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PROPERTIES OF RADIOACTIVE WASTES  
AND WASTE CONTAINERS

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PROGRESS REPORT No. 7  
OCTOBER - DECEMBER 1977

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

DATE PUBLISHED - MAY 1978

NUCLEAR WASTE MANAGEMENT RESEARCH GROUP

DEPARTMENT OF NUCLEAR ENERGY BROOKHAVEN NATIONAL LABORATORY  
UPTON, NEW YORK 11973



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DEPARTMENT OF NUCLEAR ENERGY  
BROOKHAVEN NATIONAL LABORATORY  
ASSOCIATED UNIVERSITIES, INC.  
UPTON, NEW YORK 11973

PREPARED FOR THE UNITED STATES NUCLEAR REGULATORY COMMISSION  
OFFICE OF NUCLEAR REGULATORY RESEARCH  
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## SUMMARY

Trial solidifications were made using portland type II cement - sodium silicate as the solidification agent. The sodium silicate was found to produce an initial rapid set for all wastes because of the precipitation of relatively insoluble silicate compounds upon reaction with soluble multivalent cations in solution in the cement-waste mixture. Achievement of the ultimate waste form strength required time intervals similar to waste forms produced using portland cement alone. A hard waste form was not obtained within seventy-eight days with formulations used to solidify boric acid waste.

The flash points and flame points of Pioneer 221 bitumen and bitumen waste forms were determined using the Cleveland open cup method. The bitumen alone had a flash point of  $610 \pm 2^{\circ}\text{F}$  and a flame point of  $668 \pm 4^{\circ}\text{F}$ . The bitumen waste forms exhibited similar flame points but slightly higher ( $15\text{-}20^{\circ}\text{F}$ ) flash points were measured. This is presumably due to the removal of some of the more volatile bitumen components during the extruder solidification process. The flash and flame points for boric acid - bitumen waste forms could not be determined due to foaming.

Self-irradiation exposure dose curves were calculated for BWR and PWR waste forms. These curves indicate the cumulative waste form exposure dose with time and serve as the basis for radiation stability experiments. Waste form specific activities of  $0.01\text{-}100\text{ Ci/ft}^3$  were considered.

Bitumen waste forms containing sodium sulfate from the solidification of BWR chemical regenerative waste were prepared by Werner and Pfleiderer Corp. personnel and leach tested at Brookhaven National Laboratory. These specimens swelled and cracked during leach testing, exposing substantial new surface area to the leachant. This effect is due to the conversion of the contained anhydrous sodium sulfate to its decahydrate salt and was found to occur in samples containing 17.5 to 44.3 weight percent sodium sulfate. The sodium sulfate leach rate was also independent of the salt content over this range, the leachant, and specimen size with an average cumulative sodium sulfate release rate of 0.1 cm/day.

The volumetric efficiencies of urea-formaldehyde and portland type II cement for various wastes and waste/binder ratios were calculated from compression test specimen data.

## I. WASTE SOLIDIFICATION USING PORTLAND CEMENT - SODIUM SILICATE

Portland cement and sodium silicate are used in combination for the solidification of power reactor radioactive wastes. The addition of sodium silicate is reported to increase the compatibility of portland cement with problem wastes such as boric acid and to permit the solidification of increased quantities of liquid waste per unit waste form volume. (1,2) Trial solidifications of simulated wastes were made using portland type II cement with sodium silicate.

Trial solidifications utilized the formulations listed in U.S. Patent 3,988,258 for the solidification of various wastes with portland cement and sodium silicate. These formulations are listed in Table 1. Prior to solidification, the pH of the regenerative wastes was adjusted to 7 and the pH of the boric acid waste adjusted to 10 by the addition of a saturated (25°C) sodium hydroxide solution. The waste and the cement were mixed thoroughly by hand stirring in the solidification container, which was a two inch diameter by four inch high cylindrical polyethylene vial. The appropriate quantity of 40-42° Be aqueous sodium silicate solution was then added and quickly stirred into the waste and cement mixture. The formulations for each waste solidification demonstrated a rapid initial set occurring approximately two minutes after addition of the sodium silicate. The ultimate waste form strength, as determined by a qualitative hardness-penetration observation, required approximately seven days for the bead resin, powdered resin, and BWR chemical regenerative waste form formulations. The PWR chemical regenerative waste form formulation required approximately twenty-five days for ultimate strength development. Each of these formulations resulted in hard monolithic solids comparable to the ultimate hardness of cement waste forms. The boric acid waste form formulation did not produce a hard solid within seventy-eight days. A soft waste form with limited resistance to penetration was formed, similar in consistency to the waste form after the initial rapid set. Increasing the quantity of cement or sodium silicate in the initial boric acid formulation as shown in Table 1 did not produce a hard solid within seventy days. While the boric acid waste forms produced were soft solids of limited strength, they

TABLE 1

Solidification of Simulated Wastes with Portland Type II Cement - Sodium Silicate

	<u>Waste Type</u>	<u>Formulation, Parts by Weight</u>			<u>Waste/Binder Ratio, by Weight (a)</u>	<u>Comments</u>
		<u>Waste</u>	<u>Cement</u>	<u>Sodium Silicate</u>		
1a	Bead Resin Waste (slurry)	100	25	5	3.33	--
2a(1)	Powdered Resin Waste (slurry)	100	25	10	2.86	--
3a	BWR Chemical Regenerative Waste	100	50	10	1.67	waste pH adjusted to 7
3b	PWR Chemical Regenerative Waste	100	50	10	1.67	waste pH adjusted to 7
3c	Boric Acid Waste	100	70	25	1.05	waste pH adjusted to 10
	(b)	100	80	25	0.95	waste pH adjusted to 10
	(b)	100	70	35	0.95	waste pH adjusted to 10

(a) binder = cement + sodium silicate

(b) adjustments to initial formulation

were monolithic free standing solids and did not have substantial associated quantities of free standing water. None of the waste forms produced exhibited substantial free standing water.

Sodium silicate reacts with metallic ions having a valence of two or higher in solution to form precipitates with low solubilities, often resulting in a gelatinous coagulated mass. The initial rapid setting of the waste-cement-sodium silicate mixtures is probably due to this effect. Calcium and aluminum ions go into solution when portland cement is mixed with water. These ions, together with any metallic ions having a valence of +2 or higher in the waste, react with the sodium silicate which is added last. The resulting silicate precipitates cause a gellation of the waste form mass producing the initial rapid set. Care must be taken with mixing, however, since mixing after the initial set (or after formation of substantial precipitates) will break up the gelatinous mass resulting in a mixture with a consistency similar to that of waste and cement alone.

Note that the waste to binder ratios used in these trial solidifications are higher than those utilized in this program for solidification with portland cement alone. While the literature suggests that the cement-sodium silicate system produces acceptable solidified products at waste/binder ratios in excess of those used with portland cement, no such comparison has been made to date in this program. Rather, in the absence of more complete information, the waste/binder ratios suggested in the cement-sodium silicate system patent have been used. Improvements in product properties may result from changes in the waste/binder ratio and the relative additions of cement and sodium silicate.

## II. FLASH POINT AND FIRE POINT OF BITUMEN WASTE FORMS

The flash point and fire point of bitumen and bitumen waste forms were measured using the method of ASTM D 92-72, "Standard Method of Test for Flash and Fire Points by Cleveland Open Cup". In this technique, a test cup is filled to a specified level with the sample to be tested. The temperature of the sample is increased and the flash point is approached at a slow constant rate of temperature increase. At 5°F intervals, a test flame is passed across the cup. The lowest temperature at which application of the test flame causes the vapors above the specimen surface to ignite is the flash point. The test is continued and the lowest temperature at which the test flame causes the sample to ignite for at least five seconds is the fire point.

Flash and fire point measurements were made for samples of Pioneer 221 asphalt<sup>(1)</sup> and Werner and Pfleiderer Corporation extruder specimens of Pioneer 221 asphalt used to solidify water, BWR chemical regenerative wastes (23 wt.% Na<sub>2</sub>SO<sub>4</sub>) and boric acid waste (12 wt.% H<sub>3</sub>BO<sub>3</sub>). The water solidification product is nominally 99.5 wt.% bitumen while the BWR chemical regenerative waste and boric acid waste products nominally contain 50 wt.% salt. Both the BWR chemical regenerative waste and boric acid wastes were neutralized to a pH of 7 by the addition of sodium hydroxide prior to passing through the extruder. Duplicate measurements were made for each specimen. The results are shown in Table 2.

