specimen preparation vials and capping prior to compression testing.

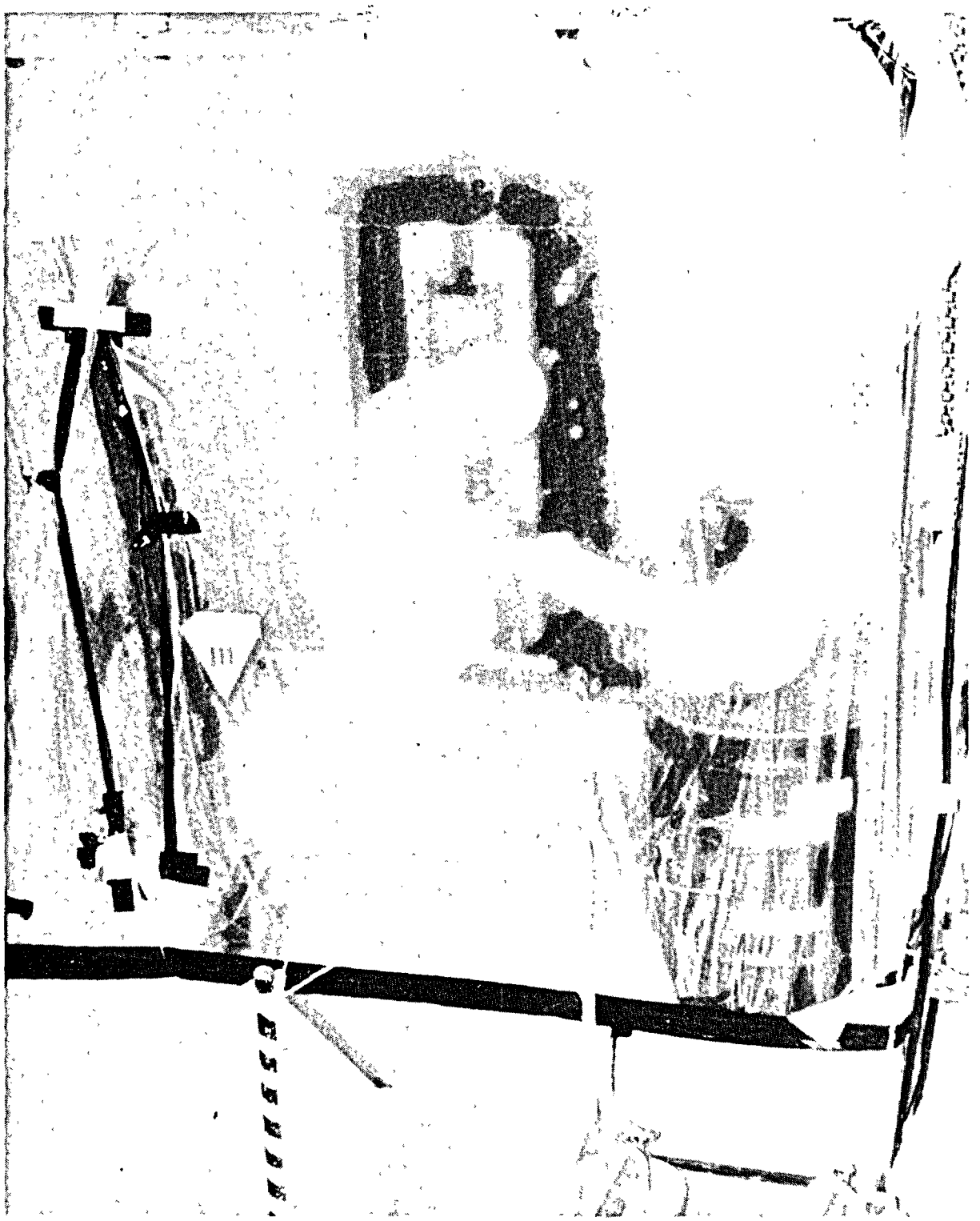


Figure 3. Compression testing of EPICOR-II waste forms in the temporary containment room.

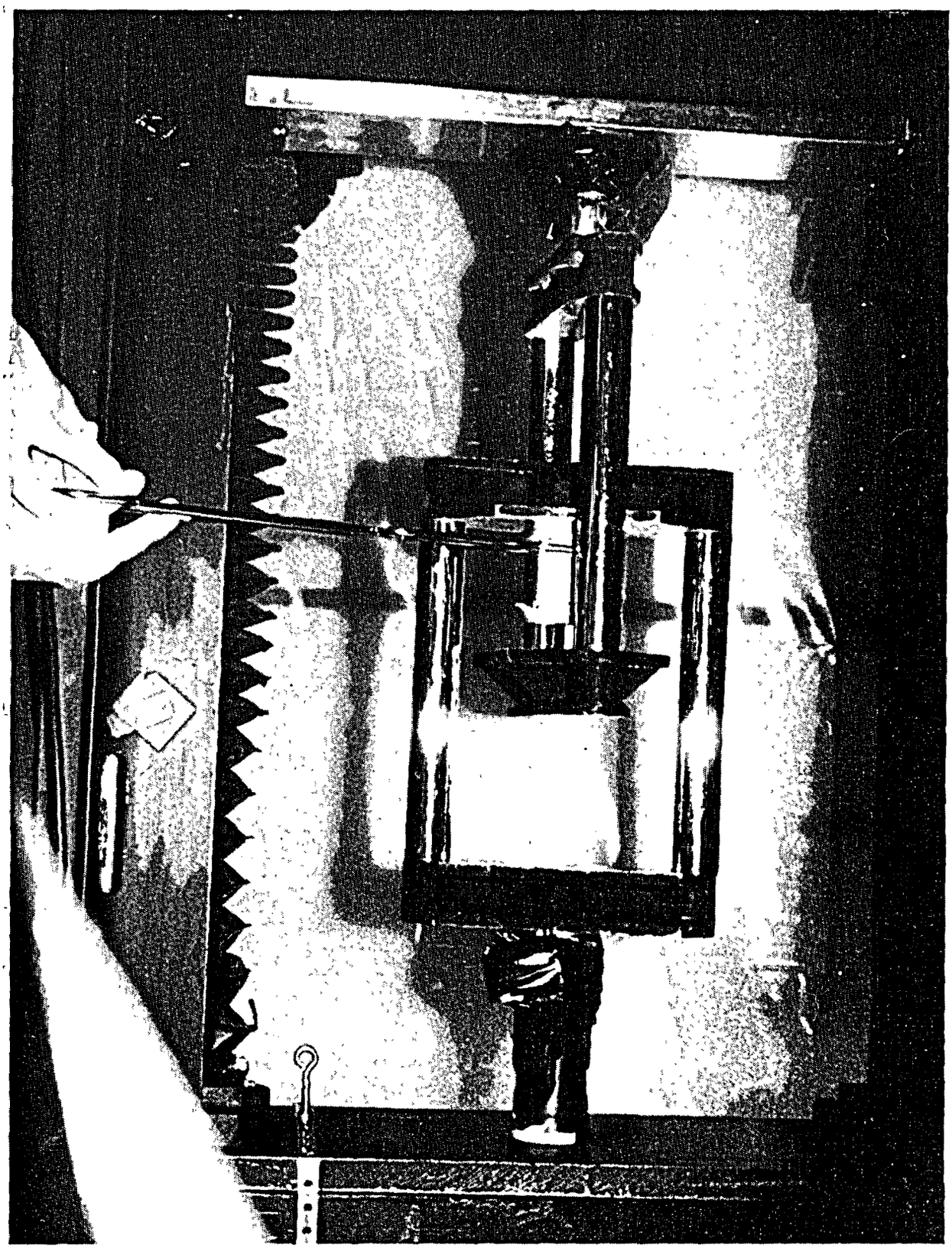


Figure 4. Compression tester frame with EPICOR-II vinyl ester-styrene waste form being placed into compression cage.

compression cage using the second enclosure method. The third method used consisted of placing the compression tester in a hot cell and conducting specimen insertion remotely. In all three methods, the compression tester control panel and data acquisition equipment were located in a clean area outside the containment.

The Technical Position on Waste Form also states:

> "Waste samples from full-scale specimens should be destructively analyzed to ensure that the product produced is homogeneous to the extent that all regions in the product can expect to have compressive strengths of at least 50 psi. Full-scale specimens may be fabricated using simulated nonradioactive products, but should be fabricated using actual solidification equipment."

Waste form fracture surfaces were examined after compression testing to verify waste form homogeneity and absence of free liquids consistent with the TP.

Thermal Stability

The Technical Position on Waste Form states:

> "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 B 553, Section 3.[10] Samples suitable for performing compressive strength tests in accordance with ASTM C 39 or ASTM D 1074 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 B 553. 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 C 39 or ASTM D 1074."

