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PROPERTIES OF RADIOACTIVE WASTES AND WASTE CONTAINERS

PROGRESS REPORT NO. 5
APRIL-JUNE 1977

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

DATE PUBLISHED - DECEMBER 1977

NUCLEAR WASTE MANAGEMENT RESEARCH GROUP

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SUMMARY

Portland type II cement decontamination factors for free standing water were determined with ^{137}Cs , ^{85}Sr , and ^{60}Co . The decontamination factor was defined as the ratio of the specific activity of the initial waste to the specific activity of the free standing water from the solidified waste form. Decontamination factors of 1, 11, and 200 were found for ^{137}Cs , ^{85}Sr , and ^{60}Co respectively, indicative of ion exchange processes occurring within the cement matrix.

An investigation of solidification agent biodegradation was initiated with an experiment to determine the quantity of organic carbon that could be removed from solidification agent matrix materials on leaching in distilled water. This is representative of the most generally operative mechanism by which nutrients can be supplied to support microbial growth. The quantity of organic carbon available will generally be related to the extent of the resulting microbial growth. No organic carbon was present in the portland type II cement leachant water after ten days while 3.4 ppm, 34.2 ppm, and 9500 ppm organic carbon were present respectively in the bitumen, Dow polymer and urea-formaldehyde leachant water (300 ml). Organic carbon removal as a function of leach time was also measured for a urea-formaldehyde specimen.

The compression strengths of portland type II cement and urea-formaldehyde waste forms containing various simulated wastes were measured. The effect of the waste/cement ratio on the compression strength of portland type II cement simulated waste forms was also studied.

The gamma radiation shielding characteristics of portland cement, urea-formaldehyde, and bitumen waste forms were studied in reference to the maximum incorporated activity permissible in 55 gallon waste packages for transportation in "non-exclusive use" vehicles under 49CFR173. Total activity levels were specified for the waste form types considering activity with gamma photon energies of 0.50 MeV and 1.0 MeV in water and 25 wt.% sodium sulfate wastes. The resultant input waste specific activities were also calculated based on the maximum activity levels in the solidified waste form and the waste/binder ratio.

Thermal conductivity, specific heat, and thermal diffusivity values were obtained for portland cement, urea-formaldehyde, bitumen, and Dow polymer waste forms.

Solidification verification studies were initiated for the Dow polymer at waste/binder ratios representative of those proposed for use by the vendor. While these studies have not been completed, successful solidifications were obtained for all waste types tested with the exception of 12 wt.% boric acid. The 12 wt.% boric acid waste was successfully solidified after a proprietary pretreatment of the waste stream.

I. PORTLAND TYPE II CEMENT DECONTAMINATION FACTORS FOR FREE STANDING WATER

An experiment was performed to measure the decontamination factors of a portland type II cement waste form for cesium-137, strontium-85, and cobalt-60 as measured from the free standing water. The decontamination factor (DF) was defined as follows for the purposes of this experiment:

$$DF = \frac{\text{specific radionuclide content of initial waste, mCi/ml}}{\text{specific radionuclide content of free standing water, mCi/ml}} \quad (1)$$

Triplicate 100 gram portland type II cement samples were prepared using distilled water for each of the above radionuclides at a water/cement weight ratio of 1.0. A one milliliter aliquot of a radionuclide tracer solution was added to each of the samples as it was being mixed. These tracer solutions were:

solution 1:	^{137}Cs , 1.00×10^{-3} mCi/ml
solution 2:	^{60}Co , 1.00×10^{-3} mCi/ml
solution 3:	^{85}Sr , 1.00×10^{-3} mCi/ml

The samples were cylindrical with a diameter of 1.88 inches and 1.25 inches in length. Counting standards were prepared from each of these solutions by taking a one milliliter aliquot from the tracer solution and diluting to ten milliliters. The specimens were allowed to cure for three days in their individual polyethylene preparation vials which were sealed to prevent evaporative water loss. After curing, the quantity of free standing water in each sample container was measured with a one milliliter aliquot taken and diluted to ten milliliters for counting by a Searle model 1185 automatic gamma counter with a three inch by three inch true well sodium iodide crystal. Comparison of the counting rate of the diluted free water samples with that for the tracer solution standards allowed calculation of the respective decontamination factors. The average quantity of water present after sample curing was 12.8 grams.

