

**Sorbent Testing for the Solidification of Organic Process Wastestreams from the Radiochemical Engineering Development Center at Oak Ridge National Laboratory**

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**ABSTRACT**

The U.S. Department of Energy (DOE) has tasked MSE Technology Applications, Inc. (MSE) with evaluating various sorbents to solidify the radioactive liquid organic waste from the Radiochemical Engineering Development Center (REDC) at Oak Ridge National Laboratory (ORNL). REDC recovers and purifies heavy elements (berkelium, californium, einsteinium, and fermium) from irradiated targets for research and industrial applications. Both aqueous and organic wastestreams are discharged from REDC. Organic waste is generated from the plutonium/uranium extraction (PUREX), Cleanex, and Pubex processes.<sup>1</sup> The PUREX waste derives from an organic-aqueous isotope separation process for plutonium and uranium fission products, the Cleanex waste derives from the removal of fission products and other impurities from the americium/curium product, and the Pubex waste is derived from the separation process of plutonium from dissolved targets. An aqueous wastestream is also produced from these separation processes. MSE has been tasked to test a grouting formula for the aqueous wastestream that includes specially formulated radioactive shielding materials developed by Science and Technology Applications, LLC. This paper will focus on the sorbent testing work.

Based on work performed at Savannah River Site (SRS) (Refs. 1, 2), ORNL tested and evaluated three sorbents capable of solidifying the PUREX, Pubex, and Cleanex wastestreams and a composite of the three organic wastestreams: Imbiber Beads® IMB230301 (Imbiber Beads), Nochar A610 Petro Bond™, and Petroset II Granular™ (Petroset II-G). Surrogates of the PUREX, Pubex, Cleanex, and a composite organic waste were used for the bench-scale testing. Recommendations resulting from the ORNL testing included follow-on testing by MSE for two of the three sorbents: Nochar Petro Bond and Petroset II-G. MSE recommended that another clay sorbent, Organoclay BM-QT-199, be added to the test sequence. The sorbent/surrogate combinations were tested at bench scale, 19-liter (L) [5-gallon (gal)] bucket scale, and 208-L (55-gal) drum scale.

The testing performed by MSE will help ORNL select the right solidification materials and wasteform generation methods for the design of a new treatment facility. The results could also be used to help demonstrate that ORNL could meet the waste acceptance criteria for the ultimate disposal site for the wasteforms. The organics will be solidified as transuranic waste for disposal at the Waste Isolation Pilot Plant, and the aqueous wastestream will be grouted and disposed of at the Nevada Test Site as low-level waste if real waste testing indicates similar results to the surrogate testing.

The objective of this work was to identify a sorbent capable of solidifying PUREX, Pubex, and Cleanex organic wastes individually and a composite of the three organic wastestreams. The sorbent and surrogate combinations must also be compatible with processing equipment and maintain stability under a variety of conditions that could occur during storage/shipment of the solidified wastes.

<sup>1</sup> The extraction process wastestreams referred to as PUREX and Cleanex are not related to the commercial products known as Purex and Kleenex.

## INTRODUCTION

The U.S. Department of Energy (DOE) tasked MSE Technology Applications, Inc. (MSE) to evaluate various sorbents to solidify the radioactive liquid organic waste from the Radiochemical Engineering Development Center (REDC) at Oak Ridge National Laboratory (ORNL). REDC recovers and purifies heavy elements (berkelium, californium, einsteinium, and fermium) from irradiated targets for research and industrial applications. The organic waste is generated from the plutonium/uranium extraction (PUREX), Cleanex, and Pubex processes. The PUREX waste derives from an organic-aqueous isotope separation process for plutonium and uranium fission products, the Cleanex waste derives from the removal of fission products and other impurities from the americium/curium product, and the Pubex waste is derived from the separation process of plutonium from dissolved targets. An aqueous wastestream, which is a mixture of the raffinate streams from the various extraction processes plus the caustic solution that is used to dissolve the aluminum cladding from the irradiated targets, is also produced from these separation processes. MSE was also tasked to test a grouting formula for the aqueous wastestream that includes radioactive shielding material as part of this project; however, the focus of this paper is the sorbent testing using the organic waste surrogates.

Based on work performed at the Savannah River Site (SRS) in 2001 (Refs. 1, 2), ORNL tested and evaluated three sorbents capable of solidifying the PUREX, Pubex, and Cleanex organic wastestreams: Imbiber Beads® IMB230301 (Imbiber Beads), Nochar A610 Petro Bond™ (Nochar), and Petroset II Granular™ (Petroset II-G). The surrogate organic wastestream formulas were developed by ORNL during this phase of testing. Recommendations resulting from the ORNL testing included follow-on testing for two of the three sorbents. The two sorbent materials recommended for evaluation and testing by ORNL during fiscal year (FY) 2006 included Nochar and Petroset II-G. MSE recommended that another clay sorbent, Organoclay BM-QT-199 (Organoclay), be added to the test sequence. The sorbent surrogate combinations were tested at bench scale and 19-L (5-gal) bucket scale and at the 208-L (55-gal) drum scale.

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

- sorbent handling;
- sorbent capacity;
- sorption rate;
- mixing requirements;
- volumetric expansion;
- time to solidify;
- final wasteform physical characteristics; and
- sorbent cost.

