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PROPERTIES OF RADIOACTIVE WASTES AND WASTE CONTAINERS

QUARTERLY PROGRESS REPORT
JANUARY - MARCH 1977

P. COLOMBO AND R.M. NEILSON, JR.

Date Published - August 1977

PROCESS TECHNOLOGY DIVISION
DEPARTMENT OF APPLIED SCIENCE
BROOKHAVEN NATIONAL LABORATORY
ASSOCIATED UNIVERSITIES, INC.
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PREPARED FOR THE UNITED STATES NUCLEAR REGULATORY COMMISSION
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SUMMARY

The densities of the simulated wastes used in this program were determined and factors reported to allow conversion of formulation data for urea-formaldehyde (UF) waste forms from weight ratios to volume ratios of waste to UF. Simulated Solka-Floc wastes were solidified with urea-formaldehyde but areas of unsolidified Solka-Floc were found in the final waste forms. The difficulty in mixing dewatered Solka-Floc waste with UF is presumed to be responsible. Attempts to solidify alkaline regenerative wastes with urea-formaldehyde were not successful. Increasing the waste/UF volume ratio to 3.0 produced acceptable solids with boric acid and decontamination waste, but not with chemical regenerative wastes.

The use of masonry cement (one part portland cement to one part anhydrous lime by weight) for the solidification of boric acid concentrate waste was investigated. Free standing solids were formed at waste/cement weight ratios of 0.6 and 1.0 but not at 1.5 or 2.0. Masonry cement was found to be superior to portland cement for the solidification of boric acid wastes.

The weight loss with temperature of urea-formaldehyde waste forms containing various simulated wastes was measured over the range of 25°C-500°C. Weight losses due to the evaporation of water, decomposition of UF and decomposition of the wastes were observed.

Static leaching experiments were conducted to determine the rate of release of cesium-137 and strontium-85 from urea-formaldehyde specimens and bead and powdered cation exchange resins. Results for the leaching of these materials in salt water, tap water, and distilled water are reported.

Hydrostatic testing of modified DOT 17H open head 55 gallon drums was performed. These drums met the required DOT specifications and ultimately failed at an average pressure of 44 psig by separation of the drum head from the drum body at the gasket.

I. SOLIDIFICATION OF SIMULATED WASTES WITH UREA-FORMALDEHYDE

A. Weight Ratio to Volume Ratio Conversion Factors

Waste to urea-formaldehyde (UF) weight ratios have generally been used to describe waste formulations in this program. Vendors and operators of urea-formaldehyde solidification systems often describe solidified waste compositions as a volume ratio of the waste and UF streams. The relation between weight ratios and corresponding volume ratios is given in Equation (1):

$$\frac{W_1}{W_2} = \frac{D_1}{D_2} \cdot \frac{V_1}{V_2} \quad (1)$$

where W = mass (g)

D = density (g/cm³)

V = volume (cm³)

let subscript 1 refer to the waste

and subscript 2 refer to UF

The waste to UF volume ratio (V_1/V_2) is equal to the weight ratio (W_1/W_2) times the inverse of the ratio of their respective densities (D_2/D_1). The density of the Borden Casco-Resin 2 urea-formaldehyde (65 wt% solids) used in this program is 1.30g/cm³ at 25°C. The densities of the simulated wastes listed in Appendix A have been determined and are listed in Table 1 with their respective weight ratio to volume ratio conversion factors (D_2/D_1) and volume ratio to weight ratio conversion factors (D_1/D_2). Bead resin and BWR precoat filter cake wastes in this table include formulations for both slurry and dewatered wastes. The slurry wastes represent formulations that are pumpable for short distances. Previous work has utilized the slurry formulations for bead resin waste and powdered resin precoat filter cake while a dewatered formulation was used for diatomaceous earth precoat filter cake.

B. Solidification of Solka-Floc

Solka-Floc* is a wood pulp derived organic material composed of a minimum of 99.5% cellulose that is used in place of diatomaceous earth at

* Grefco, Inc., Dicalite Division, Avenel, NJ 07001

TABLE 1

Volume Ratio and Weight Ratio Conversion Factors
for Waste Solidification with Urea-Formaldehyde

Waste Type	Waste Density (D_1), g/cm ³	Weight Ratio to Volume Ratio Conversion Factor (D_2/D_1)	Volume Ratio to Weight Ratio Conversion Factor (D_1/D_2)
1. Bead Resin			
a. slurry	1.05	1.24	0.81
b. dewatered	1.09	1.19	0.84
2. BWR Precoat Filter Cake			
a. powdered resin			
1. slurry	1.08	1.20	0.83
2. dewatered	1.10	1.18	0.85
b. diatomaceous earth			
1. slurry	1.10	1.18	0.85
2. dewatered	1.14	1.14	0.88
3. Forced Recirculation Evaporator Concentrates			
a. BWR chemical regenerative	1.20	1.08	0.92
b. PWR chemical regenerative	1.16	1.12	0.89
c. boric acid	1.01	1.29	0.78
d. decontamination waste	1.00	1.30	0.77

Density of UF at 25°C (D_2) = 1.30 g/cm³

some plants as a filter aid material. Several different grades are available which vary primarily in particle size. Solka-Floc grades BW-40 and BW-100 are specified for use in condensate water filtration. The average fiber length is 80 μm and 55 μm for BW-40 and BW-100 Solka-Floc respectively.

Trial solidifications of Solka-Floc wastes with urea-formaldehyde were performed. These trials employed the formulations listed for diatomaceous earth BWR precoat filter cake slurry and dewatered wastes with Solka-Floc (BW-100) substituted for diatomaceous earth. The slurry waste has a somewhat gelatinous consistency while the Solka-Floc in the dewatered waste tends to clump together. Slurry and dewatered wastes were combined with urea-formaldehyde at a waste/UF weight ratio of 2.0. This ratio has been used successfully with diatomaceous earth dewatered waste. Two volume percent of a 25 wt.% anhydrous sodium bisulfate solution was added to catalyze UF polymerization. The top surfaces of the slurry and dewatered waste-UF specimens produced were solid and quite hard within thirty minutes, however, it was evident with the dewatered waste that some settling out of the Solka-Floc had occurred. One day after mixing, the areas of settled Solka-Floc were found to be unsolidified although the remainder of each specimen was well solidified. A small amount of unsolidified settled Solka-Floc was also observed in one of the two slurry waste-UF specimens made.