The decontamination factors determined for the cement samples from the free standing water are given in Table 1. These decontaminative factors represent the average of triplicate samples for each of the indicated radionuclides. The photopeaks used for this analysis were 0.662 MeV for cesium-137, 0.513 MeV for strontium-85 and the sum of the 1.17 MeV and 1.33 MeV photopeaks for cobalt-60. A decontamination factor of 1.0 would indicate that the specific

TABLE 1

Portland Type II Cement Decontamination Factors for Free Standing Water

<u>Isotope</u>	<u>Gamma Energy, MeV</u>	<u>Decontamination Factor</u>
¹³⁷ Cs	0.662	0.91
⁸⁵ Sr	0.513	11.0
⁶⁰ Co	1.33	200
	1.17	

water/cement = 1.0 by weight

activity of the free standing water (mCi/ml) was the same as that of the initial waste prior to solidification. In this experiment, decontamination factors of 0.91, 11.0, and 200 were determined for ¹³⁷Cs, ⁸⁵Sr, and ⁶⁰Co respectively. The decontamination factor for ¹³⁷Cs is effectively 1.0 within the accuracy of the experiment. This behavior is indicative of ion exchange processes occurring within the cement matrix where ionic selectivity generally increases as the valence increases and as the ionic radii decreases for a given valence. A similar experiment was performed previously with urea-formaldehyde in which decontamination factors of 1.0-1.9 were measured. ⁽¹⁾

II. BIODEGRADATION OF SOLIDIFICATION MATRIX MATERIALS

In addition to other physical and chemical characteristics, recalcitrance or non-biodegradability is a desirable and perhaps essential feature of the solidification agents that are used in the solidification of radioactive wastes for the long-term isolation of radionuclides from the environment. Biological degradation by microorganisms can occur by either surface attached growth on the solid substrate or growth supported by the leaching of nutrients from the solid substrate, perhaps due to the infiltration of water. The second mechanism is most generally the operative one since nutrients are supplied directly in a solubilized form to support microbial growth. As such, it is useful to determine what organic constituents may be leached from a solid waste form under representative conditions, and whether this leached material will support microbial growth.

An experiment was initiated to ascertain whether organic compounds are removed from portland type II cement, Borden Casco-Resin 2 urea-formaldehyde, Pioneer 221 asphalt, and Dow Chemical Company proprietary polymer (vinyl ester-styrene) samples during leaching in distilled water. The portland type II cement sample was prepared with distilled water using a water/cement weight ratio of 0.5. A water/UF volume ratio of 1.0 was used in preparation of the urea-formaldehyde sample with two volume percent of a 25 wt.% sodium bisulfate solution added as the polymerization catalyst. The asphalt sample was made by melting asphalt from a larger block. A sample of the proprietary Dow polymer was prepared with distilled water at a water/binder ratio of 1.5 by volume using the procedure specified by the Dow Chemical Company. All samples were left undisturbed for four days before testing was begun. Samples were individually leached in 300 milliliters of distilled water for ten days, after which time an aliquot of the water was taken for analysis with a Beckman total carbon analyzer. Approximately 4.6 g of free water from the urea-formaldehyde sample was included in the leaching procedure.

Table 2 gives the results of this leaching method. A distilled water blank tested contained less than 2 ppm total carbon and less than 2 ppm inorganic carbon, which represents the lower analytical limits of the procedure. Organic carbon content of the leachant was determined by subtraction

TABLE 2

Analysis of Organic Carbon Removed from Solidification
Matrix Materials for Leaching in Distilled Water for Ten Days

	<u>Portland Type II Cement</u>	<u>Urea- Formaldehyde</u>	<u>Pioneer 221 Asphalt</u>	<u>Dow Polymer</u>
Sample mass, g.	296.0	218.3	103.1	198.2
Ratio of sample volume to geometric surface area, cm	0.98	1.1	0.75	1.1
Leachant content (300 ml)				
a. total carbon, ppm	14.8	9540	3.4	34.2
b. inorganic carbon, ppm	14.8	39	<2.0	<2.0
c. organic carbon, ppm	0.0	9500	3.4	34.2
d. organic carbon, g	0.0	2.85	0.0010	0.010

of the analyzed inorganic carbon content from the total carbon content. When the inorganic carbon content of the leachant was below the detection level (<2 ppm), it was treated as zero for organic carbon determination. No organic carbon was detected in the leachant for the portland type II cement sample. The leachant contained small amounts of organic carbon 3.4 ppm and 34.2 ppm, for the asphalt and Dow polymer samples respectively. The urea-formaldehyde sample leachant had an organic carbon content of 9,500 ppm which is equivalent to 2.85 grams carbon. The ratio of the sample volume to its geometric surface area is also listed in this table for the test specimens. The form of the organic carbon present in the leachant and whether it would support microbial growth have not yet been determined. Assuming that microbial growth could result, the extent of such growth would be related to the amount of organic carbon available.

