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Long Term Stability Testing Results for Savannah River Site Organic and Aqueous Wastestreams

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ABSTRACT

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 wastestream with a residual aqueous phase, an aqueous wastestream, and an aqueous wastestream with a residual organic phase. The Savannah River Site (SRS) legacy plutonium/uranium extraction (PUREX) process waste and the F-Canyon PUREX waste constituted the volatile organic wastes and various oils constituted the nonvolatile organic wastestream. The aqueous wastestreams included a rainwater wastestream and an aqueous organic wastestream. MSE also evaluated the PUREX wastestream with a residual aqueous component with and without aqueous-type sorbent materials.

Based on testing performed at MSE, the rainwater wastestream was successfully solidified by SRS personnel using two different sorbents. Several small oil wastes were also successfully solidified by SRS personnel using granular clay sorbents based on information provided by MSE from the oils wastestream testing and 75,706 Liters (L) [20,000 gallons (gal)] of the F-Canyon PUREX waste was solidified at Waste Consolidation Specialists (WCS).

Solidification of the various surrogate wastestreams listed above was performed from 2004 to 2006 at the MSE testing and evaluation facility located at the Mike Mansfield Advanced Technology Center in Butte, Montana. This paper summarizes the comparison of the initial liquid release testing (LRT) values with LRT results obtained over three years later in an attempt to understand the long-term stability characteristics of the solidified wastestreams. The paper also includes solidification results for B-25 box samples generated late in 2005.

INTRODUCTION

Background

The U.S. Department of Energy (DOE) has tasked MSE Technology Applications, Inc. (MSE) to evaluate various commercially available sorbent materials to solidify surrogate Legacy plutonium/uranium extraction (PUREX) process waste, F-Canyon PUREX waste, a Rainwater wastestream, an Oils wastestream, and an Aqueous Organic wastestream generated at the Savannah River Site (SRS). MSE also evaluated the PUREX wastestream with an aqueous component with and without aqueous-type sorbent materials. These radioactive wastestreams were generated at 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 the long-term stability characteristics for the solidified wastestreams. The paper also includes solidification results for B-25 box samples generated late in 2005 as well as a comparison study for four granular clay sorbent products.

The paper covers work carried over from fiscal year (FY) 2004 funding through current funding and details information about the wastestream testing listed below.

- Surrogate Legacy PUREX wasteform LRT testing for 19-L (5-gal) bucket samples generated in 2004 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 19-L (5-gal) bucket scale for samples generated in 2005.
- Surrogate Oils wasteform LRT sample testing at the 19-L (5-gal) scale for samples generated in 2005.
- Surrogate Aqueous Organic wasteform LRT sample testing at the 19-L (5-gal) scale for samples generated in 2005.
- Surrogate Legacy PUREX wasteform LRT sample testing at the 19-L (5-gal) scale with an aqueous phase using organic and aqueous-type sorbents generated in 2005.
- Surrogate F-Canyon PUREX wasteform LRT sample testing at the 19-L (5-gal) scale with an aqueous phase using Nochar Petrobond ^{TM} (an organic-type sorbent) without the addition of aqueous-type sorbents at ambient and elevated temperatures generated in 2005.
- Compatibility testing for 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 5-gal scale with an aqueous phase using Organoclay BM-QT-199 (an organic-type sorbent) without the addition of an aqueous-type sorbent at ambient temperature for samples generated in 2006.
- Surrogate PUREX wasteform testing at the B-25 box scale using two granular clay sorbents to determine scale-up ratios from the 208-L (55-gal) drum tests, LRT characteristics, and sample integrity. The scale-up samples were generated in 2005.

FULL PUREX SURROGATE SORBENT STABILITY TESTING

Liquid Release Test [1} data for the PUREX samples generated in FY04 with the full PUREX surrogate at the 19-L (5-gal) scale that have been stored outside at the MSE Test Facility are presented in Table I. 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 of the samples except those denoted in Table I as "unprotected" have been stored in UV protected overpack drums. The Nochar Petrobond samples that 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 speaking, 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. The LRTs 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 then again in September 2005, December 2006, and October 2007.