## TEST OBJECTIVES

The objective of this work was to identify a sorbent capable of solidifying PUREX, Pubex, and Cleanex organic wastes independently and then a composite of the three organic wastestreams that is also compatible with processing equipment. The sorbent and surrogate combinations must also remain stable under a variety of conditions that could occur during solidification, storage, shipment, and burial of the solidified wastes.

Specific objectives for the ORNL organic surrogate sorbent testing and evaluation were:

- verify the appropriate waste-loading ratios, addition methods, and mixing requirements during bench-scale optimization studies based on previous MSE PUREX surrogate testing with the selected sorbents;
- verify the optimum waste-loading ratio at the 19-L (5-gal) scale;
- verify the best addition order for each sorbent/surrogate combination at the 19-L (5-gal) scale;
- verify the mixing requirements for each sorbent/surrogate combination at the 19-L (5-gal) scale;
- observe and record the sorption time and volumetric expansion for each sorbent/surrogate combination at bench-scale, 19-L (5-gal) scale, and 208-L (55-gal) drum;
- verify the absence/presence of free liquid at the bench-, 19-L (5-gal), and 208-L (55-gal) scale using the Paint Filter Test (PFT) according to SW-846 Method 9095 (Ref. 3);
- verify the amount of liquid released from the wasteforms at the bench-, 19-L (5-gal), and 208-L (55-gal) scale using the Liquid Release Test (LRT) according to SW-846 Method 9096 (Ref. 4);
- perform 208-L (55-gal) drum scale-up tests with the sorbents and the composite organic surrogate based on results of the 19-L (5-gal) bucket tests to verify the optimum waste-loading ratio, addition methodology, and mixing techniques at the 208-L (55-gal) scale; and
- perform a cost evaluation for the sorbents based on the optimized 208-L (55-gal) scale test results to determine the cost to solidify 379 L (100 gal) of the composite organic wastestream.

## MATERIAL DESCRIPTIONS

The surrogate organic PUREX, Pubex, and Cleanex recipes were developed at ORNL to perform initial sorbent screening tests and should provide a representative comparison with the actual wastestreams requiring solidification. A composite organic wastestream that represents a combination of the three organic wastes was also developed by ORNL and was tested with the sorbents. The sorbents that were tested at the MSE Test Facility in Butte, Montana, were identified by ORNL and MSE.

### Surrogate Formulation Descriptions

The recipe for the four organic surrogates was developed for previously performed sorbent tests at ORNL during FY05. The surrogate recipes are presented in Tables I through IV.

Table I. PUREX Surrogate Recipe

Chemical Name	Weight
Tributyl phosphate	35.87
Petroleum naptha	64.13
Total	100

Table II. Pubex Surrogate Recipe

Chemical Name	Weight
1,4 diethylbenzene	58.17
2-ethyl-1-hexanol	17.95
2-ethylhexyl hydrogen phosphate	22.92
2,5-di-tert-butylhydroquinone	0.96
Total	100

Table III. Cleanex Surrogate Recipe

Chemical Name	Weight Percent
Petroleum naptha	62.75
1,4 diethylbenzene	6.45
HDEHP	26.97
Adogen-364	3.82
Total	100

Table IV. Composite Organic Surrogate Recipe

Chemical Name	Weight Percent
Petroleum naptha	34.13
1,4 diethylbenzene	40.96
HDEHP	17.38
Tributyl phosphate	3.99
2-ethyl-1-hexanol	2.00
Adogen-364	1.44
2,5-di-tert-butylhydroquinone	0.11
Total	100

## Sorbent Descriptions

Sorbents identified for testing with the four organic wastestreams are listed below.

- Nochar, a sorbent composed of proprietary polymer crystals. This sorbent is manufactured by Nochar, Inc.
- Petroset II-G, a modified granular clay stabilizing agent that does not require mixing during the waste solidification process. This sorbent is manufactured by Fluid Tech, Inc.
- Organoclay, a modified granular clay solidifying agent that does not require mixing during the waste solidification process. This sorbent is manufactured by M<sup>2</sup> Polymer Technologies, Inc.

## ORGANIC SURROGATE SORBENT TESTING

In summary, the objective of the experimental work was to identify a sorbent capable of solidifying the PUREX, Pubex, and Cleanex organic wastestreams and the composite organic surrogate wastestream that is cost effective and compatible with processing equipment. The sorbent/surrogate combinations must remain stable under conditions that may be encountered during solidification, storage, shipment, and burial of the wastes. The results will be used to perform a cost evaluation to project the cost (\$/379 L (100 gal) treated liquid waste) for solidifying the composite organic surrogate using each sorbent.

### Bench-Scale Organic Surrogate Sorbent Testing

Liter-sized samples of the sorbent and organic wastestreams were generated during the bench-scale sorbent testing at specific weight-based, waste-loading ratios. The initial waste-loading ratios used for the organic wastestreams were the optimized waste-loading ratios determined during MSE PUREX testing with SRS PUREX surrogates for Nochar and Petroset II-G sorbents (Refs. 5, 6). A waste-loading ratio of 1:3 sorbent to surrogate was used for the Nochar sorbent during MSE PUREX surrogate testing for the SRS PUREX wastestream. However, it has been observed that sunlight will break down the combinations over time at the 1:3 sorbent-to-surrogate ratio; consequently, a 1:2.5 and a 1:2 ratio was tested. The optimum waste-loading ratio determined during previous testing for Petroset II-G was

between 2:1 and 2.5:1, depending on specific Petroset II-G batches. For this test sequence, the 2:1 ratio was used. Another ratio corresponding to the manufacturer's recommended sorbent application of having dry sorbent material on top of the sorbed liquid waste was also tested. The optimum waste-loading ratio used for this test sequence for the Organoclay was 2:1; however, the additional ratio was also tested that corresponded to the manufacturer's recommended application of a dry layer of sorbent on top of the solidified wasteform.