As a result of the high organic carbon content in the leachant for the urea-formaldehyde sample, an additional test was run in which the distilled water leachant was sampled and changed on a daily basis (with the exception of weekends). This sample was prepared using Borden Casco-Resin 2 urea-formaldehyde and distilled water at a water/UF volume ratio of 2.0. Two volume percent of a 25 wt.% sodium bisulfate solution was added as the polymerization catalyst. This sample was allowed to cure for four days in a sealed container. Before removing the UF sample from its container, the approximately 4.3 g of free standing water present was collected for carbon analysis. This free standing water was not added to the leachant. The sample, which was cylindrical with a diameter of 7.26 cm, a length of 4.84 cm and a mass of 211.7 g, was then placed in a beaker containing 300 ml of distilled water. The ratio of the sample volume (V) to its external geometric surface area (S) was 1.04 cm. Table 3 shows the results of the leachant carbon analysis for this sample.

The organic carbon content of the free standing water was in excess of 22,000 ppm, the large majority of which was due to organic carbon. The amount of free standing water was 4.3 grams corresponding to an organic carbon content of 0.097 grams. The largest leachant organic carbon content, 4,080 ppm, was measured after the first day of leaching; the organic carbon content

TABLE 3

Analysis of Organic Carbon Removed from a Urea-Formaldehyde Sample
for Leaching in Distilled Water (Leachant replaced daily)

<u>Incremental Leach Time, t_n, days</u>	<u>Σt_n, days</u>	<u>Total Carbon, ppm</u>	<u>Inorganic Carbon, ppm</u>	<u>Organic Carbon, ppm</u>	<u>Cumulative Organic Carbon, g</u>
FREE WATER		22,680	182	22,500	0.097
1	1	4,120	42.4	4,080	1.32
1	2	3,025	25.6	3,000	2.22
1	3	1,875	12.4	1,860	2.78
1	4	1,480	6.2	1,475	3.22
3	7	1,450	<2.0	1,450	3.65
1	8	750	<2.0	750	3.88
1	9	534	<2.0	534	4.04
1	10	360	<2.0	360	4.15
1	11	270	<2.0	270	4.23
3	14	338	<2.0	338	4.33
1	15	174	<2.0	174	4.38
1	16	128	<2.0	128	4.42
1	17	91	<2.0	91	4.45
1	18	87	<2.0	87	4.48

water/UF = 2.0 by volume
sample mass = 211.7 grams
leachant volume = 300 cm³

proceeded to decrease with each successive leachant change. This is typical of leaching behavior when the quantity of leachable material in the sample decreases with time and the leachant is replaced regularly. The total organic carbon removal from this 211.7 gram sample after eighteen days of leaching was 4.48 g. While the form of the organic carbon removed in this manner is not yet known, it is interesting to note that carbon constitutes approximately 33 percent of urea-formaldehyde on a weight basis. (2)

III. COMPRESSION STRENGTH OF PORTLAND TYPE II CEMENT AND UREA-FORMALDEHYDE WASTE FORMS .

The compression strengths of waste forms resulting from the solidification of various simulated wastes with portland type II cement and Borden Casco-Resin 2 urea-formaldehyde were measured by ASTM method C-39-72, "Test for the Compression Strength of Cylindrical Concrete Specimens". Waste streams utilized included bead resin slurry, BWR precoat filter cake (with powdered resin) slurry, dewatered BWR precoat filter cake (with diatomaceous earth), BWR chemical regenerative waste (forced recirculation evaporator), PWR chemical regenerative waste (forced recirculation evaporator), and boric acid waste (forced recirculation evaporator). These waste formulations are listed in Appendix A. Bead resin and BWR precoat filter cake (with powdered resin) wastes were converted from the H^+ and OH^- cationic and anionic forms as supplied to the Na^+ and Cl^- forms prior to incorporation with a solidification agent to simulate depleted resins. Test specimens were prepared in accordance with ASTM methods C192-69 in two inch diameter by four inch long cylindrical polyethylene molds which were individually sealed to prevent evaporative water loss. Five to seven samples were prepared for each formulation and were allowed to cure for twenty-eight days at 72°F before testing.

Portland type II cement specimens were prepared using various waste/cement weight ratios for each waste type to investigate the effect of this ratio on the compressive strength. Bead resin and BWR precoat filter cake (with powdered resin) wastes were adjusted to a pH of ten by the addition of a 1 M NaOH solution prior to mixing with the cement. This was done to minimize sample swelling which may occur when ion exchange resins are solidified with cement, particularly in the presence of water in excess of that needed for cement hydration (this condition will normally be existant for resin slurry wastes solidified with cement). Some specimen swelling did occur, however, the degree of swelling was significantly less than that which occurred without prior pH adjustment of the waste which often resulted in splitting of the specimen mold. Boric acid waste/cement specimens were not tested since they lacked sufficient strength after the allotted cure time to enable their successful removal from the sample molds. The results of compression testing

cement waste forms are shown in Table 4. Note that the compression strength is normally a decreasing function of the waste/cement ratio or waste content. The packing efficiency is also listed in this table where the packing efficiency in percent is defined as the ratio of the initial waste volume to the volume of the solidified waste form x 100. The packing efficiency increases with the waste/cement ratio. Packing efficiencies of 100% and slightly above have been reported for dewatered resin and other solid wastes.