The occurrence of areas of unsolidified Solka-Floc waste appears to be related to the mixing of the waste with urea-formaldehyde. The dewatered waste when mixed with UF at a waste/UF volume ratio of 2.0 forms a viscous mixture with a gelatinous consistency not unlike that of the Solka-Floc slurry waste alone. Rather vigorous mixing is required with the dewatered waste-UF formulation and even then it is somewhat difficult to achieve a homogeneous mixture. A homogeneous mixture is considerably easier to achieve when the slurry waste is combined with urea-formaldehyde. Adequate mixing to form a homogeneous mixture is apparently required to prevent pockets of unsolidified Solka-Floc. The addition of sufficient catalyst to provide a relatively rapid solidification and complete dispersion of the catalyst throughout the waste-UF mixture is also advantageous.

C. Solidification of Alkaline Regenerative Wastes

The forced recirculation regenerative wastes listed in Appendix A have mildly acidic pH values. The pH of wastes fed to evaporators may, however, be adjusted into the alkaline range to minimize corrosion problems. When this is done, the pH of the waste entering the evaporator is normally adjusted to the range of 8-11. Although the pH of the concentrated regenerative waste stream that is ultimately solidified may revert to a near neutral pH due to absorption of carbon dioxide from the atmosphere during processing, it is desirable to determine the effect of alkaline regenerative wastes on the solidification of urea-formaldehyde waste forms.

BWR and PWR chemical regenerative wastes listed in Appendix A were adjusted to pH 10 by the addition of a saturated sodium hydroxide solution and combined with urea-formaldehyde at a waste/UF weight ratio of 1.2. A two volume percent addition of a 25 weight percent anhydrous sodium bisulfate solution was made to induce polymerization. Triplicate 100 gram samples of each formulation were prepared. These wastes did not solidify satisfactorily. In both cases, a hard bottom layer formed below a viscous but still liquid mixture apparently composed of sodium sulfate waste and some urea-formaldehyde solids. This viscous layer which did not solidify in ten days was covered by a thin membrane. When the containers were opened to the air, water in the liquid layer evaporated and a hard brittle solid resulted. These wastes were both successfully solidified with UF at this ratio when the waste pH was not adjusted into the alkaline range.

The polymerization of urea-formaldehyde is acid catalyzed. For radioactive waste applications, it is desirable to add sufficient acid catalyst to achieve a waste-UF mixture pH of 1.5 ± 0.5 .¹ When this condition is met, the time required for UF gelation and solidification is normally sufficiently short to prevent appreciable settling of wastes or urea-formaldehyde solids. However, the amount of catalyst needed to adjust the pH to this range may be considerable for alkaline or buffered wastes. If the pH is not properly adjusted, layering is likely especially at high waste/UF ratios for wastes whose densities are either low or high compared to the density of UF. This

is apparently what occurred with the alkaline sodium sulfate wastes. Since the entire mixture did not solidify quickly, settling of urea-formaldehyde solids which may have been partially polymerized resulted, producing a non-homogeneous layered waste form. The urea-formaldehyde bottom layer did completely polymerize producing a solid. The viscous liquid layer above it apparently contains some polymerized UF solids producing the increase in viscosity, but not a sufficient amount to produce a solidified waste form.

D. Waste Solidification at Waste/UF Volume Ratios of 3.0

Earlier waste-UF formulations had been derived from experiments which initially utilized low waste/UF ratios which were increased to the point which was felt to consistently provide a good solidified product for testing purposes. These formulations were not meant to represent maximum or optimized values. The literature states that liquid wastes are generally combined with urea-formaldehyde at a waste/UF volume ratio of 2.0.^{1,2} This value represents a compromise between product properties and economics. Since higher waste/UF ratios may actually be used in practice, work is proceeding to investigate the properties of products produced at waste/UF volume ratios of 3.0. The initial work reported here concerns the solidification of forced recirculation evaporator concentrates with urea-formaldehyde.

Triplicate twenty gram samples of each of the forced recirculation evaporator wastes were prepared using Borden Casco-Resin 2 at a waste to UF volume ratio of 3.0. Two volume percent of a 25 weight percent anhydrous sodium bisulfate solution was added to the waste/UF mixtures which were solidified in one inch diameter plastic vials. The vials were sealed to prevent evaporative water loss.

The specimens produced using BWR and PWR regenerative waste concentrates did not solidify after twenty days. Some settling of urea-formaldehyde is evidenced by a thin solid layer at the bottom of the solidification vial. Difficulties have been encountered in using urea-formaldehyde for the solidification of sodium sulfate wastes for waste form formulations containing in excess of 10 weight percent sodium sulfate.³ The boric acid and decontamination wastes were solidified by urea-formaldehyde at a waste/UF ratio

of 3.0. Complete solidification required two days. The resistance to probe penetration at this ratio was less than that for these wastes solidified at lower waste/UF ratios. No free standing water was observed for the boric acid waste specimens although water was easily squeezable from the formulation. Six days after solidification, the cumulative free standing water observed for the decontamination waste solidifications was $16 \pm 2\%$ based on the total formulation weight.

II. SOLIDIFICATION OF BORIC ACID WASTE FROM A FORCED RECIRCULATION EVAPORATOR USING MASONRY CEMENT

Boric acid waste is difficult to solidify using portland cement. Portland cement may be employed for boric acid waste solidification by either the use of low waste/cement ratios or by the incorporation of additives. The use of masonry cement which is composed of one part portland cement to one part anhydrous lime by weight has been suggested. Trial solidifications were made to determine the utility of masonry cement for the solidification of boric acid waste from a forced recirculation evaporator.