To test addition methods, the Petroset II-G and Organoclay sorbent/surrogate mixtures were initially prepared in two different ways: 1) by adding the dry sorbents to the liquid surrogates without mixing and 2) by adding the liquid surrogates to the solid sorbents without mixing. The Nochar samples were prepared by adding the sorbent to the surrogate without mixing per the manufacturer's recommendation and by adding the surrogate to the sorbent with mixing as recommended by MSE.

Table V shows the weight-based, waste-loading ratios; addition methods; and mixing methodologies used during the bench-scale testing. The sorption times and the PFT and LRT results for the samples are also presented in Table V. SW-846 Method 9095A, *Paint Filter Free Liquids Test Procedure*, was used to determine if free liquids exist in the final wasteforms, and SW-846 Method 9096, *Liquids Release Test Procedure*, was used to determine the amount of liquid released from the solidified wasteforms. Only samples that passed the PFT were subjected to LRT because any loaded sorbent that fails the PFT is assumed to release liquids if subjected to the LRT. The Waste Isolation Pilot Plant (WIPP) Waste Acceptance Criteria (WAC) of 1% liquid release by volume was the criteria used to determine if a sample passed the LRT. The LRT values that are bolded in Table V failed the WIPP LRT WAC.

As seen in Table V, the best addition methodology for the Petroset II-G and the Organoclay sorbents was to add the sorbent material to the liquid surrogate waste as indicated by the lower liquid release numbers and the one failed Organoclay sample that used the alternative addition method. The Nochar sorbent required vigorous mixing to ensure a uniform wasteform as shown by the free liquid evident in the samples after the 14-day curing period for the samples that were not mixed (Fig. 1). The liquid surrogate waste should be added to the sorbent while the sorbent is being mixed so the liquid is incorporated as quickly as possible since the wasteform sets up to a rubbery consistency very quickly.

The Nochar samples generated with the PUREX organic surrogate that were mixed are shown on the left-hand side of the photograph presented in Fig. 1, and the samples that were not mixed are shown on the right-hand side of the photograph. This figure shows why mixing is required when using Nochar with a volatile organic liquid.

Figures 2a and 2b show samples of the Petroset II-G sorbent and the PUREX surrogate at 2 hours (hr) after sample generation using the method of adding the surrogate liquid waste to the sorbent material. Figure 2a shows the 3.1:1 ratio and the 2:1 ratio samples, and Fig. 2b shows the back of the 3.1:1 sample. As can be seen from the photographs, excess liquid remains on top of the 3.1:1 sample with isolated pockets of dry sorbent material. A soft gummy layer results on the top of the 2:1 and the 3.1:1 ratio samples as the excess liquid sorbs into the sorbent material as seen in the samples shown in Fig. 2a.

Figure 3 shows a pair of the Organoclay and surrogate PUREX samples at the 2.6:1 ratio solidified using the two different addition methods after the 14-day cure time. The sample on the left was generated using the favored addition method of pouring the dry sorbent material into the liquid surrogate waste, and the sample on the right was generated using the alternative addition method of pouring the liquid waste onto the solid sorbent material. As can be seen in the photograph, the sample on the left has a small layer of dry sorbent material on top, and the sample on the right has isolated pockets of dry material throughout the sample. The top section of the sample on the right also resulted in a softer gummy layer where the excess surrogate liquid sorbed into the Organoclay sorbent.

Table V. Bench-Scale Test Matrix and Test Results for the Organic Surrogate Sorbent Combinations