Pioneer 221 asphalt is represented as having a minimum flash point of 550°F.<sup>(3)</sup> Cleveland open cup measurements indicated a flash point of 610±2°F with a flame point of 668±4°F. The samples from the solidification of water and sodium sulfate wastes showed a small increase in the flash point, however, no effect on the flame point was noted. The increase in the flash point may be due to the removal of some volatile components from the bitumen during the extruder solidification process. A small quantity of residual water was present in the extruder samples as indicated by the presence of water bubbles on the specimen surface during heating. Flash point and flame point determinations could not be made using neutralized boric acid waste

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(1) Witco Chemical Corporation, Perth Amboy, New Jersey 08862

forms. This was due to substantial foaming which occurred during specimen heating.

TABLE 2

Cleveland Open Cup Flash and Flame Point  
Determinations for Bitumen Waste Forms  
(ASTM D 92-72)

<u>Waste Type</u> <sup>(a)</sup>	<u>Flash Point, °F</u>	<u>Flame Point, °F</u>
None	610±2	668±4
Water	630±10	665±3
BWR Chemical Regenerative Waste	625±5	670±10
Boric Acid Waste	(b)	(b)

(a) specimens prepared using Pioneer 221 asphalt

(b) determinations could not be made due to specimen foaming

### III. EXPOSURE DOSE OF SOLIDIFIED WASTE FORMS

As a prelude to additional waste form radiation stability experiments, the cumulative radiation exposure dose received by BWR and PWR solidified waste forms was calculated as a function of time. The radiation doses were calculated for a range of waste form specific activity of 0.01-100 Ci/ft<sup>3</sup> and were based on the relative percentage of each radionuclide present in BWR and PWR solid wastes as listed in Tables 3 and 4. It is assumed that solidification takes place 180 days after waste generation. The dose contributions of each radionuclide as a function of time were calculated individually, including decay, to arrive at the cumulative self-radiation exposure dose curves shown in Figures 1 and 2 for BWR and PWR solidified waste forms respectively. The calculations also assumed that the activity was uniformly distributed in a waste form with a density of 2.0 g/cm<sup>3</sup>. For both the BWR and PWR waste forms, the cumulative dose is proportional to the waste form specific activity at any given time.

Ranges of observed activity levels in BWR and PWR solidified radwaste are shown in Table 5 together with typical generation rates. The activity content and hence the radiation dose to which a waste form is exposed is dependent upon the type of waste solidified. Doses for the observed activity concentration ranges are spanned by the curves given in Figures 1 and 2. Because of the half-lives of the contained radionuclide species, greater than 90% of the total cumulative dose is received in the first 100 years. The cumulative doses calculated here are comparable to the 10<sup>7</sup> rad integrated dose over 100 years determined by Burns for waste forms with a specific activity of the order of 10<sup>-1</sup> curies/liter (2.8 curies/ft<sup>3</sup>).<sup>(6)</sup>

TABLE 3

Calculated Radioactivity Content of Radioactive  
Materials in Solid Wastes - BWR<sup>4</sup>

<u>Nuclide</u>	<u>Half-Life</u>	<u>Decay Energy, MeV</u>	<u>Activity<sup>(a,b)</sup> Ci/Yr/Reactor (Cases 2-5)</u>	<u>% Total</u>
Sr-89	52.7d	1.47	49	3.16
Sr-90	27.7y	0.546	90	5.81
Zr-95	65d	1.12	1.1	0.07
Ru-106	368d	0.04	2.1	0.14
Te-127m	109d	0.089	1.6	0.10
Cs-134	2.0y	2.06	130	8.39
Cs-137	30.0y	1.18	120	7.75
Ce-141	32.5d	0.58	0.25	0.02
Ce-144	284d	0.32	5.1	0.33
Cr-51	27.8d	0.75	0.60	0.04
Mn-54	303d	1.38	13	0.84
Fe-55	2.6y	0.231	800	51.63
Fe-59	45.6d	1.56	6.6	0.43
Co-58	71.4d	2.31	130	8.39
Co-60	5.3y	2.82	200	12.91
TOTAL			1.6x10 <sup>3</sup>	100.0

(a) After 180-day decay.

(b) Two 3500 MW(t) reactors per station in each case.

TABLE 4

Calculated Radioactivity Content in Solid Waste - PWR<sup>4</sup>

<u>Nuclide</u>	<u>Half-Life</u>	<u>Decay Energy, MeV</u>	<u>Activity<sup>(a,b)</sup> Ci/Yr/Reactor (Cases 1-6)</u>	<u>% Total</u>
Sr-89	52.7d	1.47	2.0	0.03
Sr-90	27.7y	0.546	2.7	0.04
Zr-95	65d	1.12	0.64	0.01
Ru-103	39.5d	0.75	0.10	0.00
Ru-106	368d	0.04	1.7	0.03
Te-127m	109d	0.089	11	0.18
Te-129m	34.1d	0.106	1.3	0.02
Cs-134	2.0y	2.06	2.8x10 <sup>3</sup>	45.54
Cs-137	30.0y	1.18	2.7x10 <sup>3</sup>	43.92
Ce-144	284d	0.32	4.2	0.07
Cr-51	27.8d	0.75	0.35	0.01
Mn-54	303d	1.38	22	0.36
Fe-55	2.6y	0.231	200	3.25
Fe-59	45.6d	1.56	1.8	0.03
Co-58	71.4d	2.31	120	1.95
Co-60	5.3y	2.82	280	4.55
			6.1x10 <sup>3</sup>	100.0

(a) After 180-day decay.

(b) Two 3500 Mw(t) reactors per station in each case.

TABLE 5  
LWR Solidified Radwaste<sup>5</sup>

	<u>Evaporator Bottoms</u>	<u>Filter/ Demineralizer Resins</u>	<u>Filter/ Demineralizer Sludge</u>
<u>BWR</u> Generation rate (ft <sup>3</sup> /MWe)	25.5	4.0	19.5
Activity content (Ci/ft <sup>3</sup> )			
Range	0.0003-0.14	0.001-6.5	0.004-20.0
Average	0.039	2.0	0.2
Average Total Activity (Ci/MWe)	1.0	8.0	3.9
<u>PWR</u> Generation rate (ft <sup>3</sup> /MWe)	30.7	2.7	-
Activity content (Ci/ft <sup>3</sup> )			
Range	0.002-0.4	0.03-37.0	-
Average	0.045	4.5	-
Average Total Activity (Ci/MWe)	1.4	12	-

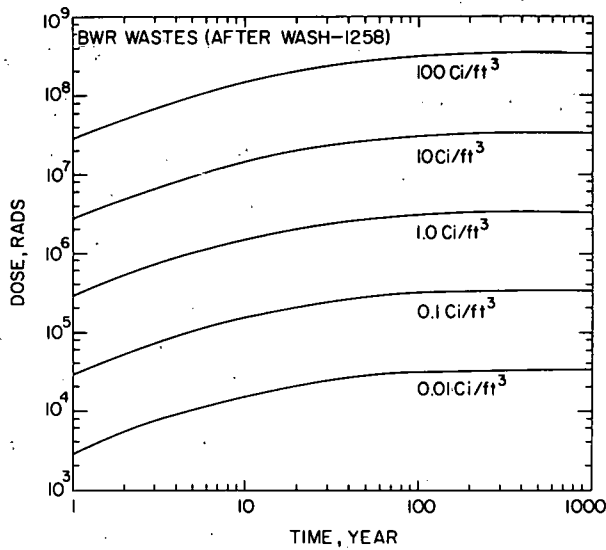


Figure 1. Waste Form Self-Irradiation Exposure Dose with Time, BWR Waste.

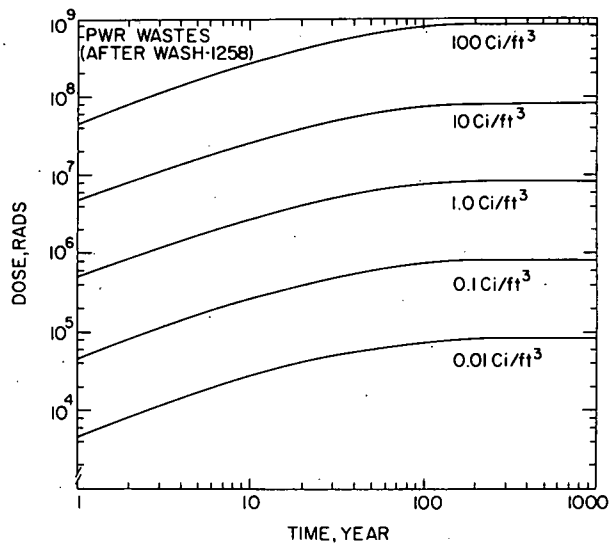


Figure 2. Waste Form Self-Irradiation Exposure Dose with Time, PWR Waste.

