Solidification Testing for Several Savannah River Low Level Wastestreams Using Grouts, Sorbents and Gamma Radiation Shielding Materials - 9378

J. Bickford
MSE Technology Applications, Inc.
200 Technology Way, Butte, MT 59701

H. Burns
Washington Savannah River Company
Savannah River Site, Building 999W, Aiken, SC 29802

ABSTRACT

This paper describes the results for the solidification testing using surrogate formulas for several Savannah River Site wastes using grout and sorbents with and without the incorporation of a gamma radiation shielding material. The surrogate wastestreams used during the test sequence are representative of Legacy Plutonium Uranium Extraction (PUREX) process waste, a Rainwater waste, an Aqueous Organic waste, and three different oils. Samples were generated at bench scale to investigate the density distribution of the radiation shielding material within the solidified surrogate wastes and the ability of the shielding material to attenuate gamma radiation. The gamma radiation shielding material used for this testing is a proprietary, newly patented product aquired from Science and Technology Applications, LLC called Gamma Guard II (GG II).

INTRODUCTION

The U.S. Department of Energy (DOE) tasked MSE Technology Applications, Inc. (MSE) with evaluating various sorbent and grout formulations with the addition of radiation shielding components to solidify Savannah River Site (SRS) radioactive liquid organic and liquid aqueous wastestreams. MSE has previously tested several SRS surrogate wastes for solidification with different commercially available sorbent materials. Radiation shielding materials were added to the surrogate/sorbent or surrogate/grout combinations to determine if the shielding components were effective at controlling gamma radiation when combined with the liquid surrogate wastestreams and solidification materials.

Based on work performed at MSE [1, 2, 3, 4, 5] to solidify different SRS surrogate wastestreams, several sorbent/surrogate combinations have been identified as successful treatment options. MSE has tested surrogate Legacy and F-Canyon plutonium-uranium extraction (PUREX) organic wastestreams, a surrogate aqueous Rainwater wastestream, a surrogate Aqueous Organic wastestream consisting of approximately 6% oil, and a surrogate Oils Wastestream. Several sorbent surrogate and/or grout surrogate mixtures were tested using the weight-based, waste-loading ratios determined during previous MSE testing. The ratios varied when incorporating the radiation shielding materials; however, initial waste-loading ratios were based on previous MSE test results. Clay and polymer sorbents and Portland cement mixtures were tested during this test sequence to solidify the surrogate formulations both with and without radiation shielding materials.

The surrogate/sorbent and surrogate/grout combinations should be capable of withstanding conditions similar to those experienced during shipping and storage and be compatible with the radiation shielding materials and with solidification processing equipment. Sorbent and grout process evaluation criteria for the organic and aqueous surrogate wastestream testing at MSE included:

- sorbent and grout formulations;
- sorbent and grout handling;
- sorbent and grout curing rates;
- grout and sorbent mixing requirements;
- time to solidify;
- radiation shielding material physical and chemical compatibility;
- radiation shielding material distribution within the solidified sample;

- final wasteform physical characteristics; and
- effectiveness of the radiation shielding material to attenuate gamma radiation.

This work was performed at the MSE Test Facility located at the Mike Mansfield Advanced Technology Center in Butte, Montana. Samples were sent to Oak Ridge National Laboratory (ORNL) for radioactive bombardment after identifying several surrogate/sorbent and surrogate/grout combinations during the bench-scale test sequence.

TEST OBJECTIVES

The objective of this work was to identify surrogate/sorbent and surrogate/grout formulations that were compatible with the radiation shielding materials. The sorbent/surrogate combinations were tested using routine solidification tests including the Paint Filter Test (PFT) and the Liquid Release Test (LRT). The combinations were also tested for the distribution of the radiation shielding materials within the solidified surrogate wasteforms after chemical and physical compatibility was proven for the surrogate/sorbent and surrogate/ grout combinations with and without the inclusion of the shielding materials.

Specific objectives for the SRS organic and aqueous surrogate/sorbent bench-scale testing and evaluation were:

- verify appropriate waste-loading ratios and radiation shielding compatibility during small-scale compatibility studies;
- generate bench-scale samples based on the compatibility testing;
- verify the absence/presence of free liquid for the bench-scale surrogate/sorbent wasteforms using the PFT according to SW-846 Method 9095 [6];
- verify the amount of liquid released from the bench-scale surrogate/sorbent wasteforms using the LRT according to SW-846 Method 9096 [7]; and
- determine the density distribution of the radiation shielding materials within the bench-scale surrogate/sorbent wasteforms both visually and by weight and volume measurements.

Specific objectives for the SRS aqueous and organic surrogate/grout bench-scale testing and evaluation were:

- verify the waste-loading ratios and chemical and physical compatibility for the radiation shielding materials, grouts, and surrogate wastes;
- develop grout formulations at bench scale using the aqueous and organic surrogates and shielding materials using Portland cement based on the compatibility testing;
- verify the absence/presence of free liquid by observing the grouted surrogate wasteforms during the curing period; and
- determine the density distribution within the surrogate/grout wasteforms both visually and by weight and volume measurements.

The solidified organic and aqueous surrogate wasteforms were sent to ORNL for radioactive bombardment to test and evaluate the effectiveness of the radiation shielding materials to attenuate radiation within the solidified samples. ORNL provided MSE with the results of the testing for inclusion in this paper.

MATERIAL DESCRIPTIONS

The surrogate organic Legacy PUREX recipe was developed during bench-scale testing at SRS in 2001 [8]. SRS provided MSE with the surrogate recipes for the Rainwater surrogate, the Aqueous Organic surrogate, and the Oils Wastestream surrogate during 2005 testing [3, 4, 5]. The sorbents that were tested during this test sequence were identified by MSE as proven solidification techniques during previous sorbent testing for the solidification of the SRS wastestreams. Grouting experiments were completed at MSE during 2007 for a surrogate ORNL aqueous wastestream, and initial grout formulations for this work were based on the previous ORNL work.

Surrogate Formulations

The surrogate recipes for the SRS wastestreams are presented in Tables I through III. Three oils were also used as surrogate wastestreams: circulating oil, utility oil, and a thick oil. The circulating oil was Texaco Regal R&O 68; the utility oil was Chevron utility oil LVI, ISO 22; and the thick oil was Chevron Maropa 460.

Table I. Legacy PUREX Surrogate Recipe

Chemical Name	Weight Percent
Tributyl phosphate	17.60
Aliphatic Hydrocarbons	
Undecane	8.45
Dodecane	8.45
Tridecane	8.45
Tetradecane	8.45
Aromatic Hydrocarbons	
Diethylbenzene	21.00
Di-isopropylbenzene	21.00
Aliphatic Amine	
Di-n-octylamine	6.60
Total	100

Table II. Rainwater Surrogate Recipe

Chemical Constituent	Desired Concentration (mg/L)	Chemical Constituent	Desired Concentration (mg/L)
Aluminum (Al)	1.1	Ni	0.02
Barium (Ba)	0.03	P	3
Beryllium (Be)	0.01	K	43
Boron (B)	0.35	Se	0.02
Bromine (Br-)	0.34	Si	3
Cadmium (Cd)	0.01	Na	62
Calcium (Ca)	11	Sr	0.04
Chlorine (Cl)	14	Ti	0.01
Chromium (Cr)	0.02	U	0.2
Cobalt (Co)	0.04	V	0.01
Copper (Cu)	0.06	Zn	0.3
Fluoride	0.7	Acetone	2
Iron (Fe)	103	Xylene	0.1
Lead (Pb)	0.02	pH	5.3
Magnesium (Mg)	2	TOC[a]	2,000
Manganese (Mn)	1.5	TSS[b]	71
Molybdenum (Mo)	0.01		

[a] TOC = total organic carbon
[b] TSS = total suspended solids

Table III. Aqueous Organic Surrogate Recipe

Chemical Constituent	Weight Percent	Desired Conc. (mg/L)
Al	.0043	43.6
Barium carbonate (as Ba)	.0002	1.69
Beryllium acetate (as Be)	<.0001	0.23
Orthoboric acid (as B)	.0020	20.4
Ca	.0002	1.68
Calcium carbonate (as Ca)	.0874	883

Table III. Aqueous Organic Surrogate Recipe

Potassium dichromate (as Cr)	.0001	1.13
Co	<.0001	0.58
Cupric sulfate, pentahydrate (as Cu)	.0004	3.97
Iron sulfate, 7-hydrate (as Fe)	.7678	7762
Lead carbonate (as Pb)	.0004	4.45
Magnesium nitrate (as Mg)	.0077	78.2
Manganese sulfate (as Mn)	.0080	81.1
Mo	.0002	2.15
Nickel sulfate, 6-hydrate [as nickel (Ni)]	.0002	2.46
Potassium Nitrate [as potassium (K)]	.0286	289
Sodium selenate, decahydrate [as selenium (Se)]	.0004	4.01
Sodium silicate [as silicon (Si)]	.0010	9.76
Sodium nitrate [as sodium (Na)]	.0613	619
Strontium nitrate [as strontium (St)]	.0006	6.06
Ti	<.0001	0.65
Uranium (U)	.0002	2.27
Zinc oxide [as zinc (Zn)]	.0410	414
Acetone	.0229	232
phthalate	.0043	43.4
TOC	.0419	424
Water	98.9	
pH		5.4

Sorbent Descriptions

Sorbents identified for testing with the surrogate organic formulations are listed below.