The ASTM B 553 method requires circulating air heating and cooling chambers (separate or combined) that can control the temperature to ±3°C of the set temperature and permit a consistent rate of heating and cooling of the test specimens. This method, in conjunction with the temperature limits set by the TP, defines one full thermal cycle by the following procedure:

1. Expose the waste forms for 1 h at 60°C.
2. Allow the waste forms to return to 20±3°C and maintain this temperature for 1 h.
3. Expose the waste forms for 1 h at -40°C.
4. Allow the waste forms to return to 20±3°C and maintain this temperature for 1 h.

After thirty thermal cycles, the waste specimens should have compressive strengths greater than 50 psi as tested using ASTM C 39.[8]

Sixteen waste form specimens were selected for thermal stability testing. They included eight waste form specimens with Portland Type I-II cement as the binder and eight waste form specimens with vinyl ester-styrene as the binder. For both types of binders, four waste form specimens containing Type 1 waste and four specimens containing Type 2 waste were tested. The specimen cure time was approximately 17 months. A Statham Model SD51-1 Environmental Chamber (Statham Instruments, Inc., Oxhard, CA), which conforms to the requirements of ASTM B 553, was used. Prior to testing, waste form specimens in their preparation vials were double-bagged in individual "zip-lock" type polyethlene bags and placed on a plastic tray. Then, the tray, containing eight specimens, was bagged. The extensive bagging was required to minimize the possible spread of contamination during thermal cycling, because the outside surfaces of the waste form preparation vials had been contaminated during a sorting operation in the hot cell. This procedure was repeated for the second set of eight specimens. A dummy cement specimen and a dummy vinyl ester-styrene specimen, each containing an axial thermocouple, were added to the second set.

The thermocouples in the dummy specimens were used to determine when the specimens had equilibrated to the environmental chamber temperature. [Note that equilibration to the test temperatures was used (as opposed to exposure to the test temperatures), representing the most restrictive interpretation of the test method.] Because of the extensive bagging of specimens and resultant detrimental effect on heat transfer, one complete thermal cycle required approximately 42 h. Specimens were exposed to 30 complete thermal cycles.

After thermal cycling, the waste form specimens were removed from the polyethylene bags and preparation vials for compressive strength testing per ASTM C 39 (described previously).

Leachability

The Technical Position on Waste Form specifies the following regarding waste form leachability:

> "Leach testing should be performed for a minimum of 90 days in accordance with the procedure in ANS 16.1.[11] Specimen sizes should be consistent with the samples prepared for the ASTM C 39 or ASTM D 1074 compressive strength tests. In

addition to the demineralized water test specified in ANS 16.1, additional testing using other leachants specified in ANS 16.1 should also be performed to confirm the solidification agents leach resistance in other leachant media. It is preferred that the synthesized sea water leachant also be tested. In addition, it is preferable that radioactive tracers be utilized in performing the leach tests. The leachability index, as calculated in accordance with ANS 16.1 should be greater than 6."

The ANS 16.1 method specifies a detailed test procedure and method of data analysis to provide meaningful and consistent results. Since many factors can interact to influence leach testing results, the procedure specifies a defined leachant, a set leachant renewal schedule, a fixed leachant temperature, and other test conditions. The following is a summary of this procedure.

The leachant specified is demineralized water with an electrical conductivity of less than 5 μmho/cm at 25°C and a total organic carbon content of less than 3 ppm. The leachant solution is sampled and entirely replaced at designated time intervals. The leachant temperature during testing must be in the range of 17.5 to 27.5°C. Leaching is conducted in a vessel which is constructed of unreactive material. The dimension of the leach vessel and the method of specimen support must expose essentially the entire external geometric surface area of the specimen to the leachant. The vessel must prevent excessive evaporation of the leachant (defined as >2% over 24 h). Sufficient leachant is used so that the ratio:

$$\frac{\text{leachant volume, cm}^3}{\text{specimen external geometric surface area, cm}^2} = 10 \pm 0.2 \text{ cm} \qquad (1)$$

is maintained during the leaching interval.

Before initiating the leach test, the specimen and its container are rinsed by immersion in demineralized water for 30 s. These rinses are collected for subsequent analysis. The specimen is then transferred into the leachant and the leach test begun. The leachate is removed and replaced with fresh leachant after each of ten incremental leach time intervals. These incremental leach times are: 2 h, 5 h, 17 h, 24 h, 24 h, 24 h, 24 h, 336 h, 672 h, and 1032 h, providing a total cumulative leach time of 90 ± 1 day. An aliquot of the leachate is taken at the end of each leach interval to determine, by a suitable method, the amounts of the species of interest present in the leachant volume.

Sixteen waste form specimens were selected for leachability testing. They included eight waste form specimens with Portland Type I-II cement as the binder and eight waste form specimens with vinyl ester-styrene as the binder. For both types of binders, four waste form specimens containing Type 1 waste and four specimens containing Type 2 waste were tested. The elapsed time between specimen preparation and initiation of leach testing was approximately 20 months.

Waste form specimens were weighed and measured after removal from their preparation vials. The specimens were then placed into individual teflon netting "baskets" which were suspended from leachant container lids. Two-liter, wide-mouthed polyethylene bottles with screw-top lids were used as leachant containers. Leachant volumes ranged from 1420 to 1590 mL, depending upon specimen dimensions, to provide a leachant volume-to-specimen external geometric surface area ratio (V_L/S) of 10 ± 0.2 cm. Both demineralized and synthetic sea water leachants were used. The synthetic sea water was prepared to the composition listed in Appendix D of the ANS 16.1 test method. Two specimens of each waste form type were leach-tested in each leachant. Leach specimens were rinsed by immersion in demineralized water for 30 s prior to initiation of the leach test. Leach-testing was then initiated. The leachant was removed and replaced after each of ten incremental leach time intervals as specified by ANS 16.1. The cumulative leach time was 89 days.

Because of the high radiation dose rate associated with handling these specimens, leach-testing was conducted in a HEPA-filtered fume hood behind a lead brick shield; and separate leaching bottles were used for each leach interval. The lead-brick-shielded leachability test arrangement is shown in Figure 5. This permitted leachate aliquots to be removed and processed in a low radiation environment. Each leachate was stirred and four 25-mL aliquots removed and placed in individual 30-mL polyethylene containers. One aliquot was used to measure leachate pH; one was used for gamma spectroscopy; and two aliquots were reserved for possible Sr-90 analysis and other potential uses. All aliquots, with the exception of aliquots taken for pH determination, were acidified with nitric acid. Analysis for gamma-emitting radionuclides was performed using a gamma spectrometer with a Ge(Li) detector. At the end of the leach test, each waste form specimen was placed into and stored in a "zip-lock" type polyethylene bag.

Figure 5. Lead-brick-shielded leachability test set-up in HEPA-filtered fume hood.

Immersion Testing

The Technical Position on Waste Form states:

> "Waste specimens should maintain a minimum compressive strength of 50 psi as tested using ASTM C 39 or ASTM D 1074, following immersion for a minimum period of 90 days. Immersion testing may be performed in conjunction with the leach testing."

Immersion testing utilized the sixteen waste form specimens that had undergone leach testing. Compressive strength testing was conducted by the method of ASTM C 39 as described previously. The elapsed time between specimen preparation and compression testing after immersion was 24 months. Compression testing was conducted approximately one month after completion of leachability testing.

Radiation Stability

The Technical Position on Waste Form states:

> "The specimens for each proposed waste stream formulation should remain stable after being exposed in a radiation field equivalent to the maximum level of exposure expected from the proposed wastes to be solidified. Specimens for each proposed waste stream formulation should be exposed to a minimum of 10^8 Rads in a gamma irradiator or equivalent. If the maximum level of exposure is expected to exceed 10^8 Rads, testing should be performed at the expected maximum accumulated dose. The irradiated specimens should have a minimum compressive strength of 50 psi following irradiation as tested in accordance with ASTM C 39..."

Sixteen waste forms were subjected to radiation stability testing. They included eight waste form specimens with Portland Type I-II cement as the binder and eight specimens with vinyl ester-styrene as the binder. For both binder types, four waste form specimens containing Type 1 waste and four specimens containing Type 2 waste were tested. The elapsed time between specimen fabrication and the initiation of radiation stability testing was approximately 27 months.

Radiation stability specimens were gamma-irradiated in the Advanced Test Reactor (ATR) Irradiation Facility located in the ATR spent fuel pool (Figure 6). This facility has three irradiation tubes; two of the tubes were used in these radiation stability experiments. Each irradiation tube is 247 in. long and has a 5.2-in. ID. An assemblage of spent ATR fuel is configured around the base of each irradiation tube to provide a controlled gamma flux in the bottom 4 ft of the tube. The resulting gamma flux is approximately constant (within 10%) at any given time over a length of approximately 20 in. near the bottom of the tube. The bottom of each irradiation tube is sealed. The interior of the irradiation tubes are not filled with water; they are "dry" tubes in which specimens are irradiated in air. Due to the low temperature of the spent fuel pool water, there is some condensation water on the interior walls of the irradiation tubes.