Urea-formaldehyde samples were prepared using the various simulated wastes at weight/UF ratios that have been published in earlier reports.⁽¹⁾ These ratios are not meant to represent maximum or optimized values, but rather to provide formulations for experimental purposes that may typify those actually used. Polymerization was initiated by the addition of 2.0 volume percent of a 25 weight percent anhydrous sodium bisulfate solution. The test data obtained from these samples is given in Table 5. Note that waste/UF weight ratio to volume ratio conversion factors for these wastes are listed in Appendix B. Compression strength is assumed to decrease with increasing waste/UF ratios for a given waste although this effect was not investigated. The packing efficiency of urea-formaldehyde for the simulated wastes at the waste/UF ratios used are also tabulated.

While compression strength may not be a primary criterion for waste solidification, the solidified product must have sufficient compression strength to be free standing under its own weight. This compression strength can be calculated for the critical volume element at the base of a waste form from the waste package size and waste form density. The required compression strengths are low. For example, a cement waste form with a density of 140 lbs/ft³ (2.24 g/cm³) must have a compression strength of 0.97 psi per foot of waste form height to be free standing under its own weight. Obviously the necessary compression strength decreases as the waste form density decreases. Compression strengths in excess of this minimum requirement to be free standing are beneficial when potential waste form loading situations in transportation and burial are considered.

TABLE 4

Compression Strength of Portland Type II Cement Waste Forms

<u>Waste Type</u> ⁽¹⁾	<u>Waste/Cement Weight Ratio</u>	<u>Waste Form Density, g/cm³</u>	<u>Packing Efficiency, %</u> ⁽²⁾	<u>Compression Strength, psi±1σ</u>
1	2.0	1.29	81.8	48± 5
1	2.4	1.33	90.2	68± 10
1	2.6	1.35	92.8	41± 8
2A	1.8	1.23	72.8	48± 4
2A	2.0	1.22	75.7	45± 7
2B	1.6	1.65	89.4	482± 98
2B	2.0	1.57	92.1	420± 17
2B	2.4	1.53	94.3	103± 25
3A	0.6	2.03	63.2	3271±262
3A	1.2	1.84	83.4	576±106
3A	1.7	1.74	91.7	177± 61
3B	0.6	2.09	67.4	3161±257
3B	1.2	1.77	83.2	72± 27
3B	1.7	1.67	90.7	40± 22

(1) Waste Types

1. Bead resin waste slurry
- 2A. BWR precoat filter cake, slurry (with powdered resin)
- 2B. BWR precoat filter cake, dewatered (with diatomaceous earth)
- 3A. BWR chemical regenerative waste (forced recirculation evaporator)
- 3B. PWR chemical regenerative waste (forced recirculation evaporator)

(2) packing efficiency = $\frac{\text{initial waste volume}}{\text{waste form volume}} \times 100$

TABLE 5

Compression Strength of Urea-Formaldehyde Waste Forms

<u>Waste Type</u> ⁽¹⁾	<u>Waste/UF Weight Ratio</u>	<u>Waste Form Density, g/cm³</u>	<u>Packing Efficiency, %</u> ⁽²⁾	<u>Compression Strength psi±1σ</u>
1	2.6	1.13	77.8	78±5
2A	2.0	1.18	72.9	384±47
2B	2.0	1.21	70.9	387±53
3A	1.2	1.23	56.1	67±12
3B	1.2	1.25	58.3	61±22
3C	2.0	1.13	73.4	95±43

(1) Waste Types

1. Bead resin waste slurry
- 2A. BWR precoat filter cake, slurry (with powdered resin)
- 2B. BWR precoat filter cake, dewatered (with diatomaceous earth)
- 3A. BWR chemical regenerative waste (forced recirculation evaporator)
- 3B. PWR chemical regenerative waste (forced recirculation evaporator)
- 3C. Boric acid waste (forced recirculation evaporator)

(2) packing efficiency = $\frac{\text{initial waste volume}}{\text{waste form volume}} \times 100$

IV. SHIELDING CHARACTERISTICS OF CEMENT, UREA-FORMALDEHYDE, AND BITUMEN WASTE FORMS

The relative shielding characteristics of waste forms produced by solidification using various agents is of interest due to regulations which restrict the radiation dose level at the surface of the waste package. As a result, the amount of activity incorporated in a solidification matrix with given shielding qualities may be either restricted or require the use of over-packs or "exclusive use" transport vehicles. These regulations are listed in Title 49 of the Code of Federal Regulations, Part 173 and are summarized in Table 6. In this section, the allowable activity that can be incorporated within 55 gallon drum cement, urea-formaldehyde, and bitumen waste packages for transport in non-exclusive use vehicles has been calculated under the most limiting condition of the regulations listed in Table 6.