#### IV. LEACHING OF BITUMEN WASTE FORMS

A test program was conducted in December 1977 during which various simulated waste streams listed in Appendix A were solidified in bitumen using a Werner and Pfleiderer Corporation<sup>(a)</sup> Model ZSKT-53 twin screw extruder. The extruder was set up and operated for this test program by Werner and Pfleiderer personnel in the presence of observers from Brookhaven National Laboratory. Waste and asphalt feed rates were selected to produce an acceptable product containing approximately 50 wt.% waste solids. The extruder screw speed and barrel temperature profile were adjusted to allow essentially complete evaporation of the water from the waste. This machine has three heat zones and four steam domes. Six thermocouples were located along the length of the extruder barrel for monitoring purposes. Each waste stream was adjusted to pH  $\sim$ 7 by the addition of sodium hydroxide when necessary to minimize corrosion effects in the extruder. Detailed operating parameters for this test sequence are found in Appendix B. Samples obtained were taken to Brookhaven National Laboratory for testing. In this section, the leach properties of bitumen waste forms produced during the solidification of BWR chemical regenerative waste will be described.

In the screw extruder solidification technique, bitumen and liquid or slurry wastes are continuously pumped into one end of an extruder which may contain one or multiple screws. The design and operation of the extruder is such that the bitumen and waste are intimately mixed and spread into a thin film on the heated surface of the extruder barrel. Depending on particle size and screw clearance, solid particles in the waste are comminuted. This mechanical processing together with the maintenance of a temperature approaching 200°C can affect the almost complete (99.5%) evaporation of the water contained in the waste and provides a homogeneous product. Final product water content is a function of waste water content, extruder barrel temperature profile and residence time. The evaporated water is vented through large disengaging sections called steam domes and passed through an oil separator before being

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<sup>(a)</sup> Werner and Pfleiderer Corporation, Waldwick, New Jersey 07463

condensed. Carryover of activity to the condensate is reported to be of the order of 0.01%.<sup>(7)</sup> Typical waste residence time in the extruder is a few minutes. The bitumen waste mixture is discharged directly into solidification containers at the end of the extruder and allowed to cool. An excellent description of this process is found in the literature.<sup>(7)</sup>

Leach samples of the  $\text{Na}_2\text{SO}_4$  - bitumen product produced by the solidification of BWR chemical regenerative waste were collected in silicone rubber coated cardboard tubes with a diameter of two inches and four inches in length. A few samples with a three inch diameter and six inches in length were also obtained by a similar technique. The bitumen waste forms exhibited substantial shrinkage after cooling and solidification. As such, each solidification container was "topped off" by a second pass under the extruder discharge port after the initial fill had cooled. This procedure is routinely used for full scale bitumen waste forms to more completely utilize the solidification container volume. Based on the average waste and asphalt feed rates, these samples contained 46.0 wt.%  $\text{Na}_2\text{SO}_4$  and had a waste solids/bitumen weight ratio of 1.02.

Leach samples were tested by the static method described in Appendix C. In this method, samples are leach tested with their entire surface area exposed to the leachant solution. A leachant volume to sample surface area ratio of 10 cm was employed. The leachant was replaced daily (except weekends) with a sample taken for analysis. Leachant analysis determined the quantity of sodium sulfate released from the specimens by a colorimetric technique for  $\text{SO}_4^{-2}$  using a Technicon autoanalyzer.

Leaching is expressed as the cumulative fraction sodium sulfate release  $\times$  (V/S) where V is the leach specimen volume and S is its external geometric surface area (based on the original specimen dimensions). Two inch diameter by four inch cylindrical specimens were tested in triplicate in distilled water (1A series) and duplicate in ground water (1C series). One three inch diameter by six inch long specimen was leached in distilled water (1D2).

The  $\text{Na}_2\text{SO}_4$  - bitumen samples exhibited a tendency to swell, crack, and break up during leach testing. This is shown in Figures 3 and 4 for sample 1A1 and Figures 5-7 for sample 1D1. The control sample in these figures indicates the initial specimen dimensions and geometry. This effect occurred in all leachants, however, severe sample degradation required a somewhat longer time for the larger leach specimen. The degradation process was essentially complete after seven days for the two inch diameter by four inch specimens and fourteen days for the three inch diameter by six inch specimen. This behavior is due to the tendency of the sodium sulfate (initially present in an anhydrous form) to combine with water to form  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ . Anhydrous sodium sulfate has a density of  $2.68 \text{ g/cm}^3$  while the decahydrate salt has a density of  $1.46 \text{ g/cm}^3$ . As such, one mole of the anhydrous salt occupies a volume of  $53.0 \text{ cm}^3$  while a mole of the decahydrate salt has a volume of  $220 \text{ cm}^3$ . This increase in the sodium sulfate volume causes an expansion of the waste form and ultimately its disintegration since the bitumen cannot elastically expand to accommodate the volume increase. Since this degradation process increases the specimen surface area exposed to the leachant, it is expected to have an affect on the leach rate.

After leach testing was completed, the leach specimens were dissolved in carbon tetrachloride and a solvent extraction process used to remove the remaining sodium sulfate. Subsequent analysis showed that essentially no sodium sulfate was present in the samples after leaching. The quantity of sodium sulfate removed during leaching revealed that the samples did not contain the assumed 46.0 wt.% sodium sulfate, but rather the sodium sulfate content varied from 17.5 to 44.3 wt.%. This was presumably due to variation in the waste stream feed rate during sample preparation. However, it did serve to allow observation of leach rates over a range of compositions.

Figure 8 illustrates the cumulative fraction  $\text{Na}_2\text{SO}_4$  release for leaching in distilled water for the two inch diameter by four inch long leach specimens. These specimens had initial sodium sulfate contents of from 17.5 to 44.3 percent by weight. The rate of release during leaching was found to be independent of the initial sodium sulfate content. This is also true for the 1C

series samples containing 24.5 and 33.6 wt.% sodium sulfate which were leached in groundwater as shown in Figure 9. The average leach rates were essentially equal for the two inch diameter by four inch long specimens independent of the leachant. For the first nine days of leaching, the average cumulative fraction  $\text{Na}_2\text{SO}_4$  release x (V/S) was 0.108 cm/day and 0.104 cm/day for leaching in distilled water and groundwater respectively. The sodium sulfate releases were approximately linear with time from the initiation of leaching until day nine, at which time little sodium sulfate remained in the specimens. The leach rate in distilled water for the three inch diameter by six inch long cylindrical sample is shown in Figure 10. The sodium sulfate release rate for this sample was 0.121 cm/day over the first nine days and 0.0960 cm/day over the first fifteen days (96.5%  $\text{Na}_2\text{SO}_4$  release). As such, the leach rate for the three inch diameter samples is essentially the same as the leach rates determined for the two inch diameter by four inch long specimens. Preparation information and tabulated leaching data for series 1A, 1C, and 1D specimens are found in Appendix D.

