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Long-Term Stability Testing Results Using Surrogates and Sorbents for Savannah River Site Organic and Aqueous Wastestreams - 10016

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The U.S. Department of Energy (DOE) has tasked MSE Technology Applications, Inc. (MSE) with evaluating the long-term stability of various commercially available sorbent materials to solidify two organic surrogate wastestreams (both volatile and nonvolatile), a volatile organic surrogate with a residual aqueous phase, an aqueous surrogate, and an aqueous surrogate with a residual organic phase. The Savannah River Site (SRS) Legacy and F-Canyon plutonium/uranium extraction (PUREX) process waste surrogates constituted the volatile organic surrogates, and various oils constituted the nonvolatile organic surrogates. The aqueous surrogates included a rainwater surrogate and an aqueous organic surrogate. MSE also evaluated the PUREX surrogate with a residual aqueous component with and without aqueous-type sorbent materials. Solidification of the various surrogate wastestreams listed above was performed from 2004 to 2006 at the MSE Test Facility located in Butte, Montana. This paper summarizes the comparison of the initial liquid release test (LRT) values with LRT results obtained during subsequent sampling events in an attempt to understand and define the long-term stability characteristics for the solidified wastestreams.

INTRODUCTION

The U.S. Department of Energy (DOE) tasked MSE Technology Applications, Inc. (MSE) with evaluating various commercially available sorbent materials to solidify surrogate wastestreams representative of the Legacy plutonium/uranium extraction (PUREX) process waste, F-Canyon PUREX waste, a Rainwater wastestream, an Oils wastestream, and an Aqueous Organic wastestream. MSE also evaluated a surrogate PUREX wastestream with an aqueous component with and without aqueous-type sorbent materials. The actual radioactive wastestreams were generated at the Savannah River Site (SRS) during several different processes.

Solidification of the various surrogate wastestreams listed above was performed from 2004 to 2006 at the MSE Test Facility located in Butte, Montana. This paper summarizes the comparison of the initial liquid release test (LRT) values with LRT results obtained during subsequent sampling events in an attempt to understand and define the long-term stability characteristics for the solidified wastestreams. The paper also includes surrogate Legacy PUREX LRT testing for four granular clay sorbents that were compared.

The paper covers work completed with fiscal year (FY) 2004 funding through the present and details information about the surrogate wastestream testing listed below.

- Surrogate Legacy PUREX wasteform LRT testing for 22-liter (L) [5-gallon (gal)] bucket samples generated in 2004 both with and without ultraviolet (UV) protected overpack drums to determine the effects of sunlight on the solidified Legacy PUREX surrogate samples.
- Surrogate Rainwater wasteform LRT sample testing at the 22-L (5-gal) bucket scale for samples generated in 2005.
- Surrogate Oils wasteform LRT sample testing at the 22-L (5-gal) scale for samples generated in 2005.

- Surrogate Aqueous Organic wasteform LRT sample testing at the 22-L (5-gal) scale for samples generated in 2005.
- Surrogate Legacy PUREX wasteform LRT sample testing at the 22-L (5-gal) scale with an aqueous phase using organic- and aqueous-type sorbents generated in 2005.
- Surrogate Legacy PUREX used for compatibility testing with four granular clay sorbent products since the first generation products that were tested are no longer available from the vendors. Samples were generated in 2004, 2005, and 2006.
- Surrogate F-Canyon PUREX wasteform LRT sample testing at the 22-L (5-gal) scale with an aqueous phase using Organoclay BM-QT-199 (an organic-type sorbent) without the addition of aqueous-type sorbents at ambient temperature for samples generated in 2006.

FULL PUREX SURROGATE SORBENT STABILITY TESTING

The LRT [1] data is presented in Table I for the surrogate Legacy PUREX and sorbent samples generated in FY04 at the 22-L (5-gal) scale that have been stored outside at the MSE Test Facility. This sample set has been LRT tested every year since sample generation to determine if exposure to sunlight would break down any of the sorbent and PUREX samples [2, 3]; the samples were initially LRT tested 2 weeks after sample generation. All samples except those denoted in Table I as "unprotected" have been stored in UV protected overpack drums. The Nochar Petrobond samples, which were not UV protected, released more liquid each year than the samples generated at the same waste-loading ratios that were UV protected. The Nevada Test Site (NTS) Waste Acceptance Criteria (WAC) of < 0.5% liquid release by volume was used as evaluation criteria for the LRT values. In Table I, the bolded LRT values show failure of the NTS LRT WAC, and the bolded free liquid numbers in the last column show failure of the paint filter test (PFT), which indicates the presence of free liquid in a sample [4]. Generally, when a sample fails the PFT, an LRT is not performed since the sample is considered to have failed the initial liquid release-type of test. However, for this test sequence, LRTs were performed on samples that failed the PFT in an effort to obtain a better understanding of the solidified wasteforms that were failing. Liquid release tests were performed after the free liquid was removed from the samples, resulting in LRT values that are much lower than the values would be if the liquid was not removed. The samples were initially sampled for liquid release in May 2004 and again in September 2005; December 2006; and October 2007, 2008, and 2009. Samples that failed the LRT criteria at any time during the test sequence shows the LRT values bolded for the years following even if the LRT values were below the NTS WAC of < 0.5%. This indicates the sample was not stable at some point during the test sequence.