- Petroset II Granular TM (Petroset II-G) is a modified granular clay-based stabilization agent that does not require mixing during the waste solidification process. This sorbent is distributed by Fluid Tech, Inc., and is designed to sorb organic liquids.
- Petroset II Powder TM (Petroset II-P) is a modified clay-based, fine-grained, powder-form sorbent that is distributed by Fluid Tech, Inc., and is designed to sorb organic liquids.
- Organoclay BM-QT-199 Granular (Organoclay-G) is a modified granular clay-based stabilization agent that does not require mixing during the waste solidification process. This sorbent is distributed by M^2 Polymer Technologies, Inc., and is designed to sorb organic liquids.
- Organoclay BML-QT-199 Powder (BML-QT-199) is a modified powdered clay-solidifying agent used to solidify organic liquids. This sorbent is distributed by M^2 Polymer Technologies, Inc.
- Organoclay LM-QT-Plus (LM-QT-Plus) is a powdered clay-solidifying agent used to solidify oils. This sorbent is distributed by M^2 Polymer Technologies, Inc.

Sorbents identified for testing with the surrogate aqueous formulations are listed below.

- Aquaset is a light grey-colored, water-activated, granular, clay-based solidification agent used for the treatment of aqueous liquids. The sorbent is distributed by Fluid Tech, Inc., and is designed to sorb aqueous-type liquids with up to 5% organics.
- Petroset is a light grey-colored, water-activated, powdered, clay-based solidification agent used for the treatment of aqueous liquids. The sorbent is distributed by Fluid Tech, Inc., and is designed to sorb aqueous-type liquids.

Grout Description

The grout material chosen for application in the testing scheme was Portland cement.

Radiation Shielding Material Description

One gamma radiation shielding material was tested during this test sequence. The product is a proprietary, newly patented shielding material aquired from Science and Technology Applications, LLC and is called Gamma Guard II (GG II).

COMPATIBILITY TESTING

Small samples were generated early in the test sequence to determine the chemical and physical compatibility of the sorbent and grout surrogate mixtures with the inclusion of the gamma shielding material, referred to as GG II. The organic surrogates, PUREX, and the three oils were not chemically compatible with the Portland cement; therefore, bench-scale samples were not generated using the organic surrogates with grout. The organic surrogates were chemically compatible with the granular and powdered clay sorbents; however, the granular clay products were not physically compatible with the dense GG II. The granular clay sorbent products are designed to eliminate mixing and could not evenly incorporate the gamma radiation shielding material into the solidified matrix; therefore, the granular clays (Petroset II-G, Organoclay-G,) were eliminated from the test matrix.

In summary, based on the compatibility testing, Petroset II-P sorbent was used to solidify the Legacy PUREX surrogate and all three oils. BML-QT-199 was used to solidify the Legacy PUREX surrogate, and LM-QT-Plus was used to solidify the oils. These three sorbents are all powdered clays and were the only sorbent or grout materials used to solidify the organic surrogate wastestreams when using the GG II shielding material in an attempt to generate denser samples.

Small samples were also generated using the Aqueous Organic and Rainwater surrogates with sorbents and GG II and grout and GG II for compatibility testing. The Portland cement was compatible both physically and chemically with these two aqueous surrogates. Sorbents were selected for this part of the test sequence based on long-term stability testing performed at the MSE Test Facility. Samples that initially passed all of the liquid release-type testing performed in 2004 and that continue to pass LRT testing in subsequent years were used to generate the sorbent samples that included the GG II shielding material. One sorbent that produced a dense wasteform and passed long-term stability LRT testing was Aquaset II. However, Aquaset II was not compatible with the shielding material; consequently, Petroset (a sister product) was substituted.

BENCH-SCALE TESTING

Two sample sets were generated for each of the sorbent or grout mixtures with each of the surrogates for radioactive bombardment testing. One sample set included the gamma radiation shielding material, and one set did not contain the shielding material. The sorbent and grout samples were weighed and measured to determine density values, and the sorbent samples were PFT and LRT tested. The sample sets were prepared for bombardment and sent to ORNL for radiation measurements. Sliced samples of the solidified wasteforms were placed in front of a collimated cesium-137 source, and the degree of gamma radiation attenuation was measured. For comparative studies, an empty box was bombarded with the selected radiation source strength of 175 roentgens per hour (R/hr) to determine the actual radiation reading provided by the selected source strength through the empty box; that value was 172.5 R/hr. The percent radiation attenuation for each sample was calculated using the radiation source strength of 172.5 R/hr.

Legacy PUREX Surrogate Sorbent Testing with GG II Shielding Material

Quart-sized samples of the sorbent and Legacy PUREX surrogate waste forms were generated during the bench-scale sorbent testing at specific weight-based, total solids-to-surrogate ratios and differing weight-based ratios of shielding material to sorbent. The Legacy PUREX surrogate was combined with Petroset II-P and BML-QT-199 at 1:1, 1.5:1, and 2:1 total solid-to-surrogate ratios with GG II-to-sorbent ratios that varied from 1:1 up to 2.5:1 (determined early in the compatibility testing).

A neat sample (a sample without GG II) was also generated during testing to compare with the shielded samples containing GG II. The test matrix for the bench-scale PUREX surrogate sorbent work with and without the inclusion of GG II is presented in Table IV. Note that the neat samples do not reflect the total solid-to-surrogate ratios for the samples generated with GG II. The maximum amount of sorbent that could be incorporated into the surrogate liquid was used in this application for the neat samples. All of the sorbent, PUREX, and shielding samples were mixed, and the shielding material was easily incorporated at bench scale.

After a 2-week curing period, the samples were removed from the sample containers, and sub samples were collected for density, PFT, and LRT data. Density values for the top and bottom sections of the bench-scale samples were collected to help determine if the dense GG II shielding material was evenly incorporated into the sample matrix. The sample consistency was also checked when the samples were removed from the containers. Table IV also presents the density and LRT data collected from the SRS Legacy PUREX and sorbent samples with and without the incorporation of the gamma shielding material.

Table IV. Test Matrix, Density, and LRT Data Collected from the Legacy PUREX Surrogate Sorbent Samples with and without the Inclusion of GG II Shielding Material

Sorbent Name	Surrogate Name	Total Solids-to-Surrogate Ratio (solid:liquid)	GG II-to-Sorbent Ratio (shielding:sorbent)	Sample Section	Density Values per Sample Section (g/mL)	LRT % Liquid Released by Volume (NTS WAC < 0.5%)
Legacy PUREX – Petroset II-G – GG II						
Petroset II-P	Legacy PUREX	1:1	1:1	Top	1.29	0.371
				Bottom	1.28	
Petroset II-P	Legacy PUREX	1.5:1	2:1	Top	1.59	0.368
				Bottom	1.59	
Petroset II-P	Legacy PUREX	2:1	2:1	Top	1.68	0.319
				Bottom	1.68	
Petroset II-P	Legacy PUREX	2:1	2.5:1	Top	1.77	0.346
				Bottom	1.76	
Petroset II-P	Legacy PUREX	0.7:1	NA - Neat Sample	Top	1.03	0.308
				Bottom	1.03	
Legacy PUREX – BML-QT-199 – GG II						
BML-QT-199	Legacy PUREX	1:1	1:1	Top	1.36	0.380
				Bottom	1.35	
BML-QT-199	Legacy PUREX	1.5:1	2:1	Top	1.52	0.389
				Bottom	1.51	
BML-QT-199	Legacy PUREX	2:1	2:1	Top	1.66	0.306
				Bottom	1.67	
BML-QT-199	Legacy PUREX	2:1	2.5:1	Top	1.74	0.356
				Bottom	1.75	
BML-QT-199	Legacy PUREX	0.7:1	NA - Neat Sample	Top	1.00	0.320
				Bottom	1.01	

SW-846 Method 9095A, *Paint Filter Free Liquids Test Procedure* [6], was used to determine if free liquids existed in the final sorbent/surrogate wasteforms, and SW-846 Method 9096, *Liquids Release Test Procedure* [7], was used to determine the amount of liquid released from the final wasteforms. Only samples that passed the PFT were subjected to LRT because any loaded sorbent that fails the PFT was assumed to release liquids if subjected to pressure during the LRT.