Triplicate twenty gram mixtures were made using simulated boric acid waste from a forced recirculation evaporator and masonry cement at waste/cement weight ratios of 0.6, 1.0, 1.5, and 2.0. These mixtures were placed into individual one inch diameter plastic vials which were sealed to prevent water loss by evaporation. At daily intervals the samples were probed to determine if the mixture had solidified. At waste/cement weight ratios of 0.6 and 1.0 a solidified product resulted after one and two days respectively with no measurable free standing water. No solidification occurred after thirty days for waste/cement ratios of 1.5 and 2.0. As a comparison, boric acid waste solidified with portland type II cement at a waste/cement weight ratio of 0.6 required approximately forty days to solidify when no additives were used. The masonry cement product formed at a waste/cement ratio of 0.6 was quite hard and resisted penetration by a probe. A ratio of 1.0 resulted in a monolithic free standing solid, but it could be penetrated after solidification to a depth of a few millimeters by a wooden probe which was also capable of breaking off small pieces of the solid indicating a lower product integrity. The hardness of this product did increase with time, but after fourteen days it was not as resistant to probe penetration as was the solid formed at a ratio of 0.6 after one day.

The anhydrous lime in the masonry cement produces a more alkaline media when water is added then portland cement alone. Thus it has a greater ability to raise the pH of a boric acid waste-cement mixture into the alkaline range that is favored for the setting and ultimate curing of portland cement.

III. THERMOGRAVIMETRIC ANALYSIS OF UREA-FORMALDEHYDE FORMULATIONS

Thermogravimetric analyses were performed on urea-formaldehyde waste forms containing each of the simulated wastes listed in Appendix A. These tests were conducted using a Perkin Elmer TGS-2 thermogravimetric analyzer and specimens prepared using Borden Casco-Resin 2 urea-formaldehyde. A $10^{\circ}\text{C}/\text{minute}$ temperature scanning rate was employed with a nitrogen purge gas.

Curves of percent initial weight as a function of temperature are shown in Figures 1-12. In deriving these figures, three samples of each waste form formulation were tested. The solid curve designates the average of the data while the dashed lines indicate the data spread envelope. The samples tested were small ($\sim 10\text{ mg}$) and the difficulty in obtaining representative samples of that size is indicated by the width of some data envelopes, particularly those in Figures 5 and 10. Figure 1 illustrates the weight loss of urea-formaldehyde made with distilled water at a water/UF weight ratio of 2.0, which corresponds to a water/UF volume ratio of 2.6. This curve has two distinct features, a rapid weight loss between 100°C and 140°C due to evaporation of contained water and a weight loss with a maximum rate at approximately 290°C corresponding to thermal decomposition of the urea-formaldehyde. These features also predominate the thermogravimetric curves for the simulated wastes solidified with urea-formaldehyde, although other features also appear. For bead resin waste forms, an additional weight loss occurs with a maximum rate at 400°C due to decomposition of the resin beads. Boric acid loses one-half molecule of water at 300°C which combined with the urea-formaldehyde decomposition produces the large weight loss at 300°C shown in Figures 7 and 11. Ammonium sulfate, present in PWR regenerative waste decomposes at 235°C , but this decomposition is not apparent in the corresponding curves, due to the relatively small quantity present in these waste forms.

Figure 1. Thermogravimetric weight loss of water-urea-formaldehyde ($H_2O/UF = 2.0$ by weight).

Figure 2. Thermogravimetric weight loss of a bead resin slurry waste (1a) - UF product (waste/UF = 2.2 by weight).

Figure 3. Thermogravimetric weight loss of a powdered resin slurry waste (2a(1)) - UF product (waste/UF = 2.0 by weight).

Figure 4. Thermogravimetric weight loss of a dewatered diatomaceous earth precoated filter cake (2b(2)) - UF product (waste/UF = 1.5 by weight).

Figure 5. Thermogravimetric weight loss of a forced recirculation evaporator BWR chemical regenerative waste (3a) - UF product (waste/UF = 1.2 by weight),

Figure 6. Thermogravimetric weight loss of a forced recirculation evaporator PWR chemical regenerative waste (3b) - UF product (waste/UF = 1.2 by weight).

Figure 7. Thermogravimetric weight loss of a forced recirculation evaporator boric acid waste (3c) - UF product (waste/UF = 1.0 by weight).

Figure 8. Thermogravimetric weight loss of a forced recirculation decontamination waste (3d) - UF product (waste/UF = 1.1 by weight).

Figure 9. Thermogravimetric weight loss of a thin film evaporator BWR chemical regenerative waste (4a) - UF product (waste/UF = 1.0 by weight).

Figure 10. Thermogravimetric weight loss of a thin film evaporator PWR chemical regenerative waste (4b) - UF product (waste/UF = 1.0 by weight).

Figure 11. Thermogravimetric weight loss of a thin film evaporator boric acid waste (4c) - UF product (waste/UF = 1.0 by weight).

Figure 12. Thermogravimetric weight loss of a thin film evaporator decontamination waste (4d) - UF product (waste/UF = 1.5 by weight).