The LRT values for the Petroset II-G samples did not significantly change during the 5-1/2-year storage period. In fact, comparable amounts of liquid were released from the samples during the initial and the 5-1/2-year sampling events. Sunlight did not affect the sample integrity for the Petroset II-G/PUREX samples as indicated by the LRT values of 0.034% liquid release for the UV unprotected sample and 0.012% for the UV protected sample at the 2:1 ratio in FY09. No free liquid was detected during the 5-1/2-year storage period for any of the Petroset II-G samples, and the sample consistency remained a hard paste.

The UV protected Nochar Petrobond samples did not significantly change during the storage period for the samples generated at the 1:2 and 1:2.5 waste-loading ratios, showing similar LRT values for all of the sampling events. The sample consistency also remained rubbery, which is similar to the consistency after sample generation in 2004.

The UV protected Nochar Petrobond sample generated at the 1:4 waste-loading ratio released more liquid in 2007 (when it failed the LRT WAC) than during the 2008 and 2009 sampling events while the 1:3 UV

protected Nochar Petrobond sample released the most liquid during 2008 with less liquid released in 2009. The sample's consistency was rubbery, which is consistent to the initial sample consistency after generation in 2004.

Table I. LRT Data for PUREX Full Surrogate and Nochar Petrobond and Petroset II-G Samples Generated in FY04.

Sorbent Name	Weight-Based, Waste-Loading Ratio (wt sorbent: wt PUREX)	FY04 LRT % Release by Volume NTS WAC < 0.05%	FY05 LRT % Release by Volume NTS WAC < 0.5%	FY06 LRT % Release by Volume NTS WAC < 0.5%	FY07 LRT % Release by Volume NTS WAC < 0.5%	FY08 LRT % Release by Volume NTS WAC < 0.5%	FY09 LRT % Release by Volume NTS WAC < 0.5%	Total Free Liquid Removed from Sample (mL)[a]
Petroset II-G	2:1	0.006	0.005	0.014	0.012	0.011	0.012	---[b]
Petroset II-G Unprotected[c]	2:1	0.010	0.006	0.013	0.012	0.012	0.034	---
Petroset II-G	1.9:1	0.023	0.028	0.009	0.008	0.008	0.012	---
Petroset II-G	1.8:1	0.010	0.003	0.007	0.027	0.006	0.013	---
Petroset II-G	1.7:1	0.026	0.003	0.011	0.016	0.010	0.006	---[a]
Petroset II-G	1.6:1	0.017	0.012	0.011	0.012	0.011	0.011	---
Petroset II-G	1.5:1	0.059	0.019	0.010	0.013	0.011	0.020	---
Nochar Petrobond	1:4	0.416	0.377	0.357	**0.538**	**0.425**	**0.332**	---
Nochar Petrobond Unprotected[c]	1:4	0.403	**0.568**	**1.049**	**1.291**	**1.360**	**0.274**	**405**
Nochar Petrobond	1:3	0.139	0.110	0.135	0.240	0.278	0.101	---
Nochar Petrobond Unprotected[c]	1:3	0.140	**0.150**	**0.450**	**0.124**	**0.128**	**0.076**	**424**
Nochar Petrobond	1:2.9	0.131	0.124	0.121	0.378	0.202	0.084	---
Nochar Petrobond	1:2.8	0.091	0.132	0.114	0.229	0.181	0.110	---
Nochar Petrobond	1:2.7	0.084	0.085	0.057	0.105	0.101	0.065	---
Nochar Petrobond	1:2.6	0.066	0.067	0.085	0.092	0.094	0.066	---
Nochar Petrobond	1:2.5	0.043	0.062	0.054	0.064	0.068	0.046	---
Nochar Petrobond Unprotected[c]	1:2.5	0.063	0.072	**0.244**	**0.444**	**0.464**	**0.041**	**461**
Nochar Petrobond	1:2.4	0.029	0.037	0.058	0.173	0.166	0.041	---
Nochar Petrobond	1:2.3	0.031	0.052	0.049	0.152	0.131	0.041	---
Nochar Petrobond	1:2.2	0.023	0.020	0.037	0.042	0.050	0.026	---
Nochar Petrobond	1:2.1	0.006	0.015	0.011	0.023	0.015	0.005	---
Nochar Petrobond	1:2	0.009	0.020	0.010	0.038	0.041	0.019	---
Nochar Petrobond Unprotected[c]	1:2	0.009	0.023	**1.363**	**1.173**	**0.472**	**0.063**	**451**

[a] mL = milliliters.
[b] --- No free liquid was discovered in the solidified wasteforms.
[c] The unprotected samples were not put into UV protected overpack drums.