The Legacy PUREX samples all passed the PFT and LRT requirements for this test sequence. All of the LRT values were below the Nevada Test Site (NTS) Waste Acceptance Criteria (WAC) for LRT of < 0.5% release by volume. The sample density values were the same or within 0.01 grams per milliliter (g/mL) from the top and bottom sections of the bench-scale samples, which indicates that the heavy GG II radiation shielding material can be evenly incorporated into the PUREX and clay sorbent matrices. The sample densities increased as more of the GG II shielding material was added to the samples. The sample densities ranged from approximately 1 g/mL for the two neat sorbent samples up to approximately 1.77 g/mL for the samples with the largest amount of shielding material incorporated. All of the Legacy PUREX samples had the same sample consistency of a soft paste resembling room temperature peanut butter.

Table V presents the gamma attenuation data collected by ORNL for the Legacy PUREX surrogate sample set. The percent of gamma attenuation for each sample set increased as the amount of shielding material increased in the samples and ranged from approximately 25% to 36%. The neat samples (samples without GG II shielding material) provided attenuation values of approximately 18%. The samples that included GG II shielding material attenuated gamma radiation approximately 8% to 22% better than the neat sorbent samples. As expected, the attenuation values trend with the density data presented in Table IV (i.e., as the density values increase the samples provide better gamma attenuation). The density and attenuation data prove that the dense GG II material can be evenly incorporated into a surrogate Legacy PUREX and powdered clay matrix. Fig. 1 shows the neat BML-QT-199 sample on the left, the 2:1 to 2:1 shielded sample in the middle, and the 2:1 to 2.5:1 shielded sample on the right before sample bombardment.

Table V. Legacy PUREX Surrogate and Sorbent Gamma Attenuation Results

Sorbent Name	Total Solids-to-Surrogate Ratio (solid:liquid)	GG II-to-Sorbent Ratio (shielding:sorbent)	Radiation Source Strength (R/hr)	Radiation Reading (R/hr)	Percent Attenuation for Sample	Percent Attenuation Shielded to Neat Sample
Empty Box	---	---	175	172.5	---	---
Legacy PUREX – Petroset II-P – GG II						
Petroset II-P	1:1	1:1	172.5	126.9	26.4	9.3
Petroset II-P	1.5:1	2:1	172.5	116.7	32.3	16.6
Petroset II-P	2:1	2:1	172.5	115.1	33.3	17.7
Petroset II-P	2:1	2.5:1	172.5	111.3	35.5	20.4
Petroset II-P	0.7:1	NA - Neat Sample	172.5	139.9	18.9	0
Legacy PUREX – BML-QT-199 – GG II						
BML-QT-199	1:1	1:1	172.5	129.6	24.9	8.2
BML-QT-199	1.5:1	2:1	172.5	120.1	30.4	14.9
BML-QT-199	2:1	2:1	172.5	112.0	35.1	20.6
BML-QT-199	2:1	2.5:1	172.5	110.4	36.0	21.8
BML-QT-199	0.7:1	NA - Neat Sample	172.5	141.1	18.2	0

Fig. 1. Neat BML-QT-199 sample (left), the 2:1 to 2:1 sample (middle), and the 2:1 to 2.5:1 sample (right) before bombardment.

Oil and Sorbent Testing with GG II Shielding Material

Quart-sized samples of the sorbent and oil wastestreams were generated during the bench-scale sorbent testing at specific weight-based total solids-to-surrogate ratios and differing weight-based total solids ratios of shielding material to sorbent. The oil wastestreams were combined with Petroset II-P and LM-QT-Plus at 2:1, 2.5:1, and 3:1 total solid-to-surrogate ratios with GG II-to-sorbent ratios that varied from 1:1 up to 3:1 determined during compatibility testing. The test matrix and density and LRT data for the Oil Wastestream sorbent testing with and without the addition of GG II shielding material is presented in Table VI.

Table VI. Test Matrix, Density, and LRT Data Collected from the Surrogate Oil and Sorbent Samples with and without the Inclusion of GG II Shielding Material

Sorbent Name	Surrogate Name	Total Solids to Surrogate Ratio (solid:liquid)	GG II to Sorbent Ratio (shielding:sorbent)	Sample Section	Density Values per Sample Section (g/mL)	LRT % Liquid Released by Volume (NTS WAC < 0.5%)
Utility Oil – Petroset II-P – GG II						
Petroset II-P	Utility Oil	2:1	1:1	Top	1.65	0.313
				Bottom	1.66	
Petroset II-P	Utility Oil	2:1	2:1	Top	1.77	0.261
				Bottom	1.76	
Petroset II-P	Utility Oil	2.5:1	2:1	Top	1.88	0.231
				Bottom	1.89	
Petroset II-P	Utility Oil	3:1	3:1	Top	2.13	0.231
				Bottom	2.12	
Petroset II-P	Utility Oil	0.83:1	NA – Neat Sample	Top	1.15	0.344
				Bottom	1.17	
LM-QT-Plus	Utility Oil	2:1	1:1	Top	1.54	0.333
				Bottom	1.55	
LM-QT-Plus	Utility Oil	2:1	2:1	Top	1.71	0.414
				Bottom	1.71	
LM-QT-Plus	Utility Oil	2.5:1	2:1	Top	1.89	0.424
				Bottom	1.89	
LM-QT-Plus	Utility Oil	3:1	3:1	Top	2.12	0.283
				Bottom	2.13	
LM-QT-Plus	Utility Oil	0.83:1	NA – Neat Sample	Top	1.14	0.374
				Bottom	1.15	
Regal Oil – Petroset II-P – GG II						
Petroset II-P	Regal Oil	2:1	1:1	Top	1.63	0.326
				Bottom	1.64	
Petroset II-P	Regal Oil	2:1	2:1	Top	1.74	0.321
				Bottom	1.74	
Petroset II-P	Regal Oil	2.5:1	2:1	Top	1.85	0.410
				Bottom	1.85	
Petroset II-P	Regal Oil	3:1	3:1	Top	2.09	0.409
				Bottom	2.10	
Petroset II-P	Regal Oil	1.08:1	NA – Neat Sample	Top	1.16	0.355
				Bottom	1.17	
LM-QT-Plus	Regal Oil	2:1	1:1	Top	1.55	0.347
				Bottom	1.55	
LM-QT-Plus	Regal Oil	2:1	2:1	Top	1.73	0.408
				Bottom	1.72	
LM-QT-Plus	Regal Oil	2.5:1	2:1	Top	1.88	0.306
				Bottom	1.88	
LM-QT-	Regal Oil	3:1	3:1	Top	2.11	0.440

Table VI. Test Matrix, Density, and LRT Data Collected from the Surrogate Oil and Sorbent Samples with and without the Inclusion of GG II Shielding Material

Sorbent Name	Surrogate Name	Total Solids to Surrogate Ratio (solid:liquid)	GG II to Sorbent Ratio (shielding:sorbent)	Sample Section	Density Values per Sample Section (g/mL)	LRT % Liquid Released by Volume (NTS WAC < 0.5%)
Plus				Bottom	2.12	
LM-QT-Plus	Regal Oil	1.25:1	NA – Neat Sample	Top	1.15	0.362
				Bottom	1.15	
Maropa Oil – Petroset II-P – GG II						
Petroset II-P	Maropa Oil	2:1	1:1	Top	1.62	0.307
				Bottom	1.60	
Petroset II-P	Maropa Oil	2:1	2:1	Top	1.78	0.410
				Bottom	1.79	
Petroset II-P	Maropa Oil	2.5:1	2:1	Top	1.82	0.293
				Bottom	1.81	
Petroset II-P	Maropa Oil	3:1	3:1	Top	2.15	0.284
				Bottom	2.15	
Petroset II-P	Maropa Oil	1.03:1	NA – Neat Sample	Top	1.19	0.367
				Bottom	1.20	
LM-QT-Plus	Maropa Oil	2:1	1:1	Top	1.63	0.317
				Bottom	1.63	
LM-QT-Plus	Maropa Oil	2:1	2:1	Top	1.80	0.417
				Bottom	1.80	
LM-QT-Plus	Maropa Oil	2.5:1	2:1	Top	1.88	0.286
				Bottom	1.89	
LM-QT-Plus	Maropa Oil	3:1	3:1	Top	2.12	0.262
				Bottom	2.12	
LM-QT-Plus	Maropa Oil	1.1:1	NA – Neat Sample	Top	1.17	0.358
				Bottom	1.18	

A neat sample (a sample without GG II) was also generated during testing to compare with the shielded samples containing GG II. Note that the neat samples do not reflect the total solid-to-surrogate ratios for the samples generated with the gamma shielding material. The maximum amount of sorbent that could be incorporated into the surrogate liquid was used in this application for the neat samples.