IV. LEACHABILITY

A. Cesium-137 and Strontium-85 Release from Urea-Formaldehyde in Static Leaching

The ^{137}Cs and ^{85}Sr leachability of urea-formaldehyde for static leaching in distilled water, tap water, and sea water was measured using the test method described in Appendix B. These specimens were prepared with distilled water at a water to UF volume ratio of 2.0. Leachability measurements made with distilled water and sea water leachants are believed to span the range of expected release rates. Distilled water provides a convenient method for interlaboratory comparisons while leaching in sea water illustrates the effect of high salt concentrations (ion exchange interactions) and also may provide data relevant to ocean disposal. Sea water used in leaching experiments was obtained from the Atlantic Ocean at Shinnecock Inlet, Long Island, New York. The rate of radionuclide release from waste forms in shallow land burial trenches is of primary interest. Shallow land burial sites are located such that the buried wastes are disposed above local water tables. As such, water that comes in contact with buried wastes is not ground water, but rather percolate rain water that contains dissolved species acquired in its downward migration from the surface. Due to the relatively short time this percolate rain water is exposed to soluble materials in geologic strata before entering a burial trench, it should have a lower dissolved solids content than the ground water present in the area. Water analyses at the existing commercial shallow land burial sites are being collected and analyzed. At this time, however, leaching experiments are being conducted using local tap water as the leachant. Long Island tap water is derived from ground water aquifer supplies but has a relatively low dissolved solids content due to the geologic nature of the area. The local geology and hence the expected ground water chemistry is similar to that at the Barnwell, SC shallow land burial site. The tap water used in these experiments does undergo some pretreatment. Analyses of this tap water will be presented in a subsequent report.

Urea-formaldehyde (UF) leach specimens were prepared by solidifying distilled water with Borden Casco-Resin 2 at a water-UF volume ratio of 2.0.

Specimens were prepared in one inch I.D. lucite tubes which were sealed with a removable teflon plug at one end. The amount of distilled water and urea-formaldehyde necessary to provide a cylindrical sample approximately two inches in length had been previously determined. An addition of 0.01 mCi of ^{137}Cs as $^{137}\text{CsCl}$ (carrier free) and 0.01 mCi ^{85}Sr as $^{85}\text{SrCl}$ (27.6 mCi/mg) was made to each specimen by addition to the distilled water prior to mixing with urea-formaldehyde. After the urea-formaldehyde was added, the contents of the specimen tube were thoroughly mixed. Two volume percent of a 25 wt% anhydrous sodium bisulfate solution was added as a polymerization catalyst, after which the water-UF mixture was again stirred and the specimen tube sealed to prevent water loss by evaporation. The urea-formaldehyde specimens were allowed to cure for three days before leach testing was begun.

Screw closure wide mouth polymethylpentene leachant solution containers with a nominal capacity of $1,000\text{ cm}^3$ were employed. Any free standing water present in the specimen preparation tube as the specimen was removed was added to the leachant solution container. Specimen mass and dimensions were recorded. Each specimen was placed on a small lucite support in its leachant container which allowed the entire surface of the specimen to be exposed to the leachant. Sufficient leachant was added to provide a leachant volume (V_L) to external geometric specimen surface area ratio of 10 cm. At specified intervals, the specimens were removed from their leach containers and a 10 ml aliquot of the stirred leachant taken for analysis by gamma spectrometry.

Gamma spectrometry leachant analysis employed a Searle Model 1185 automatic counter with a 3x3 inch true well sodium iodide crystal. The analyzing channels were adjusted to detect the gamma photon peaks at 0.662 MeV for cesium-137 and 0.513 MeV for strontium-85 with window widths adjusted to provide a high count rate with minimal peak overlap. A ten minute counting time was used for most samples. Corrections were made to the data to compensate for background, peak overlap and Compton scattering into the strontium-85 channel. Fraction activity releases were obtained by comparison with prepared standards. This release was checked by dissolution of the specimens in 3M nitric acid at the end of the leaching experiments.

The measured leach data is reported in Figures 13-20. This data is expressed as a (cumulative fraction release) $\times(V/S)$ where V is the specimen volume (cm^3) and S is its external geometric surface area (cm^2). The factor (V/S) is included so that the leach rate is independent of specimen geometry and size for leach times which are short relative to a complete release of the species of interest. When this criteria is met, fraction activity releases can be determined for waste forms of differing (V/S) ratio. The ordinate in these figures is also expressed as a cumulative fraction activity release. This release is derived using the (V/S) value for the individual specimens which do vary somewhat and as a consequence the expressed cumulative fraction release is approximate. Figures 13 and 14 illustrate the release of cesium-137 and strontium-85 respectively for static leaching in seawater, tap water and distilled water. Each sample series consisted of three specimens leached in a different leachant: series 67 - seawater, series 68 - tap water, series 69 - distilled water. The curves shown are plotted for the average of the three samples in each series with error bars employed to indicate data spread. Where no error bar is indicated, the data spread falls within the data point. The data for the individual samples are shown in Figures 15-20. The data spread between similar samples was found to be generally small although a substantial spread was found for some samples at the initial sampling (100 minutes). The cesium release was rapid and essentially complete after one day of leaching. The rate of strontium release from urea-formaldehyde in static leaching was lower and more greatly dependent upon the leachant. Leaching in sea water produced the most rapid strontium release followed in decreasing order by leaching in tap water and distilled water. Although the initial releases occurring in the first several days were large, the strontium release was not complete after in excess of one hundred days of leaching.

Figure 13. (Cumulative fraction release ^{137}Cs) $\times (V/S)$ for static leaching of urea-formaldehyde samples in various leachants.

Figure 14. (Cumulative fraction release ^{85}Sr) $\times (V/S)$ for static leaching of urea-formaldehyde samples in various leachants.

Figure 15. Release of Cesium-137 for static leaching of urea-formaldehyde samples in salt water.

Figure 16. Release of Strontium-85 for static leaching of urea-formaldehyde samples in salt water.

Figure 17. Release of Cesium-137 for static leaching of urea-formaldehyde specimens in tap water.

Figure 18. Release of Strontium-85 for static leaching of urea-formaldehyde samples in tap water.

Figure 19. Release of Cesium-137 for static leaching of urea-formaldehyde samples in distilled water.

Figure 20. Release of Strontium-85 for static leaching of urea-formaldehyde samples in distilled water.