The LRT values for the unprotected Nochar Petrobond duplicate samples significantly changed during the storage period as reflected by the LRT and free liquid values presented in Table I for samples at 1:4, 1:3, 1:2.5, and 1:2 waste-loading ratios. It was noted during the monthly sample checks that the samples solidified with Nochar Petrobond at waste-loading ratios of 1:4 and 1:3 stored without UV protected overpacks began to exhibit a breakdown in stability during September and December 2005, respectively. This breakdown in stability was in the form of small quantities of free liquid appearing on the surface of the samples. The unprotected 1:2.5 ratio Nochar Petrobond sample started releasing free liquid in July 2006 while the unprotected 1:2 ratio Nochar Petrobond sample started releasing liquid in October 2006. All of the Nochar Petrobond samples without UV protection released liquid during the 5-1/2-year storage time. If more than a few drops of liquid were discovered in a sample, the free liquid was drained and measured in the month the liquid was discovered. The 1:4 ratio Nochar Petrobond sample had released 253 mL of free liquid by the end of 2006 with a total of 405 mL by the sampling event in October 2007. The 1:3 ratio Nochar Petrobond sample had released 235 mL of free liquid by the end of 2006 and another 189 mL in 2007 for a total of 424 mL of free liquid. The 1:2.5 ratio released 228 mL by the end of 2006 and 256 mL during 2007, resulting in 483 mL of total liquid released from the sample. The 1:2 ratio Nochar Petrobond sample released 205 mL by December 2006 and another 246 mL by the October 2007 sampling event, resulting in a total of 451 mL of total free liquid released. This indicates that sunlight does impact sample stability over time for the samples generated with Nochar Petrobond and the full PUREX surrogate. Sample consistency remained rubbery but softer than after sample generation.

Since the samples had been stored outside at the MSE Test Facility for 5-1/2 years, some additional information can be determined concerning the solidified full surrogate PUREX samples. Thermal cycling and freeze-thaw cycling does not seem to have an impact on the UV protected and unprotected Petroset II-G samples and the UV protected Nochar samples as indicated by the sample stability during the 5-1/2-year outdoor storage period. No free liquid was released from these samples, nor did the LRT values fluctuate significantly since sample generation.

RAINWATER WASTESTREAM SURROGATE SORBENT STABILITY TESTING

The Rainwater samples were generated in January and February 2005 and initially sampled during the same months in 2005. Additional LRT testing was performed in November 2006 and October 2007, 2008, and 2009. The samples were tested for liquid release using the PFT and LRT during the 2005 sampling event and again in 2006, 2007, 2008, and 2009. The samples listed in red text in Table II indicate the samples that passed all of the liquid release-type testing performed in 2005 [5]. This testing included ambient and elevated temperature LRT testing and shaker testing. Table II presents the ambient temperature LRT values for the 22-L (5-gal) bucket Rainwater samples. The NTS WAC of < 0.5% liquid release by volume was used as evaluation criteria for the LRT values. The bolded LRT values in Table II show failure of the NTS WAC for LRT.

Most of the aqueous-type sorbents tested were polymers, except the Aquaset products, which are modified clays (one is granular and the other is powdered). All of the clay samples passed the LRT for each of the three sampling events at both surrogate pH values. All of the polymers passed the LRT for the samples generated at the waste-loading ratio of 1:5; however, some of the polymers did not pass the LRT requirements for samples generated at the 1:10 ratio recommended by the sorbent manufacturers. This information suggests that the polymer sorbents should use at least a 1:5 weight-based, waste-loading ratio when solidifying fairly clean aqueous solutions while the clay products should be used at a minimum ratio of 1:1 sorbent to waste when used at the 22-L (5-gal) scale.

Table II. LRT Data for the Surrogate Rainwater Samples Generated in FY05.