All of the sorbent, oil, and shielding samples were cured for 3 weeks instead of the standard cure time of 2 weeks based on the long-term stability results for oils samples stored at the MSE Test Facility [9]. All of the oil samples had the consistency of chilled to cold peanut butter; however, the Maropa oil produced samples that were very sticky.

The oil samples all passed the PFT and LRT requirements for this test sequence. All of the LRT values were below the NTS WAC for LRT of < 0.5% release by volume. The sample density values were the same or within 0.02 g/mL from the top and bottom sections of the bench-scale samples, which indicates that the heavy GG II radiation shielding material can be evenly incorporated into the oil and powdered clay sorbent matrices. The sample densities increased as more of the GG II shielding material was added to the samples, as expected.

The sample density for the neat utility oil samples ranged from 1.15 to 1.17 g/mL for the Petroset II-P samples and from 1.14 to 1.15 g/mL for the LM-QT-Plus samples. The utility oil sorbent sample densities with the inclusion of the GG II shielding material ranged from 1.54 to 2.13 g/mL, depending on the amount of shielding material that was added. The sample density for the neat Regal oil samples ranged from 1.16 to 1.17 g/mL for the Petroset II-P samples and was 1.15 g/mL for both the top and bottom LM-QT-Plus sample sections. The Regal oil sorbent sample with the inclusion of GG II shielding material had densities that ranged from 1.55 to 2.12 g/mL. The sample densities for the neat Maropa oil samples ranged from 1.19 to 1.20 g/mL for the Petroset II-P samples and from 1.17 to 1.18 g/mL for the LM-QT-Plus samples. The Maropa oil sorbent samples with the inclusion of the GG II shielding material ranged from 1.60 to 2.12 g/mL.

Table VII presents the gamma attenuation data collected by ORNL for the oil sample set. The powdered clay sorbents when combined with the three different oils provided a good matrix to incorporate the GG II shielding material. The data show a good trend between the density values presented in Table VI and the gamma attenuation data presented in Table VII. As more of the gamma shielding material was incorporated into the sample matrix, the density values increased and provided better gamma attenuation. The neat oil samples all provided approximately 18% gamma attenuation while the shielded samples provide gamma attenuation values ranging from around 29% to approximately 40%. The shielded samples provide gamma attenuation values ranging from 12% to 27% better than the neat oil sorbent samples. The data indicate that the dense radiation shielding material can be evenly incorporated into an oil and powdered clay matrix and that it provides good gamma attenuation.

Table VII. Oils Surrogate Sorbent Gamma Attenuation Results

Sorbent Name	Total Solids-to-Surrogate Ratio (solid:liquid)	GG II-to-Sorbent Ratio (shielding:sorbent)	Radiation Source Strength (R/hr)	Radiation Reading (R/hr)	Percent Attenuation for Sample	Percent Attenuation Shielded to Neat Sample
Empty Box	---	---	175	172.5	---	---
Utility Oil – Sorbents – GG II						
Petroset II-P	2:1	1:1	172.5	120.6	30.1	14.9
Petroset II-P	2:1	2:1	172.5	116.1	32.7	18.1
Petroset II-P	2.5:1	2:1	172.5	113.5	34.2	19.9
Petroset II-P	3:1	3:1	172.5	102.6	40.5	27.6
Petroset II-P	0.83:1	NA – Neat Sample	172.5	141.7	17.9	0
LM-QT-Plus	2:1	1:1	172.5	120.9	29.9	14.2
LM-QT-Plus	2:1	2:1	172.5	114.6	33.6	18.7
LM-QT-Plus	2.5:1	2:1	172.5	112.9	34.6	19.9
LM-QT-Plus	3:1	3:1	172.5	107.7	37.6	23.6
LM-QT-Plus	0.83:1	NA – Neat Sample	172.5	140.9	18.3	0
Regal Oil – Sorbents – GG II						
Petroset II-P	2:1	1:1	172.5	121.3	29.7	13.4
Petroset II-P	2:1	2:1	172.5	113.8	34.0	18.7
Petroset II-P	2.5:1	2:1	172.5	114.5	33.6	18.2
Petroset II-P	3:1	3:1	172.5	103.4	40.1	26.1
Petroset II-P	0.83:1	NA – Neat Sample	172.5	140.0	18.8	0
LM-QT-Plus	2:1	1:1	172.5	118.7	31.2	15.3
LM-QT-Plus	2:1	2:1	172.5	114.3	33.7	18.4
LM-QT-Plus	2.5:1	2:1	172.5	114.3	33.7	18.4
LM-QT-Plus	3:1	3:1	172.5	103.9	39.8	25.8
LM-QT-Plus	0.83:1	NA – Neat Sample	172.5	140.1	18.8	0
Maropa Oil – Sorbents – GG II						
Petroset II-P	2:1	1:1	172.5	122.0	29.3	12.3
Petroset II-P	2:1	2:1	172.5	116.0	32.8	16.6
Petroset II-P	2.5:1	2:1	172.5	113.0	34.5	18.8
Petroset II-P	3:1	3:1	172.5	102.4	40.6	26.4
Petroset II-P	0.83:1	NA – Neat Sample	172.5	139.1	19.4	0
LM-QT-Plus	2:1	1:1	172.5	123.0	28.7	12.5

Table VII. Oils Surrogate Sorbent Gamma Attenuation Results

Sorbent Name	Total Solids-to-Surrogate Ratio (solid:liquid)	GG II-to-Sorbent Ratio (shielding:sorbent)	Radiation Source Strength (R/hr)	Radiation Reading (R/hr)	Percent Attenuation for Sample	Percent Attenuation Shielded to Neat Sample
LM-QT-Plus	2:1	2:1	172.5	115.3	33.2	18.0
LM-QT-Plus	2.5:1	2:1	172.5	110.6	35.9	21.3
LM-QT-Plus	3:1	3:1	172.5	105.6	38.8	24.9
LM-QT-Plus	0.83:1	NA – Neat Sample	172.5	140.6	18.5	0

Rainwater Surrogate, Sorbent, and Grout Testing with GG II Shielding Material

Quart-sized samples of the sorbent and Rainwater surrogate wastestream were generated during the bench-scale sorbent testing at specific weight-based total solids-to-surrogate ratios and differing weight-based total solids ratios of shielding material to sorbent. The Rainwater surrogate wastestream was combined with Petroset at 1:1 and 1.5:1 total solid-to-surrogate ratios with GG II-to-sorbent ratios that varied from 1.5:1 up to 4:1 determined during compatibility testing. The Rainwater surrogate wastestream and grout samples were generated in 1-gallon containers and were checked daily for the presence of free liquid. The samples were generated at total solids-to-surrogate ratios of 2:1, 2.5:1, and 3:1 with different GG II-to-Portland cement ratios that ranged from 2:1 to 4:1. The test matrix for the Rainwater surrogate wastestream sorbent and grout testing with and without the addition of GG II shielding material is presented in Table VIII.