Waste forms were irradiated in their low-density polyethylene preparation vials (Nalge Division of Sybron Corp., Rochester, NY). All waste form specimens with the same binder (either Portland cement or vinyl ester-styrene) were stacked on a carousel which was placed into a stainless steel basket. This basket was then lowered into the desired position in the irradiation tube. Each basket contained four levels of three specimens each (twelve total[a]) stacked on the carousel. Since the specimen vial height was 4 in., the length of the specimen column in the basket was 16 in. The stacking of the specimens in each basket was carefully documented as an alternate means of post-irradiation specimen identification.

Irradiation tube dosimetry measurements were made before waste form irradiation to determine the dose rate as a function of position, decay rate, and field uniformity. The specimen basket was positioned in the irradiation tube and the irradiation time selected based upon these data. The loaded specimen baskets were lowered into position and their positions fixed by a support chain (Figure 7). Both support chains broke during irradiation, and the specimen baskets fell to the bottom of the irradiation tubes. The vinyl ester-styrene specimen basket support chain apparently broke during insertion into the irradiation tube due to stress concentration at a kink in the chain. This was determined by examination of oxidation on the chain and its fracture surface. The Portland cement specimen basket support chain was sheared by an appendage of the fuel pool canal bridge approximately one week after basket insertion. This basket was subsequently repositioned in the irradiation tube. Despite chain breakage, the tube dosimetry data allowed calculation of the waste form gamma doses.

After irradiation for the desired time, the specimen baskets were removed from the gamma field, withdrawn from the irradiation tubes, and placed into a lead cave. The waste form specimens were then removed from the baskets, identified by position in the baskets, and placed into a shielded cask for transport to the compression testing location. Figure 8 shows a waste form being removed from the specimen basket carousel after irradiation.

Compression testing of gamma-irradiated waste forms was conducted according to the method of ASTM C 39, as described previously.

Biodegradation Testing

Biodegradation testing is an on-going effort in this program. It will be the subject of a subsequent report after testing is completed.

a. Four additional waste forms of each binder type, half with Type 1 waste and half containing Type 2 waste, were irradiated for possible leaching after irradiation testing. These eight total specimens were returned to storage after irradiation.

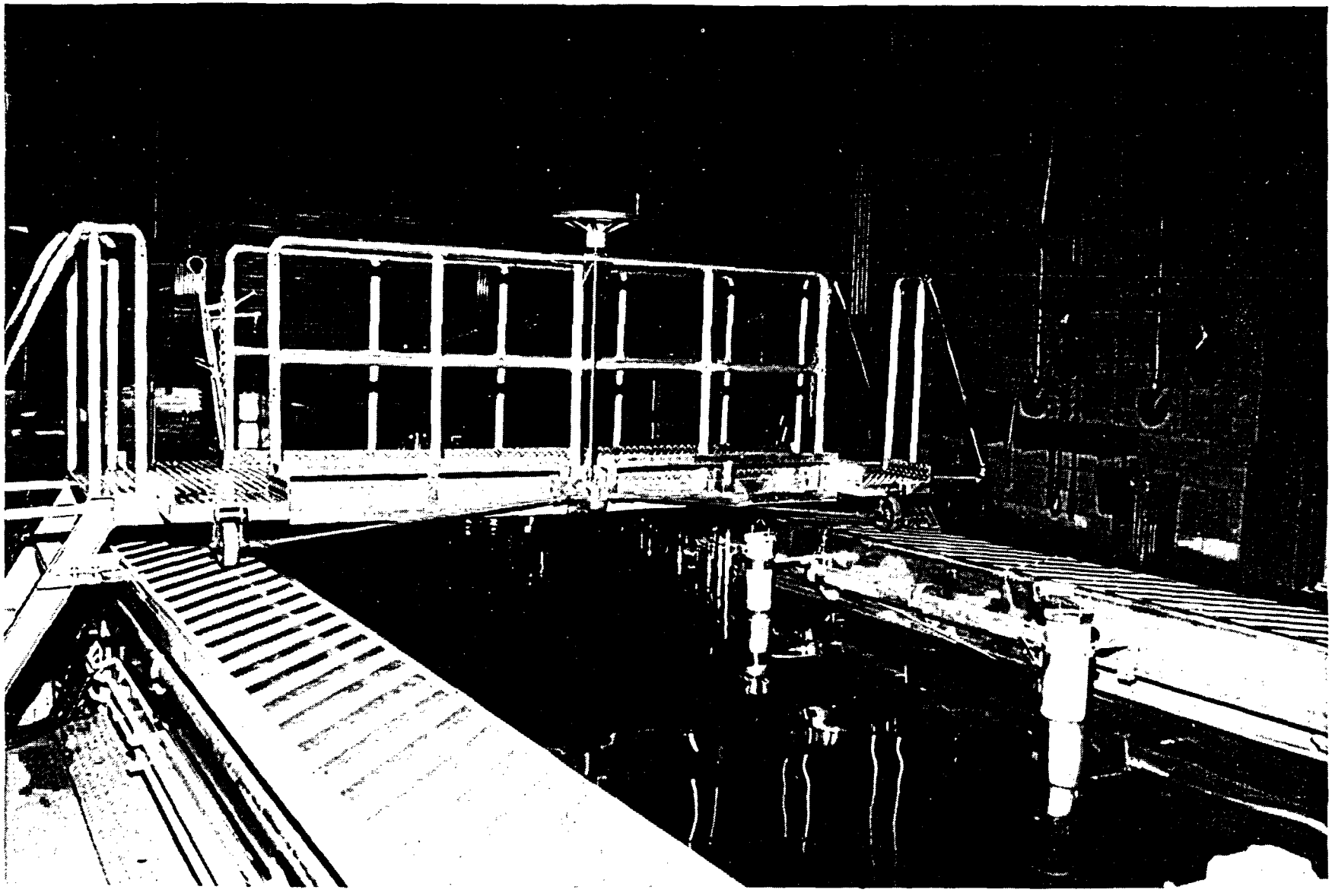


Figure 6. Gamma irradiation facility located in the Advanced Test Reactor (ATR) spent fuel pool.

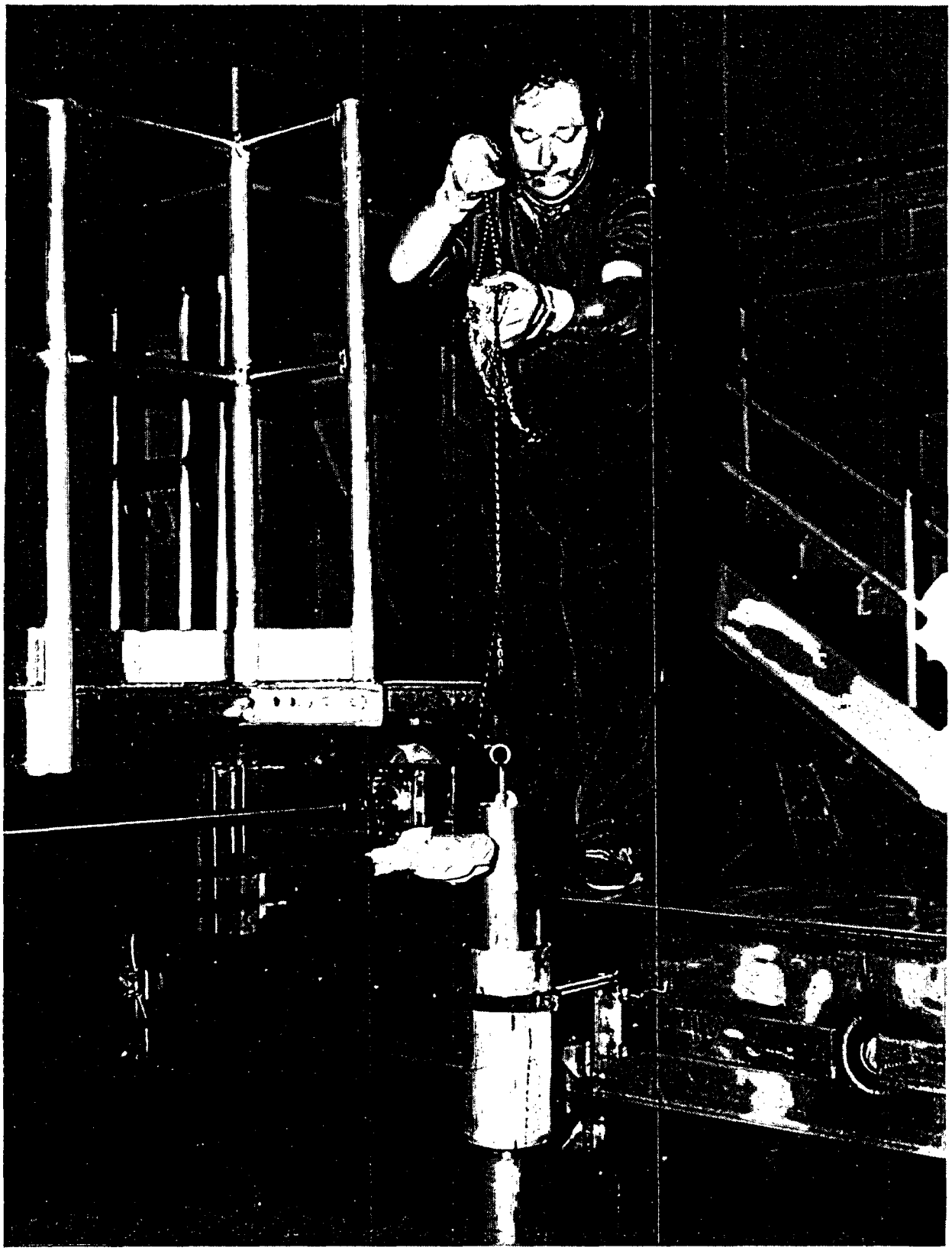


Figure 7. EPICOR-II specimen test basket being lowered into its irradiation tube in the ATR gamma irradiation facility.