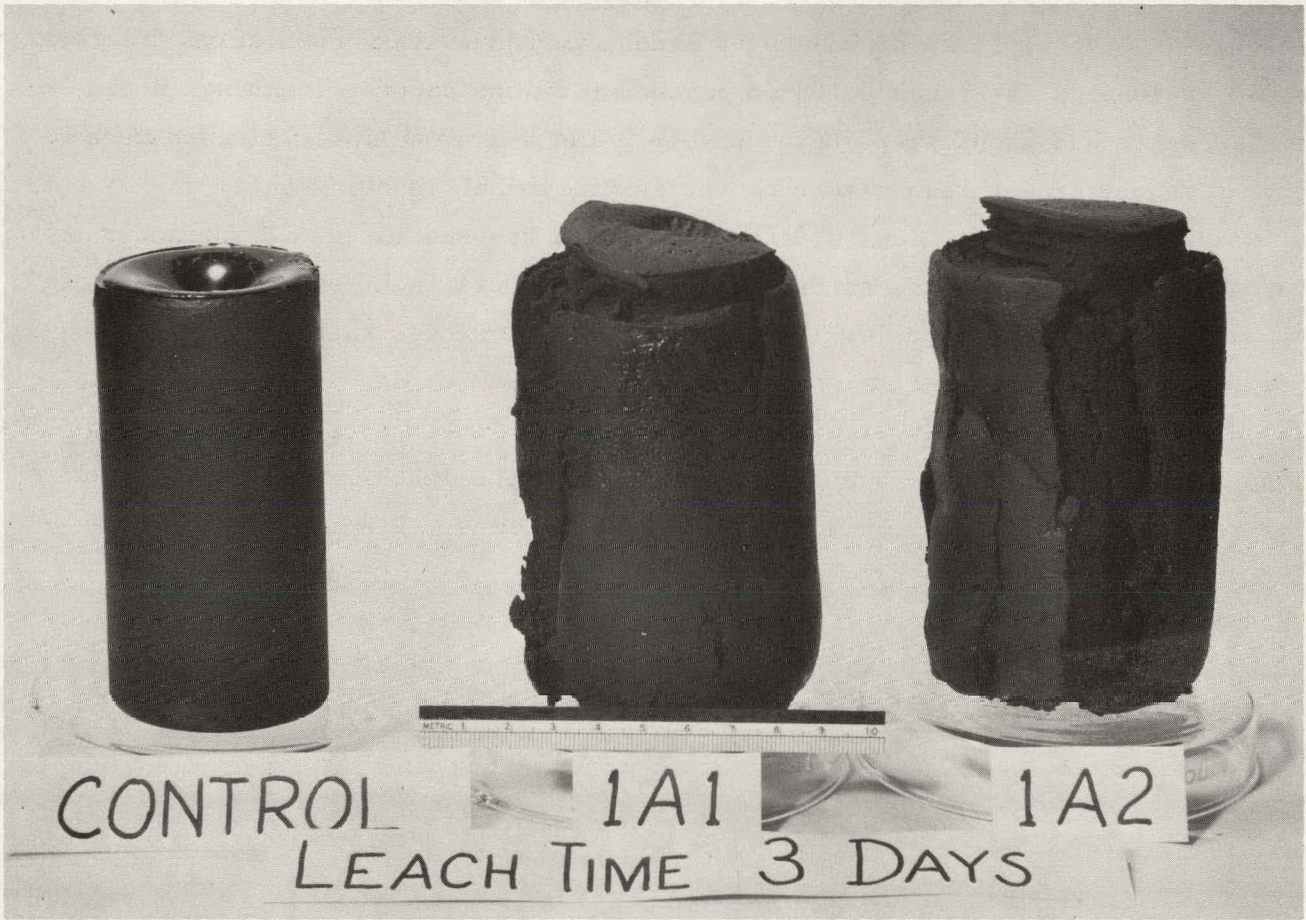


Figure 3. Bitumen Waste Form 1A1 (28.5 Wt.%  $\text{Na}_2\text{SO}_4$ ) after Three Days of Leaching in Distilled Water.



Figure 4. Bitumen Waste Form 1A1 (28.5 Wt.%  $\text{Na}_2\text{SO}_4$ ) after Seven Days of Leaching in Distilled Water.

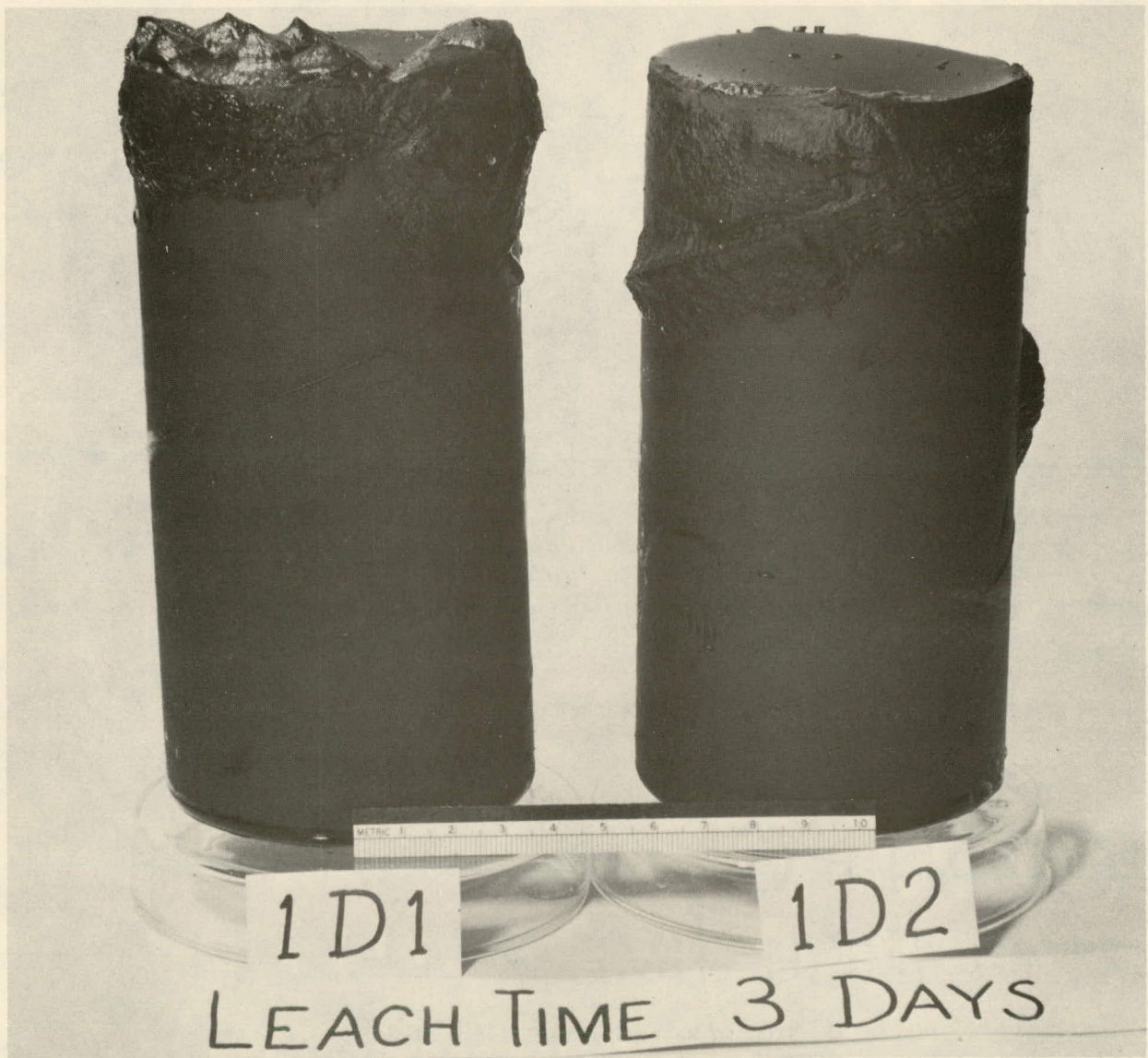


Figure 5. Bitumen Waste Form 1D2 (41.4 Wt.%  $\text{Na}_2\text{SO}_4$ ) after Three Days of Leaching in Distilled Water.



Figure 6. Bitumen Waste Form ID2 (41.4 Wt.%  $\text{Na}_2\text{SO}_4$ ) after Seven Days of Leaching in Distilled Water.