Table VIII. Test Matrix, Density, and LRT Data Collected from the Rainwater Surrogate, Sorbent, and Grout Samples with and without the Inclusion of GG II or RS II Shielding Material

Sorbent Name	Surrogate Name	Total Solids-to-Surrogate Ratio (solid:liquid)	GG II- or RS II-to-Sorbent or Grout Ratio (shielding:sorbent or grout)	Sample Section	Density Values per Sample Section (g/mL)	LRT % Liquid Released by Volume (NTS WAC < 0.5%)
Rainwater – Petroset – GG II						
Petroset	Rainwater	1:1	1.5:1	Top	1.58	0.336
				Bottom	1.58	
Petroset	Rainwater	1:1	2:1	Top	1.65	0.407
				Bottom	1.65	
Petroset	Rainwater	1:1	3:1	Top	1.75	**0.582**
				Bottom	1.75	
Petroset	Rainwater	1:1	4:1	Top	1.78	0.332
				Bottom	1.77	
Petroset	Rainwater	1.5:1	2:1	Top	1.88	0.396
				Bottom	1.89	
Petroset	Rainwater	1.5:1	3:1	Top	2.04	**0.574**
				Bottom	2.02	
Petroset	Rainwater	1.5:1	4:1	Top	2.16	0.350
				Bottom	2.17	
Petroset	Rainwater	1:2	NA – Neat Sample	Top	1.24	0.489
				Bottom	1.24	
Rainwater – Portland Cement – RS II						
Portland cement	Rainwater	2:1	3:1	Top	2.22	---
				Bottom	2.22	
Portland	Rainwater	2.5:1	3:1	Top	2.58	---

Table VIII. Test Matrix, Density, and LRT Data Collected from the Rainwater Surrogate, Sorbent, and Grout Samples with and without the Inclusion of GG II or RS II Shielding Material

Sorbent Name	Surrogate Name	Total Solids-to-Surrogate Ratio (solid:liquid)	GG II- or RS II-to-Sorbent or Grout Ratio (shielding:sorbent or grout)	Sample Section	Density Values per Sample Section (g/mL)	LRT % Liquid Released by Volume (NTS WAC < 0.5%)
cement				Bottom	2.50 [a]	
Portland cement	Rainwater	2.5:1	4:1	Top	2.51	---
				Bottom	2.49	
Portland cement	Rainwater	3:1	3:1	Top	2.80	---
				Bottom	2.77 [b]	
Portland cement	Rainwater	3:1	4:1	Top	2.85	---
				Bottom	2.87	
Portland cement	Rainwater	2:1	NA – Neat Sample	Middle	1.80	---
Portland cement	Rainwater	2.5:1	NA – Neat Sample	Middle	1.95	---
Portland cement	Rainwater	3:1	NA – Neat Sample	Middle	1.97	---

--- = No LRT value for cement samples only for sorbent samples
[a] = sample was thinner
[b] = sample had a hole
Bold - Failed NTS liquid release WAC

A neat sample (a sample without GG II) was also generated during testing for Petroset and Portland cement to compare with the shielded samples containing GG II. Note that the neat Petroset sorbent sample does not reflect the total solid-to-total liquid ratios for the samples generated with the gamma shielding material. The maximum amount of sorbent that could be incorporated into the surrogate liquid was used in this application for the neat samples. Three neat samples were generated using Portland cement and the Rainwater surrogate waste at solids-to-surrogate ratios of 2:1, 2.5:1, and 3:1, which represent the three total solids-to-liquid ratios used for the grout samples that included the GG II shielding material. An additional component was added to the grout and GG II samples to help keep the dense gamma shielding material evenly incorporated within the grout matrix, and this grouting formulation is referred to as Radioactive Shielding II (RS II). MSE identified this formulation during previous ORNL grout testing.

The Rainwater sorbent and GG II samples were allowed to cure for the standard 2-week curing period while the Rainwater grout and shielding material samples were allowed to cure for 1 week before being removed from the sample containers. At the end of the 2-week curing period, the sorbent samples were tested for PFT, LRT, and density. The grout samples were tested for density after they were removed from the sample containers and cut into sample sections. Grout samples were not tested using the PFT or LRT since these tests only apply to sorbent samples; however, they were visually inspected for free liquids on top of the grouted surface during the curing period. None of the grout samples had any free liquid after the curing period. The density and LRT data collected from the Rainwater samples are also presented in Table VIII.

All of the Rainwater sorbent and gamma shielding samples passed the PFT; however, the 1:1 to 3:1 and the 1.5:1 to 3:1 Petroset samples failed the NTS LRT WAC of < 0.5% release by volume. The samples that failed the LRT criteria have their LRT values bolded in Table VIII. The neat Rainwater Petroset samples had a top and bottom density of 1.24 g/mL while the shielded sorbent samples have density values that range from 1.58 to 2.17 g/mL. The top and bottom density values were within 0.02 g/mL, indicating that the GG II shielding material can be evenly incorporated into the surrogate Rainwater and Petroset sorbent matrix.

The Rainwater and Portland cement samples all cured without the presence of free liquid on top of the samples. After a 1-week cure time, the samples were removed from the sample containers and cut into sections for top and bottom density measurements. The neat 2:1 Portland cement sample had a density value of 1.80 g/mL while the shielded 2:1 sample had a density value of 2.22 g/mL for both the top and bottom sample sections. The 2.5:1 neat sample had a density value of 1.95 g/mL while the 2.5:1 shielded samples had density values that ranged from 2.49 to 2.58 g/mL. The 3:1 neat sample had a density value of 1.97 g/mL while the shielded samples had density values that ranged from 2.77 to 2.87 g/mL.

The 2.5:1 to 3:1 Portland cement sample had a bottom density value of 2.50 g/mL, which is low when compared to the top density value of 2.58 g/mL. The 3:1 to 3:1 bottom sample had a sizable hole in the sample, and the value of 2.77 g/mL is an estimate of the sample density based on the size of the hole discovered in the sample. Although the density values for these two samples have a wider density range than the rest of the grouted samples, MSE feels the density values are closer than the two values reported in the table. The top and bottom density value range was between 0 g/mL and 0.02 g/mL for the rest of the grouted samples, which shows good incorporation of the RS II shielding formulation in the grouted sample matrices.

Table IX presents the gamma attenuation data collected by ORNL for the Rainwater sample set. Petroset (the powdered clay sorbent) when combined with the Rainwater surrogate waste provided a good matrix to incorporate the GG II shielding material. The data show a good trend between the density values presented in Table VIII and the gamma attenuation data presented in Table IX with the exception of the 1:1 to 3:1 sample, which failed the LRT. The samples that failed the LRT criteria have bolded LRT and attenuation values. As more of the gamma shielding material was incorporated into the sample matrix, the density values increased and provided better gamma attenuation. The neat Rainwater sample provided approximately 20% gamma attenuation while the shielded samples provide gamma attenuation values ranging from around 30% to approximately 38%. The shielded samples provide gamma attenuation values ranging from 13% to 22% better than the neat Rainwater sorbent samples.

The data indicate that the dense radiation shielding material can be evenly incorporated into the Rainwater surrogate and powdered clay matrix and it does provide good gamma attenuation. Fig. 2 shows the Rainwater Portland cement 3:1 neat sample on the left, the top sample sections for the 3:1 to 3:1 shielded sample in the middle, and the 3:1 to 4:1 shielded sample on the right.

The Portland cement and Rainwater samples provided a good matrix to incorporate the GG II shielding material. The data show a decent trend between density values presented in Table VIII and the gamma attenuation results. As expected, as the shielding amounts were increased, the density values increased and provided better attenuation results. The neat Rainwater Portland cement samples had attenuation values of approximately 28% while the shielded samples had attenuation values that ranged from 42% to 55%. The shielded samples provided attenuation values ranging from approximately 20% to 37% better than the neat Rainwater Portland cement samples.

Table IX. Rainwater Surrogate, Sorbent, and Grout Gamma Attenuation Results

Sorbent or Grout Name	Total Solids-to-Surrogate Ratio (solid:liquid)	GG II-to-Sorbent or Grout Ratio (shielding:sorbent or grout)	Sample Section	Radiation Source Strength (R/hr)	Radiation Reading (R/hr)	Percent Attenuation for Sample	Percent Attenuation Shielded to Neat Sample
Empty Box	---	---		175	172.5	---	---
Rainwater – Petroset – GG II							
Petroset	1:1	1.5:1	Middle	172.5	119.9	30.5	13.0
Petroset	1:1	2:1	Middle	172.5	119.2	30.9	13.5
Petroset	1:1	3:1	Middle	172.5	120.8	**30.0**	**12.3**
Petroset	1:1	4:1	Middle	172.5	114.6	33.6	16.8
Petroset	1.5:1	2:1	Middle	172.5	115.5	35.4	19.1
Petroset	1.5:1	3:1	Middle	172.5	107.7	**37.6**	**21.8**
Petroset	1.5:1	4:1	Middle	172.5	106.9	38.0	22.4
Petroset		NA – Neat Sample	Middle	172.5	137.8	20.1	0
Rainwater – Portland Cement – RS II							
Portland cement	2:1	3:1	Top	172.5	99.8	42.1	20.2

			Bottom	172.5	100.5	41.7	19.7
Portland cement	2.5:1	3:1	Top	172.5	87.7	49.2	29.6
			Bottom	172.5	93.5	45.8	25.0
Portland cement	2.5:1	4:1	Top	172.5	91.4	47.0	26.6
			Bottom	172.5	92.6	46.3	25.7
Portland cement	3:1	3:1	Top	172.5	87.1	49.5	29.2
			Bottom	172.5	87.7	49.2	28.7
Portland cement	3:1	4:1	Top	172.5	89.6	48.1	27.1
			Bottom	172.5	77.2	55.2	37.2
Portland cement	2:1	NA – Neat Sample	Middle	172.5	125.1	27.5	0
Portland cement	2.5:1	NA – Neat Sample	Middle	172.5	124.6	27.8	0
Portland cement	3:1	NA – Neat Sample	Middle	172.5	122.9	28.7	0

Bold - Failed NTS liquid release WAC

Fig. 2. The Rainwater Portland cement 3:1 neat sample (left) and the top sample sections for the 3:1 to 3:1 shielded sample (middle) and the 3:1 to 4:1 shielded sample (right).