Figure 8. Removing irradiated EPICOR-II waste forms from specimen basket carousel in shielded cave.

RESULTS AND DISCUSSION

Free Liquids

The TP specifies that "Waste specimens should have less than 0.5 percent by volume of the waste specimen as free liquids as measured using the method described in ANS 55.1." This corresponds to less than 0.68 cm^3 free liquid for the nominal specimen dimensions. Since the method described in ANS 55.1 is meant for application to full-scale waste packages (55-gal drums or larger), the observation of any liquid during removal of the waste form from its preparation vial was deemed sufficient to violate the free liquid requirement. However, no free liquid was observed for any of the Portland cement or vinyl ester-styrene waste forms examined. Since there was no observable free liquids, it was not possible to determine the free liquid pH.

Although the free liquid determination method in ANS 55.1 applies to full-scale waste forms, procedure modifications made in this work for small individual specimens met the intent of the TP. Testing suggests that the ANS 55.1 free liquid determination procedure is adequate for determining the compliance of full-scale waste forms with 10 CFR 61. Testing and test results indicated that administrative procedures can be successfully implemented with this procedure to minimize contamination and personnel exposure.

Compression Strength and Homogeneity

Compressive strength data for the sixteen waste form specimens tested (not including compressive testing conducted as part of other stability test) are shown in Table 4.

When the two Portland cement specimens with insufficiently parallel caps are excluded, average compressive strengths for Portland cement specimens and vinyl ester-styrene specimens incorporating the same waste type are approximately equal. Waste forms containing Type 2 waste (organic resin with zeolite) exhibited a higher average compressive strength (3,600 psi) than those prepared with Type 1 waste (2,900 psi). The inorganic zeolite in the Type 2 waste acts as an effective aggregate in strengthening the waste form specimens. Note that the D2A specimens have a higher average compressive strength (3,700 psi) than the D2 specimens (3,460 psi). This is due to the higher binder content in the D2A batch formulation (see Table 2). The compressive strengths of all waste forms tested greatly exceeded the 50-psi minimum strength required by the TP. The high measured compressive strengths and visual observations of specimens after failure indicated that the waste forms were sufficiently homogeneous on a macroscopic level that all regions within them had compressive strengths in excess of 50 psi. Small quantities of free liquid were observed on vinyl ester-styrene waste form fracture surfaces. This liquid results from fracture surfaces intersecting water microcells from the emulsion and water squeezed out of such microcells during compression.

Testing indicates that the C 39 compressive strength testing method (and presumably, by extension, the D 1074 method) is appropriate for verifying compliance with the requirements of the TP. Administrative procedures implemented with this procedure were successful in minimizing contamination and personnel exposure. These administrative procedures, (i.e., use of long tools, working behind a shielding wall, and minimization of handling time) did result in some low strength values due to fabrication of insufficiently parallel caps. However, this would only be problematic in regard to compliance with the TP when waste forms have low compressive strengths (near the 50-psi requirement) and/or when very small numbers of specimens are tested.

Thermal Stability

Compression test data for the thermally cycled waste form specimens are shown in Table 5. The thermal stability specimens exhibited compressive strengths of 2,600 to 6,400 psi, far in excess of the minimum 50 psi required by the TP.

The compression strengths of Portland cement specimens were higher than the vinyl ester-styrene specimens. Specimens containing Type 2 resin waste had higher compression strengths than those containing Type 1 resin. As mentioned previously, inorganic zeolite in the Type 2 waste acts as an effective aggregate in strengthening the waste form specimens. The compression strengths of the thermally cycled Portland cement waste form specimens were significantly higher (by ~60%) than the corresponding as-prepared specimens (Table 4). An increase in compressive strength due to additional cement hydration is expected due to the long cure time before compression testing. However, the resultant strength increase appears to be too large to be accounted for by this explanation. Also contributing are the substantial time at high temperature during thermal cycling (approximately 30 days above ambient temperature) and possible variation of

Table 4. Compressive strengths of EPICOR-II resin waste forms

Specimen Designation[a]	Cure Time (days)		Compressive Strength (psi)
C1-1	49		2,720
C1-2	49		2,590
C1-3	49		3,480
C1-4	49		1,630[b]
		Average[c]	2,930
		Std. dev.[c]	480
C2A-1	35		2,260[b]
C2A-2	35		2,900
C2A-3	35		3,630
C2B-29	34		4,330
		Average[c]	3,620
		Std. dev.[c]	720
D1A-17	43		2,960
C1A-18	43		3,060
D1A-19	43		2,700
D1A-20	43		2,870
		Average	2,900
		Std. dev.	150
D2-2B	38		3,310
D2-29	38		3,600
D2A-1	30		3,770
D2A-2	30		3,620
		Average	3,580
		Std. dev.	190

a. First digit refers to the solidification agent: C = Portland Type I-II cement, D = vinyl ester-styrene. Second digit refers to the EPICOR resin waste type: 1 = Type 1, 2 = Type 2. Number after the hyphen refers to the specimen number within a batch.

b. Caps were not sufficiently parallel for proper seating of the compression tester. Compressive strengths measured were lower than the actual strengths.

c. These calculations do not include data for specimens with insufficiently parallel caps.

Table 5. Compressive strengths of thermally cycled EPICOR-II resin waste forms

Specimen Designation[a]	Cure Time (months)		Compressive Strength (psi)
C1A-3	17		4,820
C1A-5	17		4,640
C1A-6	17		4,750
C1A-7	17		2,210[b]
		Average[c]	4,740
		Std. dev.[c]	90
C2A-18	17		5,340
C2A-19	17		5,250
C2A-20	17		6,420
C2A-21	17		2,500[b]
		Average[c]	5,670
		Std. dev.[c]	650
D1A-25	17		2,790
D1A-26	17		2,640
D1A-27	17		2,430
D1A-28	17		3,200
		Average	2,770
		Std. dev.	330
D2A-8	17		4,130
D2A-9	17		4,040
D2A-10	17		3,970
D2A-11	17		4,110
		Average	4,060
		Std. dev.	70

a. First digit refers to solidification agent: C = Portland Type I-II cement, D = vinyl ester-styrene. Second digit refers to EPICOR-II resin type 1: 1 = Type 1, 2 = Type 2. Number after the hyphen refers to the specimen number within a batch.

b. Caps not sufficiently parallel for proper seating in the compression tester. Measured compressive strengths are lower than actual strengths.

c. These calculations do not include data for specimens with insufficiently parallel caps.

individual specimen compositions from the batch composition.

The compression strengths of the thermally cycled vinyl ester-styrene specimens were 4% lower and 13% higher than the strength of as-prepared specimens for the Type 1 and Type 2 resins, respectively. The average compressive strength difference between as-prepared and thermally cycled vinyl ester-styrene waste forms containing Type 1 resin is small relative to the standard deviation of the measurements. The average compression strength difference between as-prepared and thermally cycled waste forms containing Type 2 resin is larger than the standard deviation of the measurements and may suggest increased cross-linking of the binder. Free liquid in excess of 0.5% by volume has been reported for vinyl ester-styrene waste forms subjected to thermal stability testing.[12] No free liquid was observed during thermal stability testing of either vinyl ester-styrene or Portland cement waste forms incorporating EPICOR-II resin waste.

The general applicability of the ASTM B 533 test procedure to verify compliance with the requirements of the TP was demonstrated. It was also shown that administrative procedures can be employed successfully to minimize contamination and personnel exposure. However, minor modifications to the TP are required to assure that evaporative water loss from specimens does not occur before or during thermal cycling and that times at temperature are sufficient to cause specimen temperatures to cycle between desired limits.

The lack of sufficient thermal stability of low-level waste forms for environmental temperature conditions usually results from freezing and thawing of chemically uncombined water in the waste form. Consequently, degradation results from the expansion of water upon freezing. As a result, thermal stability testing should additionally require that the specimen be containerized immediately after preparation and through thermal cycling to prevent evaporative water loss.