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 < 0.5%	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%	Total Free Liquid Removed from Sample (mL)**
Petroset II-G	2:1	0.006	0.005	0.014	0.012	---
Petroset II-G *Unprotected	2:1	0.010	0.006	0.013	0.012	---
Petroset II-G	1.9:1	0.023	0.028	0.009	0.008	---
Petroset II-G	1.8:1	0.010	0.007	0.007	0.027	---
Petroset II-G	1.7:1	0.026	0.003	0.011	0.016	---
Petroset II-G	1.6:1	0.017	0.012	0.011	0.012	---
Petroset II-G	1.5:1	0.059	0.019	0.010	0.013	---
Nochar Petrobond	1:4	0.416	0.377	0.357	**0.538**	---
Nochar Petrobond *Unprotected	1:4	0.403	**0.568**	**1.049**	**1.291**	**405**
Nochar Petrobond	1:3	0.139	0.110	0.135	0.240	---
Nochar Petrobond *Unprotected	1:3	0.140	0.150	0.450	**0.124**	**424**
Nochar Petrobond	1:2.9	0.131	0.124	0.121	0.378	---
Nochar Petrobond	1:2.8	0.091	0.132	0.114	0.229	---
Nochar Petrobond	1:2.7	0.084	0.085	0.057	0.105	---
Nochar Petrobond	1:2.6	0.066	0.067	0.085	0.092	---
Nochar Petrobond	1:2.5	0.043	0.062	0.054	0.064	---
Nochar Petrobond *Unprotected	1:2.5	0.063	0.072	0.244	**0.444**	**461**
Nochar Petrobond	1:2.4	0.029	0.037	0.058	0.173	---
Nochar Petrobond	1:2.3	0.031	0.052	0.049	0.152	---
Nochar Petrobond	1:2.2	0.023	0.020	0.037	0.042	---
Nochar Petrobond	1:2.1	0.006	0.015	0.011	0.023	---
Nochar Petrobond	1:2	0.009	0.020	0.010	0.038	---
Nochar Petrobond *Unprotected	1:2	0.009	0.026	**1.363**	**1.173**	**451**

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

The LRT values for the Petroset II-G samples did not significantly change during the 3-1/2-year storage period. In fact, comparable amounts of liquid were released from the samples during the initial and the 3-1/2-year sampling events. Sunlight did not affect the sample integrity for the Petroset II-G/PUREX samples as indicated by the LRT value of 0.012% liquid release for both the UV protected and unprotected samples at the 2:1 ratio in FY07. No free liquid was detected during the 3-1/2-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 samples generated at the 1:4 and 1:3 waste-loading ratios show a trend of releasing more liquid after the 2006 sampling event. The 1:3 waste-loading ratio sample had an LRT value that increased from 0.135% release by volume to 0.240%, and the 1:4 ratio sample had an LRT value that increased from 0.357 to 0.538, which fails the NTS LRT criteria. However, the samples remained a rubbery consistency.

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 3-1/2-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. All four of the unprotected Nochar Petrobond samples released free liquid from April through August 2007 when the weather was much warmer and no free liquid when the weather was colder. Sample consistency remained rubbery but slightly softer than after sample generation.

Since the samples had been stored outside at the MSE Test Facility for 3-1/2 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 as indicated by the sample stability during the 3-1/2-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 liquid release testing was performed in October 2005, November 2006, and October 2007. The samples were tested for ambient temperature liquid release values using the PFT and LRT during the second 2005 sampling event and in 2006 and 2007. 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 19-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.

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

Sorbent Name	Weight-Based, Waste-Loading Ratio (wt sorbent: wt PUREX)	Initial 2005 LRT % Release by Volume NTS WAC < 0.5%	End of 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%
pH 5.5					
Waste Lock 770	1:10	0.360	0.897	**0.884**	**0.774**
Waste Lock 770	1:5	0.135	0.232	0.265	0.202
Quick Solid	1:10	0.364	0.408	0.358	0.360
Quick Solid	1:5	0.151	0.204	0.309	0.315
WaterWorks	1:10	0.359	0.356	**0.709**	**0.719**
WaterWorks	1:5	0.219	0.142	0.250	0.254
Aquaset II	1.1:1	0.142	0.075	0.100	0.103
Aquaset II	1.2:1	0.269	0.050	0.084	0.090
Aquaset	1:1	0.089	0.082	0.1941	0.178
Aquaset	1:1.25	0.477	0.148	0.189	0.195
Aquasorb	1:10	0.212	0.203	0.319	0.323
Aquasorb	1:5	0.222	0.158	0.213	0.173
Nochar A660 Acid Bond	1:10	0.208	0.292	**0.507**	0.317
Nochar A660 Acid Bond	1:5	0.227	0.198	0.176	0.275
pH 10.9					
Waste Lock 770	1:5	0.259	0.189	0.345	0.305
WaterWorks	1:5	0.269	0.216	0.222	0.248
Aquaset	1.25:1	0.056	0.059	0.098	0.099

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 19-L (5-gal) scale.