The 2.5:1 to 3:1 Portland cement sample had density and attenuation values that were not consistent from the top to the bottom of the sample. When comparing density values for that sample, the top sample had a larger density of 2.58 g/mL compared to the bottom sample section that had a density of 2.50 g/mL, which resulted in a better top section attenuation value of 49.2% compared to 45.8% for the bottom sample section. The 3:1 to 3:1 Portland cement sample had density values that varied due to a hole in the bottom sample section, which resulted in a slightly lower attenuation value for the bottom section. The 3:1 to 4:1 Portland cement sample had density values that were within 0.02 g/mL; however, the attenuation value for the top sample section was 48.1% compared to 55.2%. The explanation for the differing attenuation values for that sample resulted from a smear of the shielding material in the middle of the bottom sample section. A lower speed was used to cut the samples than was used for previous work, which produced the melted metal smear shown in Fig. 3. The sample labeled 57 is the top sample section, and the sample labeled 58 is the bottom sample section. Note the dark center section shown on sample 58–this is a smear of the melted shielding material. The person cutting the samples alerted the Project Manager when this phenomenon occurred during the cutting process. This phenomenon happened only when cutting the samples that had the largest shielding loading ratio of 4:1 (shielding material to Portland cement).

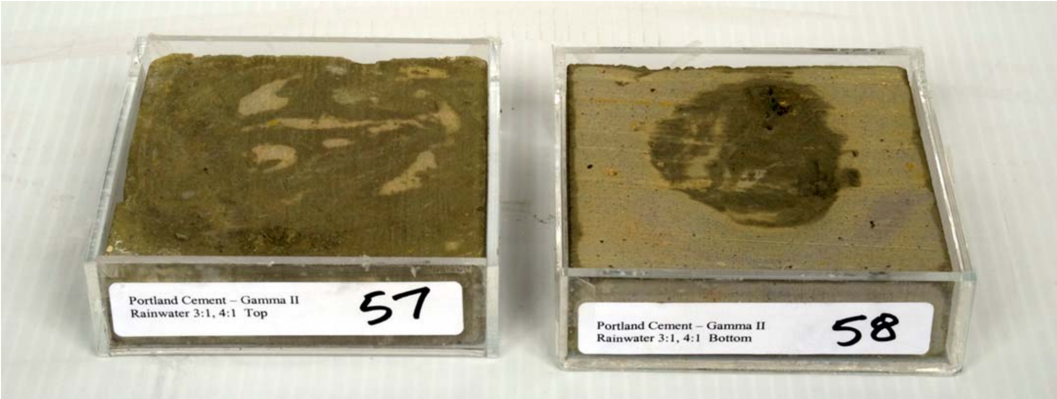

Fig. 3. Sample 58 shows the melted metal smear of the shielding material.

Aqueous Organic Surrogate, Sorbent, and Grout Testing with GG II Shielding Material

Quart-sized samples of the sorbent and Aqueous Organic surrogate wastestream were generated during the bench-scale sorbent testing at specific weight-based total solids-to-surrogate ratios and differing weight-based total solids ratios of shielding material to sorbent. The Aqueous Organic surrogate wastestream was combined with Petroset at 1:1 and 1.5:1 total solid-to-total surrogate liquid ratios with GG II to sorbent ratios that varied from 1.5:1 up to 4:1 determined during compatibility testing. The Aqueous Organic surrogate wastestream and grout samples were generated in 1-gallon containers and were checked daily for the presence of free liquid. The samples were generated at total solids-to-surrogate ratios of 2:1, 2.5:1, and 3:1 with differing amounts of RS II to Portland cement. The test matrix for the Aqueous Organic surrogate wastestream sorbent grout testing with and without the addition of GG II or RS II shielding material is presented in Table X.

A neat sample (a sample without GG II) was also generated during testing for Petroset and Portland cement to compare with the shielded samples containing GG II or RS II shielding formulation. Note that the neat Petroset sorbent sample does not reflect the total solid-to-surrogate ratios for the samples generated with the gamma shielding material. The maximum amount of sorbent that could be incorporated into the surrogate liquid was used in this application for the neat sample, which correlates to a total solid-to-surrogate ratio of 1:1.25. Three neat samples were generated using Portland cement and the Aqueous Organic surrogate waste at total solids-to-surrogate ratios of 2:1, 2.5:1, and 3:1, which represent the three total solids-to-surrogate ratios used for the grout samples that included the RS II shielding formulation.

The Aqueous Organic sorbent and shielding samples were allowed to cure for the standard 2-week curing period while the Aqueous Organic grout and shielding material samples were allowed to cure for 1 week before being removed from the sample containers. At the end of the 2-week curing period, the sorbent samples were tested for PFT, LRT, and density. The grout samples were tested for density after they were removed from the sample containers and cut into sample sections. Grout samples were not tested using the PFT or LRT since these tests only apply to sorbent samples; however, the grout samples were visually inspected for free liquids on top of the grouted surface. None of the grout samples had any free liquid after the curing period. The density and LRT data collected from the Aqueous Organic samples are also presented in Table X.

All of the Aqueous Organic surrogate, sorbent, and GG II samples passed the PFT and LRT. The neat Aqueous Organic surrogate and Petroset samples had a top and bottom density of 1.17 and 1.18 g/mL, respectively, while the shielded sorbent samples have density values that ranged from 1.51 to 2.09 g/mL. The top and bottom density values were within 0.01 g/mL, indicating that the GG II shielding material can be evenly incorporated into the Aqueous Organic surrogate and Petroset sorbent matrix.

Table X. Test Matrix, Density, and LRT Data Collected from the Aqueous Organic Surrogate, Sorbent, and Grout Samples with and without the Inclusion of GG II or RS II Shielding Material