For the purpose of these calculations, it is assumed that the activity is evenly distributed throughout a homogeneous cylindrical waste form which has the inner dimensions of a 0.050 inch thick (18 gauge) mild steel 55 gallon drum. Shielding by the drum is included. Equations for the solution of the gamma flux (gammas/(cm²-sec)) for the exterior of cylindrical sources and graphical aids for the solution of these equations have been compiled.⁽³⁾ These equations were solved for the center of the circular face of the cylindrical waste form both at the surface (case 1) and at a perpendicular distance of three feet from this point (case 2) and for the side of the cylinder at the midpoint between the upper and lower faces for both the surface (case 3) and at a point three feet perpendicular from this midpoint on the surface (case 4). Once the gamma flux at a point is known, a conversion to dose rate (rads/hr) can be made for gamma photons of a given energy.⁽⁴⁾ Since the quality factor for gamma photons is one, the equivalence of rads/hr to rem/hr is noted. The number of curies of activity in a water waste containing either 0.5 MeV or 1.0 MeV gamma emitters that can be incorporated in a cement waste form with a density of 2.0 g/cm³ producing a surface dose rate of 200 mrem/hr (cases 1 and 3) or a dose rate of 10 mrem/hr at three feet (cases 2 and 4) were calculated and are listed in Table 7. The attenuation coefficient used for the cement waste form was calculated from

TABLE 6

Radioactive Materials Maximum Radiation Level Limitations
(49 CFR 173.393)

Radiation level (dose) rate at any point on external surface of any package of radioactive material may not exceed:

- A. 200 millirem per hour.
- B. 10 millirem per hour at three feet (transport index may not exceed 10).

Unless the packages are consigned to a "sole use" or "exclusive use" closed transport vehicle (except aircraft) then the maximum radiation levels may be:

- A. 1000 millirem per hour at 3 feet from external package surface.
- B. 200 millirem per hour at external surface of the vehicle.
- C. 10 millirem per hour at 6 feet from external surface of the vehicle.
- D. 2 millirem per hour in any position of the vehicle which is occupied by a person.

TABLE 7

Activity in 55 Gallon Drum Portland Type I Cement Waste Packages
Meeting 49 CFR 173 Regulations for Non-Exclusive Use Vehicles

<u>Calculational Case</u> ⁽¹⁾	<u>Activity, Curies</u>	
	<u>0.5 MeV</u>	<u>1.0 MeV</u>
Case 1 (200 mrem/hr)	0.61	0.20
Case 2 (10 mrem/hr)	0.70	--
Case 3 (200 mrem/hr)	0.60	--
Case 4 (10 mrem/hr)	0.40	0.15

water/cement = 1.0 by weight

concrete density = 2.0 g/cm^3

waste container = 0.050 inch thick (18 gauge) mild steel drum

(1) Calculational Cases (External to a Cylinder)

Case 1: Center of circular face.

Case 2: Three feet perpendicular from the point in Case 1.

Case 3: Side surface at the mid-point between the upper and lower circular faces.

Case 4: Three feet perpendicular from the point in Case 3.

the normalized formulation of type I portland cement⁽⁵⁾ shown in Table 8 which was mixed with an aqueous waste, providing a water/cement weight ratio of 1.0. From Table 7 is apparent that the case in which a maximum dose rate of 10 mrem/hr measured three feet perpendicular from the side of the cylinder is the most restrictive one, i.e., highest dose rate/unit activity incorporated in the waste form. As would be expected, a larger quantity of activity emitting 0.5 MeV gamma photons can be incorporated into the waste form to produce a given dose rate than with 1.0 MeV gamma emitting activity.