A total of 2498 L (660 gal) of the Rainwater wastestream was solidified at SRS, with 1249 L (330 gal) solidified using Aquaset at a waste-loading ratio of 1:1, and 1249 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 sorbents can solidify liquids with aqueous and organic phases and were tested since the Oils wastestream had a small aqueous component. The samples were tested for liquid release using the PFT and LRT after generation in 2005, and in November 2006 and October 2007. Table III presents the LRT values for the 19-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 < 0.5%	FY 2006 LRT % Release by Volume NTS WAC < 0.5%	FY 2007 LRT % Release by Volume NTS WAC < 0.5%
Organic Sorbents				
Petroset II	1:1.5	**0.519**	0.257	0.274
Petroset II + 5% Methanol	1:1.5	0.262	0.149	0.071
Petroset II-G Batch 17 + 5% Methanol	2:1	0.098	0.013	0.017
Petroset II-G Batch 17 + 0.5% Methanol	2.5:1	0.415	0.022	0.027
Petroset II-G Batch 18	2.5:1	0.013	0.012	0.018
Petroset II-G Batch 18 + 5% Methanol	2.5:1	0.003	0.025	0.021
Nochar Petrobond	1:1	0.035	0.029	0.045
Nochar Petrobond	1:2	0.199	0.059	0.055
Zorbent Metamatrix	1:2.5	**15.704**	**13.664**	**8.411**
Imbiber Beads	2.5:1	0.235	0.279	0.069
Petrol Sorb	1.5:1	0.020	0.018	0.050
ABZORBIT	1:2	0.036	0.022	0.034
Combination Sorbents				
Quick Solid 50	1:1	0.441	**1.095**	**0.562**
Sorbond Loc 40	2:1	**4.768**	0.035	0.011
Instasorb C	1.5:1	**11.577**	**10.469**	**11.401**
Liquisorb 1000	1:1	**0.512**	0.331	0.256
Organoclay TA-11	2.15:1	0.012	0.009	0.023
Additional Organic Sorbent				
Organoclay BM-QT-199 - A	2:1	**8.125**	0.009	0.016
Organoclay BM-QT-199 – A +0.5% Methanol	2:1	**5.817**	0.010	0.013
Organoclay BM-QT-199 – A +5% Methanol	2:1	**0.637**	0.014	0.015

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 and October 2007 than after the initial 2-week curing period when the samples were generated in 2005. The 2006 and 2007 polymer sorbent (Nochar Petrobond, Zorbent Metamatrix, Imbiber Beads, Petrol Sorb, and ABZORBIT) LRT values seemed to remain consistent with the initial LRT values determined 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. The samples will be tested for liquid release again at the end of FY08 to determine if the samples continue to release less liquid than the NTS WAC of < 0.5%. Additional testing should be performed in future work that will give a better indication of a realistic curing time for different oils wastestreams when combined with modified clay sorbents.

AQUEOUS ORGANIC SURROGATE WASTESTREAM 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, and the third data set was collected in September 2007. The LRT data are presented in Table IV. All of the 19-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 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%
Aqueous Sorbents				
Aquaset	1.5:1	0.260	0.148	0.147
Aquaset	2:1	0.033	0.037	0.059
Nochar A660 Acid Bond	1:5	0.293	----	----
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**
Quick Solid	1:3	0.219	**0.679**	----
Quick Solid	1:2	0.047	0.087	0.053
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**
WaterWorks SP-400	1:3	0.200	**0.541**	0.492
Combination Sorbents				
Quick Solid 50	1:1.5	0.074	0.053	0.074
Quick Solid 50	1:1	0.035	0.032	0.043
Instasorb C	1.5:1	0.055	0.058	0.083
Sorbond Loc-40	1:1	0.041	0.030	0.040
Sorbond Loc-40	1.5:1	0.048	0.031	0.029
Sorbond Loc-40	2:1	0.047	0.022	0.028
Liquisorb 1000	1:2	0.319	0.254	0.254
Liquisorb 1000	1:1	0.037	0.037	0.056

--- Failed PFT; therefore, no LRT was performed.

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]. Aquasorb and Quick Solid samples at the 1:3 ratio that passed all previous testing in 2005 showed a liquid release value higher than the NTS LRT WAC of < 0.5% release when tested in 2006 and 2007.

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 and 2007. This indicates that the combination-type sorbent materials are capable of solidifying aqueous liquids with a 6% oil phase, at least, over the short term of 2 years.

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 Nochar Petrobond sorbent and a 1:2 waste-loading ratio for the aqueous phase and the Nochar Acid Bond sorbent. The 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 are 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 < 0.5%	FY06 LRT % Release by Volume NTS WAC < 0.5%	FY07 LRT % Release by Volume NTS WAC < 0.5%
Petroset II-G	1.7:1	5	0.027	0.010	0.011
Aquaset	1.5:1				
Petroset II-G	1.7:1	10	0.022	0.020	0.012
Aquaset	1.5:1				
Petrobond	1:3	5	0.156	0.110	0.113
Acid Bond	1:2				
Petrobond	1:3	10	0.176	0.115	0.148
Acid Bond	1:2				

As seen in Table V, all of the LRT values decreased from the initial LRT values after the 29-month storage time, which indicates the 19-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. 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.

NOCHAR PETROBOND AND PUREX SURROGATE SORBENT STABILITY TESTING WITH AN AQUEOUS COMPONENT WITHOUT THE ADDITION OF AN AQUEOUS-TYPE SORBENT

At the request of the SRS client, tests were performed during FY06 to determine the impact that an aqueous component within the PUREX wastestream would produce when solidified using only organic-type sorbents without the addition of aqueous-type sorbents. These tests were undertaken in an effort to understand the wasteform breakdown characteristics of F-Canyon PUREX when combined with Nochar Petrobond sorbent at the 1:3 weight-based, waste-loading ratio of sorbent to surrogate with a water phase.