B. Cesium-137 and Strontium-85 Release from Bead and Powdered Cation Resins in Static Leaching

The rates of cesium-137 and strontium-85 release from Amberlite IRN-77* and Powdex PCH** cation exchange resins were determined for static leaching in distilled water, tap water, and sea water. Amberlite IRN-77 is a strongly acidic polystyrene base nuclear grade cation exchange resin with sulfonic acid functionality and supplied in the H⁺ ionic form. This resin has an exchange capacity of 4.7 meq/g (dry), a maximum moisture content as received of 55% and an effective size of 0.45-0.60 mm. Powdex PCH is also a strongly acidic sulfonated styrene-divinylbenzene copolymer supplied in the H⁺ ionic form. However, it has a much higher specific surface area to provide more rapid ion exchange rates. The resin particle size is 100-400 mesh (0.149-0.037 mm) with most of the particles in the 200-400 mesh (0.074-0.037 mm) range and provides an ion exchange capacity of 4.2-4.6 meq/g (dry) with an average moisture content as received of 56%.

The resins were statically leached using a leachant volume to sample geometric surface area ratio (V_L/S) of 1.0 cm. Since the (V_L/S) ratio was kept constant, the quantity of resin leached varied; Amberlite IRN-77 bead resin samples were 10.95 gram aliquots (as received) while 0.97 gram aliquots of the Powdex PCH resin were used. This variation is a consequence of the smaller particle size and hence higher surface area per unit volume for the powdered resin. Prior to the incorporation of activity in the resins, they were converted to Na⁺ form with sodium chloride. The individual resin aliquots were loaded with 0.1 mCi each of cesium-137 and strontium-85 by a batch technique. The resin aliquots were statically leached individually in one liter of distilled water, tap water, or sea water leachant. Three aliquots of each resin were leached in sea water and distilled water while two aliquots of each were leached in tap water. The leachant solutions were replaced daily with aliquots taken for gamma spectrographic analysis.

The average static leaching results are shown in Figures 21-25 for the Amberlite IRN-77 bead resin and in Figures 26-30 for the Powdex PCH powdered resin. These results are expressed as (cumulative fraction release) $\times(V/S)$ where V is the geometric resin volume and S is its external

* Rohm and Haas Company, Philadelphia, PA 19105

** Ecodyne Corporation, Union, NJ 07083

geometric surface area. The ratio (V/S) is 8.75×10^{-3} cm for the bead resin and 9.25×10^{-4} cm for the powdered resin. The ordinate of these curves are also labeled in cumulative fraction activity release. Figures 21 and 22 respectively illustrate the release of cesium-137 and strontium-85 from bead resin in all leachants. Leaching in salt water was the most severe condition for the bead resin with 88% of the initially contained cesium and 56% of the initially contained strontium being released in one day. This eluting action of the seawater is due to the replacement of radioactive ions held on the resin with dissolved species in the seawater. As expected, the release in distilled water was the lowest for the three leachants, after an initial first day cesium release of 1.85×10^{-5} cm (2.1×10^{-3} fraction release), the average rate of release was 1.4×10^{-6} cm/day (1.6×10^{-4} fraction release/day). The average strontium release rate was 2.4×10^{-8} cm/day (2.8×10^{-6} fraction release/day) after an initial release of 8.0×10^{-6} cm (9.1×10^{-4} fraction release) in the first six days of leaching. Leaching of bead resin in tap water gave average release rates of 3.5×10^{-5} cm/day (4.0×10^{-3} fraction release/day) for cesium and 2.3×10^{-6} cm/day (2.7×10^{-4} fraction release/day) for strontium. This data is more clearly shown in Figures 23-25 which are plotted for the individual leachants. Figures 26 and 27 show respectively the release of cesium-137 and strontium-85 from powdered resin in the various leachants. Seawater leaching was again the most rapid, with 65% of the initially contained cesium and 71% of the strontium released in the first day. The cesium release in tap water was approximately linear with time with the release essentially complete after ten days while the average rate of strontium release was 2.7×10^{-5} cm/day (2.9×10^{-2} fraction release/day). The average rate of cesium release for static leaching in distilled water was 1.9×10^{-6} cm/day (2.1×10^{-3} fraction release/day) after an initial first day release of 7.9×10^{-5} cm (8.5×10^{-2} fraction release). The average rate of strontium release over the initial twenty one days of leaching was 9.9×10^{-6} cm/day (1.1×10^{-2} fraction release/day) which decreased to 2.4×10^{-7} cm/day (2.5×10^{-4} fraction release per day). Figures 28-30 illustrate the release of cesium and strontium from powdered resin in seawater tap water and distilled water respectively.

Figure 21. Cesium-137 release from IRN-77 bead resin for static leaching in various leachants.

Figure 22. Strontium-85 release from IRN-77 bead resin for static leaching in various leachants.

Figure 23. Cesium-137 and Strontium-85 release from IRN-77 bead resin for static leaching in salt water.

Figure 24. Cesium-137 and Strontium-85 release from IRN-77 bead resin for static leaching in tap water.

Figure 25. Cesium-137 and Strontium-85 release from IRN-77 bead resin for static leaching in distilled water.

Figure 26. Cesium-137 release from PCH powdered resin for static leaching in various leachants.

Figure 27. Strontium-85 release from PCH powdered resin for static leaching in various leachants.

Figure 28. Cesium-137 and Strontium-85 release from PCH powdered resin for static leaching in salt water.

Figure 29. Cesium-137 and Strontium-85 release from PCH powdered resin for static leaching in tap water.

Figure 30. Cesium-137 and Strontium-85 release from PCH powdered resin for static leaching in distilled water.