Similarly, testing should require that the temperature of the *specimens* cycle from below freezing and then above thawing (not just be exposed to required temperatures). The use of multiple bagging to prevent spread of contamination during testing can minimize specimen temperature changes due to insulating effects. For this reason, the testing reported herein used thermocouples to monitor specimen centerline temperatures. The instrumented specimens were maintained at required temperatures for sufficient times to allow their centerlines to attain thermal equilibrium. The test temperatures were maintained for a minimum of 1 h after thermal equilibrium was achieved. Maintaining test temperatures for more than 1 h was not detrimental to the test results and was necessary because the required increased thermal cycle time conflicted with normal working hours. (A programmable controller was not available.)

Leachability

Leach data calculations were performed in the manner detailed in the ANS 16.1 leach test method standard. This method allows calculation of a leachability index (L) for a given radionuclide, i. This index is defined by

$$L_i = \frac{1}{10} \sum_{n=1}^{10} [\log (\beta/D_i)]_n \qquad (2)$$

where

β = 1 cm^2/s (defined constant), and

D_i = effective diffusivity for leach interval n, cm^2/s.

The effective diffusivity, D_i, is calculated from the leach test data in the following manner:

$$D_i = \pi \left[\frac{a_n/A_o}{(\Delta t)_n}\right]^2 (V/S)^2 T \qquad (3)$$

where

a_n/A_o = fraction activity release in leach interval n,

V = specimen volume, cm^3

S = specimen external geometric surface area, cm^2

Δt_n = duration of the n-th leach interval, and

T = $[1/2(t_n^{1/2} + t_{n-1}^{1/2})]^2$, mean time of leach interval n, s.

Table 6 lists the leachant sampling (and replacement) intervals specified by the ANS 16.1 method. The ANS/16.1 procedure does not specify which radionuclides are used to calculate the leachability index.

The relationship presented in Equation (3) represents the solution of the mass transport equation for diffusion from a semi-infinite medium. The ANS 16.1 procedure applies this relationship when the cumulative fraction activity release from the waste form is less than 20%. A shape-dependent solution based upon diffusion from a finite medium is used in the ANS 16.1 leach test method standard when the cumulative

Table 6. ANS 16.1 test method leachant sampling intervals

Sampling Interval, n	Incremental Leach Time, Δt_n (h)	Cumulative Leach Time, t_n (h)	Cumulative Leach Time, t_n (s)	Mean Time of Leach[a] Interval, T (s)
1	2	2	7.20E+03	1.80E+03
2	5	7	2.52E+04	1.48E+04
3	17	24	8.64E+04	5.12E+04
4	24	48	1.73E+05	1.26E+05
5	24	72	2.59E+05	2.14E+05
6	24	96	3.46E+05	3.01E+05
7	24	120	4.32E+05	3.88E+05
8	336[b]	456	1.64E+06	9.39E+05
9	672[b]	1128	4.06E+06	2.72E+06
10	1008[b]	2136	7.69E+06	5.73E+06

a. $T = [1/2\ (t_n^{1/2} + t_{n-1}^{1/2})]^2$.

b. The incremental leach times for these intervals may vary by ±1 day.

fraction release exceeds 20%. All of the EPICOR-II resin waste forms tested had cumulative fraction releases of less than 20%.

The ANS 16.1 method also determines the 99.9% confidence range (c) and correlation coefficient (r) for the calculated leachability index. The 99.9% confidence range is defined by:

$$C = L_i \pm 4.781\ \sigma_L\ n^{-1/2} = L_i \pm 1.51\ \sigma_L. \qquad (4)$$

where

C = the 99.9% confidence range of L_i, dimensionless,

L_i = the mean of the ten values of L_n [i.e., Eq. (2)], dimensionless,

L_n = the value of L at the end of the n-th leaching interval, dimensionless, and

$$\sigma_L = \frac{1}{3}\left[\sum_1^{10} (L_n - L_i)^2\right]^{1/2}, \qquad (5)$$

the standard deviation of the ten values of L_n, dimensionless.

The correlation coefficient between L and t is defined by the relationship:

$$r = \frac{\sigma_{Lt}}{\sigma_L\ \sigma_t} \qquad (6)$$

where

r = the correlation coefficient, dimensionless,

$$\sigma_{Lt} = \frac{1}{9}\sum_1^{10} (L_n - L_i)\ (t_n - t_m), \qquad (7)$$

= the covariance of the ten sets of L and t, s,

t_n = the value of t at the end of the n-th leaching interval, s,

$$t_m = \frac{1}{10}\sum_1^{10} t_n \qquad (8)$$

= the mean of the ten values of t_n, s, and

$$\sigma_t = \frac{1}{3}\left[\sum_1^{10} (t_n - t_m)^2\right]^{1/2}, \qquad (9)$$

= the standard deviation of the ten values of t_n, s.

The correlation coefficient varies from -1 to +1. The sign indicates whether L_n is tending to increase (+r) or to decrease (−r) as t_n increases. Calculation of the 99% confidence range, C, of L_i and the correlation coefficient, r, can be used to identify intermediate-term deviations from diffusion as the rate-determining mechanism of radionuclide release.

The three most important sources of error associated with the leach testing conducted can be attributed to (a) the manner in which the test method is implemented (different operators, temperature control, measurement of leachant volumes, etc.), (b) the determination of the specimen activity content, and (c) the analysis of the leachate solutions.

Errors associated with implementation of the leach test method are difficult to quantify, because the effect of variation of most of the experimental variables on leachability is not known. For example, the change in the leachability index as measured by this test method resulting from variation in leachant volume or temperature has not been determined. However, the development of the ANS 16.1 leach test method considered "acceptable" variation in experimental conditions; and the test method was conducted within the acceptable ranges of these variables. Since the ANS 16.1 procedure uses incremental release data rather than cumulative data (which couples data points), an error in testing (or analysis) is not propagated throughout the test. Operator error was minimized by use of a single operator for specific tasks for the duration of leach testing.

Determination of the specimen activity content is probably the major source of error in the experiment. The specimen activity content in each waste form specimen was calculated from the measured average activity content of dry homogenized EPICOR-II resin wastes, the average water content of the as-received EPICOR-II wastes, and the batch solidification formulations (Tables 1 and 2).[1] Activity content and water content for the as-received EPICOR-II wastes are listed in Table 7. These analyses were performed using two small aliquots (<0.3 g dried resin for activity content and ~5 g as-received resin for water content) of resin from each prefilter (after resin homogenization before the solidification operation described in Reference 1). Only small resin aliquots could be used for activity content measurement due to their high radionuclide loading. This results in only a small counting error associated with each resin aliquot (since the number of counts is high relative to background) but potentially a larger error in the assumed as-supplied resin content due to the small size and number of aliquots counted. However, the standard deviation calculated for the average activity content for each waste type is relatively small. In the worst case, one

Table 7. Activity and water content of EPICOR-II resin wastes

Prefilter	Activity Content[a,b] (Ci/g (dry resin) ± 1 σ) Cs-134	Cs-137	Sr-90	Water Content (wt %)
PF-7	7.75E-05 ± 1.47E-06	1.10E-03 ± 5.28E-06	6.41E-05 ± 3.49E-06	36.1
	7.71E-05 ± 1.38E-06	1.24E-03 ± 5.59E-06	7.43E-05 ± 4.05E-06	37.1
PF-7 Average	7.73E-05 ± 2.82E-07	1.17E-03 ± 9.90E-05	6.92E-05 ± 7.21E-06	36.6
PF-24	2.89E-04 ± 5.48E-06	4.77E-03 ± 2.23E-05	1.13E-05 ± 1.04E-06	42.9
	3.71E-04 ± 6.97E-06	5.20E-03 ± 2.34E-05	1.22E-05 ± 1.10E-06	40.4
PF-24 Average	3.30E-04 ± 5.80E-05	4.99E-03 ± 3.04E-04	1.18E-05 ± 6.36E-07	41.7

a. Cs-134 and Cs-137 as of 9/20/83; Sr-90 as of 10/25/83.

b. Gamma spectroscopy revealed no gamma-emitting radionuclides other than Cs-134 and Cs-137.

standard deviation is 17.6% of the average calculated activity content (Cs-134 in prefilter PF-24); it is only 0.4% in the best case (Cs-134 in prefilter PF-7). While these data suggest that the prefilter wastes were reasonably homogeneous prior to solidification, the waste form activity content calculations also assume that EPICOR-II wastes were uniformly distributed in each formulation batch. This assumption is probably good for Portland cement waste forms, but was not for vinyl ester-styrene specimens. Ion exchange resin settling in the batch formulation was noted during solidification.[1] However, since the amount of EPICOR-II resin waste in each formulation was easily observed after preparation vial filling (and before promoter addition), only individual waste specimens containing a representative amount of prefilter waste were selected for leach testing. The visual observations described above were supplemented by contact gamma dose measurements of solidified waste forms to verify that those selected for leaching had similar activity contents and activity contents representative of the batch activity content.