Samples were generated with several different amounts of water incorporated into the liquid F-Canyon Surrogate PUREX wastestream. Two sample sets were generated using 2%, 5%, 10%, 15%, and 20% water as part of the liquid surrogate organic phase that was solidified using Nochar Petrobond sorbent. One sample set was stored at ambient temperature, and the other sample set was stored at an elevated temperature of 37.8 °C (100 ºF). The samples were thoroughly mixed when generated and have been stored at the MSE Test Facility since January 2006. Liquid release tests were performed twice on the samples after 3 to 4 days and then weekly for approximately 1 month and then intermittently for almost 1 year during 2006. The samples were LRT tested three more times during 2007. Table VI presents the data collected during the 21-month storage period. The bolded LRT values in Table VI show failure of the NTS WAC for LRT.

Table VI. LRT Data for F-Canyon Surrogate PUREX and Nochar Petrobond Sorbent Generated at the 1:3 Weight-Based, Waste-Loading Ratio (Sorbent To Surrogate)

Sorbent Name	Sample Dates	LRT % Release by Volume NTS WAC < 0.5%	Total Free Liquid Released (mL)	Sorbent Name	Sample Dates	LRT % Release by Volume NTS WAC < 0.5%	Total Free Liquid Released (mL)
2% Ambient Temperature Sample				2% Elevated Temperature Sample			
Petrobond	1-13-06	0.233	0	Petrobond	1-13-06	0.275	0
Petrobond	1-17-06	0.334	0	Petrobond	1-17-06	0.291	0
Petrobond	1-24-06	0.341	0	Petrobond	1-24-06	0.296	0
Petrobond	1-31-06	0.449	0	Petrobond	1-31-06	0.342	0
Petrobond	2-7-06	0.460	0	Petrobond	2-7-06	0.379	0
Petrobond	2-14-06	**0.527**	0	Petrobond	2-14-06	0.407	0
Petrobond	3-23-06	**0.523**	0	Petrobond	3-23-06	0.482	0
Petrobond	9-13-06	0.262	60	Petrobond	9-13-06	**0.572**	5
Petrobond	12-4-06	0.405	200	Petrobond	12-4-06	**0.642**	5
Petrobond	4-24-07	0.391	225	Petrobond	4-24-07	**0.797**	30
Petrobond	8-10-07	**0.596**	15	Petrobond	8-10-07	**0.769**	10
Petrobond	10-3-07	**0.675**	200	Petrobond	10-3-07	**0.769**	30
Total Free Liquid			700 mL	Total Free Liquid			80 mL
5% Ambient Temperature Sample				5% Elevated Temperature Sample			
Petrobond	1-13-06	0.316	0	Petrobond	1-13-06	0.211	0
Petrobond	1-17-06	0.324	0	Petrobond	1-17-06	0.229	0
Petrobond	1-24-06	0.393	0	Petrobond	1-24-06	0.346	0
Petrobond	1-31-06	0.485	0	Petrobond	1-31-06	0.460	0
Petrobond	2-7-06	**0.501**	0	Petrobond	2-7-06	0.474	0
Petrobond	2-14-06	**0.533**	0	Petrobond	2-14-06	**0.523**	0
Petrobond	3-23-06	**0.545**	0	Petrobond	3-23-06	**0.530**	0
Petrobond	9-13-06	**0.534**	180	Petrobond	9-13-06	**0.659**	35
Petrobond	12-4-06	**0.624**	500	Petrobond	12-4-06	**0.837**	50
Petrobond	4-24-07	0.208	450	Petrobond	4-24-07	**0.809**	10
Petrobond	8-10-07	**0.511**	35	Petrobond	8-10-07	**0.764**	15
Petrobond	10-3-07	**0.571**	300	Petrobond	10-3-07	**0.768**	12
Total Free Liquid			1465 mL	Total Free Liquid			122 mL
10% Ambient Temperature Sample				10% Elevated Temperature Sample			
Petrobond	1-9-06	0.321	0	Petrobond	1-9-06	0.349	0
Petrobond	1-12-06	0.385	0	Petrobond	1-12-06	0.407	0
Petrobond	1-19-06	0.372	0	Petrobond	1-19-06	**0.522**	0
Petrobond	1-26-06	**0.503**	0	Petrobond	1-26-06	**0.544**	2
Petrobond	2-2-06	**0.740**	0	Petrobond	2-2-06	**0.474**	2