<table>
<tr><th>Sorbent Name</th><th>Surrogate Name</th><th>Total Solids-to-Surrogate Ratio (solid:liquid)</th><th>GG II- or RS II-to-Sorbent or Grout Ratio (shielding:sorbent or grout)</th><th>Sample Section</th><th>Density Values per Sample Section (g/mL)</th><th>LRT % Liquid Released by Volume (NTS WAC < 0.5%)</th></tr>
<tr><td colspan="7">Aqueous Organic – Petroset – GG II</td></tr>
<tr><td rowspan="2">Petroset</td><td rowspan="2">Aqueous Organic</td><td rowspan="2">1:1</td><td rowspan="2">1.5:1</td><td>Top</td><td>1.52</td><td rowspan="2">0.311</td></tr>
<tr><td>Bottom</td><td>1.51</td></tr>
<tr><td rowspan="2">Petroset</td><td rowspan="2">Aqueous Organic</td><td rowspan="2">1:1</td><td rowspan="2">2:1</td><td>Top</td><td>1.59</td><td rowspan="2">0.368</td></tr>
<tr><td>Bottom</td><td>1.60</td></tr>
<tr><td rowspan="2">Petroset</td><td rowspan="2">Aqueous Organic</td><td rowspan="2">1:1</td><td rowspan="2">3:1</td><td>Top</td><td>1.74</td><td rowspan="2">0.364</td></tr>
<tr><td>Bottom</td><td>1.73</td></tr>
<tr><td rowspan="2">Petroset</td><td rowspan="2">Aqueous Organic</td><td rowspan="2">1:1</td><td rowspan="2">4:1</td><td>Top</td><td>1.74</td><td rowspan="2">0.307</td></tr>
<tr><td>Bottom</td><td>1.74</td></tr>
<tr><td rowspan="2">Petroset</td><td rowspan="2">Aqueous Organic</td><td rowspan="2">1.5:1</td><td rowspan="2">2:1</td><td>Top</td><td>1.85</td><td rowspan="2">0.472</td></tr>
<tr><td>Bottom</td><td>1.86</td></tr>
<tr><td rowspan="2">Petroset</td><td rowspan="2">Aqueous Organic</td><td rowspan="2">1.5:1</td><td rowspan="2">3:1</td><td>Top</td><td>2.02</td><td rowspan="2">0.417</td></tr>
<tr><td>Bottom</td><td>2.01</td></tr>
<tr><td rowspan="2">Petroset</td><td rowspan="2">Aqueous Organic</td><td rowspan="2">1.5:1</td><td rowspan="2">4:1</td><td>Top</td><td>2.09</td><td rowspan="2">0.336</td></tr>
<tr><td>Bottom</td><td>2.09</td></tr>
<tr><td rowspan="2">Petroset</td><td rowspan="2">Aqueous Organic</td><td rowspan="2">1:1.25</td><td rowspan="2">NA – Neat Sample</td><td>Top</td><td>1.17</td><td rowspan="2">0.472</td></tr>
<tr><td>Bottom</td><td>1.18</td></tr>
<tr><td colspan="7">Aqueous Organic – Portland Cement – RS II</td></tr>
<tr><td rowspan="2">Portland cement</td><td rowspan="2">Aqueous Organic</td><td rowspan="2">2:1</td><td rowspan="2">3:1</td><td>Top</td><td>2.20</td><td rowspan="2">---</td></tr>
<tr><td>Bottom</td><td>2.22</td></tr>
<tr><td rowspan="2">Portland cement</td><td rowspan="2">Aqueous Organic</td><td rowspan="2">2.5:1</td><td rowspan="2">3:1</td><td>Top</td><td>2.40</td><td rowspan="2">---</td></tr>
<tr><td>Bottom</td><td>2.40</td></tr>
<tr><td rowspan="2">Portland cement</td><td rowspan="2">Aqueous Organic</td><td rowspan="2">2.5:1</td><td rowspan="2">4:1</td><td>Top</td><td>2.50</td><td rowspan="2">---</td></tr>
<tr><td>Bottom</td><td>2.47</td></tr>
<tr><td rowspan="2">Portland cement</td><td rowspan="2">Aqueous Organic</td><td rowspan="2">3:1</td><td rowspan="2">3:1</td><td>Top</td><td>2.64</td><td rowspan="2">---</td></tr>
<tr><td>Bottom</td><td>2.61</td></tr>
<tr><td rowspan="2">Portland cement</td><td rowspan="2">Aqueous Organic</td><td rowspan="2">3:1</td><td rowspan="2">4:1</td><td>Top</td><td>2.80 [a]</td><td rowspan="2">---</td></tr>
<tr><td>Bottom</td><td>2.89</td></tr>
<tr><td>Portland cement</td><td>Aqueous Organic</td><td>2:1</td><td>NA – Neat Sample</td><td>Middle</td><td>1.79</td><td>---</td></tr>
<tr><td>Portland cement</td><td>Aqueous Organic</td><td>2.5:1</td><td>NA – Neat Sample</td><td>Middle</td><td>1.81</td><td>---</td></tr>
<tr><td>Portland cement</td><td>Aqueous Organic</td><td>3:1</td><td>NA – Neat Sample</td><td>Middle</td><td>1.93</td><td>---</td></tr>
</table>

--- = No LRT value for cement samples only for sorbent samples
[a] = Sample had several voids

The Aqueous Organic and Portland cement samples all cured without the presence of free liquid on top of the samples. After a 1-week cure time, the samples were removed from the sample containers and cut into sections that were used for sample bombardment and to collect top and bottom density measurements. The neat 2:1 Portland cement sample had a density value of 1.79 g/mL while the shielded 2:1 sample had a top density value of 2.22 g/mL and a bottom density value of 2.20 g/mL. The 2.5:1 neat sample had a density value of 1.81 g/mL while the 2.5:1 shielded samples had density values that ranged from 2.40 to 2.50 g/mL. The 3:1 neat sample had a density value of 1.93 g/mL while the 3:1 shielded samples had density values that ranged from 2.61 to 2.89 g/mL. The top and bottom density values ranged from 0 to 0.03 g/mL for all of the grout samples except the 3:1 to 4:1 Aqueous Organic sample. The top sample for the 3:1 to 4:1 sample had several voids in the sample resulting in a lower bulk density value and in a 0.08-g/mL difference for the top and bottom samples. Even though the 3:1 to 4:1 samples had a wider density range than the rest of the grouted samples, the dense radiation shielding material appears to be evenly incorporated into the Aqueous Organic surrogate and the Portland cement sample set.

Table XI presents the gamma attenuation data collected for the Aqueous Organic sample set. Petroset (the powdered clay sorbent) when combined with the Aqueous Organic surrogate waste provided a good matrix to incorporate the GG II shielding material. The data show a good trend between the density values presented in Table X and the gamma attenuation data presented in Table XI with the exception of the 1:1 to 2:1 sample, which seems to have a low attenuation value. As more of the gamma shielding material was incorporated into the sample matrix, the density values increased and provided better gamma attenuation. The neat Aqueous Organic sample provided approximately 22% gamma attenuation while the shielded samples provided gamma attenuation values ranging from around 15% to approximately 27%. The shielded samples provide gamma attenuation values ranging from 13% to 22% better than the neat Aqueous Organic sorbent samples. The data indicate that the dense radiation shielding material can be evenly incorporated into the Aqueous Organic surrogate and powdered clay matrix and that it provides good gamma attenuation.

Table XI. Aqueous Organic Surrogate, Sorbent, and Grout Gamma Attenuation Results

Sorbent or Grout Name	Total Solids-to-Surrogate Ratio (solid:liquid)	GG II-to-Sorbent or Grout Ratio (shielding:sorbent or grout)	Sample Section	Radiation Source Strength (R/hr)	Radiation Reading (R/hr)	Percent Attenuation for Sample	Percent Attenuation Shielded to Neat Sample
Empty Box	---	---		175	172.5	---	---
Petroset	1:1	1.5:1	Middle	172.5	112.0	35.1	16.8
Petroset	1:1	2:1	Middle	172.5	114.7	33.5	14.8
Petroset	1:1	3:1	Middle	172.5	111.6	35.3	17.1
Petroset	1:1	4:1	Middle	172.5	108.3	37.2	19.5
Petroset	1.5:1	2:1	Middle	172.5	104.9	39.2	22.1
Petroset	1.5:1	3:1	Middle	172.5	101.8	41.0	24.4
Petroset	1.5:1	4:1	Middle	172.5	97.7	43.4	27.4
Petroset	1.25:1	NA – Neat Sample	Middle	172.5	134.6	22.0	0
Portland cement	2:1	3:1	Top	172.5	101.2	41.3	20.1
			Bottom	172.5	97.8	43.3	22.7
Portland cement	2.5:1	3:1	Top	172.5	95.3	44.8	24.2
			Bottom	172.5	94.3	45.3	25.0
Portland cement	2.5:1	4:1	Top	172.5	88.9	48.5	29.3
			Bottom	172.5	94.3	45.3	25.0
Portland cement	3:1	3:1	Top	172.5	89.7	48.0	27.3
			Bottom	172.5	94.5	45.2	23.4
Portland cement	3:1	4:1	Top	172.5	88.1	48.9	28.9
			Bottom	172.5	70.5	59.1	42.9
Portland cement	2:1	NA – Neat Sample	Middle	172.5	126.6	26.6	0
Portland cement	2.5:1	NA – Neat Sample	Middle	172.5	125.8	27.1	0
Portland cement	3:1	NA – Neat Sample	Middle	172.5	123.4	28.5	0

The gamma attenuation values for the Aqueous Organic samples did not trend as well with the density data as the other surrogate waste and Portland cement samples. Even small differences in density produced fairly large differences in the gamma attenuation numbers. For example, a 0.02-g/mL density difference resulted in a 2.0% difference in the attenuation data for the 2:1 to 3:1 Portland cement sample and a 0.03 density difference for the 2.5:1 to 4:1, and the 3:1 to 3:1 samples produced attenuation data with an approximately 3% difference. The 3:1 to

4:1 sample had the largest difference in density and attenuation data. The density values were 2.80 g/mL for the top sample section and 2.89 g/mL for the bottom sample section, which resulted in attenuation values of 48.9% and 59.1%, over a 10% attenuation difference with a density difference of 0.09 g/mL. The 3:1 to 4:1 sample had a melted shielding smear similar to the Rainwater sample that was generated at the same ratios. Fig. 4 shows the surrogate Rainwater and Aqueous Organic samples generated with Portland cement during the cutting process.

Fig. 4. Rainwater and Aqueous Organic and GG II samples solidified with Portland cement during the cutting process.