The maximum activity that could be incorporated in homogeneous 55 gallon drum urea-formaldehyde and bitumen waste packages were also determined for the case in which the maximum acceptable dose rate is 10 mrem/hr measured three feet perpendicular from the side of the waste form (case 4). These calculations were made for wastes containing 0.5 MeV or 1.0 MeV gamma emitting activity. For the urea-formaldehyde (UF) waste form, the attenuation coefficient was calculated assuming a waste/UF weight ratio of 2.0 while for bitumen, the attenuation coefficient used was that of bitumen alone since liquid waste is evaporated to dryness in this process. Calculated attenuation coefficients are tabulated in Table 9 for the solidification agent atomic weight percentage compositions shown in Table 8. The activity providing 10 mrem/hr at three feet from the side of bitumen and urea-formaldehyde waste forms are listed in Table 10. The effects of using a 25 weight percentage sodium sulfate solution rather than water as the waste on the activity that can be incorporated in cement and urea-formaldehyde waste forms at waste/binder weight ratios of 1.0 and 2.0 respectively and for a bitumen waste form containing 50 weight percent sodium sulfate salt are also shown in Table 10.

The relative shielding of the waste package (waste form and container) and the waste/binder ratio employed will determine the maximum specific activity of the waste that can be incorporated and meet the provisions of 49 CFR 173. The maximum waste specific activity (mCi/liter) has been tabulated in Table 11 for the allowable curie limits shown in Table 10 for 55 gallon portland cement, urea-formaldehyde and bitumen waste packages. Waste specific activity limits are dependent upon the volume of waste incorporated in the waste form and hence are decreased from the values shown in Table 11 for

TABLE 8

Solidification Agent Compositions (Exclusive of Waste)

A. Portland Type I Cement (Normalized) ⁽⁵⁾

<u>Constituent</u>	<u>Weight Percentage</u>
Ca	0.481
O	0.355
Si	0.109
Al	0.034
Fe	0.020

B. Urea-Formaldehyde ⁽²⁾

<u>Constituent</u>	<u>Weight Percentage</u>
C	33
H	3
O	44
N	20

C. Bitumen (Blown) ⁽⁶⁾

<u>Constituent</u>	<u>Weight Percentage</u>
C	80
H	8
O	4
S	8

TABLE 9
Attenuation Coefficients

<u>Solidification Agent</u>	<u>Waste</u> ⁽¹⁾	<u>Waste Form</u> <u>Density, g/cm³</u>	<u>Attenuation Coefficient, cm⁻¹</u>	
			<u>0.5 MeV</u>	<u>1.0 MeV</u>
portland cement (waste/cement = 1.0 by wt.)	A	2.0	0.184	0.132
	B	2.0	0.193	--
urea-formaldehyde (waste/UF = 2.0 by wt.)	A	1.1	0.103	0.072
	B	1.2	0.099	--
bitumen (water evaporated)	-- (2)	1.06	0.098	0.072
	B (3)	1.4	0.129	--

-
- (1) Waste types are A: water
 B: 25 wt.% Na₂SO₄ in water
- (2) Pure bitumen
- (3) Waste form contains 50 wt.% Na₂SO₄

TABLE 10

Activity in 55 Gallon Drum Cement, Urea-Formaldehyde, and Bitumen
Waste Packages⁽¹⁾ Producing 10 mrem/hr at Three Feet

<u>Solidification Agent</u>	<u>Waste⁽²⁾</u>	<u>Waste Form³ Density, g/cm³</u>	<u>Activity, Curies</u>	
			<u>0.5 MeV</u>	<u>1.0 MeV</u>
portland cement (waste/cement = 1.0 by wt.)	A	2.0	0.40	0.15
	B	2.0	0.41	--
urea-formaldehyde (waste/UF = 2.0 by wt.)	A	1.1	0.28	0.11
	B	1.2	0.27	--
bitumen (water evaporated)	-- ⁽³⁾	1.06	0.26	0.12
	B ⁽⁴⁾	1.4	0.30	--

(1) 18 gauge (0.050 inch) mild steel container

(2) Waste types are A: water
 B: 25 wt.% Na₂SO₄ in water

(3) Pure bitumen

(4) Waste form contains 50 wt.% Na₂SO₄

TABLE 11

Waste Specific Activity Producing 10 mrem/hr at Three Feet for
55 Gallon Drum Cement, Urea-Formaldehyde and Bitumen Waste Packages⁽¹⁾

<u>Solidification Agent</u>	<u>Waste⁽²⁾ Type</u>	<u>Waste/Binder by Weight</u>	<u>Waste Form Density g/cm³</u>	<u>Waste Volume ℓ</u>	<u>Activity, Curies</u>	<u>Waste Specific Activity mCi/ℓ</u>
portland cement	A	1.0	1.8	187	0.40 (0.5 MeV) 0.15 (1.0 MeV)	2.1 0.80
	B	1.0	2.0	173	0.41 (0.5 MeV)	2.4
urea-formaldehyde	A	2.0	1.1	153	0.28 (0.5 MeV) 0.11 (1.0 MeV)	1.8 0.72
	B	2.0	1.2	139	0.27 (0.5 MeV)	1.9
bitumen	A	(3)	1.06	771	0.26 (0.5 MeV) 0.12 (1.0 MeV)	0.34 0.16
	B	(4)	1.4	485	0.30 (0.5 MeV)	0.62