Sorbent Name	Sample Dates	LRT % Release by Volume NTS WAC < 0.5%	Total Free Liquid Released (mL)	Sorbent Name	Sample Dates	LRT % Release by Volume NTS WAC < 0.5%	Total Free Liquid Released (mL)
Petrobond	3-8-06	**0.682**	0	Petrobond	3-8-06	**0.655**	4
Petrobond	9-13-06	0.349	19	Petrobond	9-13-06	**0.669**	50
Petrobond	12-4-06	**1.055**	30	Petrobond	12-4-06	**0.514**	85
Petrobond	4-24-07	0.419	55	Petrobond	4-24-07	**0.612**	0
Petrobond	8-10-07	**0.600**	50	Petrobond	8-10-07	**0.627**	0
Petrobond	10-3-07	**0.531**	1	Petrobond	10-3-07	**0.639**	200
Total Free Liquid			155 mL	Total Free Liquid			341 mL
15% Ambient Temperature Sample				15% Elevated Temperature Sample			
Petrobond	1-9-06	**0.530**	0	Petrobond	1-9-06	**1.270**	6
Petrobond	1-12-06	**1.383**	30	Petrobond	1-12-06	**1.388**	9
Petrobond	1-19-06	**1.083**	8	Petrobond	1-19-06	**1.071**	3
Petrobond	1-26-06	**1.147**	0	Petrobond	1-26-06	**1.054**	9
Petrobond	2-2-06	**1.062**	1	Petrobond	2-2-06	**1.167**	4
Petrobond	3-8-06	**1.172**	6	Petrobond	3-8-06	**1.220**	88
Petrobond	9-13-06	**1.261**	26	Petrobond	9-13-06	**1.048**	46
Petrobond	12-4-06	**1.311**	31	Petrobond	12-4-06	**1.064**	6
Petrobond	4-24-07	**0.889**	40	Petrobond	4-24-07	**0.676**	0
Petrobond	8-10-07	**0.825**	49	Petrobond	8-10-07	**0.770**	0
Petrobond	10-3-07	**0.820**	8	Petrobond	10-3-07	**0.898**	295
Total Free Liquid			199 mL	Total Free Liquid			466 mL
20% Ambient Temperature Sample				20% Elevated Temperature Sample			
Petrobond	1-9-06	**1.209**	58	Petrobond	1-9-06	**1.303**	49
Petrobond	1-12-06	**1.322**	69	Petrobond	1-12-06	**1.539**	164
Petrobond	1-19-06	**1.212**	23	Petrobond	1-19-06	**1.199**	81
Petrobond	1-26-06	**1.123**	32	Petrobond	1-26-06	**1.241**	5
Petrobond	2-2-06	**1.207**	77	Petrobond	2-2-06	**1.239**	72
Petrobond	3-8-06	**0.983**	46	Petrobond	3-8-06	**1.185**	51
Petrobond	9-13-06	**1.096**	50	Petrobond	9-13-06	**1.130**	10
Petrobond	12-4-06	**1.289**	37	Petrobond	12-4-06	**0.679**	0
Petrobond	4-24-07	**1.203**	29	Petrobond	4-24-07	0.188	0
Petrobond	8-10-07	**1.069**	42	Petrobond	8-10-07	0.116	0
Petrobond	10-3-07	**1.043**	5	Petrobond	10-3-07	0.169	0
Total Free Liquid			468 mL	Total Free Liquid			431 mL

For the 2% and 5% water F-Canyon PUREX and Petrobond Nochar samples, the samples stored at ambient temperature failed the LRT criteria faster than the samples stored at the elevated temperature of 37.8 °C (100 ºF) and released substantially more liquid than the samples stored at the elevated temperature. After 21 months of storage, the 2% water samples released 700 mL and 80 mL of free liquid from the ambient temperature sample and the elevated temperature sample, respectively. The initial amount of water added to the 2% water F-Canyon samples was 195 mL. The free liquid collected from the 2% samples was organic with only a few drops of water. The elevated temperature sample stayed mostly dry with jelly-like masses appearing on the top and bottom of the sample and free liquid only showing up at the bottom of the sample during the last two sampling events. The ambient temperature sample stayed mostly dry until the September 2006 sampling date when 60 mL of organic was collected from the top of the sample. All of the free liquid collected from the 2% water samples was removed from the sample buckets prior to running the LRTs, and no free liquid was released from the bottom of the LRT test apparatus during testing. The ambient temperature sample was very gelatinous by December 2006

with freestanding organic liquid in the sample. The ambient temperature sample then became somewhat more solid by the August 2007 sampling event; however, by October 2007, the sample once again was very gelatinous in consistency with 200 mL of organic liquid on top of the sample. The elevated temperature sample stayed rubbery in consistency until April 2007 when the sample consistency became jelly like with no freestanding liquid and remained the same throughout the 21-month storage time.

The 5% water F-Canyon PUREX and Nochar Petrobond samples showed the same trend as the 2% water samples with the ambient temperature sample failing the LRT criteria before the elevated temperature sample and again releasing much more free liquid than the sample stored at the elevated temperature of 37.8 °C (100 ºF). The amount of water added to the 5% water F-Canyon PUREX samples was 486 mL. Just as with the 2% water samples, the ambient temperature 5% water sample released more free liquid than the amount of water that was initially added while the elevated temperature sample released less liquid than the water amount initially added to the samples. The ambient temperature 5% water sample released 1,465 mL, and the elevated temperature sample released 122 mL of free liquid during the 21-month storage period. Of the 1,465 mL of free liquid released from the ambient temperature sample, 1,450 mL was organic, and 15 mL was water. The 5% water elevated temperature sample released only organic liquid and no water. The sample consistency for the 5% water samples was similar to the 2% water samples with the ambient temperature sample breaking down into a gelatinous form and the elevated temperature sample becoming jelly like.