Sorbent Name	Surrogate Name	Weight-Based, Waste-Loading Ratio (wt sorbent: wt surrogate)	Sorbent/Surrogate Addition Method and Effectiveness	Mixing/ No Mixing	Sorption Time	PFT	LRT (Percent Liquid Released by Volume)
Petroset II-G	PUREX	2:1	Sorbent into surrogate	No Mixing	4 hr	Pass	0.191
Petroset II-G	PUREX	3.1:1	Sorbent into surrogate	No Mixing	15 min	Pass	0.013
Petroset II-G	Cleanex	2:1	Sorbent into surrogate	No Mixing	1-1/2 hr	Pass	0.181
Petroset II-G	Cleanex	3.6:1	Sorbent into surrogate	No Mixing	17 min	Pass	0.012
Petroset II-G	Pubex	2:1	Sorbent into surrogate	No Mixing	15 min	Pass	0.041
Petroset II-G	Pubex	2.3:1	Sorbent into surrogate	No Mixing	5 min	Pass	0.041
Petroset II-G	Composite	2:1	Sorbent into surrogate	No Mixing	2-1/2 hr	Pass	0.028
Petroset II-G	Composite	2.6:1	Sorbent into surrogate	No Mixing	5 min	Pass	0.016
Petroset II-G	PUREX	2:1	Surrogate into sorbent	No Mixing	36 hr	Pass	0.268
Petroset II-G	PUREX	3.1:1	Surrogate into sorbent	No Mixing	4-1/2 hr	Pass	0.396
Petroset II-G	Cleanex	2:1	Surrogate into sorbent	No Mixing	1/2 hr	Pass	0.030
Petroset II-G	Cleanex	3.6:1	Surrogate into sorbent	No Mixing	15 min	Pass	0.010
Petroset II-G	Pubex	2:1	Surrogate into sorbent	No Mixing	5 min	Pass	0.229
Petroset II-G	Pubex	2.3:1	Surrogate into sorbent	No Mixing	28 min	Pass	0.229
Petroset II-G	Composite	2:1	Surrogate into sorbent	No Mixing	1 hr 40 min	Pass	0.162
Petroset II-G	Composite	2.6:1	Surrogate into sorbent	No Mixing	5 hr	Pass	0.435
Organoclay	PUREX	2:1	Sorbent into surrogate	No Mixing	1 hr 15 min	Pass	0.013
Organoclay	PUREX	2.6:1	Sorbent into surrogate	No Mixing	15 min	Pass	0.005
Organoclay	Cleanex	2:1	Sorbent into surrogate	No Mixing	2-1/2 hr	Pass	0.674
Organoclay	Cleanex	2.8:1	Sorbent into surrogate	No Mixing	10 min	Pass	0.007
Organoclay	Pubex	2:1	Sorbent into surrogate	No Mixing	2-1/2 hr	Pass	0.074
Organoclay	Composite	2.2:1	Sorbent into surrogate	No Mixing	1/2 hr	Pass	0.043
Organoclay	Composite	2:1	Sorbent into surrogate	No Mixing	15 min	Pass	0.020
Organoclay	PUREX	2:1	Surrogate into sorbent	No Mixing	36 hr	Pass	0.345
Organoclay	PUREX	2.6:1	Surrogate into sorbent	No Mixing	32 hr	Pass	0.077
Organoclay	Cleanex	2:1	Surrogate into sorbent	No Mixing	15 hr	Pass	<b>1.053</b>
Organoclay	Cleanex	2.8:1	Surrogate into sorbent	No Mixing	12 hr	Pass	0.009
Organoclay	Pubex	2:1	Surrogate into sorbent	No Mixing	15 hr	Pass	0.496
Organoclay	Composite	2.2:1	Surrogate into sorbent	No Mixing	20 hr	Pass	0.422
Organoclay	Composite	2:1	Surrogate into sorbent	No Mixing	18 hr	Pass	0.429
Nochar	PUREX	1:2	Surrogate into sorbent	Mixing	NA	Pass	0.023
Nochar	PUREX	1:2.5	Surrogate into sorbent	Mixing	NA	Pass	0.081
Nochar	Cleanex	1:2	Surrogate into sorbent	Mixing	NA	Pass	0.627
Nochar	Cleanex	1:2.5	Surrogate into sorbent	Mixing	NA	Pass	<b>1.449</b>
Nochar	Pubex	1:2	Surrogate into sorbent	Mixing	NA	Pass	0.639
Nochar	Pubex	1:2.5	Surrogate into sorbent	Mixing	NA	Pass	<b>1.416</b>
Nochar	Composite	1:2	Surrogate into sorbent	Mixing	NA	Pass	0.039
Nochar	Composite	1:2.5	Surrogate into sorbent	Mixing	NA	Pass	0.014
Nochar	PUREX	1:2	Sorbent into surrogate	No Mixing	Never Sorbed	Fail	NA
Nochar	PUREX	1:2.5	Sorbent into surrogate	No Mixing	Never Sorbed	Fail	NA
Nochar	Cleanex	1:2	Sorbent into surrogate	No Mixing	3 hr	Pass	<b>1.054</b>
Nochar	Cleanex	1:2.5	Sorbent into surrogate	No Mixing	2 hr	Pass	<b>1.477</b>
Nochar	Pubex	1:2	Sorbent into surrogate	No Mixing	Never Sorbed	Fail	NA
Nochar	Pubex	1:2.5	Sorbent into surrogate	No Mixing	Never Sorbed	Fail	NA
Nochar	Composite	1:2	Sorbent into surrogate	No Mixing	Never Sorbed	Fail	NA
Nochar	Composite	1:2.5	Sorbent into surrogate	No Mixing	Never Sorbed	Fail	NA

NA Not applicable sample failed the PFT so no LRT was performed.



Fig. 1. Mixed and unmixed Nochar and PUREX surrogate samples.

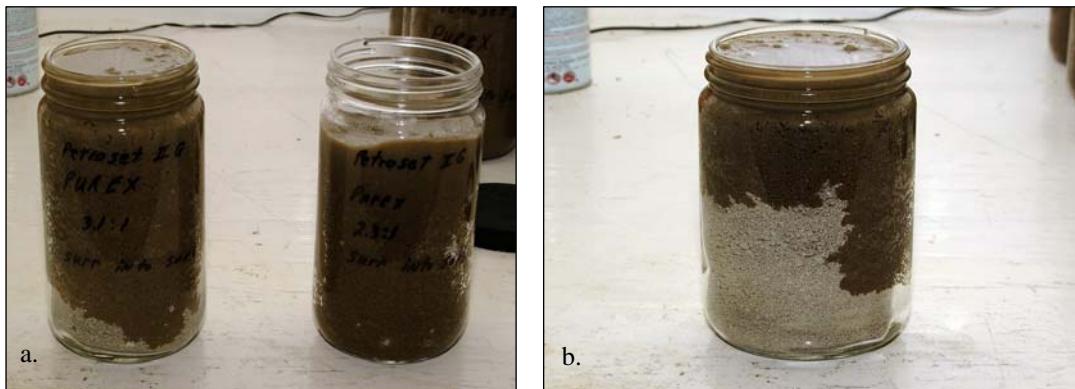


Fig. 2. a) Petroset II-G and b) PUREX samples when the liquid surrogate was added to the solid sorbent material using the alternative addition method.