The Portland cement and Aqueous Organic samples also provided a good matrix to incorporate the GG II shielding material. The data show a trend between density values presented in Table X and the gamma attenuation results. As expected, as the shielding amounts were increased, the density values increased and provided better attenuation results. The neat Aqueous Organic Portland cement samples had attenuation values of approximately 27% while the shielded samples had attenuation values that ranged from 41% to 59%. The shielded samples provide attenuation values ranging from approximately 20% to 42% better than the neat Aqueous Organic Portland cement samples.

CONCLUSIONS AND RECOMMENDATIONS

The testing sequence proved that the dense GG II radiation shielding material can be evenly incorporated into a sorbent or grout matrix and provide good gamma attenuation results. Four different types of SRS surrogate waste formulations were combined with the GG II and sorbents or grout. The Legacy PUREX surrogate waste was combined with GG II and two powdered clay sorbents but was not compatible with Portland cement. Three different oils were combined with two other powdered clay sorbents and the GG II shielding material but not with Portland cement. The Rainwater wastestream and GG II shielding material was combined with one of the powdered clay sorbents and Portland cement. An Aqueous Organic surrogate was also combined using GG II with a powdered clay sorbent and Portland cement.

The Legacy PUREX surrogate (when combined with the Petroset II powdered clay sorbent and GG II) produced samples with gamma attenuation results that ranged from 26.4% to 35.5% and a neat sample (a sample without the gamma shielding material) with a gamma attenuation of 18.9%. When compared to the neat Petroset II sample, the other samples that included GG II provided 9.3% to 20.4% better gamma attenuation results. The Legacy PUREX surrogate waste, BML-QT-199 powdered clay sorbent, and GG II shielding produced samples with gamma attenuation results that ranged from 24.9% to 36% and a neat sample with gamma attenuation results of 18.2%. The GG II samples produced attenuation results that were 8.2% to 21.8% better than the neat BML-QT-199 sample. Both powdered clay sorbent samples passed the LRT criteria and produced similar gamma attenuation results, and the attenuation results trended well with the sample density values. This proved that the GG II shielding material can be incorporated into a powdered clay sorbent and surrogate PUREX matrix.

Utility oil, Regal oil, and Maropa oil were used as the three surrogate oil wastestreams during this testing, and they were all combined with GG II and the two different powdered clays, Petroset II-P and LM-QT-Plus. All three oils produced similar results when combined with the GG II shielding material and the two powdered clay sorbents. The six neat oil and sorbent samples produced gamma attenuation values that ranged from 17.9% to 19.4%, and the samples that included the GG II gamma shielding material had gamma attenuation values that ranged from 28.7% to 40.5%. The shielded samples produced gamma attenuation values that were 12.5% to 27.6% better than the neat samples for the oil sample set. All of the samples in the oil surrogate sample set passed the LRT criteria and showed a good correlation between density and gamma attenuation values, proving that the GG II shielding material can be incorporated into an oil and powdered clay sorbent wasteform.

The Rainwater surrogate was combined with the GG II shielding material and Petroset sorbent or Portland cement. The Rainwater, GG II, and Petroset sorbent samples produced attenuation values that ranged from 30% to 38% with a neat sample attenuation value of 20.1%. The shielded Rainwater and Petroset samples provided attenuation values that ranged from 12.3% to 22.4% better than the neat sample. Six of the eight samples passed the LRT criteria, and the density values correlated with the gamma attenuation values. This proved that the GG II shielding material can be incorporated into the Rainwater and Petroset matrix.

The Rainwater surrogate was also combined with GG II and Portland cement, but the dense shielding material could not be incorporated. Consequently, MSE added a component to the mixture to incorporate the shielding material into the grout matrix (based on previous work performed for ORNL), and this mixture is referred to as RS II. The Rainwater, RS II, and Portland cement samples had density values that could be correlated with the attenuation results; however, that did not trend as well as the sorbent samples. The neat Rainwater and Portland samples had gamma attenuation results that ranged from 27.5% to 28.7%, and the shielded samples had gamma attenuation values that ranged from 41.7% to 55.2% with the shielded samples performing 19.7% to 37.2% better than the neat samples. All of the Portland cement and Rainwater samples cured in 3 days with no bleed water.

The Aqueous Organic surrogate was combined with GG II and Petroset, a powdered clay sorbent, or Portland cement. The Aqueous Organic waste, GG II, and Petroset sorbent samples produced attenuation values that ranged from 33.5% to 43.4% with a neat sample attenuation value of 22%. The shielded Aqueous Organic wastestream and Petroset samples provided attenuation values that ranged from 14.8% to 27.4% better than the neat sample. All of the samples passed the LRT criteria, and the density values correlated with the gamma attenuation values. This proved that the GG II shielding material can be incorporated into the Aqueous Organic surrogate waste and Petroset matrix.

The Aqueous Organic surrogate was also combined with GG II and Portland cement, but the dense shielding material could not be incorporated. Therefore, MSE added a component to the mixture to incorporate the shielding material into the grout matrix (based on previous work performed for ORNL); this mixture is referred to as RS II. The Aqueous Organic, RS II, and Portland cement samples had density values that could be correlated with the attenuation results; however, that did not trend as well as the sorbent samples. The neat Aqueous Organic and Portland samples had gamma attenuation results that ranged from 26.6% to 28.5%, and the shielded samples had gamma attenuation values that ranged from 41.3% to 59.1% with the shielded samples performing 20.1% to 42.9% better than the neat samples. All of the Portland cement and Aqueous Organic surrogate samples cured in 3 days with no bleed water.

All of the surrogate waste formulations tested could incorporate the dense GG II shielding material with sorbents and/or Portland cement. The results showed that the organic wastestreams could be solidified using sorbents with the inclusion of the GG II shielding material and that the aqueous wastestreams could be solidified using sorbents and Portland cement with the GG II shielding material by adding an extra component identified by MSE. Portland cement samples provided better gamma attenuation results than the sorbent samples for the aqueous wastestreams; however, the total solid-to-liquid ratios were much higher than those for the sorbent samples. The sorbent samples were generated at 1:1 and 1.5:1 total solid-to-liquid ratios while the Portland cement samples were generated using 2:1. 2.5:1, and 3:1 total solid-to-liquid ratios. Depending on the application, either sorbents or Portland cement could be used as an effective media to incorporate the GG II shielding material.

REFERENCES

1. MSE TECHNOLOGY APPLICATIONS, INC., "Mixed Waste Technology Program Preliminary Test Report–Phase III Sorbent Testing for Solidification of Plutonium/Uranium Extraction Process Waste," PTP-129 (May 2004).
2. MSE TECHNOLOGY APPLICATIONS, INC., "Accelerated Site Cleanup Programs Final Report–Phase IV Sorbent Testing for Solidification of Plutonium/Uranium Extraction Process Waste," MSE-102 (July 2006).
3. MSE TECHNOLOGY APPLICATIONS, INC., "Accelerated Site Cleanup Program Final Report–Sorbent Testing for Solidification of Savannah River Site Rainwater," MSE- 98 (February 2006).
4. MSE TECHNOLOGY APPLICATIONS, INC., "DOE Program Final Report–Sorbent Testing for Solidification of Savannah River Site Oils Wastestreams," MSE-99 (April 2006).
5. MSE TECHNOLOGY APPLICATIONS, INC., "DOE Program Final Report–Sorbent Testing for Solidification of Savannah River Site Aqueous Organic Wastestream," DPWTR0*1*, MSE-100 (June 2006).
6. U.S. ENVIRONMENTAL PROTECTION AGENCY, "Test Methods for Evaluating Solid Waste—Physical/ Chemical Methods, Method 9095A, Paint Filter Liquids Test Procedure," U.S. EPA, Washington D.C., Office of Solid Waste web site: http://www.epa.gov/epaoswer/hazwaste/test/main.htm.
7. U.S. ENVIRONMENTAL PROTECTION AGENCY, "Test Methods for Evaluating Solid Waste—Physical/ Chemical Methods, Method 9096, Liquid Release Test (LRT) Procedure," U.S. EPA, Washington D.C., Office of Solid Waste web site: http://www.epa.gov/epaoswer/hazwaste/test/main.htm.
8. LANGTON, CHRISTINE A., GARY M. IVERSEN, FERNANDO FONDEUR, GREGGORY D. CREECH, AND LAWRENCE N. OGI, "PUREX Organic Waste Solidification," Westinghouse Savannah River Company and Savannah River Technology Center (December 3, 2002).
9. WESTINGHOUSE SAVANNAH RIVER COMPANY AND SAVANNAH RIVER TECHNOLOGY CENTER, "PUREX Waste Solidification," (November 14, 2001).

ACKNOWLEDGMENT

Work was conducted through the DOE Environmental Management Consolidated Business Center at the Western Environmental Technology Office under DOE Contract Number DE-AC09-96EW96405.