(1) 18 gauge (0.050 inch) mild steel container

(2) Waste type A: water, density = 1.0 g/cm³
 B: 25 wt.% Na₂SO₄, density = 1.2 g/cm³

(3) Waste form contains 50 wt.% waste solids, initial waste 14.3 wt.% solids

(4) Waste form contains 50 wt.% Na₂SO₄

increasing waste/binder ratios. The waste/binder ratios used in this table are not necessarily optimum ratios or those employed in practice. In the bitumen solidification process, volume reduction results from the evaporation of water from the input waste stream. The extent of volume reduction in this process is dependent upon the solids content of the waste with values of 3 to 20 reported for the volume reduction.⁽⁷⁾ The calculations in Table 11 consider bitumen waste forms containing weight percent waste solids, produced from water base waste containing 14.3 wt.% solids and a sodium sulfate waste containing 25 wt.% Na_2SO_4 . While the volume reduction occurring in this process may permit a larger volume of these initial wastes to be incorporated into a 55 gallon waste form than with other solidification agents, the waste specific activity must be lower to produce the same 10 mrem/hr dose rate at three feet. As such, it is particularly important with volume reduction systems to control the specific activity of the waste in order to avoid the use of shielding overpacks or exclusive use vehicles in complying with transportation regulations.

V. THERMAL PROPERTIES OF SOLIDIFIED WASTE FORMS

A. Thermal Conductivity

The thermal conductivity of cement, urea-formaldehyde, bitumen, and Dow polymer waste forms were determined at 45°C with a Dynatech TCHM-G undirectional heat flow thermal conductance analyzer. Cylindrical test specimens with a diameter of 1.8 inches and a thickness of 0.50 inch were prepared using each of the solidification agents listed above. The cement specimens were prepared with portland type II cement and distilled water at water/cement waste ratio of 0.5. Casco-Resin 2 urea-formaldehyde specimens employed a water/UF weight ratio of 2.0. Bitumen specimens were cast from a larger block of Pioneer 221 asphalt. Dow chemical polymer samples were made using a water/binder volume ratio of 1.5 by techniques specified by Dow Chemical Company personnel. Specimens were cured in sealed containers for one week prior to testing. The results of thermal conductivity testing are shown in Table 12. All specimens tended to lose water as a result of drying during testing (with the exception of bitumen which contains no water) which resulted in decreases in the thermal conductivity as shown in this table. The thermal conductivity of the Dow polymer has been previously measured for other waste/binder ratios.⁽¹⁾ In actual waste forms, the thermal conductivity will be dependent upon the type of waste solidified and the waste to binder ratio.

B. Specific Heat

The specific heats at 25°C portland type II cement, Borden Casco Resin-2 urea-formaldehyde, Pioneer 221 asphalt, and Dow polymer specimens were determined with a Perkin Elmer DSC-2 differential scanning calorimeter. Portland type II cement and urea-formaldehyde samples were prepared with water at water/binder weight ratios of 0.5 and 2.0 (2.6 by volume) respectively. Urea-formaldehyde polymerization was induced by the addition of 2.0 volume percent of a 25 wt.% sodium bisulfate solution. These specimens were cured at 72°F for one week prior to testing. The Pioneer 221 asphalt samples were taken from a larger cast block of asphalt. Dow polymer samples were prepared at a waste/binder volume ratio of 1.5. This sample was cured at 72°C for one day. The samples utilized for specific heat determinations are quite small, averaging

TABLE 12

Thermal Conductivity of Waste Forms at 45°C

<u>Solidification Agent</u>	<u>Water/Binder Ratio</u>		<u>Percentage Initial Weight</u>	<u>Thermal Conductivity cal/(cm-sec-°C)</u>
	<u>Weight Ratio</u>	<u>Volume Ratio</u>		
portland type II cement	0.5	1.0 ⁽¹⁾	100.0	0.0029
			90.5	0.0021
urea-formaldehyde (Casco-Resin 2)	2.0	2.6	100.0	0.0036
			63.1	0.0029
			33.0	0.0019
bitumen (Pioneer 221)	0.0	0.0	100.0	0.0011
Dow Polymer (vinyl ester-styrene)	--	1.5	100.0	0.0023
			90.0	0.0018
			80.0	0.0014

(1) Based on bulk cement density of 2.0 g/cm³; this value is dependent upon the packing of the dry cement powder.

approximately twenty milligrams. As a result, difficulties can be encountered in obtaining homogeneous samples for testing. The specific heat was determined in comparison to a sapphire calibration standard for three samples of each waste form formulation; the range of values obtained are listed in Table 13. Note that in actual practice the specific heat will be a function of the waste/binder ratio and the type of waste solidified.