V. CONTAINER TESTING

A. Hydrostatic Testing of DOT 17H Modified 55 Gallon Removable Head Steel Drums

DOT 17H 55 gallon carbon steel drums are used for Type A packaging at many power reactors. These drums have removable heads which are secured by means of bolted clamp ring. Specifications for these drums are found in 49 CFR 178. Minimum body sheet and bottom head sheet thicknesses are 18 gauge (nominal 0.0478 inch) with a minimum 16 gauge (nominal 0.0598 inch) removable head sheet. These drums are closed with a 12 gauge bolted ring with drop forged lugs, one of which is threaded, and 5/8 inch bolt and nut. DOT 17H drums must be able to sustain 15 psi for five minutes in hydrostatic testing. The results determined for hydrostatic testing of commercially procured DOT 17H drums have been reported previously.⁴ Modified design 17H drums manufactured by the U.S. Steel Corp.* were claimed to provide substantial improvements in pressure retention capability. These modified drums have 18 gauge body sheets 16 gauge removable head sheets with improved gaskets, head sheets and clamp rings. The clamp ring, gasket and inside of the removable head sheet of one of these drums is shown in Figure 31. Two of these drums were hydrostatically tested by the following procedure. A thirty psig pressure gauge was attached to the drum at a bung hole. The removable head sheet was secured and the drum placed bottom head sheet downward on a level concrete pad and filled with water. Water was then introduced into the drum at a constant rate producing a pressurization of approximately one-half psig per minute. When fifteen psig was reached the water supply was turned off for five minutes to determine if this pressure could be sustained. The water supply was then turned on once again until a pressure of approximately thirty psig was attained. At this point, the pressure was bled off and the thirty psig pressure gauge replaced with a 100 psig gauge. Pressurization was begun at zero and increased at a rate of approximately one-half psig per minute. The location and extent of all leaks were noted during testing. Testing continued until

* U.S. Steel Products Div., Dolton, IL. 60419

Figure 31. Removable drum head, gasket, and clamp ring of the modified 55 gallon DOT 17H drum.

the rate of leakage exceeded the rate of water input resulting in a continuing decrease in pressurization.

Both of the two DOT 17H drums tested satisfied the hydrostatic test requirement of fifteen psig sustained for five minutes. The first drum tested exhibited incipient slow leaks located at the gasket at pressures of 19 and 20 psig. These leaks were not sufficient to prevent continued pressurization of the drum. The drum failed at 40 psig as the top head separated from the drum body at the clamp ring closure as shown in Figures 32 and 33. In testing of the second drum, slow leaks was observed beginning at 11 and 18 psig located at the gasket. Drum failure occurred at 47 psig when the drum head separated from the body as shown in Figures 34 and 35.

B. Corrosion Testing of Mild Steel Containers

Corrosion testing of mild steel containers is being initiated. Container corrosion is interesting from both the aspect of interaction of the container with the burial environment and the interaction of the waste form with the container. Beginning efforts will be directed towards the waste form - container interaction, with a particular emphasis on the effect of acidic free standing water on mild steel corrosion.

Figure 32. Side view of the first test drum showing the location of the drum failure at the gasket in the bolt ring gap which occurred at 40 psig.

Figure 33. Top view of the first test drum showing the relative locations at the gasket of the slow leaks (SL) observed at 19 and 20 psig and the drum failure at 40 psig.

Figure 34. Side view of the second test drum showing the separation of the drum head from the body which resulted in the drum failure at 47 psig.

Figure 35. Top view of the second test drum showing the relative locations at the gasket of the slow leaks (SL) at 11 and 18 psig and the drum failure at 47 psig.

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3. H.W. Heacock and J.W. Riches, "Waste Solidification - Cement or Urea Formaldehyde", American Society of Mechanical Engineers, 74-WA/NE-9, 1974.
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Appendix A

Waste Formulations

The following are the chemical formulations of the waste types included in the experimental program. The temperature and pH listed are typical values prior to solidification at reactor sites. Slurry and dewatered formulations are listed for bead resin and BWR precoat filter cake wastes. The slurry formulations are meant to represent wastes that are pumpable for short distances.

1a. BEAD RESIN WASTE (Slurry)

<u>Material</u>	<u>Weight Percent, %</u>
Water	50.
Bead Resin (IRN-150) ^a	50.
Temperature	70°F
pH	7

1b. BEAD RESIN WASTE (Dewatered)

<u>Material</u>	<u>Weight Percent, %</u>
Water	35.
Bead Resin (IRN-150) ^a	65.
Temperature	70°F
pH	7

2a(1), BWR PRECOAT FILTER CAKE WITH POWDERED RESIN (Slurry)

<u>Material</u>	<u>Weight Percent in Filter Cake, %</u>
Water	50.
Anion Powdered Resin (PAO) ^b	20.
Cation Powdered Resin (PCH) ^b	20.
Crud ^c	5.
Sodium Chloride	5.
Temperature	70°F
pH	7

2a(2). BWR PRECOAT FILTER CAKE WITH POWDERED RESIN (Dewatered)

<u>Material</u>	<u>Weight Percent in Filter Cake, %</u>
Water	32.
Anion Powdered Resin (PAO) ^b	30.
Cation Powdered Resin (PCH) ^b	30.
Crud ^c	6.
Sodium Chloride	2.
Temperature	70°F
pH	7

2b(1). BWR PRECOAT FILTER CAKE WITH DIATOMACEOUS EARTH (Slurry)

<u>Material</u>	<u>Weight Percent in Filter Cake</u>
Water	75.0
Diatomaceous Earth	20.0
Crud ^c	5.0
Temperature	70°F
pH	7

2b(2) BWR PRECOAT FILTER CAKE WITH DIATOMACEOUS EARTH (Dewatered)

<u>Material</u>	<u>Weight Percent in Filter Cake</u>
Water	60.
Diatomaceous Earth	30.
Crud ^c	10.
Temperature	70°F
pH	7

3a. BWR CHEMICAL REGENERATIVE WASTE OF A FORCED RECIRCULATION EVAPORATOR

<u>Material</u>	<u>Weight Percent In Evaporator Bottoms, %</u>
Water	75.
Sodium Sulfate	22.9
Sodium Chloride	2.0
Crud ^c	0.1
Temperature	170°F
pH	6