Fig. 3. Organoclay samples generated using different addition methods at the 2.6:1 ratio.

The sorption times varied from 5 minutes (min) to 4 hr for the Petroset II-G samples using the favored addition method and from 15 min to 2-1/2 hr for the Organoclay samples using the favored addition method (Table V). For the samples that were generated using the alternative addition method of pouring the liquid surrogate waste into the sorbent material, the Petroset II-G sorption times ranged from 5 min to 36 hr and from 12 to 36 hr for the Organoclay samples. All of the Petroset II-G and Organoclay samples eventually sorbed the organic surrogates regardless of addition methods. Sorption time is not applicable for the Nochar samples that were mixed; however, the two Cleanex surrogate samples were sorbed without mixing, resulting in sorption times of 2 and 3 hr. The remainder of the Nochar samples that were not mixed did not sorb any of the other organic wastestreams. In fact, the Nochar and surrogate waste formed a translucent rubbery interface layer between the solid and liquid phases before complete sorption could take place as seen in Fig. 1.

All of the bench-scale Petroset II-G samples passed the PFT and LRT. All but one of the bench-scale Organoclay samples passed LRT; however, that sample was generated using the alternate addition method. All of the mixed Nochar samples passed PFT; however, the Cleanex and Pubex samples generated at the 1:2.5 ratios failed the LRT criteria. With the exception of the two Cleanex samples, all of the Nochar samples that were not mixed failed the PFT due to the presence of free liquid remaining in the samples after the 14-day curing period. The two Cleanex Nochar samples that were not mixed failed the LRT, proving that mixing is necessary for the Nochar and organic surrogates used during this test sequence. Any samples that failed the PFT due to free liquids were not tested in the LRT device. All of the bench-scale samples that passed PFT were tested in the LRT, and all but five of these samples passed the WIPP WAC of less than 1% liquid release by volume.

Based on the results from the bench-scale testing, the clay sorbents, Organoclay, and Petroset II-G used the favored method of adding the dry solid sorbent materials into the liquid surrogate wastestreams when generating the 19-L (5-gal) scale-up samples. The polymer sorbent, Nochar, was mixed by adding the liquid into the dry sorbent with vigorous mixing.

### **19-Liter Scale Organic Surrogate Sorbent Testing**

For the 19-L (5-gal) bucket tests, sorbents were evaluated at the waste-loading ratios and addition and mixing methods determined during the bench-scale testing with the organic surrogates. The larger scale tests were used to verify waste-loading ratios, volumetric expansion, sorption rate, final wasteform physical characteristics, liquid release characteristics, and curing behavior.

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

The surrogate liquid/sorbent combinations for the sorbent property/mixing verification tests were prepared using approximately 8 L (2 gal) of each organic surrogate and adding an appropriate amount of sorbent to achieve the optimum waste-loading ratios determined during the bench-scale testing.

Table VI provides the test matrix of the applicable tests for each sorbent surrogate combination and details the test parameters and data collected. The bold numbers in the LRT column of Table VI show the samples that failed the WIPP LRT WAC of less than 1% release by volume. The clay samples were generated at two waste-loading ratios: 1) the optimum ratio that results in a layer of dry sorbent material on top of the sample and 2) the 2:1 ratio that results in a soft gummy layer on top of the sample. Both waste-loading ratios were tested to determine if the soft gummy layer that results from excess liquid sorbing into the clay sorbent materials could pass the LRT criteria when scaling up to the 19-L (5-gal)

size since some of the samples generated at the 2:1 ratio during the bench-scale testing passed the WIPP LRT criteria. Figures 4 and 5 show the two Petroset II-G samples generated with the composite surrogate. Figure 4 shows the sample that was made using the 2:1 waste-loading ratio that generates a soft gummy layer on top of the sample where the excess surrogate is sorbed into the top section of the sample. Figure 5 shows the sample made using the 2.6:1 waste-loading ratio that generates a sample with excess sorbent on top.

Table VI. 19-L (5-Gal) Scale Test Matrix and Results for the Organic Surrogate Sorbent Combinations