Sorbent Name	Weight-Based, Waste-Loading Ratio (wt sorbent: wt PUREX)	2005 LRT % Release by Volume NTS WAC < 0.5%	2006 LRT % Release by Volume NTS WAC < 0.5%	2007 LRT % Release by Volume NTS WAC < 0.5%	2008 LRT % Release by Volume NTS WAC < 0.5%	2009 LRT % Release by Volume NTS WAC < 0.5%
pH – 5.5						
Waste Lock 770	1:10	0.360	**0.884**	**0.774**	**0.854**	**0.648**
Waste Lock 770	1:5	0.135	0.265	0.202	0.238	0.285
Quick Solid	1:10	0.364	0.358	0.360	0.405	0.403
Quick Solid	1:5	0.151	0.309	0.315	0.323	0.272
WaterWorks	1:10	0.359	**0.709**	**0.719**	**0.747**	**0.629**
WaterWorks	1:5	0.219	0.250	0.254	0.264	0.270
Aquaset II	1.1:1	0.142	0.100	0.103	0.104	0.139
Aquaset II	1.2:1	0.269	0.084	0.090	0.086	0.065
Aquaset	1:1	0.089	0.191	0.178	0.180	0.120
Aquaset	1:1.25	0.477	0.189	0.195	0.191	0.374
Aquasorb	1:10	0.212	0.319	0.323	0.336	0.329
Aquasorb	1:5	0.222	0.213	0.173	0.262	0.286
Nochar A660 Acid Bond	1:10	0.208	**0.507**	0.317	0.488	0.462
Nochar A660 Acid Bond	1:5	0.227	0.176	0.275	0.290	0.237
pH – 10.9						
Waste Lock 770	1:5	0.259	0.345	0.305	0.330	0.407
WaterWorks	1:5	0.269	0.222	0.248	0.260	0.212
Aquaset	1.25:1	0.056	0.098	0.099	0.102	0.077

A total of 2,498 L (660 gal) of the actual Rainwater wastestream was solidified at SRS, with 1,249 L (330 gal) solidified using Aquaset at a waste-loading ratio of 1:1, and 1,249 L (330 gal) solidified using Waste Lock 770 at a waste-loading ratio of 1:5. Both of the solidified wasteforms were buried at SRS.

OILS WASTESTREAM SURROGATE SORBENT STABILITY TESTING

The Oils wastestream surrogate samples were generated using several organic-type sorbents in May 2005 [6]. During July and September 2005, combination-type sorbents and additional organic-type sorbents were identified and added to the test matrix. The combination-type sorbents can solidify liquids with both aqueous and organic phases. These were tested since the Oils wastestream did have a small aqueous component; however, the sorbents all failed the NTS LRT criteria at some point in time except the ABZORBIT sample. The Oils sample sets were tested for liquid release using the PFT and LRT after sample generation in 2005 and then in November 2006, October 2007, November 2008, and October 2009. Table III presents the LRT values for the 22-L (5-gal) bucket Oils wastestream samples. The NTS WAC of < 0.5% liquid release by volume was used as the evaluation criteria for the LRT values.

Table III. LRT Data for the Surrogate Oils Wastestream Samples Generated in FY05.

Sorbent Name	Weight-Based, Waste-Loading Ratio (wt sorbent: wt PUREX)	Initial LRT % Release by Volume NTS WAC < 0.05%	FY06 LRT % Release by Volume NTS WAC < 0.5%	FY07 LRT % Release by Volume NTS WAC < 0.5%	FY08 LRT % Release by Volume NTS WAC < 0.5%	FY09 LRT % Release by Volume NTS WAC < 0.5%
Organic-Type Sorbents						
Petroset II	1:1.5	**0.519**	0.257	0.274	0.273	0.139
Petroset II + 5% Methanol	1:1.5	0.262	0.149	0.071	0.104	0.169
Petroset II-G Batch 17 + 5% Methanol	2:1	0.098	0.013	0.017	0.018	0.039
Petroset II-G Batch 17 + 0.5% Methanol	2.5:1	0.415	0.022	0.027	0.028	0.019
Petroset II-G Batch 18	2.5:1	0.013	0.012	0.018	0.016	0.020
Petroset II-G Batch 18 + 5% Methanol	2.5:1	0.003	0.025	0.021	0.025	0.014
Nochar Petrobond	1:1	0.035	0.029	0.045	0.040	0.041
Nochar Petrobond	1:2	0.199	0.059	0.055	0.061	0.048
Imbiber Beads	2.5:1	0.235	0.279	0.069	0.148	0.013
Petrol Sorb	1.5:1	0.020	0.018	0.050	0.044	0.023
Combination-Type Sorbents						
ABZORBIT	1:2	0.036	0.022	0.034	0.033	0.052
Quick Solid 50	1:1	0.441	**1.095**	**0.562**	**0.591**	**0.519**
Sorbond Loc 40	2:1	**4.768**	0.035	0.011	0.013	0.027
Instasorb C	1.5:1	**11.577**	**10.469**	**11.401**	**6.000**	**0.916**
Additional Organic-Type Sorbents						
Liquisorb 1000	1:1	**0.512**	0.331	0.256	0.033	0.101
Organoclay TA-11	2.15:1	0.012	0.009	0.023	0.014	0.038
Organoclay BM-QT-199 – A	2:1	**8.125**	0.009	0.016	0.018	0.023
Organoclay BM-QT-199 – A +0.5% Methanol	2:1	**5.817**	0.010	0.013	0.016	0.027
Organoclay BM-QT-199 – A +5% Methanol	2:1	**0.637**	0.014	0.015	0.015	0.021

Bolded LRT values indicate failure of NTS WAC for liquid released.