Activity content in leachate aliquots was sufficiently high for good counting statistics. The standard duration associated with the measured leachate activity content was typically only 2 to 3% of the measured activity. This is somewhat larger than the error associated with pipeting a known volume of leachate for analysis.

While the assumed specimen activity content probably accounts for the largest source of error in leach testing, the probable difference between actual and calculated activity contents is not large. While this and other errors associated with testing and analysis may have statistical significance relative to the calculated leachability index, they are small and have no practical significance in interpreting the results. [The standard deviation is "small" relative to the measurement.]

Leach test data are shown in Tables 8 and 9 for Cs-134 and Cs-137 releases, respectively. Although Sr-90 leachability was not determined, strontium release is typically lower than that of the cesium radionuclides from Portland cement waste forms and about equal for vinyl ester-styrene waste forms.[5,13,14] Each of the leach specimens exhibited a leachability index greater than six, as recommended by the TP. Since the leachability index is a logarithmic function of the fractional activity release, the measured releases were far below the acceptable amounts for the specimen geometry used.

The leachability of Portland cement waste forms was generally higher than comparable vinyl ester-styrene waste forms in the same leachant. This is indicated by lower leachability indices for the cement waste forms. Both Portland cement and vinyl ester-styrene waste forms containing Type 1 waste exhibited higher leachability than those containing Type 2 waste. Leaching in sea water resulted in higher leachability than when demineralized water was employed as the leachant (much higher for vinyl ester-styrene waste forms). Cesium-137 and Cs-134 leach indices, confidence ranges, and correlation coefficients were comparable for replicate waste forms. The relative leaching behavior of Portland cement and vinyl ester-styrene waste forms is consistent with other studies in the literature.[4,5,13,14]

Significantly lower leach indices (5.8 to 9.3) for Cs-134 and -137 were determined in a leaching investigation of full-scale commercial waste forms.[15] While that study included Portland cement and vinyl ester-styrene waste forms, their compositions were significantly different from those discussed here, since they resulted from the solidification of different waste streams and utilized higher waste loadings representative of commercial practice. The closest waste stream (in composition) to that of EPICOR-II waste was a mixture of sodium sulfate concentrate with ion exchange resins. This waste was solidified with Portland Type III cement and exhibited a leachability index of 6.8 for Cs-134 and 6.7 for Cs-137 in demineralized water. The highest leachability index determined (i.e., lowest leachability) was for a vinyl ester-styrene waste form containing sodium sulfate concentrate. A lower leachability index for both Cs-134 and -137 in demineralized water (6.8) for a Portland Type III cement waste form incorporating sodium sulfate concentrate waste agrees with the general observation in the current work that the leachability of vinyl ester-styrene waste forms is generally lower than that of Portland cement waste forms. The higher leachabilities (lower leachability indices) determined from the BNL study,[15] as compared to the current work, is attributed to the solidification of different waste streams and higher waste loadings rather than any size effects. It should be noted that the BNL leach procedure used a more frequent leachant sampling/replacement frequency than suggested by ANS 16.1. The data analysis also employed a longer cumulative leach time (99 days) and an increased number of data points (18).

Although the EPICOR-II resin waste forms had high associated dose rates and large activity contents, administrative procedures were successful in minimizing contamination and personnel exposure.

Leach-testing and results suggest that the experimental procedure specified in the ANS 16.1 leach test

Table 8. Cesium-134 leachability from EPICOR-II resin waste forms

Specimen[a] Designation	Leachant[b]	Initial Activity Content (μCi)	Cumulative Activity Release (%)	Leachibility: Index[c] (L)	Leachibility: Confidence Range (C)[d]	Leachibility: Correlation Coefficient (r)[e]
C1-7	DI	1.17 E+03	4.0	10.3	9.1 - 10.4	-0.40
C1-8	DI	1.13 E+03	3.4	10.2	9.4 - 11.1	-0.59
C1-10	SW	1.16 E+03	7.6	9.6	9.0 - 10.2	-0.74
C1-11	SW	1.14 E+03	7.5	9.6	9.0 - 10.1	-0.71
C2B-4	DI	4.72 E+03	1.9	10.6	10.1 - 11.0	-0.21
C2B-5	DI	4.91 E+03	1.7	10.6	10.1 - 11.1	0.01
C2B-6	SW	4.73 E+03	2.4	10.3	10.1 - 10.6	-0.30
C2B-7	SW	4.56 E+03	2.0	10.5	10.3 - 10.8	-0.58
D1-3	DI	1.79 E+03	0.22	12.2	11.0 - 13.3	0.76
D1-4	DI	1.80 E+03	0.13	12.5	11.6 - 13.4	0.84
D1-5	SW	1.74 E+03	5.5	9.4	8.4 - 10.5	0.51
D1-6	SW	1.81 E+03	5.4	9.4	8.5 - 10.2	0.59
D2-17	DI	6.86 E+03	0.031	13.9	13.4 - 14.3	0.76
D2-18	DI	7.00 E+03	0.026	14.1	13.7 - 14.5	0.35
D2-19	SW	7.03 E+03	0.91	11.1	10.6 - 11.5	0.26
D2-20	SW	6.78 E+03	1.4	10.7	10.4 - 11.0	0.26

a. First digit refers to solidification agent: C = Portland Type I-II cement, D = vinyl ester-styrene. Second digit refers to EPICOR-II resin type: 1 = Type 1, 2 = Type 2. Number after hyphen refers to the specimen number within a batch.

b. DI = demineralized water, SW = synthetic sea water.

c. $L = \sum_{n=1}^{10} [\log (\beta/D_i)]_n/10.$

d. C = 99.9% confidence range of L.

e. $r = \frac{\sigma_{Lt}}{\sigma_L \sigma_t}$

Table 9. Cesium-137 leachability from EPICOR-II resin waste forms

Specimen[a] Designation	Leachant[b]	Initial Activity Content (μCi)	Cumulative Activity Release (%)	Leachibility: Index[c] (L)	Leachibility: Confidence Range (C)[d]	Leachibility: Correlation Coefficient (r)[e]
C1-7	DI	2.79 E+04	4.8	10.3	9.1 - 11.4	-0.40
C1-8	DI	2.70 E+04	4.7	10.3	9.1 - 11.4	-0.40
C1-10	SW	2.76 E+04	9.0	9.5	8.9 - 10.1	-0.74
C1-11	SW	2.73 E+04	9.1	9.5	8.9 - 10.1	-0.72
C2B-4	DI	1.12 E+05	2.2	10.4	10.0 - 10.8	-0.25
C2B-5	DI	1.17 E+05	2.3	10.4	10.0 - 10.8	-0.24
C2B-6	SW	1.13 E+05	2.9	10.2	10.0 - 10.5	-0.09
C2B-7	SW	1.08 E+05	2.4	10.4	10.2 - 10.6	-0.20
D1-3	DI	4.27 E+04	0.26	12.0	10.9 - 13.2	0.76
D1-4	DI	4.29 E+04	0.15	12.4	11.4 - 13.3	0.84
D1-5	SW	4.15 E+04	6.5	9.3	8.2 - 10.3	0.50
D1-6	SW	4.33 E+04	6.4	9.2	8.4 - 10.1	0.58
D2-17	DI	1.69 E+05	0.037	13.7	13.3 - 14.2	0.73
D2-18	DI	1.72 E+05	0.031	13.9	13.6 - 14.3	0.62
D2-19	SW	1.73 E+05	1.1	10.9	10.4 - 11.4	0.34
D2-20	SW	1.67 E+03	1.6	10.6	10.3 - 10.9	0.23

a. First digit refers to solidification agent: C = Portland Type I-II cement, D = vinyl ester-styrene. Second digit refers to EPICOR-II resin type: 1 = Type 1, 2 = Type 2. Number after hyphen refers to the specimen number within a batch.

b. DI = demineralized water, SW = synthetic sea water.

c. $L = \sum_{n=1}^{10} [\log (\beta/D_i)]_n/10.$

d. 99.9% confidence range of L.

e. $r = \frac{\sigma_{Lt}}{\sigma_L \sigma_t}$

method is satisfactory for leachability testing. The time, temperature, leachant, and materials requirements are reasonable and applicable. It should be noted that the TP requires leach testing for a minimum of 90 days, while the ANS 16.1 test method is based upon a cumulative leach time of 89 $\pm$ 1 days. This difference is not significant. The TP should specify the immersion fluid type(s) (demineralized water and sea water, demineralized water alone, or other leachants) required for immersion testing. Since the leachability index calculated can vary depending upon the radionuclides measured in leachate analysis, the TP should provide guidance on selection of the radionuclides of interest.

Immersion Testing

Compression strength results for EPICOR-II waste forms subjected to immersion as part of the leachability test procedure are listed in Table 10. One specimen had caps which were insufficiently parallel for proper seating in the compression tester. Average compression strengths for each waste form type measured after immersion are approximately the same as those determined for as-prepared waste forms, indicating no significant degradation of strength as a result of immersion. Although average compression strengths measured for waste forms immersed in sea water are somewhat higher than for specimens leached in demineralized water, the number of specimens tested and the strength differences are too small to suggest any significant effect of leachant type on compressive strength after immersion. All immersion-tested waste forms exhibited compressive strengths far in excess of the 50 psi required by the TP.