Sorbent Name	Surrogate Name	Weight-Based, Waste-Loading Ratio (sorbent to surrogate)	Sorbent & Surrogate Addition and Mixing Method	Sorption Times	Percent Volume Increase	Sample Location Within Bucket	PFT (Pass / Fail)	LRT (Percent Liquid Release by Volume)
Petroset II-G	PUREX	2:1	Sorbent into surrogate - no mixing	156 hr	100%	Top	Pass	<b>1.030</b>
						¾ Down	Pass	0.035
Petroset II-G	PUREX	3.5:1	Sorbent into surrogate - no mixing	2 hr	260%	Top	Pass	0.013
						¾ Down	Pass	0.003
Petroset II-G	Cleanex	2:1	Sorbent into surrogate - no mixing	6 hr	110%	Top	Pass	0.600
						¾ Down	Pass	0.457
Petroset II-G	Cleanex	4.3:1	Sorbent into surrogate - no mixing	2 hr	300%	Top	Pass	0.011
						¾ Down	Pass	0.005
Petroset II-G	Pubex	2:1	Sorbent into surrogate - no mixing	60 hr	113%	Top	Pass	<b>1.031</b>
						¾ Down	Pass	0.012
Petroset II-G	Pubex	3.4:1	Sorbent into surrogate - no mixing	2 hr	300%	Top	Pass	0.008
						¾ Down	Pass	0.060
Petroset II-G	Composite	2:1	Sorbent into surrogate - no mixing	45 hr	125%	Top	Pass	<b>1.271</b>
						¾ Down	Pass	0.022
Petroset II-G	Composite	4.1:1	Sorbent into surrogate - no mixing	2 hr	300%	Top	Pass	0.009
						¾ Down	Pass	0.011
Organoclay	PUREX	2:1	Sorbent into surrogate - no mixing	35 hr	125%	Top	Pass	0.352
						¾ Down	Pass	0.010
Organoclay	PUREX	2.6:1	Sorbent into surrogate - no mixing	2 hr	181%	Top	Pass	0.005
						¾ Down	Pass	0.006
Organoclay	Cleanex	2:1	Sorbent into surrogate - no mixing	60 hr	130%	Top	Pass	<b>1.237</b>
						¾ Down	Pass	0.281
Organoclay	Cleanex	2.8:1	Sorbent into surrogate - no mixing	2 hr	140%	Top	Pass	0.004
						¾ Down	Pass	0.003
Organoclay	Pubex	2:1	Sorbent into surrogate - no mixing	2.5 hr	138%	Top	Pass	0.242
						¾ Down	Pass	0.123
Organoclay	Composite	2:1	Sorbent into surrogate - no mixing	40 hr	125%	Top	Pass	0.193
						¾ Down	Pass	0.043
Organoclay	Composite	2.3:1	Sorbent into surrogate - no mixing	2 hr	150%	Top	Pass	0.018
						¾ Down	Pass	0.013
Nochar	PUREX	1:2	Surrogate into sorbent - with mixing	NA	94%	Middle	Pass	0.043
Nochar	Cleanex	1:2	Surrogate into sorbent - with mixing	NA	178%	Middle	Pass	<b>1.707</b>
Nochar	Pubex	1:2	Surrogate into sorbent - with mixing	NA	94%	Middle	Pass	0.029
Nochar	Composite	1:2	Surrogate into sorbent - with mixing	NA	94%	Middle	Pass	0.060
Nochar	Composite	1:2.5	Surrogate into sorbent - with mixing	NA	53%	Middle	Pass	0.466

NA Not applicable; samples were mixed for 3 min



**Fig. 4.** Composite surrogate and Petroset II-G sample at the 2:1 waste-loading ratio after the 14-day curing period.



**Fig. 5.** Composite surrogate and Petroset II-G sample at the 2.6:1 waste-loading ratio after the 14-day curing period.

One sample was taken from the middle for each of the Nochar samples. Two samples were taken from each of the 19-L (5-gal) Organoclay and Petroset II-G samples: one from the top of the sample and one from approximately three-quarters down from the top of the sample. The Petroset II-G samples generated using the 2:1 waste-loading ratio failed the LRT criteria for the PUREX, Pubex, and composite wasteforms for the top gummy layer of the samples. All of the other Petroset II-G samples collected from the 19-L (5-gal) samples passed the PFT and LRT criteria. The Organoclay sample generated with the Cleanex surrogate also failed the LRT criteria for the top gummy layer; however, the remainder of the samples taken from the 19-L (5-gal) Organoclay samples passed the PFT and LRT criteria. All of the Nochar samples passed the PFT and LRT, except the Nochar Cleanex sample that failed the LRT criteria.

The sorption rates for the Organoclay and Petroset II-G samples generated at the 2:1 waste-loading ratio that resulted in a soft top layer had a wide range of sorption times from 2-1/2 to 60 hr and 2 to 156 hr, respectively. All of the clay samples generated with the optimum waste-loading ratios (resulting in a dry layer of sorbent on top of the samples) had sorption times ranging from 2 to 2-1/2 hr. Since the Nochar samples were mixed, a sorption time was not an applicable criterion. However, the samples were mixed for 3 min, which produced a uniform wasteform.

The volumetric expansion values ranged from 100% to 300% for the Petroset II-G samples, from 125% to 181% for the Organoclay samples, and from 94% to 178% for the Nochar samples, depending on the waste-loading ratios. The Petroset II-G and Organoclay sorbents produced samples with the consistency of a thick paste with the exception of the soft gummy layer on top of the 2:1 ratio samples. The Nochar sorbent produced samples that had a rubbery consistency.