C. Thermal Diffusivity

In the treatment of heat conduction problems, a derived property called the thermal diffusivity is useful. The thermal diffusivity α is defined as:

$$\alpha = \frac{k}{\rho c} \quad (2)$$

where k = thermal conductivity

ρ = density

c = specific heat

The values obtained for the thermal conductivity and specific heat for simulated waste forms in the two prior sections have been used to calculate the thermal diffusivities listed in Table 14. Note that these calculations utilize the thermal conductivity values at 45°C for 100% initial sample weight and the average value of the respective specific heat values at 25°C. Although the thermal conductivity values used in these calculations were obtained at 45°C (because the instrument would not be used to obtain measurements at 25°C), the thermal conductivity is not a strong function of temperature so that the resultant thermal diffusivity values are approximately representative of those at 25°C.

TABLE 13Specific Heat of Waste Forms at 25°C

<u>Solidification Agent</u>	<u>Waste/Binder Weight Ratio</u>	<u>Specific Heat at 25°C, cal/(g-°C)</u>
portland type II cement	0.5	0.385-0.510
urea-formaldehyde (Borden Casco Resin-2)	2.0	0.798-0.808
bitumen (Pioncer 221)	0.0	0.331-0.360
Dow polymer	1.5 ⁽¹⁾	0.44 -0.49

(1) volume ratio

TABLE 14Calculated Thermal Diffusivities of Waste Forms at 25°C

<u>Solidification Agent</u>	<u>Waste/Binder Weight Ratio</u>	<u>Density, g/cm³</u>	<u>Thermal Conductivity, (1) cal/(cm-sec-°C)</u>	<u>Specific Heat, (2) cal/(g-°C)</u>	<u>Thermal Diffusivity, cm²/sec</u>
cement	0.5	2.2	0.0029	0.43	0.0031
urea-formaldehyde	2.0	1.1	0.0036	0.80	0.0041
bitumen	0.0	1.0	0.0011	0.35	0.0031
Dow polymer	1.5 ⁽³⁾	1.0	0.0023	0.47	0.0049

(1) values indicated were measured at 45°C

(2) average value

(3) volume ratio

VI. SOLIDIFICATION OF SIMULATED WASTES WITH THE DOW POLYMER

Dow Industrial Service (DIS) a division of the Dow Chemical Company has developed a proprietary vinyl ester-styrene polymer system (Nuclear Binder 101) for the solidification of radioactive wastes. Polymerization is achieved by an addition mechanism using a promoter-catalyst system which permits curing without the addition of external heat. Wastes are entrapped in the polymer matrix; no chemical reaction between the waste and the solidification agent takes place. Solidification verification studies were performed at Brookhaven National Laboratory for the Dow polymer system with various simulated wastes. These specimens were prepared by the proprietary procedure as recommended by agents of the Dow Chemical Company. The waste/binder ratios employed for this testing were also recommended by Dow and are said to be representative the use of this product for the respective power reactor wastes. These tests have not been concluded to date and have not been performed using some of the simulated wastes utilized in other phases of this program. Samples were each made using 100 grams of the Dow resin monomer. Curing was accomplished in an open container. Solidified products were obtained with all wastes at their respective waste/binder volume ratios as listed in Table 15. No free standing water was noted for any of the waste forms produced. The simulated boric acid waste, 3c, which contains 12 wt.% boric acid required a proprietary pretreatment to form an acceptable waste solid with the Dow polymer. Gel times were measured from the time mixing of the waste with the polymer system components had been completed.

TABLE 15

Solidification Verification Studies Using the
Dow Polymer (Nuclear Binder 101)

<u>Waste Type</u>	<u>Waste/Binder Volume Ratio</u>	<u>Gel Time, Minutes</u>
1a. Bead Resin Waste (slurry)	2.0	8
3a. BWR Chemical Regenerative Waste	1.65	3
3c. Boric Acid Waste ⁽¹⁾	1.5	4
Boric Acid Waste (8 wt.% H_3BO_3)	1.5	3
Diatomaceous Earth Waste (40 wt.% in water)	1.5	4

(1) Required waste pretreatment to produce an acceptable solid.

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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.
Nutek 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

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

<u>Waste Type</u>	<u>Waste Density (D_1), g/cm³</u>	<u>Weight Ratio to Volume Ratio Conversion Factor (D_2/D_1)</u>	<u>Volume Ratio to Weight Ratio Conversion Factor (D_1/D_2)</u>
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

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