It is interesting to note that all of the clay-based organic-type sorbents (Petrosets and Organoclays) except the Petroset II-G Batch 18 samples released less liquid in November 2006, October 2007, November 2008, and October 2009 than after the initial 2-week curing period when the samples were generated in 2005. The 2006, 2007, 2008, and 2009 polymer sorbent (Nochar Petrobond, Imbiber Beads, Petrol Sorb, and ABZORBIT) LRT values seemed to remain consistent with or were lower than the initial LRT values obtained during 2005. In Table III, the bolded LRT numbers indicate failure to meet the NTS WAC of less than < 0.5% liquid release by volume.

The LRT numbers seem to indicate that the initial curing period of 2 weeks was not sufficient for some of the clay sorbent materials when combined with the surrogate oils waste and that methanol is not necessary for solidification as recommended by the manufacturer. The samples will be tested for liquid release again at the end of 2010 or 2011 to determine if the samples continue to release less liquid than the NTS

WAC of < 0.5%. Additional testing was performed for another project using a 3-week curing time for oil solidified using the granular clay sorbents, and those samples all passed the NTS LRT criteria of < 0.5% liquid released by volume with the additional week curing time.

AQUEOUS ORGANIC WASTESTREAM SURROGATE SORBENT STABILITY TESTING

The Aqueous Organic wastestream samples were generated during September 2005 using a surrogate that simulates the SRS wastestream DPWTR01 [7]. The surrogate was an aqueous-based liquid with a 6% oil phase. Initial ambient temperature LRTs were conducted on the samples after the 2-week curing period. The second set of ambient temperature LRT data was collected in November 2006, the third data set was collected in September 2007, the fourth data set was collected in October 2008, and the fifth set was collected in October 2009. The LRT data is presented in Table IV. All of the 22-L (5-gal) bucket samples, except the two Waste Lock 770 samples and the Nochar A660 Acid Bond sample at the 1:5 ratio that had free standing liquid, were tested in the LRT apparatus in November 2006. The same samples with the addition of the Quick Solid sample at the 1:3 ratio were not tested in 2007 for liquid release since there was freestanding liquid in the samples, resulting in failure of the PFT. The bolded LRT values in Table IV show samples that failed the NTS LRT criteria.

Table IV. LRT Data for the Surrogate Aqueous Organic Samples Generated in FY05.

Sorbent Name	Weight-Based, Waste-Loading Ratio (wt sorbent: wt PUREX)	FY 2005 LRT % Release by Volume NTS WAC < 0.5%	FY 2006 LRT % Release by Volume NTS WAC < 0.5%	FY 2007 LRT % Release by Volume NTS WAC < 0.5%	FY 2008 LRT % Release by Volume NTS WAC < 0.5%	FY 2009 LRT % Release by Volume NTS WAC < 0.5%
Aqueous Sorbents						
Aquaset	1.5:1	0.260	0.148	0.147	0.169	0.116
Aquaset	2:1	0.033	0.037	0.059	0.065	0.072
Nochar A660 Acid Bond	1:5	0.293	----[a]	----	----	----
Nochar A660 Acid Bond	1:3	0.296	0.476	0.317	----	----
Aquasorb	1:5	0.336	**0.789**	**1.106**	----	----
Aquasorb	1:3	0.172	**0.664**	**0.675**	**2.035**	**0.298**
Quick Solid	1:3	0.219	**0.679**	----	----	----
Quick Solid	1:2	0.047	0.087	0.053	0.138	0.57
Waste Lock 770	1:4	0.133	----	----	----	----
Waste Lock 770	1:3	0.111	----	----	----	----
WaterWorks SP-400	1:5	0.295	0.359	**1.737**	**2.658**	**1.854**
WaterWorks SP-400	1:3	0.200	**0.541**	0.492	**0.713**	**0.273**
Combination Sorbents						
Quick Solid 50	1:1.5	0.074	0.053	0.074	0.076	0.156
Quick Solid 50	1:1	0.035	0.032	0.043	0.052	0.055
Instasorb C	1.5:1	0.055	0.058	0.083	0.113	0.153
Sorbond Loc-40	1:1	0.041	0.030	0.040	0.034	0.049
Sorbond Loc-40	1.5:1	0.048	0.031	0.029	0.029	0.057
Sorbond Loc-40	2:1	0.047	0.022	0.028	0.027	0.025
Liquisorb 1000	1:2	0.319	0.254	0.254	0.311	0.118
Liquisorb 1000	1:1	0.037	0.037	0.056	0.066	0.045

[a] ---- Samples with free-standing liquid were not tested for LRT. Bolded LRT values failed the NTS WAC for liquid released.