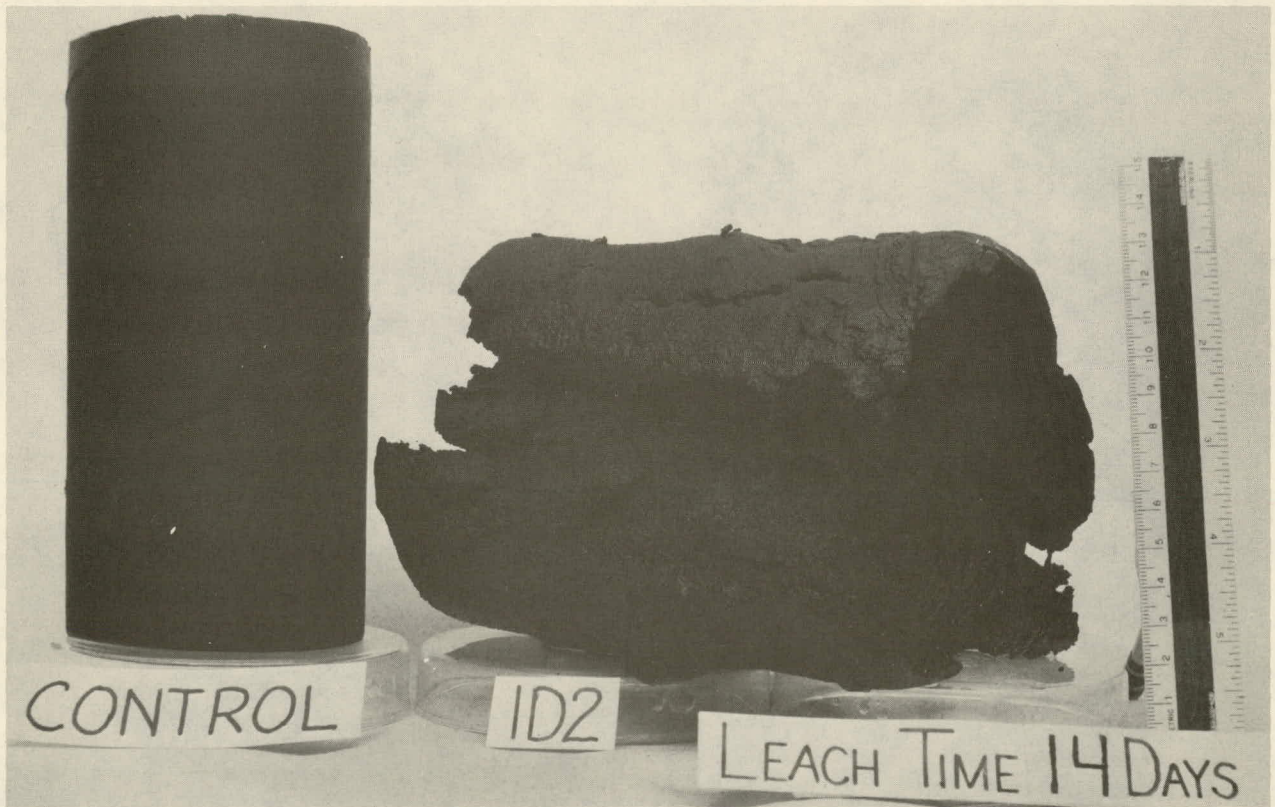


Figure 7. Bitumen Waste Form ID2 (41.4 Wt.%  $\text{Na}_2\text{SO}_4$ ) after Fourteen Days of Leaching in Distilled Water.

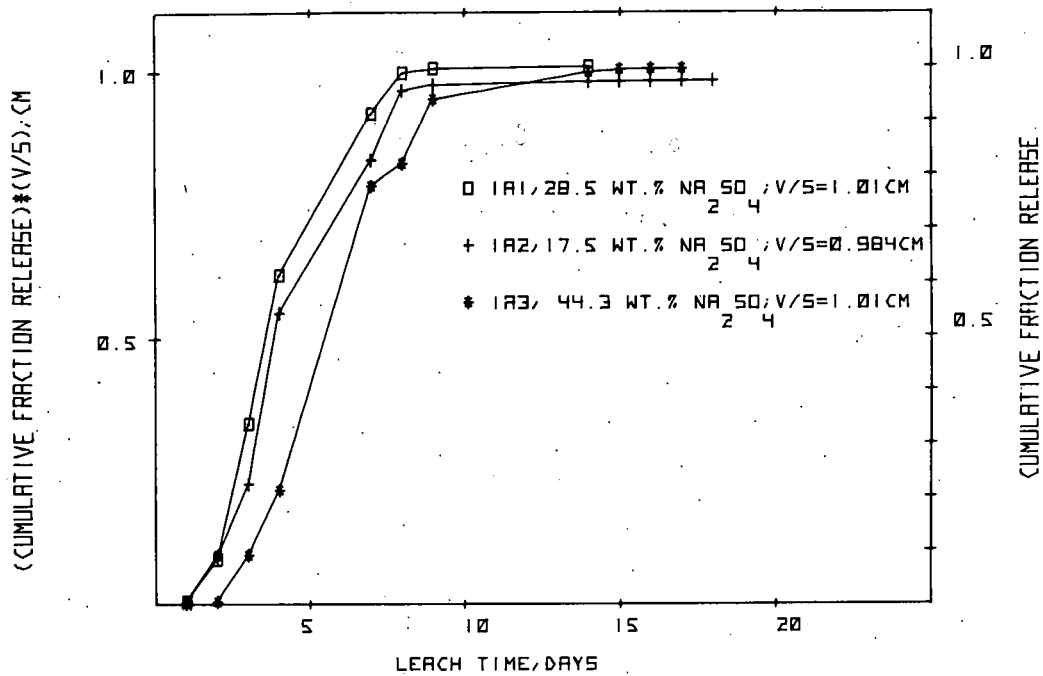


Figure 8. Cumulative Sodium Sulfate Fraction Release x (V/S), Static Leaching of Series 1A Waste Forms in Distilled Water.

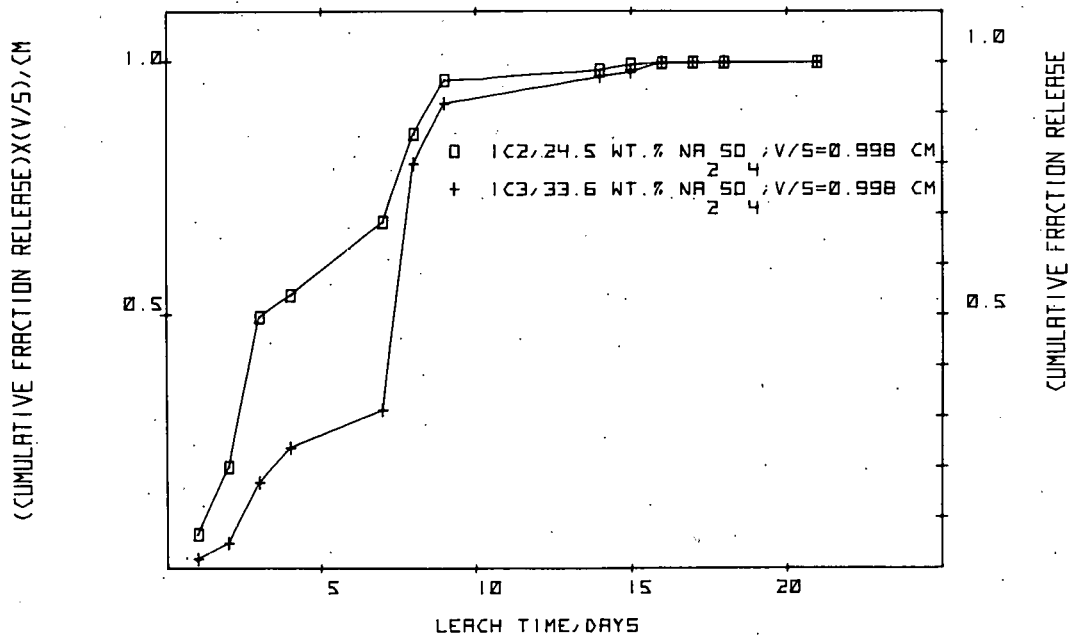


Figure 9. Cumulative Sodium Sulfate Fraction Release x (V/S), Static Leaching of Series 1C Waste Forms in Ground Water.

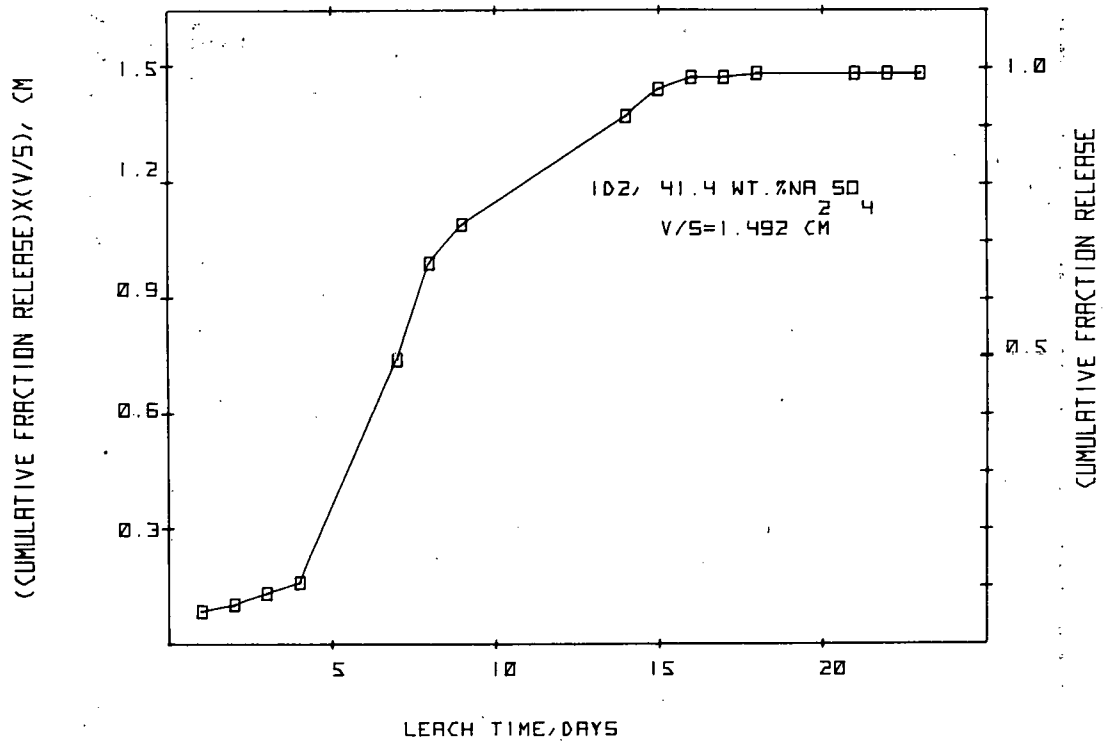


Figure 10. Cumulative Sodium Sulfate Fraction Release x (V/S), Static Leaching of Waste Form 1D2 in Ground Water.