The trend changed for the 10% water F-Canyon and Nochar Petrobond sample set, with the elevated temperature sample failing the LRT criteria 1 week before the ambient temperature sample and releasing more free liquid than the ambient temperature sample. The 10% water samples were generated by adding 973 mL of water to the organic PUREX phase. The ambient temperature sample released 155 mL of free liquid (140 mL of organic and 15 mL of water) while the elevated temperature sample released 341 mL of free liquid (141 mL of water and 200 mL of organic). The 10% water samples released much more water and much less organic liquid than the 2% and 5% water samples during the 21-month storage period. Small amounts of free liquid were released during the LRT testing, but most of the free liquid was recovered from the sample buckets prior to performing the LRTs. This phenomenon did not occur for the 2% and 5% samples, which only released free liquid from the sample buckets and none from the LRT device during testing. Both samples generated with 10% water remained rubbery at the top section of the samples with the bottom sample sections becoming softer and more gelatinous in nature by the end of the storage period.

The 15% water F-Canyon PUREX and Nochar Petrobond samples at both temperatures failed the LRT criteria every time they were sampled during the 21-month storage period and followed the same trend as the 10% water samples with the ambient temperature sample releasing less free liquid than the elevated temperature sample. The initial amount of water in the 15% water samples was 1,459 mL. A total of 199 mL of free liquid was released from the ambient temperature sample with 143 mL being water and 56 mL being organic. The elevated temperature sample released a total of 466 mL of free liquid with 338 mL being water and the remaining 128 mL being organic. Both samples released more water than organic liquid during the 21-month storage period, and neither sample released any organic liquid until September 2006. The 15% water samples released free liquid from the LRT test apparatus during most tests as well as the free liquid that was collected from the buckets during the sampling events. Sample consistency after the 21-month storage period for the 15% water elevated temperature sample was turning jelly-like on the top surface but was still rubbery for the bottom three-fourths of the sample. The 15% water ambient temperature sample was starting to soften up in the center volume of the bucket but was still somewhat rubbery around the outside edges of the sample.

The 20% water sample sets performed differently than the other sample sets with the ambient temperature and elevated temperature samples releasing similar amounts of free liquid over the storage period. The ambient temperature sample released 468 mL of free liquid with only 10 mL of the total being organic and the rest being water. The elevated temperature sample released 431 mL of free liquid with the total amount being water with no organic liquid released during the storage period. The ambient temperature sample released a more consistent amount of water throughout the storage period, and the elevated temperature sample released more water earlier during the storage period with no free liquid released since December 2006. The sample consistency of the 20% water ambient temperature sample is more spongy than rubbery, and the consistency of the elevated temperature sample has been dry and rubbery since December 2006.

Once the ambient temperature 2% and 5% F-Canyon PUREX and Nochar Petrobond water samples start releasing liquid, they did so at a high rate, and the sample consistency broke down from a rubbery mass to a much softer gelatinous form with chunks of rubbery material as shown in Fig. 1. These photos were taken at the last sampling event in 2006.

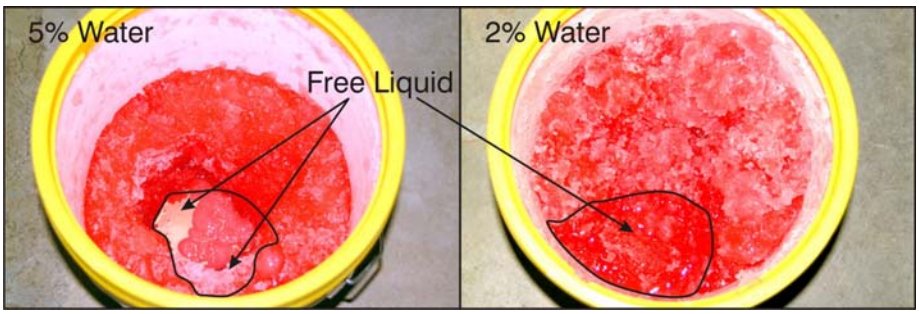

Fig 1. Pictures of the ambient temperature F-Canyon PUREX and Nochar Petrobond 2% (left) and 5% (right) water samples.

The 2% ambient temperature water sample is shown in Fig. 1 on the left-hand side, and the picture of the 5% ambient temperature waster sample is shown on the right. The 2% sample shows free liquid toward the bottom of the photo with drier chunks of the sample toward the top of the photo. The 5% sample shows free liquid in the depression of the sample surrounded by a very gelatinous mass. In all, 700 mL of free liquid was removed from the 2% water sample, and 1,465 mL of organic and water was removed from the 5% sample by the end of the 21-month storage period. The 10%, 15%, and 20% water samples stored at ambient temperature released much less free liquid than the 2% and the 5% ambient temperature samples. The samples stored at the elevated temperature show a more normal trend by releasing higher amounts of liquid over time as the original percent of water increased in the samples.