This work indicates that immersion testing can be satisfactorily performed in conjunction with ANS 16.1 leach testing. In this manner, the immersion fluid and volume (in relation to the specimen geometric surface area) is specified. Immersion testing performed in conjunction with leachability testing also decreases personnel exposure. The TP itself does not specify the conditions to be imposed during immersion testing (other than the minimum 90-day duration). The TP should either (a) specify that immersion testing be performed in conjunction in ANS 16.1 leach testing or (b) specify relevant immersion testing conditions. Relevant immersion testing conditions are expected to include immersion fluid composition, specification of immersion fluid volume to specimen external geometric surface area (to assure complete immersion and avoid concentration effects), and a requirement that essentially the entire specimen surface be exposed to the immersion fluid.

Radiation Stability

Based upon the calculated activity inventory of EPICOR-II prefilters PF-7 and -24 as of April 1982 (given in Table 11), the total waste form self-irradiation dose to infinity was determined for each waste form type (Table 12). (Only gamma and beta radiation is given, since there are no significant alpha emitters in the EPICOR-II wastes.) Radionuclide decay from the prefilter activity inventory to waste form fabrication and testing was not considered. The waste form self-irradiation doses shown in Table 12 (calculated using the method of Reference 16) consider both the activity content of the prefilter waste and the waste form resin content.

Waste form radiation doses to infinity vary from 2.85×10^8 to 4.22×10^8 rad depending upon waste form type. The major portion of the dose (56%) for waste forms containing Type 1 waste is due to the high Sr-90 content of the PF-7 prefilter. In contrast, only 2.8% of the dose to waste forms containing Type 2 waste in PF-24 is due to Sr-90 decay; the majority of the dose is due to the gamma emitters Cs-137 and Cs-134. Based upon these data, a desired gamma dose of 5×10^8 rad was selected for radiation stability tests. [The self-irradiation dose acquired by the waste forms since preparation was not considered; however, it represents only a small fraction (approximately 5%) of the expected accumulated dose.] That desired gamma irradiation dose exceeds the total dose (gamma and beta) to infinity for all waste form types. The dose to infinity is essentially equal to the 300-yr dose, since Cs-137 and Sr-90 (with half-lives of 39 and 29 yr, respectively) dominate the EPICOR-II resin activity inventory.

The doses received by the EPICOR-II waste forms in the ATR gamma irradiation facility are shown in Table 13. The dose received varied as a function of position (level) in the specimen basket carousel as shown in that Table. Despite support chain breakage in the irradiation tubes (described previously), all waste forms received gamma irradiation doses exceeding the calculated dose to infinity (Table 12) except vinyl ester-styrene waste forms DIA-21, -22, and -23. The 2.8×10^8 rad dose received by those waste forms did approximate the calculated gamma dose to infinity (3.13×10^8 rad). One vinyl ester-styrene waste form containing Type 1 waste (DIA-24) did receive a dose (5.7×10^8 rad) exceeding the calculated total dose to infinity.

During removal of EPICOR waste forms from the specimen baskets after gamma irradiation, the specimen preparation vials were observed to be strongly

Table 10. Compressive strengths of immersion-tested EPICOR-II resin waste forms

Specimen Designation[a]	Cure Time (months)	Leachant		Compressive Strength (psi)
C1-7	24	DI		2,160
C1-8	24	DI		3,260
C1-10	24	SW		2,500
C1-11	24	SW		3,910
			Average[c]	2,960
			Std. dev.[c]	780
C2B-4	24	DI		5,200
C2B-5	24	DI		3,410
C2B-6	24	SW		2,930
C2B-7	24	SW		1,450[b]
			Average[c]	3,850
			Std. dev.[c]	1,200
D1-3	24	DI		2,860
D1-4	24	DI		2,320
D1-5	24	SW		2,900
D1-6	24	SW		2,990
			Average[c]	2,770
			Std. dev.[c]	300
D2-17	24	DI		3,620
D2-18	24	DI		2,890
D2-19	24	SW		3,150
D2-20	24	SW		3,410
			Average[c]	3,270
			Std. dev.[c]	320

a. First digit refers to solidification agent: C = Portland Type I-II cement, D = vinyl ester-styrene. Second digit refers to EPICOR-II resin type: 1 = Type 1, 2 = Type 2. Number after hyphen refers to the specimen number within a batch.

b. Caps not sufficiently parallel for proper seating in the compression tester. Measured compressive strengths are lower than actual strengths.

c. These calculations do not include data for specimens with insufficiently parallel caps.

Table 11. Activity inventory for EPICOR-II prefilters PF-7 and -24

	Prefilter PF-7		Prefilter PF-24	
Radionuclide	(Ci)	(%)	(Ci)	(%)
Co-60	1.27E−01	0.01	2.08E−01	0.01
Sr-90	3.45E+02	25.18	1.75E+01	0.93
Cs-134	4.99E+01	3.64	9.16E+01	4.85
Cs-137	9.71E+02	70.88	1.78E+03	94.18

Table 12. Calculated total (gamma and beta) self-irradiation doses to infinity for EPICOR-II waste forms

		Total Dose to Infinity, (rad)	
Liner	Waste Type	Portland Cement	Vinyl Ester-Styrene
PF-7	1	3.38E+08	4.22E+08
PF-24	2	2.85E+08	3.56E+08

Table 13. Gamma irradiation doses received by EPICOR-II forms in the ATR gamma irradiation facility

Binder	Position	Specimen Designations[a,b]	Gamma Dose, (rad)
Portland cement	Level 1	C1A-1, C1A-2, C1A-4	4.9E+08
	Level 2	C2A-11, C2A-12, C2A-13	5.3E+08
	Level 3	C2A-16, C2B-2, C2B-3	5.4E+08
	Level 4	C1A-10, C1-5, C1-6	5.3E+08
Vinyl ester-styrene	Level 1	D1A-21, D1A-22, D1A-23	2.8E+08
	Level 2	D2-22, D2-24, D2-25	3.9E+08
	Level 3	D2-26, D2-21, D2-23	4.9E+08
	Level 4	D1A-24, D1-1, D1-7	5.7E+08

a. First digit refers to solidification agent: C = Portland Type I-II cement, D = vinyl ester-styrene. Second digit refers to EPICOR-II resin type: 1 = Type 1, 2 = Type 2. Number after hyphen refers to the specimen number within a batch.

b. Specimens C2B-2, C2B-3, C1-5, C1-6, D2-21, D2-23, D1-1, and D1-7 were exposed in the ATR gamma irradiation facility for leach after irradiation testing.

discolored (brown) and the specimen identification numbers had been eradicated (Figure 9). (Documentation of specimen location in the baskets allowed the waste forms to be identified; the specimen identification numbers were replaced.) The low-density polyethylene preparation vials were very brittle, as determined during specimen removal for capping. (The specimen vials were retained for subsequent examination.) The vinyl ester-styrene waste form specimens were slightly discolored (weak brownish tint to the initially white polymer) with a small amount of a brownish "oil" on the surface. No visible change was observed for Portland cement waste forms. Figure 10 shows the appearance of Portland cement and vinyl ester-styrene waste forms after gamma irradiation.