## V. UREA-FORMALDEHYDE AND PORTLAND TYPE II CEMENT WASTE FORM VOLUME EFFICIENCY

The volume efficiency of urea-formaldehyde and portland type II cement waste forms has been calculated based upon volumetric data from compression test specimens. Volumetric efficiency is defined as the ratio of the solidification system input waste volume to the solidified waste form volume. Volume efficiency is a function of the weight/binder weight ratio and the waste and binder densities. The average volume efficiencies are shown in Tables 6 and 7 for urea-formaldehyde and portland type II cement compression test specimens respectively for selected waste/binder weight ratios. These weight ratios are not meant to represent optimized formulations, but simply represent formulations that have been utilized in the course of this program.

TABLE 6

Urea-Formaldehyde Waste Form Volume Efficiency

	<u>Waste Type</u>	<u>Waste/Binder Weight Ratio</u>	<u>Volume Efficiency</u>
1a	Bead Resin Slurry	2.6	0.792±0.012
2a(1)	Powdered Resin Slurry	2.0	0.726±0.005
2b(2)	Diatomaceous Earth (Dewatered)	2.0	0.715±0.009
3a	BWR Chemical Regenerative Waste	1.2	0.564±0.007
3c	Boric Acid Waste	2.0	0.735±0.013

TABLE 7

Portland Type II Cement Waste Form Volume Efficiency

	<u>Waste Type</u>	<u>Waste/Binder Weight Ratio</u>	<u>Volume Efficiency</u>
1a	Bead Resin Slurry	2.4	0.895±0.017
		2.6	0.944±0.024
2a(1)	Powdered Resin Slurry	1.8	0.732±0.004
		2.0	0.759±0.002
2b(1)	Diatomaceous Earth Slurry	1.6	0.926±0.006
3a	BWR Chemical Regenerative Waste	0.6	0.635±0.003
		1.2	0.842±0.007
		1.7	0.922±0.003
3c	Boric Acid Waste	0.6	0.771±0.011

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1. Heacock, H.W. and Riches, J.W., Waste Solidification - Cement or Urea Formaldehyde, American Society of Mechanical Engineers, 74-WA/NE-9, 1974.
2. U.S. Patent 3,988,258, Radwaste Disposal by Incorporation in Matrix, October 26, 1976.
3. John E. Stewart, private communication.
4. Final Environmental Statement - ALAP - LWE Effluents, WASH-1258, July 1973.
5. National Environmental Studies Project, A Survey and Evaluation of Handling and Disposing of Solid Low-Level Nuclear Fuel Cycle Wastes, AIF/NESP-008, Atomic Industrial Forum, Inc., October 1976.
6. Burns, R.H., Atomic Energy Rev., 9(3), 571, (1971).
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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) <sup>a</sup>	50.
Temperature	70°F
pH	7

1b. BEAD RESIN WASTE (Dewatered)

<u>Material</u>	<u>Weight Percent, %</u>
Water	35.
Bead Resin (IRN-150) <sup>a</sup>	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) <sup>b</sup>	20.
Cation Powdered Resin (PCH) <sup>b</sup>	20.
Crud <sup>c</sup>	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) <sup>b</sup>	30.
Cation Powdered Resin (PCH) <sup>b</sup>	30.
Crud <sup>c</sup>	6.
Sodium Chloride	2.
Temperature	70 <sup>o</sup> 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 <sup>c</sup>	5.0
Temperature	70 <sup>o</sup> 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 <sup>c</sup>	10.
Temperature	70 <sup>o</sup> 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 <sup>c</sup>	0.1
Temperature	170 <sup>o</sup> 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 <sup>C</sup>	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 <sup>C</sup>	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 <sup>d</sup>	9.4
EDTA	5.
Citric Acid	5.
Crud <sup>C</sup>	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 <sup>C</sup>	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 <sup>c</sup>	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 <sup>c</sup>	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.
Nutek NT-700 <sup>d</sup>	20.
EDTA	9.8
Citric Acid	19.
Crud <sup>c</sup>	0.2
Hydraulic Oil No. 2	0.5
Lubricating Oil No. 20	0.5
Temperature	150 to 250°F
pH	5

<sup>a</sup> Rohm and Haas Co., Philadelphia, Pa. 19105

<sup>b</sup> Ecodyne Corp., Union, N.J. 07083

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

<sup>d</sup> Nuclear Technology Corp., Amston, Conn. 06231

## Appendix B

Bitumen Solidification Test Program Run Data  
(conducted at Werner and Pfleiderer Corp., Waldwick, NJ)

1. BWR Chemical Regenerative Waste of a Forced Recirculation Evaporator  
(Run 10/27/77).

A. Feedstream Composition

<u>Material</u>	<u>Weight Percent</u>	<u>Weight, lbs.</u>
Water	75.0	198.0
Sodium Sulfate	22.9	60.5
Sodium Chloride	2.0	5.3
Crud (Ferrous Oxide)	0.1	<u>0.26</u>
		264.06 lb. total waste

The W&P heated waste tank and Triplex pump were used for this waste. Because of the limited capacity of this tank, it was necessary to use three individual waste batches, each one-third of the total waste. These waste batches were each neutralized to pH $\sim$ 7 by the addition of  $\sim$ 10M NaOH (400g NaOH/750g H<sub>2</sub>O, providing  $\sim$  1 liter) and cesium chloride was added for leaching purposes as listed below.

<u>Waste Batch</u>	<u><math>\sim</math> 10M NaOH, g</u>	<u>pH</u>	<u>CsCl, g</u>
1	4.0	7	49.0
2	20.0*	7.6	48.5
3	5.0	8.6	49.0

\* Over addition, excess neutralized with 13 g 37% HCl

B. Operating Conditions (ZSKT-53)

Screw speed 250 rpm

Asphalt feed temperature 200°F

Barrel temperature profile, °F

<u>Thermocouple</u>	<u>1130 hrs</u>	<u>1400 hrs</u>
t <sub>1</sub>	215	215
t <sub>2</sub>	250	300
t <sub>3</sub>	285	290
t <sub>4</sub>	340	335
t <sub>5</sub>	375	375
t <sub>6</sub>	290	290
discharge	330	340

Waste feed rate:	44.6 lbs/hr
Asphalt feed rate:	11.0 lbs/hr
Output	23.5 lbs/hr (1130 hrs)
	22.0 lbs/hr (1400 hrs)
Product ratio (weight)	50.5/49.5 (solids/asphalt)
	46.0 wt.% Na <sub>2</sub> SO <sub>4</sub>

C. Distillate Samples

Distillate samples were taken from each of the four steam domes while this waste was being solidified. The plastic garden hoses were removed from steam domes one and four to prevent organic contamination. Note that the interiors of steam domes three and four were painted just prior to the beginning of this run.

2. Boric Acid Waste of a Forced Recirculation Evaporation (Run 10/28/77)

A. Feedstream Composition

<u>Material</u>	<u>Weight Percent</u>	<u>Weight, lbs.</u>
Water	87.9	197.8
Boric Acid	12.0	27.0
Crud (Ferrous Oxide)	0.1	0.23
		225.03 lb total waste

The W&P heated waste tank and Triplex pump were used for this waste. Because of the limited capacity of this tank, it was necessary to make three individual waste batches, each one-third of the total waste. These waste batches were neutralized to pH $\approx$ 7 by the addition of  $\approx$ 10M NaOH (400g NaOH/750g H<sub>2</sub>O) as shown below:

<u>Waste Batch</u>	$\approx$ 10M NaOH,	
	<u>g</u>	<u>pH</u>
1	1,684	7.5
2	1,492	7.2
3	1,550	7.3

B. Operating Conditions

Screw speed 250 rpm  
 Asphalt feed temperature 200°F  
 Barrel temperature profile, °F

<u>Thermocouple</u>	<u>1215 hrs</u>	<u>1400 hrs</u>	<u>1545 hrs</u>
t <sub>1</sub>	225	225	225
t <sub>2</sub>	225	225	225
t <sub>3</sub>	280	290	285
t <sub>4</sub>	365	365	365
t <sub>5</sub>	370	365	365
t <sub>6</sub>	275	280	280
discharge	310	310	310

Waste feed rate: 37.6 lb/hr  
 Asphalt feed rate: 4.5 lb/hr  
 Output 7.8 lb/hr (1215 hrs)  
 7.0 lb/hr (1400 hrs)  
 7.1 lb/hr (1545 hrs)  
 Product ratio (weight) 52.2/47.8 (solids/asphalt)  
 45.7 wt.% boric acid

C. Distillate Samples

Distillate samples were taken from each of the four steam domes during this run. The steam domes were cleaned prior to this run.