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 that was lower in price. 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 had similar performance characteristics to each other and to the original Petroset II-G Batch 17. Bench-scale LRT data for the Petroset II-G Batch 17 and the full PUREX surrogate waste was available from 2004 testing. The 19-L (5-gal) bucket samples were generated in 2005 using the two granular clays and the full Legacy PUREX surrogate waste 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 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 19-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 VII represent 19-L (5-gal) samples that had not been generated for testing at that point in time or bench-scale sampling events where no sample was available for future testing due to their small size.

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

Table VII. 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%
Batch 17 – Bench-Scale Samples					
Petroset II-G	2:1	0.006	-	-	-
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	-	-	-
Batch 18 – 19-L (5-Gal) Bucket Samples					
Petroset II-G	2:1	-	0.009	0.012	0.015
Petroset II-G	1.9:1	-	0.010	0.013	0.017
Petroset II-G	1.8:1	-	0.012	0.011	0.020
Petroset II-G	1.7:1	-	0.024	0.018	0.027
Petroset II-G	1.6:1	-	0.039	0.015	0.031
Petroset II-G	1.5:1	-	0.043	0.026	0.031
Batch 1 – 19-L (5-Gal) Bucket Samples					
Organoclay	2:1	-	0.015	0.008	0.008
Organoclay	1.9:1	-	0.014	0.013	0.019
Organoclay	1.8:1	-	0.013	0.016	0.024
Organoclay	1.7:1	-	0.008	0.017	0.027
Organoclay	1.6:1	-	0.151	0.017	0.027
Organoclay	1.5:1	-	0.209	0.063	0.099
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	-
- no sample available for testing					

As seen in previous PUREX surrogate data sets, the clay samples generated in FY05 continue to pass LRT requirements after 26 months 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 show 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 19-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 aqueous phases without sample breakdown over time. The liquid organic phase included 64% kerosene and 36% tributyl phosphate by weight. The samples were made using 8 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 VIII presents the LRT data collected until October 2007.

Table VIII. 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 < 0.5%
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	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	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

The samples passed all of the LRTs performed during the 17-month testing period with comparable amounts of liquid released. 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 2008 to see if any sample breakdown occurs at a later point in time.

B-25 BOX SAMPLES GENERATED WITH PETROSET II-G AND ORGANOCLAY

Two large-scale samples were generated in November 2005 using kerosene as the simple F-Canyon PUREX surrogate and two granular clay sorbents [Petroset II-G and Organoclay Batch 1 (no longer available)], to determine if the ratios used during previous 208-L (55-gal) drum tests would scale up to the B-25 box size. The successful weight-based, waste-loading ratio used in the 208-L (55-gal) drum tests was 2.35:1 sorbent to surrogate for both sorbents. A total of 757 L (200 gal) of kerosene was used to generate each of the B-25 box samples. The waste-loading ratio for the Organoclay Batch 1 sample scaled up to a 2:1 ratio, and the Petroset II-G sample ratio scaled up to 3:1. The Organoclay sample used much less sorbent to solidify 757 L (200 gal) of organic than the Petroset II-G sample did. The samples were allowed to cure for 2 weeks; small samples from within the boxes were then taken, and an LRT was performed on each of the small samples. The B-25 box samples were stored outside at the MSE Test Facility for 1 year, at which time, another subset of samples was collected and subjected to LRT. The LRT data for both sampling events is included in Table IX.

Both of the B-25 box samples generated in November 2005 passed all of the initial LRTs performed on the small samples taken from various locations within the boxes. The sample boxes were stored outside for 1 year, after which they were cut open to identify sample integrity and consistency, check for void spaces in the samples, and to collect small samples for LRT. Figures 2 through 4 show MSE personnel destroying and removing the B-25 boxes to check the samples. Figure 5 shows the kerosene and Organoclay and Petroset II-G samples after the B-25 boxes were removed.

The Organoclay sample is gray in color while the Petroset II-G sample is brown. The Organoclay sample was waxy to the touch, and the Petroset II-G sample was more like dry dirt in consistency. Neither of the samples had any void spaces as shown in Fig. 5, nor were any void spaces discovered within the samples when they were physically destroyed to collect the smaller samples. Both of the granular clay products produced a hard freestanding wasteform with the same consistency for each of the materials as when the samples were generated.

Figure 6 shows the cutting tool and sampling approach used to destroy the samples, to check for void spaces and sample consistency, and to cut the B-25-scale samples for smaller samples used for the LRT.

These large-scale samples demonstrated that Organoclay and Petroset II-G could sorb organic liquids and retain that liquid for 1 year. These granular clay products do not require mixing (i.e., liquid is poured into the box and then the sorbents are poured into the liquid already in the box until a small amount of dry solid is present on top of the solidified liquid). The labor saved would be considerable compared to products that need to be mixed to solidify the waste, and worker exposure to radioactive liquids would be much less for this procedure.