The samples listed in red text in Table IV passed all of the liquid release-type tests during testing in 2005. Additional tests performed in 2005 included elevated temperature LRT tests, shaker tests, and freeze-thaw tests for samples cured at ambient and elevated temperatures [7]. For the samples generated using the aqueous-type sorbents (Aquaset and Quick Solid), only three continue to pass the LRT WAC of < 0.5% release by volume, and they are presented in blue font. This indicates that Aquaset and Quick Solid sorbents are capable of sorbing aqueous liquids with up to a 6% organic phase.

The only samples that passed all initial testing in 2005 and continue to pass LRT testing were generated using combination-type sorbents designed to sorb liquids with both aqueous and organic phases. Sorbond Loc-40, Liquisorb 1000, and Quick Solid 50 passed all initial testing in 2005 and continued to pass the ambient temperature LRT test criteria when tested in 2006, 2007, 2008, and 2009. This indicates that the combination-type sorbent materials are capable of solidifying aqueous liquids with a 6% oil phase.

FULL LEGACY PUREX SURROGATE/AQUEOUS SORBENT STABILITY TESTING

The full Legacy PUREX surrogate samples with an aqueous phase were solidified using the combination of an organic-type and an aqueous-type sorbent in FY05 [2]. The liquid surrogates were generated using a 5% and a 10% water component with the full Legacy PUREX surrogate. The sorbent combinations used to solidify the surrogate PUREX liquid wastestream were Aquaset combined with Petroset II-G and Nochar Acid Bond combined with Nochar Petrobond. The Nochar samples were generated using a 1:3 weight-based, waste-loading ratio for the organic liquid phase with the Nochar Petrobond sorbent and a 1:2 waste-loading ratio for the aqueous phase and the Nochar Acid Bond sorbent. The granular clay samples were generated using a 1.7:1 weight-based, waste-loading ratio for Petroset II-G sorbent with the organic liquid phase and a 1.5:1 waste-loading ratio for the Aquaset with the liquid water phase. The LRT data for these samples is presented in Table V.

Table V. LRT Data for Aqueous and Full PUREX Surrogate Sorbent Samples.

Sorbent Name	Weight-Based Waste-Loading Ratio (wt sorbent: wt PUREX)	Percent Water	Initial LRT % Release by Volume NTS WAC < 0.05%	FY06 LRT % Release by Volume NTS WAC < 0.5%	FY07 LRT % Release by Volume NTS WAC < 0.05%	FY08 LRT % Release by Volume NTS WAC < 0.05%	FY09 LRT % Release by Volume NTS WAC < 0.05%
Petroset II-G	1.7:1	5	0.027	0.010	0.011	0.019	0.011
Aquaset	1.5:1						
Petroset II-G	1.7:1	10	0.022	0.020	0.012	0.038	0.012
Aquaset	1.5:1						
Petrobond	1:3	5	0.156	0.110	0.113	0.122	0.065
Acid Bond	1:2						
Petrobond	1:3	10	0.176	0.115	0.148	0.168	0.138
Acid Bond	1:2						

As seen in Table V, most of the LRT values decreased from the initial LRT values after the 3-year storage time, which indicates the 22-L (5-gal) bucket samples remained stable over the storage period. However, the 208-L (55-gal) drum samples generated in FY05 did not pass LRT testing at the waste-loadings shown in Table V after a 3-month storage period. A water phase within the organic PUREX waste presents a problem when solidifying the surrogate wastestream at the larger scale. Testing indicates that the water phase should be removed prior to solidification of the organic phase of the PUREX waste when using sorbents to avoid the unstable combinations generated when a water phase is present.

COMPARATIVE GRANULAR CLAY TESTING

In 2005, the SRS client requested that MSE identify another source for granular clay products that had characteristics similar to Petroset II-G Batch 17 but was less expensive. Petroset II-G Batch 17 was no longer available and was replaced with Petroset II-G Batch 18, which had different characteristics than Batch 17. MSE identified another granular clay named Organoclay BM-QT-199 (Organoclay) in 2005, which was similar to the Petroset II-G Batch 17. Comparison testing was initiated to determine if the Petroset II-G Batch 18 and Organoclay clays had similar performance characteristics to each other and to the original Petroset II-G Batch 17. Only bench-scale LRT data for the Petroset II-G Batch 17 and the full PUREX surrogate was available from 2004 testing. However, 22-L (5-gal) bucket samples were generated in 2005 using the two granular clays (Petroset II-G Batch 18 and Organoclay) and the full Legacy PUREX surrogate at the same weight-based, waste-loading ratios that were used during testing in 2004, which ranged from 1.5:1 up to 2:1 (sorbent to surrogate).