3. Blank Run - Water and Asphalt Samples (Run 10/31/77, 1:30 p.m.)

A. Feedstream Composition

<u>Material</u>	<u>Weight Percent</u>
Water	100.0

Water was heated up to 140°F at the inlet of the feedline.

B. Operating Conditions (ZSK-53)

Screw speed 250 rpm  
Asphalt feed temperature 235°F, water feed temp. 140°F

Barrel Temperature profile, °F

Thermocouple	1330 hrs	1400 hrs
t <sub>1</sub>	213.5	215.0
t <sub>2</sub>	222.5	220.0
t <sub>3</sub>	280.0	280.0
t <sub>4</sub>	335.0	335.0
t <sub>5</sub>	360.0	360.0
t <sub>6</sub>	290.5	290.0
discharge	300.0	301.0
Waste feed rate:	45.0 lb/hr	
Asphalt feed rate:	14.7 lb/hr	
Output	14.3 lb/hr (1330 hrs)	
	14.5 lb/hr (1400 hrs)	
Product ratio (weight)	0/100 (solid/asphalt)	

C. Distillate Samples

Distillate samples were taken from each of the four steam domes while this product was being solidified. The steam domes were cleaned before this run. These samples will represent distillation blanks for asphalt alone to be used as a background in comparison with other distillate samples.

4. BWR Precoat Filter Cake with Diatomaceous Earth

A. Feedstream Composition

<u>Material</u>	<u>Weight Percent</u>	<u>Weight, lbs</u>
Water	75	270.0
Diatomaceous earth	20	72.0
Crud (ferrous oxide)	5	18.0
		360.0 lbs total water

The W&P heated waste tank and triplex pump were used for this water. Because of the limited capacity of the tank, it was necessary to use individual waste batches. These waste batches were each neutralized to a pH<sup>~</sup>7 by the addition of 10M NaOH when necessary. In addition, small quantities of sodium iodide and sodium chloride were added for leaching purposes.

Difficulties arose during the attempted pumping of this waste. As a result, the waste batches differed somewhat in composition. The asphalt feed rate for batch 4 was varied to produce a different solid/asphalt ratio in the final product. The batch 2 run was ended after 9 samples were collected due to Triplex pump outlet clogging.

Batches 1 and 2 were run on 10/31/77, batches 3 and 4 on 11/1/77. No NaOH solution was added to batches 2, 3, or 4. 0.5.g of 10M NaOH was added to batch 1.

	<u>Weight Percentage</u>					<u>Total Weight, lbs</u>
	<u>Water</u>	<u>DE</u>	<u>Crud</u>	<u>NaI</u>	<u>NaCl</u>	
Batch 1	79.9	11.8	5.3	1.5	1.5	84.5
Batch 2	73.0	19.4	4.8	1.4	1.4	92.5
Batch 3	82.6	12.5	3.1	0.87	0.87	108.7
Batch 4	81.6	12.4	4.3	0.86	0.86	45.6

B. Operating Conditions

Screw speed	250 rpm
Asphalt feed temperature	235°F (Batches 1, 2, 3) 215°F (Batch 4)
Waste feed temperature	140°F (Batch 1) 160°F (Batch 2) 68°F (Batches 3, 4)

Barrel temperature profile, °F

	Batch 1 (1500 hrs)	Batch 1 (1600 hrs)	Batch 2 (1650 hrs)	Batch 3 (1500 hrs)	Batch 3 (1540 hrs)	Batch 4 (1600 hrs)
t <sub>1</sub>	206	205	206	226	226	225
t <sub>2</sub>	216	215	215	227	227	225
t <sub>3</sub>	256	255	255	283	288	290
t <sub>4</sub>	346	345	346	362	361	365
t <sub>5</sub>	367	365	365	371	372	375
t <sub>6</sub>	297	300	300	288	288	295
discharge	310	330	325	335	340	340

Waste feed rate	60.0 lb/hr
asphalt feed rate	15.2 lb/hr (Batches 1, 2, 3) 10.0 lb/hr (Batch 4)
Output	28.0 lb/hr (Batch 1) 27.8 lb/hr (Batch 2) 25.0 lb/hr (Batch 3, 1500 hrs) 24.8 lb/hr (Batch 3, 1540 hrs) 21.0 lb/hr (Batch 4)
Product ratio (weight)	44.3/55.7 (solids/asphalt, Batch 1) 51.6/48.4 (Batch 2) 40.7/59.3 (Batch 3) 52.5/47.5 (Batch 4)

C. Distillate Samples

Distillate samples were taken during the runs of batch 1 from each of the four steam domes.

5. BWR Precoat Filter Cake (with powdered resin) (Run 11/2/77)

A. Feedstream Composition

<u>Material</u>	<u>Weight Percent</u>	<u>Weight, lbs</u>
Water	50.0	88.60
Anion Resin (58% water)	20.0	35.46
Cation Resin (62% water)	20.0	35.46
Crud (Ferrous oxide)	5.0	8.86
Sodium Chloride	5.0	8.86
		177.24 lbs total waste

The W&P heated waste tank and Triplex pump were used for this waste. Because of the limited capacity of this tank, it was necessary to make two individual waste batches, each one-half of the total waste. These waste batches were each neutralized to pH<sup>v</sup>7 by the addition of 10M NaOH. Sodium iodide and cobaltous chloride ( $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ ) were added for leaching purposes as follows:

<u>Waste Batch</u>	<u>10M NaOH, g</u>	<u>pH</u>	<u>NaI, lb</u>	<u><math>\text{CoCl}_2 \cdot 6\text{H}_2\text{O}</math>, lb</u>
1	500	7.6	2.13	2.0
2	597	7.8	2.13	2.0

B. Operating Conditions

Screw speed	250 rpm
Asphalt feed temperature	225°F
Waste feed temperature	68°F
Barrel temperature profile, °F	

<u>Thermocouple</u>	<u>(0945 hrs)</u>	<u>(1230 hrs)</u>
t <sub>1</sub>	223	223
t <sub>2</sub>	228	228
t <sub>3</sub>	337	337
t <sub>4</sub>	367	367
t <sub>5</sub>	373	373
t <sub>6</sub>	294	294
discharge	340	340

Waste feed rate	44.6 lb/hr
Asphalt feed rate	11.6 lb/hr
Output	23.0 lb/hr
Product ratio (weight)	55.5/44.5 (solids/asphalt)

C. Distillate Samples

Distillate samples were taken from all four steam domes. The steam domes were cleaned prior to running this waste.

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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

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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 ( $\text{cm}^2$ ).

$V$  = volume of the specimen ( $\text{cm}^3$ ).