3b. PWR CHEMICAL REGENERATIVE WASTE OF A FORCED RECIRCULATION EVAPORATOR

<u>Material</u>	<u>Weight Percent in Evaporator Bottoms, %</u>
Water	73.4
Sodium Sulfate	14.9
Ammonium Sulfate	9.6
Sodium Chloride	2.0
Crud ^C	0.1
Temperature	170°F
pH	2.5 to 4.0

3c. BORIC ACID WASTE OF A FORCED RECIRCULATION EVAPORATOR

<u>Material</u>	<u>Weight Percent in Evaporator Bottoms, %</u>
Water	87.9
Boric Acid	12.0
Crud ^C	0.1
Temperature	170°F
pH	3.5

3d. DECONTAMINATION WASTE OF A FORCED RECIRCULATION EVAPORATOR

<u>Material</u>	<u>Weight Percent in Evaporator Bottoms, %</u>
Water	80.
Nutek NT-700 ^d	9.4
EDTA	5.
Citric Acid	5.
Crud ^C	0.2
Hydraulic Oil No. 2	0.2
Lubricating Oil No. 20	0.2
Temperature	170°F
pH	5

4a. BWR CHEMICAL REGENERATIVE WASTE OF A THIN FILM EVAPORATOR

<u>Material</u>	<u>Weight Percent in Evaporator Bottoms, %</u>
Water	50.
Sodium Sulfate	45.8
Sodium Chloride	4.0
Crud ^C	0.2
Temperature	150 to 250°F
pH	6

4b. PWR CHEMICAL REGENERATIVE WASTE OF A THIN FILM EVAPORATOR

<u>Material</u>	<u>Weight Percent in Evaporator Bottoms, %</u>
Water	50.
Sodium Sulfate	29.
Ammonium Sulfate	16.8
Sodium Chloride	4.0
Crud ^c	0.2
Temperature	150 to 250°F
pH	1.8 to 4.0

4c. BORIC ACID WASTE OF A THIN FILM EVAPORATOR

<u>Material</u>	<u>Weight Percent in Evaporator Bottoms, %</u>
Water	50.
Boric Acid	49.8
Crud ^c	0.2
Temperature	150 to 250°F
pH	2.5 to 3.5

4d. DECONTAMINATION WASTE OF A THIN FILM EVAPORATOR

<u>Material</u>	<u>Weight Percent in Evaporator Bottoms, %</u>
Water	50.
Nutec NT-700 ^d	20.
EDTA	9.8
Citric Acid	19.
Crud ^c	0.2
Hydraulic Oil No. 2	0.5
Lubricating Oil No. 20	0.5
Temperature	150 to 250°F
pH	5

^a Rohm and Haas Co., Philadelphia, Pa. 19105

^b Ecodyne Corp., Union, N.J. 07083

^c fine air cleaner test dust no. 1543094, AC Spark Plug Division, General Motors Corp., Flint, Michigan 48556

^d Nuclear Technology Corp., Amston, Conn. 06231

Appendix B

Proposed Standard Static Leach Test Procedure for Solidified Radioactive Wastes

(This procedure is based on that proposed by the IAEA with minor modifications)

I. INTERCOMPARISON METHOD

1. Requirements for Specimen and Leachant Containers

1.1 Materials of Construction

1.1.1 Specimen Container

The specimen container shall be constructed of a material which:

- (a) will not react chemically with the specimen
- (b) is not sorbent towards the ions extracted from the sample by the leachant

- (c) will withstand the conditions involved in sample preparation

If the leach test is performed with the specimen in the specimen container, the material from which the specimen container is constructed must meet these additional criteria:

- (d) it will not react chemically with the leachant
- (e) it will not be affected by the radiation dose received during testing
- (f) it is not sorbent towards the ions originally present in the leachant

It is recommended, whenever possible, that the specimen not be leach tested in its specimen container.

1.1.2 Leachant Container

The container for the leachant shall be constructed of a material which:

- (a) will not react chemically with the leachant
- (b) is not sorbent towards the ions extracted from the sample by the leachant
- (c) will not be affected by the radiation dose received during the test

1.1.3 A blank run will be made to determine the leach rate of the apparatus.

1.2 Dimensions and Form

- 1.2.1 The specimen container shall be of cylindrical form open at one end, or in the form of a support or envelope with sufficient openings and so shaped that maximum contact between the leachant and all exposed specimen surfaces is achieved. The specimen container dimension and specimen size may vary and will be selected so that the procedure being used for analyzing the leached material in the leachant will have satisfactory accuracy. In the event that a granulated specimen is required for a material of very low leachability, the size of the individual granules must be large enough so that an appreciable portion of each specimen particle will maintain its original composition throughout the tests. This requirement will be assumed to be met if the data from replicate analyses agree.
- 1.2.2 The dimensions of the leachant (outer) container shall be such that the exposed surface of the sample be covered by at least 2.54cm of leachant. If the specimen is not covered to this depth with leachant, the depth used along with the reason for deviation shall be reported.

2. Requirements for Specimen Preparation

2.1 Sampling

Suitable precautions shall be taken to ensure that the sample is representative of the process material, and that the homogeneity of the test specimen is the same as that of the process material.

2.2 Sample Preparation

2.2.1 Cement or Concrete/Waste Mixtures

The mixtures shall be cast in a convenient form which facilitates surface area and volume calculations. The specimen shall be cured in a manner representative of the conditions expected to be used in actual waste processing. The history of the specimen during preparation and between the time of preparation and leaching

shall be recorded, including temperature, humidity, storage atmosphere, and cure time. It is preferable that the specimen be removed from its container for leaching, although it may be leached in the container in which it was cast. If desired, new surfaces may be exposed by sawing or other techniques, or part of the surface may be coated with a waterproofing material. In any case, the surface area exposed to the leachant shall be known. Whenever possible representative specimens should be obtained from actual waste forms.