During later testing in 2006, the original Organoclay (Batch 1) sorbent source was no longer available, and another source was identified for testing. Bench-scale samples were then generated using the new product at the same weight-based, waste-loading ratios as the previous granular clay samples. There was not enough of the new Organoclay to generate 22-L (5-gal) samples; consequently, bench-scale samples were generated. For clarity, the original Organoclay will be referred to as Batch 1, and the second Organoclay will be referred to as Batch 2. The dashes (-) in Table VI represent bench-scale sampling events where no sample was available for future testing due to the limited amount.

Table VI. LRT Data for Granular Clay Sorbents and Full Legacy PUREX Surrogate Samples.

Sorbent Name	Weight-Based, Waste-Loading Ratio (sorbent to PUREX)	FY04 LRT % Release by Volume NTS WAC < 0.5%	FY05 LRT % Release by Volume NTS WAC < 0.5%	FY06 LRT % Release by Volume NTS WAC < 0.5%	FY07 LRT % Release by Volume NTS WAC < 0.5%	FY08 LRT % Release by Volume NTS WAC < 0.5%	FY09 LRT % Release by Volume NTS WAC < 0.5%
Petroset II-G Batch 17 – Bench-Scale Samples							
Petroset II-G	2:1	0.006	-[a]	-	-	-	-
Petroset II-G	1.9:1	0.023	-	-	-	-	-
Petroset II-G	1.8:1	0.020	-	-	-	-	-
Petroset II-G	1.7:1	0.026	-	-	-	-	-
Petroset II-G	1.6:1	0.017	-	-	-	-	-
Petroset II-G	1.5:1	0.059	-	-	-	-	-
Petroset II-G Batch 18 – 22-L (5-Gal) Bucket Samples							
Petroset II-G	2:1		0.009	0.012	0.015	0.017	0.009
Petroset II-G	1.9:1		0.010	0.013	0.017	0.021	0.002
Petroset II-G	1.8:1		0.012	0.011	0.020	0.024	0.008
Petroset II-G	1.7:1		0.024	0.018	0.027	0.036	0.019
Petroset II-G	1.6:1		0.039	0.015	0.031	0.036	0.009
Petroset II-G	1.5:1		0.043	0.026	0.031	0.042	0.014
Organoclay Batch 1 – 22-L (5-Gal) Bucket Samples							
Organoclay	2:1		0.015	0.008	0.008	0.020	0.010
Organoclay	1.9:1		0.014	0.013	0.019	0.018	0.005
Organoclay	1.8:1		0.013	0.016	0.024	0.027	0.007
Organoclay	1.7:1		0.008	0.017	0.027	0.028	0.008
Organoclay	1.6:1		0.151	0.017	0.027	0.028	0.014
Organoclay	1.5:1		0.209	0.063	0.099	0.096	0.029

Sorbent Name	Weight-Based, Waste-Loading Ratio (sorbent to PUREX)	FY04 LRT % Release by Volume NTS WAC < 0.5%	FY05 LRT % Release by Volume NTS WAC < 0.5%	FY06 LRT % Release by Volume NTS WAC < 0.5%	FY07 LRT % Release by Volume NTS WAC < 0.5%	FY08 LRT % Release by Volume NTS WAC < 0.5%	FY09 LRT % Release by Volume NTS WAC < 0.5%
Petroset II-G Batch 17 – Bench-Scale Samples							
Organoclay Batch 2 – Bench-Scale Samples							
Organoclay	2:1			0.014	-	-	-
Organoclay	1.9:1			0.032	-	-	-
Organoclay	1.8:1			0.033	-	-	-
Organoclay	1.7:1			0.071	-	-	-
Organoclay	1.6:1			0.083	-	-	-
Organoclay	1.5:1			0.168	-	-	-
[a] - no sample available for testing							

As can be seen from the LRT results in Table VI, all of the granular clays tested produce samples with LRT data that is very comparable and range one to two orders of magnitude less than the LRT criteria for NTS, which is < 0.5% release by volume.

As seen in previous PUREX surrogate data sets, the clay samples generated in FY05 continue to pass LRT requirements after approximately 4 years of storage with LRT values that remain consistent with the initial values. Also, the LRT values for the Batch 17 and Batch 18 of Petroset II-G demonstrate a good correlation between samples generated at the same waste-loading ratios as do the two Organoclay products. The data also shows all of the granular clay sorbents perform similarly when combined with the full Legacy PUREX surrogate.