$M$  = 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) ( $\text{day}^{-1}$ ). 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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Appendix D

Individual Bitumen Sample Leach Data

Experimental Results for the Leaching of  
Sodium Sulfate from Bitumen  
Specimen 1A1

A. Description of Specimen

Waste Type	-	BWR Chemical Regenerate Waste
Na <sub>2</sub> SO <sub>4</sub> /bitumen weight ratio	-	0.399
Mass	-	284.4 g
Volume	-	202.3 cm <sup>3</sup>
Geometric Surface Area	-	200.3 cm <sup>2</sup>
(V/S)	-	1.01 cm

B. Leach Test Procedure

Leach Test	-	static
Leachant	-	distilled water
Leachant Volume	-	2027 cm <sup>3</sup>
Leachant Replacement Interval	-	daily

Sample Number 1A1

Leach Time (days)		Final pH of Leachant	(Fraction Release) X (V/S)	
Incremental	Cumulative		Incremental	Cumulative
1.000	1.000	7.00	4.79E-03	4.79E-03
1.000	2.000	7.04	7.91E-02	8.39E-02
1.000	3.000	6.96	2.56E-01	3.40E-01
1.000	4.000	6.98	2.79E-01	6.19E-01
3.000	7.000	6.95	3.03E-01	9.22E-01
1.000	8.000	6.35	7.59E-02	9.98E-01
1.000	9.000	6.37	8.75E-03	1.01E 00
5.000	14.000	6.87	3.63E-03	1.01E 00

Experimental Results for the Leaching of  
Sodium Sulfate from Bitumen  
Specimen 1A2

A. Description of Specimen

Waste Type	-	BWR Chemical Regenerate Waste
Na <sub>2</sub> SO <sub>4</sub> /bitumen weight ratio	-	0.212
Mass	-	289.1 g
Volume	-	188.6 cm <sup>3</sup>
Geometric Surface Area	-	191.7 cm <sup>2</sup>
(V/S)	-	0.984 cm

B. Leach Test Procedure

Leach Test	-	static
Leachant	-	distilled water
Leachant Volume	-	2027 cm <sup>3</sup>
Leachant Replacement Interval	-	daily

Sample number 1A2

Leach Time(days)		Final pH of Leachant	(Fraction Release)X(V/S)	
Incremental	Cumulative		Incremental	Cumulative
1.000	1.000	7.00	2.52E-03	2.52E-03
1.000	2.000	7.04	8.50E-02	8.75E-02
1.000	3.000	6.96	1.38E-01	2.26E-01
1.000	4.000	6.98	3.22E-01	5.48E-01
3.000	7.000	6.95	2.87E-01	8.35E-01
1.000	8.000	6.35	1.30E-01	9.65E-01
1.000	9.000	6.37	1.12E-02	9.76E-01
5.000	14.000	6.87	5.78E-03	9.82E-01
1.000	15.000	6.95	5.49E-04	9.83E-01
1.000	16.000	7.22	3.43E-04	9.83E-01
1.000	17.000	5.94	3.43E-04	9.83E-01
1.000	18.000	6.81	7.07E-04	9.84E-01

Experimental Results for the Leaching of  
Sodium Sulfate from Bitumen  
Specimen 1A3

---

A. Description of Specimen

Waste Type	-	BWR Chemical Regenerate Waste
Na <sub>2</sub> SO <sub>4</sub> /bitumen weight ratio	-	0.795
Mass	-	282.8 g
Volume	-	198.3 cm <sup>3</sup>
Geometric Surface Area	-	197.1 cm <sup>2</sup>
(V/S)	-	1.01 cm

B. Leach Test Procedure

Leach Test	-	static
Leachant	-	distilled water
Leachant Volume	-	2027 cm <sup>3</sup>
Leachant Replacement Interval	-	daily

Sample Number 1A3

Leach Time (days)		Final pH of Leachant	(Fraction Release) X (V/S)	
Incremental	Cumulative		Incremental	Cumulative
1.000	1.000	7.00	0.00E-00	0.00E-00
1.000	2.000	7.04	4.75E-03	4.75E-03
1.000	3.000	6.96	8.71E-02	9.18E-02
1.000	4.000	6.98	1.22E-01	2.14E-01
3.000	7.000	6.95	5.72E-01	7.86E-01
1.000	8.000	6.35	4.21E-02	8.28E-01
1.000	9.000	6.37	1.21E-01	9.49E-01
5.000	14.000	6.87	5.07E-02	1.00E-00
1.000	15.000	6.95	5.11E-03	1.00E-00
1.000	16.000	7.22	8.06E-04	1.01E-00
1.000	17.000	5.94	2.99E-04	1.01E-00

Experimental Results for the Leaching of  
Sodium Sulfate from Bitumen  
Specimen 1C2

---

A. Description of Specimen

Waste Type	-	BWR Chemical Regenerate Waste
Na <sub>2</sub> SO <sub>4</sub> /bitumen weight ratio	-	0.325
Mass	-	283.6 g
Volume	-	194.4 cm <sup>3</sup>
Geometric Surface Area	-	194.8 cm <sup>2</sup>
(V/S)	-	0.998 cm

B. Leach Test Procedure

Leach Test	-	static
Leachant	-	ground water
Leachant Volume	-	2027 cm <sup>3</sup>
Leachant Replacement Interval	-	daily

Sample number LC2

Leach Time (days)		Final pH of Leachant	(Fraction Release) X (V/S)	
Incremental	Cumulative		Incremental	Cumulative
1.000	1.000	NA	6.63E-02	6.63E-02
1.000	2.000	6.88	1.33E-01	1.99E-01
1.000	3.000	7.31	2.96E-01	4.95E-01
1.000	4.000	6.91	4.35E-02	5.38E-01
3.000	7.000	7.00	1.44E-01	6.82E-01
1.000	8.000	7.31	1.74E-01	8.56E-01
1.000	9.000	6.37	1.06E-01	9.62E-01
5.000	14.000	6.80	2.00E-02	9.82E-01
1.000	15.000	7.07	1.06E-02	9.93E-01
1.000	16.000	7.02	2.86E-03	9.96E-01
1.000	17.000	5.05	1.09E-03	9.97E-01
1.000	18.000	6.62	5.36E-04	9.97E-01
3.000	21.000	6.66	5.36E-04	9.98E-01

Experimental Results for the Leaching of  
Sodium Sulfate from Bitumen  
Specimen 1C3

---

A. Description of Specimen

Waste Type	-	BWR Chemical Regenerate Waste
Na <sub>2</sub> SO <sub>4</sub> /bitumen weight ratio	-	0.506
Mass	-	285.4 g
Volume	-	194.4 cm <sup>3</sup>
Geometric Surface Area	-	194.8 cm <sup>2</sup>
(V/S)	-	0.998 cm

B. Leach Test Procedure

Leach Test	-	static
Leachant	-	ground water
Leachant Volume	-	2027 cm <sup>3</sup>
Leachant Replacement Interval	-	daily

Sample number 103

Leach. Time (days)		Final pH of Leachant	(Fraction Release) X (V/S)	
Incremental	Cumulative		Incremental	Cumulative
1.000	1.000	NA	1.81E-02	1.81E-02
<del>1.000</del>	<del>2.000</del>	<del>6.88</del>	<del>3.09E-02</del>	<del>4.90E-02</del>
1.000	3.000	7.32	1.19E-01	1.68E-01
1.000	4.000	6.91	6.90E-02	2.37E-01
3.000	7.000	7.00	7.41E-02	3.11E-01
1.000	8.000	7.31	4.86E-01	7.97E-01
1.000	9.000	6.37	1.19E-01	9.16E-01
<del>5.000</del>	<del>14.000</del>	<del>6.80</del>	<del>5.29E-02</del>	<del>9.69E-01</del>
1.000	15.000	7.07	8.99E-03	9.78E-01
1.000	16.000	7.02	1.90E-02	9.97E-01
1.000	17.000	5.05	8.07E-04	9.97E-01
1.000	18.000	6.62	3.34E-04	9.98E-01
3.000	21.000	6.66	2.92E-04	9.98E-01

Experimental Results for the Leaching of  
Sodium Sulfate from Bitumen  
Specimen ID2

---

A. Description of Specimen

Waste Type	-	BWR Chemical Regenerate Waste
$\text{Na}_2\text{SO}_4$ /bitumen weight ratio	-	0.707
Mass	-	939.3 g
Volume	-	638.5 cm <sup>3</sup>
Geometric Surface Area	-	428.0 cm <sup>2</sup>
(V/S)	-	1.492 cm

B. Leach Test Procedure

Leach Test	-	static
Leachant	-	distilled water
Leachant Volume	-	4560 cm <sup>3</sup>
Leachant Replacement Interval	-	daily

Sample Number 1D2

Leach Time (days)		Final pH of Leachant	(Fraction Release)X(V/S)	
Incremental	Cumulative		Incremental	Cumulative
1.000	1.000	NA	8.49E-02	8.49E-02
1.000	2.000	NA	1.73E-02	1.02E-01
1.000	3.000	NA	2.89E-02	1.31E-01
1.000	4.000	NA	2.74E-02	1.59E-01
3.000	7.000	NA	5.81E-01	7.39E-01
1.000	8.000	NA	2.51E-01	9.90E-01
1.000	9.000	NA	9.87E-02	1.09E 00
5.000	14.000	NA	2.80E-01	1.37E 00
1.000	15.000	NA	7.44E-02	1.44E 00
1.000	16.000	NA	2.52E-02	1.47E 00
1.000	17.000	NA	5.65E-03	1.47E 00
1.000	18.000	NA	2.37E-03	1.48E 00
3.000	21.000	NA	2.03E-03	1.48E 00
1.000	22.000	NA	4.84E-04	1.48E 00
1.000	23.000	NA	4.31E-04	1.48E 00

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