## 208-Liter Scale Organic Surrogate Sorbent Testing

The purpose of the 208-L (55-gal) scale-up tests was to verify the sorbent-to-waste loadings and the addition methods and mixing schemes determined in the 19-L (5-gal) bucket testing sequence. Sample generation and testing was performed in the large-scale laboratory hood. The surrogate liquid/sorbent combinations for the sorbent property/mixing verification tests were prepared using 57 L (15 gal) of organic composite surrogate waste for the Organoclay and Petroset II-G sorbents and 95 L (25 gal) of the organic composite surrogate waste for the Nochar sample. Based on the 19-L (5-gal) tests, a 1:2.5 weight-based, waste-loading ratio was used for the Nochar sample, and 3.8:1 and 2.3:1 ratios were used for the Petroset II-G and Organoclay samples, respectively. Observations made during preparation of the 208-L (55-gal) surrogate/sorbent combinations included time for the sorbent to soak up the liquid, behavior of combinations at a larger scale, ease of mixing or sample generation, and wasteform physical characteristics. Data collected during the 208-L (55-gal) drum tests were similar to the 19-L (5-gal)-scale tests including volumetric expansion, mixing and addition requirements, sorption rate, final wasteform physical characteristics, liquid release measurements, and curing behavior.

The scale-up 208-L (55-gal) ratios that correspond to a small amount of dry sorbent on top of the sample were different than the optimum ratios determined during the 19-L (5-gal) testing for the clay sorbents. The actual ratios used for the 208-L (55-gal) sample generation were 3.44:1 for the Petroset II-G sample and 2.53:1 for the Organoclay sample. The Nochar sample was generated using the 1:2.5 waste-loading ratio that was determined during the 19-L (5-gal) testing for the composite organic wastestream.

A 20-horsepower (hp) mixer was used to mix the Nochar sample at the 208-L (55-gal) drum scale. The Nochar sorbent was placed into the 208-L (55-gal) drum, and mixing was initiated before the introduction of the surrogate liquid waste as shown in Fig. 6. (The sorbent clumps together; consequently, it is important to mix the sorbent until it is uniform prior to introducing the liquid phase.) The liquid surrogate waste was then poured into the 208-L (55-gal) drum as quickly as possible while the mixture was mixed vigorously as shown in Figs. 7 and 8. After the total volume of the surrogate composite organic waste was introduced into the drum, there was a 6-second (s) mixing window before the Nochar and surrogate liquid formed a very thick mixture and could no longer be mixed efficiently. Figure 9 shows the sample during the 6-s mixing window as indicated by the slight vortex in the mixing drum, and Fig. 10 shows the sample after mixing is no longer effective. The mixture did not trip the 20-hp mixer after it thickened as it did when using a 5-hp drum mixer during previous testing for SRS using Nochar and surrogate PUREX wastestreams; however, a 6-s mixing window is not an adequate mixing window for field applications. Figure 11 shows sample consistency after the mixing sequence for the Nochar and the composite surrogate at the 208-L (55-gal) drum scale.

The two granular clay sorbent products require less labor and would be much easier to use in field applications since both the Petroset II-G and Organoclay sorbent materials were simply poured into the liquid composite organic surrogate and allowed to imbibe the liquid, which took approximately 1-1/2 hr. Figures 12 and 13 show the addition sequence for the Organoclay and the composite organic surrogate. Figures 14 and 15 show the addition process for Petroset II-G and the composite organic surrogate.

*Nochar Mixing Sequence*



**Fig. 6.** Nochar sorbent being mixed prior to surrogate introduction.



**Fig. 7.** Addition of composite surrogate to the Nochar sorbent while mixing.



**Fig. 8.** Addition of composite surrogate to the Nochar sorbent while mixing.



**Fig. 9.** Composite surrogate and Nochar sorbent during the 6-s mixing window.



**Fig. 10.** Composite surrogate and Nochar sorbent after 6-s mixing window at the point where mixing stops being effective.



**Fig. 11.** Composite surrogate and Nochar sorbent sample consistency immediately after mixing.

*Organoclay Addition Sequence*



Fig. 12. Addition of Organoclay into the composite surrogate wastestream.



Fig. 13. Sample after Organoclay addition.

*Petroset II-G Addition Sequence*



Fig. 14. Addition of Petroset II-G into the composite surrogate wastestream.



**Fig. 15. Sample after Petroset II-G addition.**

The samples were allowed to cure for 14 days before performing the PFT and LRT from three distinct sampling zones within each 208-L (55-gal) drum sample. Both of the clay samples had the consistency of a hard paste, and the Nochar sample had a hard, rubbery consistency. The samples collected from the top section of the 208-L (55-gal) drum samples were located approximately 6 inches below the top surface of the sample, the samples obtained from the middle sections of the large samples were located at mid-depth of the individual sample height, and the samples obtained from the bottom of the 208-L (55-gal) drum samples were located approximately 2 inches from the bottom of the sample drum. The 208-L (55-gal) drum test matrix and results are presented in Table VII.

Table VII. 208-L (55-Gal) Scale Test Matrix and Results for the Organic Surrogate Sorbent Combinations

Sorbent Name	Surrogate Name	Weight-Based, Waste-Loading Ratio (Sorbent to Surrogate)	Sorbent & Surrogate Addition and Mixing Method	Sorption Times/ Mixing Times	Percent Volume Increase	Sample Location Within Bucket	PFT (Pass/ Fail)	LRT, Percent Liquid Release by Volume (WIPP WAC 1% Release)
Petroset II-G	Composite	3.44:1	Sorbent into surrogate with no mixing	90 min	306	Top Section	Pass	0.005
						Middle Section	Pass	0.004
						Bottom Section	Pass	0.009
Organoclay	Composite	2.53:1	Sorbent into surrogate with no mixing	90 min	253	Top Section	Pass	0.020
						Middle Section	Pass	0.006
						Bottom Section	Pass	0.008
Nochar	Composite	1:2.5	Surrogate into sorbent with extreme mixing	6-s mixing window	156	Top Section	Pass	0.143
						Middle Section	Pass	0.136
						Bottom Section	Pass	0.149

As can be seen from the results presented in Table VII, all of the samples collected from each of the large-scale 208-L (55-gal) drum samples passed the PFT and WIPP LRT requirements of 1% release by volume, proving that each of the sorbent materials tested could solidify the composite organic surrogate during this scale-up testing sequence.