ORGANOCLAY BM-QT-199 AND PUREX SURROGATE SORBENT STABILITY TESTING WITH AN AQUEOUS COMPONENT WITHOUT THE ADDITION OF AN AQUEOUS-TYPE SORBENT

During the F-Canyon PUREX proposal work in FY06, MSE prepared three 22-L (5-gal) bucket samples using the F-Canyon PUREX surrogate with various amounts of water added to the surrogate and then solidified the samples using Organoclay Batch 2 at a 2:1 weight-based, waste-loading ratio, sorbent to surrogate. The Organoclay sorbent is designed to sorb organic liquids not aqueous liquids. These samples were generated to determine if the Organoclay sorbent could sorb both the organic and a small aqueous phase without sample breakdown over time. The liquid organic phase included 64% kerosene and 36% tributyl phosphate by weight. The samples were made using 7.6 L (2 gal) of liquid, which included the organic phase and water at 1%, 5%, and 10% by weight.

The samples were generated in May 2006, and after the 2-week curing period, they were tested in the LRT apparatus to determine the liquid release characteristics of the samples. Table VII presents the LRT data collected until October 2009.

The samples passed all of the LRTs performed during the approximately 3.5-year testing period with comparable amounts of liquid released. The LRT values were all one to two orders of magnitude less than the NTS WAC of < 0.5% release by volume. The samples all had the consistency of a hard paste with no signs of sample breakdown. It appears that the Organoclay will sorb an aqueous phase up to 10% water and not break down over the short period. The samples will be kept at the MSE Test Facility for further observation and testing during 2010 or 2011 to see if any sample breakdown occurs at a later point in time.

Table VII. LRT data for Organoclay and F-Canyon surrogate PUREX samples with an aqueous phase.

Sorbent Name	Weight-Based, Waste-Loading Ratio (wt sorbent:wt F-Canyon PUREX)	Percent Water	Sample Dates	LRT % Release by Volume NTS WAC < 0.05%
Organoclay	2:1	1%	5-18-06	0.017
Organoclay	2:1	1%	6-22-06	0.017
Organoclay	2:1	1%	7-28-06	0.008
Organoclay	2:1	1%	9-13-06	0.022
Organoclay	2:1	1%	12-1-06	0.007
Organoclay	2:1	1%	5-1-07	0.016
Organoclay	2:1	1%	8-9-07	0.014
Organoclay	2:1	1%	10-3-07	0.013
Organoclay	2:1	1%	11-4-08	0.017
Organoclay	2:1	1%	10-6-09	0.006
Organoclay	2:1	5%	5-18-06	0.007
Organoclay	2:1	5%	6-22-06	0.014
Organoclay	2:1	5%	7-28-06	0.012
Organoclay	2:1	5%	9-13-06	0.015
Organoclay	2:1	5%	12-1-06	0.015
Organoclay	2:1	5%	5-1-07	0.014
Organoclay	2:1	5%	8-9-07	0.018
Organoclay	2:1	5%	10-3-07	0.019
Organoclay	2:1	5%	11-4-08	0.022
Organoclay	2:1	5%	10-8-09	0.005
Organoclay	2:1	10%	5-18-06	0.043
Organoclay	2:1	10%	6-22-06	0.035
Organoclay	2:1	10%	7-28-06	0.032
Organoclay	2:1	10%	9-13-06	0.028
Organoclay	2:1	10%	12-1-06	0.016
Organoclay	2:1	10%	5-1-07	0.024
Organoclay	2:1	10%	8-9-07	0.021
Organoclay	2:1	10%	10-3-07	0.024
Organoclay	2:1	10%	11-4-08	0.032
Organoclay	2:1	10%	10-8-09	0.060

CONCLUSIONS

Several characteristics of the solidified surrogate wastestreams that were determined from the long-term stability testing that was performed are listed below.

- Ultraviolet light does break down sample stability on full Legacy PUREX surrogate waste when solidified with Nochar Petrobond but not with the granular clay, Petroset II-G. Freeze-thaw and thermal cycling does not affect the Petroset II-G samples.
- Organoclay alone solidifies the organic F-Canyon PUREX surrogate with a water component up to 10% of the surrogate weight for the short term of 3.5 years.

- The Rainwater and Aqueous Organic surrogate wastestreams can be successfully solidified using several different sorbents and most remain solidified after more than 4 years of storage.
- The Oils wastestream surrogate waste can be successfully solidified using several sorbents, and most remain solidified after more than 4 years of storage.
- Regardless of which batch was tested, the granular clays (Petroset II-G and Organoclay) performed similarly and produced LRT values one or two orders of magnitude below the NTS LRT WAC of < 0.5% liquid release by volume.

RECOMMENDATIONS

To better characterize the solidified SRS wastestreams, MSE recommends that all solidified wastestreams at the MSE Test Facility be liquid release tested again in 2010 or 2011 to help define conclusions from FY09 and earlier testing after additional storage time.

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