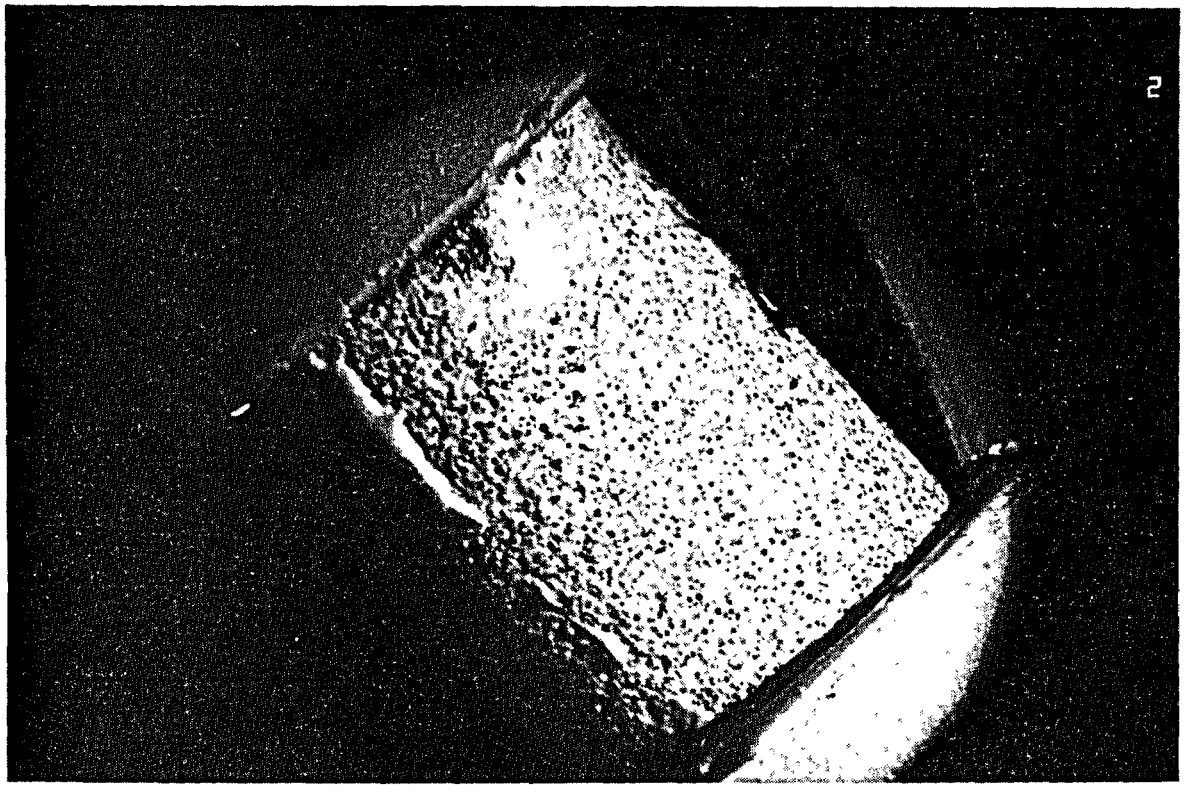
Compression test data for radiation stability specimens are listed in Table 14. All radiation stability specimens tested had compressive strengths far in excess of the 50 psi recommended by the TP. Radiation stability waste form specimens had compressive strengths in the range of 1,200 to 5,200 psi.

The compressive strengths of vinyl ester-styrene waste forms were significantly decreased as a result of gamma irradiation. Average strengths of gamma-irradiated vinyl ester-styrene waste forms were approximately 67% of the compressive strengths of as-prepared specimens. Vinyl ester-styrene waste forms containing Type 2 waste had higher strengths than those containing Type 1 waste. The strain to failure decreased significantly for vinyl ester-styrene waste forms as a result of irradiation; the gamma-irradiated waste form is brittle relative to its as-prepared counterpart. Note that the compressive strength of D1A-24, which received a dose of 5.7 x 10^8 rad, is approximately equal to those of the other D1A batch waste forms which did not receive the desired irradiation dose.

The average compressive strengths of Portland cement waste forms did not appear to have been significantly affected by gamma irradiation. The strengths of Portland cement waste forms containing Type 2 waste decreased relative to waste forms containing Type 1 waste. Note that the standard deviations of irradiated Portland cement waste form data are large. Material effects induced by gamma irradiation may be responsible for this increase in compressive strength standard deviation.

Although the EPICOR-II resin waste form specimens tested had high associated dose rates and large activity contents, administrative procedures were successful in minimizing personnel exposure and contamination during gamma irradiation and compression testing.

Figure 9. Irradiated EPICOR-II waste form being placed into polyethylene bag marked with the appropriate specimen designation.



b. Vinyl ester-styrene.

Figure 10. Appearance of EPICOR-II waste forms after gamma irradiation.

Table 14. Compressive strengths of gamma-irradiated EPICOR-II resin waste forms

Specimen Designation[a]	Cure Time (months)		Compressive Strength (psi)
C1A-1	27		5,230
C1A-2	27		3,980
C1A-4	27		3,590
C1A-5	27		1,740
		Average[c]	3,640
		Std. dev.[c]	1,440
C2A-11	27		5,200
C2A-12	27		2,880
C2A-13	27		1,200[b]
C2A-16	27		1,860
		Average[c]	3,310
		Std. dev.[c]	1,710
D1A-21	27		2,010
D1A-22	27		1,930
D1A-23	27		1,910
D1A-24	27		1,880
		Average[c]	1,930
		Std. dev.[c]	560
D2-22	27		1,230
D2-24	27		2,890
D2-25	27		2,950
D2-26	27		2,610
		Average[c]	2,420
		Std. dev.[c]	810

a. First digit refers to solidification agent: C = Portland Type I-II cement, D = vinyl ester-styrene. Second digit refers to EPICOR-II resin type: 1 = Type 1, 2 = Type 2. Number after hyphen refers to the specimen number within a batch.

b. Caps not sufficiently parallel for proper seating in the compression tester. Measured compressive strengths are lower than actual strengths.

c. These calculations do not include data for specimens with insufficiently parallel caps.

This work demonstrates that radiation stability testing as specified by the TP is generally applicable. However, the TP should specify that if the level of *total* ionizing radiation exposure (i.e., gamma, beta, and alpha radiation) is expected to exceed 10^8 rad, testing should be performed at the expected maximum *total* accumulated dose.

The effect of dose rate on the compression strength of waste forms is not clear; however, the nature of 10 CFR 61 testing requires dose rates far in excess of expected values. If dose rate effects are significant, they would normally be expected to provide conservative results in testing.

Biodegradation

Since biodegradation testing is continuing, the results of this work will be the subject of a subsequent report.

CONCLUSIONS

Procedures as specified in the TP were utilized to test Portland Type I-II cement and vinyl ester-styrene waste forms containing EPICOR-II prefilter waste. The purpose of this testing was to determine the adequacy of those procedures in determining compliance with the stability requirements for solidified Class B and C low-level waste in 10 CFR 61. Waste form performance data were also obtained as a result of this testing.

The Portland cement and vinyl ester-styrene waste forms tested were found to meet the waste form stability requirements for (a) free liquids, (b) homogeneity, (c) compressive strength, (d) resistance to thermal degradation, (e) leachability, (f) immersion, and (g) radiation stability. The formulations utilized for the solidification of actual EPICOR-II wastes have low waste loadings compared to commercial practice. This results in improved waste form performance; and, as a result, test results presented in this report may overestimate the conservativeness of commercial products. Biodegradation testing is on-going and will be the subject of a later report. This work also demonstrated that appropriate administrative controls can be implemented to minimize contamination and personnel exposure while utilizing the procedures specified by the TP.

While the procedures specified in the TP are generally satisfactory for demonstrating compliance with 10 CFR 61 stability requirements, some recommendations were developed as a result of this work:

Free Liquids

The ANS 55.1 free liquid determination procedure is adequate for determining compliance of full-scale (55-gal or larger) waste forms with 10 CFR 61. Minor procedural modifications are required and should be allowed for use with smaller waste forms.

Thermal Stability

Thermal stability testing should require that the specimen be appropriately containerized immediately after preparation and through thermal cycling to prevent evaporative water loss.

Thermal stability testing should require that the temperatures of the specimens cycle from below freezing and then above thawing (or between desired temperature limits) rather than simply specifying exposure to required temperatures.

Leachability

The TP requirement of a minimum 90-day test is in minor conflict with the specified ANL 16.1 leach procedure (89 $\pm$ 1 days) and should be revised.

Leachability guidance as provided by the TP should specify the leachant fluid type(s) (demineralized water and sea water, demineralized water alone, or other leachants).

Since the leachability index calculated for a waste form can be dependent upon the radionuclides measured in leachate analysis, the TP should provide guidance on selection of the radionuclides of interest.

Immersion Testing

Immersion testing must either require the conditions of the ANS 16.1 leach procedure or specify relevant immersion test conditions. (Only immersion time is specified in the TP.) Relevant immersion testing conditions (in addition to time) are expected to include immersion fluid composition, the ratio of immersion fluid volume to specimen external geometric surface area, and that essentially the entire specimen surface be exposed to the immersion fluid.

Radiation Stability

Radiation stability testing should require that waste forms be exposed to 10^8 rad gamma irradiation or, if the level of *total* ionizing radiation exposure (i.e., gamma, beta, and alpha radiation) is expected to exceed 10^8 rad, testing should be performed at the expected maximum total dose.

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13. ABSTRACT (200 words or less)

One goal of the EPICOR-II Resin/Liner Investigation Program is to confirm the adequacy of the test procedures specified in the U.S. Nuclear Regulatory Commission "Technical Position on Waste Form" (TP) relative to compliance with stability requirements for solidified Class B and C wastes. In previous work, sponsored by the Department of Energy under Contract No. DE-AC07-76IDO1570, formulations were developed to solidify radioactive wastes from EPICOR prefilters PF-7 (organic ion exchange resins) and PF-24 (organic ion exchange resins with zeolite) from Three Mile Island Unit-2 using Portland Type I-II cement and vinyl ester-styrene. Those waste forms were fabricated and then subjected to the specified stability test procedures. This report describes later work funded by the U.S. Nuclear Regulatory Commission. That work consisted of performing the comprehensive waste form testing specified in the TP. Test methodologies used to verify compliance with test criteria for free standing water, compressive strength, thermal stability, leachability, and radiation stability are described. The waste form performance data are presented and evaluated in this report.

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