2.2.2 Urea-Formaldehyde Waste Mixtures

The mixtures shall be cast in a convenient form which facilitates surface area and volume calculations. The specimen shall be prepared in a manner representative of the conditions expected to be used in actual waste processing. The history of the specimen during preparation and between the time of preparation and leaching shall be recorded, including temperature, humidity, storage atmosphere, and elapsed time. It is preferable that the specimen be removed from its container for leaching, although it may be leached in the container in which it was cast. If desired, new surfaces may be exposed by sawing or other techniques, or part of the surface may be coated with a waterproofing material. In any case, the surface area exposed to the leachant shall be known. Whenever possible representative specimens should be obtained from actual waste forms.

2.2.3 Bitumen Waste Mixtures

The mixtures shall be cast in a convenient form which facilitates surface area and volume calculations. The specimen shall be prepared in a manner representative of the conditions expected to be used in actual waste processing. The history of the specimen during preparation and between the time of preparation and leaching shall be recorded, including temperature, humidity, storage atmosphere, and elapsed time. It is preferable that the specimen be removed from its container for leaching, although it may be leached

in the container in which it was cast. If desired, new surfaces may be exposed by sawing or other techniques, or part of the surface may be coated with a waterproofing material. In any case, the surface area exposed to the leachant shall be known. Whenever possible representative specimens should be obtained from actual waste forms.

3. Test Method

3.1 Leachant

The leachant shall be distilled or deionized water.

3.1.1 The pH of the leachant shall be determined and recorded.

3.2 Temperature of the Test

The leaching shall be performed at a temperature of $25^{\circ}\pm 5^{\circ}\text{C}$ and the temperature of the test recorded to the nearest degree.

3.3 Procedure

3.3.1 Prior to placing in the leachant solution container, all fines and dust, such as might result from sawing or crushing a granulated sample shall be removed from the specimen by a technique that is not expected to result in a removal of the species of interest from the specimen. Free standing water, if present, will be added to the leachant solution container.

3.3.2 The specimen shall be placed in the leachant container and sufficient leachant added to ensure that the specimen is covered by at least 2.54cm. The ratio of the volume of leachant to the external geometric surface area of the specimen exposed to the leachant shall not be less than 2.5cm for monolithic specimens and shall not for any specimens exceed a ratio which results in an appreciable decrease in leachant analysis accuracy. The leachant shall be added carefully so as not to impinge directly on the exposed face of the specimen.

3.3.3 The leachant container shall be lightly covered and allowed to stand undisturbed until sampling is necessary.

3.3.4 At sampling the specimen shall be removed from the leachant container and the leachant thoroughly mixed. During sampling of the leachant, care will be taken to prevent any solid residue from following the solution.

A sample of the leachant sufficient for replicate analysis shall be taken and the remainder discarded.

Leachant shall be allowed to drain off the specimen; however, the specimen will not be rinsed.

The leachant container will be rinsed after which the specimen will be replaced and covered with a fresh portion of leachant of the same volume and the test shall proceed.

3.4 ~~Sampling~~ Frequency

3.4.1 At ten minutes after the start of the test

3.4.2 After one hundred minutes of leaching

3.4.3 After six hours of leaching

3.4.4 After twenty-four hours of leaching

3.4.5 After each successive twenty-four hour period during the work week for at least the first four weeks. Leaching should begin on Monday whenever possible.

3.4.6 Once per week during the following five months.

3.4.7 Once per month for the following six months and then,

3.4.8 Twice per year for as long as is considered necessary.

3.4.9 Sampling may be performed more frequently than expressed in this section.

3.5 Analysis

3.5.1 The leachant sample shall be mixed and allowed to settle

3.5.2 On the clear supernatant solution the following determinations shall be made and recorded:

3.5.2.1 pH value

- 3.5.2.2 Analysis of the desired constituents leached from the specimen by the best available means.
- 3.5.3 The methods of analysis used, together with the standard deviation and the rated accuracy and precision of the method shall be reported.
- 3.5.4 The same analytical methods shall be used to determine the concentrations of specific elements in the mixture of waste/immobilizing material and in the sample of the leachant. Should this not be practical, an assessment should be made of the effect on the results of the use of different methods and reported with the results.

4. Reporting of Results

- 4.1 Information concerning the specimen tested when reporting the results of leaching tests, the following information concerning the specimen tested shall be reported:
 - 4.1.1 The proportion by weight or volume of waste incorporated in the mixture;
 - 4.1.2 The type, chemical and radioisotopic composition, and specific activity of the waste incorporated in the mixture;
 - 4.1.3 The type and composition of the immobilizing material including any additives or catalyzing agent;
 - 4.1.4 Any departures from the specified methods for specimen preparation.
 - 4.1.5 The history of the specimen between preparation and leaching.
- 4.2 Information concerning the test procedure.
 - 4.2.1 The volume of leachant shall be reported.
 - 4.2.2 Any departures from the specified testing procedure shall be reported together with the reasons for the departure.
- 4.3 Reporting the leach results.

4.3.1 The amount of each element leached or radioactivity proportional to the amount leached (a_n), during each leachant renewal period (n) shall be presented in tabular form along with the duration, in days, of the leachant renewal period (t_n). The following information shall accompany the tabulated leach data:

A_0 = total amount, or radioactivity proportional to the total amount, of the element initially present in the specimen.

S = exposed geometric surface area of the specimen (cm^2).

V = volume of the specimen (cm^3).

M = mass of the specimen (g).

The method used to determine the surface area and volume of the specimen shall be described.

The intent of the foregoing is to assure that the "raw" data are made available in an accessible and unambiguous form.

It is recommended that the data be tabulated as (a_n/A_0) (V/S) ($1/t_n$) with the units of (fraction leached) (cm) (day^{-1}). It is suggested that $(\Sigma a_n/A_0)$ (V/S) also be tabulated.

4.3.2 The results may, in addition, be expressed graphically, by, for instance, $(\Sigma a_n/A_0)$ (V/S) as a function of the total time of leaching.

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