Table IX. LRT Data for the B-25 Box Samples

Sorbent Name	Weight-Based, Waste-Loading Ratio (wt sorbent: wt kerosene)	Sample Location	Sample Dates	LRT % Release by Volume NTS WAC < 0.5%
Organoclay	2:1	1 Top	11-21-05	0.163
Organoclay	2:1	1 Bottom	11-21-05	0.225
Organoclay	2:1	2 Top	11-21-05	0.142
Organoclay	2:1	2 Bottom	11-21-05	0.247
Organoclay	2:1	3 Top	11-21-05	0.304
Organoclay	2:1	3 Bottom	11-21-05	0.305
Organoclay	2:1	4 Top	11-21-05	0.145
Organoclay	2:1	4 Bottom	11-21-05	0.212
Organoclay	2:1	5 Top	11-21-05	0.128
Organoclay	2:1	5 Bottom	11-21-05	0.207
Organoclay	2:1	1	11-1-06	0.487
Organoclay	2:1	2	11-1-06	0.024
Organoclay	2:1	3	11-1-06	0.466
Organoclay	2:1	4	11-1-06	0.107
Organoclay	2:1	5	11-1-06	0.079
Organoclay	2:1	6	11-1-06	0.020
Petroset II-G	3:1	1 Top	11-28-05	0.006
Petroset II-G	3:1	1 Bottom	11-28-05	0.011
Petroset II-G	3:1	2 Top	11-28-05	0.007
Petroset II-G	3:1	2 Bottom	11-28-05	0.006
Petroset II-G	3:1	3 Top	11-28-05	0.006
Petroset II-G	3:1	3 Bottom	11-28-05	0.004
Petroset II-G	3:1	4 Top	11-28-05	0.010
Petroset II-G	3:1	4 Bottom	11-28-05	0.005
Petroset II-G	3:1	5 Top	11-28-05	0.007
Petroset II-G	3:1	5 Bottom	11-28-05	0.005
Petroset II-G	3:1	1	11-1-06	0.003
Petroset II-G	3:1	2	11-1-06	0.003
Petroset II-G	3:1	3	11-1-06	0.005
Petroset II-G	3:1	4	11-1-06	0.008
Petroset II-G	3:1	5	11-1-06	0.010

Fig 2. B-25 box being cut open.

Fig. 3. B-25 box being opened to check samples for void spaces and sample consistency.

Fig. 4. B-25 box opened.

Fig. 5. Organoclay (left) and Petroset II-G (right) samples after removal from boxes.

Fig. 6. Sampling tool and sampling approach for the B-25 box samples.

CONCLUSIONS

Several characteristics of the PUREX 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.
- A water component in the surrogate PUREX waste is not desirable for the following reasons:
  - a combination of the aqueous-type sorbents and organic sorbents tie up the aqueous organic full Legacy PUREX surrogate waste at bench scale and 19-L (5-gal) bucket scale but do not tie up the 2-phase liquid at the 208 (55-gal) drum scale;
  - Organoclay alone solidifies the organic F-Canyon PUREX surrogate with a water component up to 10% of the surrogate weight for the short term;
  - organic F-Canyon PUREX with a water component solidified with Nochar Petrobond shows sample failure later for the 2% and 5% samples than for samples generated with higher water contents, but they show severe sample breakdown with mostly organic releasing from the samples;
  - organic F-Canyon PUREX with a water component solidified with Nochar Petrobond shows sample failure for the 10% water sample with both organic and water releasing from the samples;

  - organic F-Canyon PUREX with a water component solidified with Nochar Petrobond shows sample failure almost immediately for the 15% water sample by releasing more water than organic; and
  - organic F-Canyon PUREX with a 20% water component solidified with Nochar Petrobond shows sample failure almost immediately by releasing mostly water with a small amount of organic.
- The simple F-Canyon PUREX surrogate can be successfully solidified at the B-25 box scale using Organoclay and Petroset II-G granular clay sorbents that do not require mixing.
- The Rainwater and Aqueous Organic surrogate wastestreams can be successfully solidified using several different sorbents, and most remain solidified after more than 2 years of storage.
- The Oils wastestream surrogate waste can be successfully solidified using several sorbents, and most remain solidified after more than 2 years of storage. However, additional testing should be performed to determine a sufficient cure time for some of the clay sorbents since they were able to pass LRT after more than a year but failed the LRT tests after a 2-week curing period. These samples will be tested again in FY08 to confirm sample stability after a longer storage period.
- Regardless of which batch was tested, the granular clays (Petroset II-G and Organoclay) performed similarly and produced LRT values and order of magnitudes below the NTS LRT WAC of < 0.5% liquid release by volume.

To better characterize the solidified SRS wastestreams, MSE recommends that all solidified wastestreams at the MSE Test Facility be liquid release tested in 2008 to confirm conclusions from FY07 testing after additional storage time in FY08.

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