### **Additional Liquid Release Testing**

Approximately 1 year after the 19-L (5-gal) buckets were generated, LRT testing was performed on the samples. The 208-L (55-gal) drum samples were also tested for liquid release after physically destroying the samples to check for sample consistency and void spaces within the large-scale samples.

### **19-Liter Bucket Testing**

Bucket samples that were generated in September and early October of 2006 [19-L (5-gal)] have been stored at the MSE Test Facility and were LRT tested during September 2007 to determine if the samples could still tie up the organic liquids and to check for any sample breakdown. When the buckets were retrieved for sampling, it was observed the some buckets had degraded and showed cracks and/or breaks in the lids. It was noted which sample buckets had been compromised by the sorbent and surrogate combinations when sample inspection and LRT testing was performed.

Table VIII lists the original sample dates and original LRT data and the second sampling date with the corresponding LRT data gathered approximately 1 year after sample generation. All samples were tested for liquid release even though some of the sample lids had allowed air to the samples, which would result in drier samples in Montana's dry environment. The WIPP LRT WAC of less than 1% liquid release by volume was used as the LRT criteria. The sample names followed by an asterisk were the samples that had cracks in the bucket lids, and the sample names followed by two asterisks had bucket lids that were broken with pieces that hung away from the lid. Figures 16 and 17 are pictures of the compromised bucket lids. The compromised samples were discarded after the second set of LRT testing. The samples that were not compromised will be retained at the MSE Test Facility but have been transferred to metal buckets.



**Fig. 16. Cracked bucket lid.**



**Fig. 17. Broken bucket lid.**

Table VIII. 19-L (5-Gal) Scale LRT Results for the Organic Surrogate Sorbent Combinations

Sorbent Name	Surrogate Name	Weight-Based, Waste-Loading Ratio (sorbent to surrogate)	Original Sampling Date	Original Location within the Bucket	Original LRT Data (Percent Liquid Release by Volume)	Second Sampling Date	Second Location within the Bucket	Second LRT Data (Percent Liquid Release by Volume)
Petroset II - G	PUREX	2:1	10-3-06	Top	<b>1.030</b>	9-18-07	Several Locations	0.526
				½ Down	0.035			
Petroset II -G	PUREX	3.5:1	10-3-06	Top	0.013	9-18-07	Several Locations	0.006
				½ Down	0.003			
Petroset II -G	Cleanex	2:1	10-3-06	Top	0.600	9-18-07	Several Locations	0.531
				½ Down	0.457			
Petroset II -G*	Cleanex	4.3:1	10-3-06	Top	0.011	9-18-07	Several Locations	0.001
				½ Down	0.005			
Petroset II -G	Pubex	2:1	10-3-06	Top	<b>1.031</b>	9-18-07	Several Locations	0.290
				½ Down	0.012			
Petroset II -G	Pubex	3.4:1	10-3-06	Top	0.008	9-18-07	Several Locations	0.005
				½ Down	0.060			
Petroset II -G	Composite	2:1	10-3-06	Top	<b>1.271</b>	9-18-07	Several Locations	0.261
				½ Down	0.022			
Petroset II - G *	Composite	4:1	10-3-06	Top	0.009	9-18-07	Several Locations	0.007
				½ Down	0.011			
Organoclay *	PUREX	2:1	9-27-06	Top	0.352	9-12-07	Several Locations	0.219
				½ Down	0.010			
Organoclay	PUREX	2.6:1	9-27-06	Top	0.005	9-12-07	Several Locations	0.008
				½ Down	0.006			
Organoclay *	Cleanex	2:1	9-27-06	Top	<b>1.237</b>	9-12-07	Several Locations	0.382
				½ Down	0.281			
Organoclay *	Cleanex	2.8:1	9-27-06	Top	0.004	9-12-07	Several Locations	0.002
				½ Down	0.003			
Organoclay *	Pubex	2:1	9-27-06	Top	0.242	9-17-07	Several Locations	0.087
				½ Down	0.123			
Organoclay **	Composite	2:1	9-27-06	Top	0.193	9-17-07	Several Locations	0.025
				½ Down	0.043			
Organoclay	Composite	2.3:1	9-27-06	Top	0.018	9-17-07	Several Locations	0.111
				½ Down	0.013			
Nochar	PUREX	1:2	9-27-06	Middle	0.043	9-17-07	Several Locations	0.048
Nochar**	Cleanex	1:2	9-27-06	Middle	<b>1.707</b>	9-17-07	Several Locations	0.580
Nochar *	Pubex	1:2	9-27-06	Middle	0.029	9-17-07	Several Locations	0.023
Nochar	Composite	1:2	9-27-06	Middle	0.060	9-17-07	Several Locations	0.026
Nochar *	Composite	1:2.5	9-27-06	Middle	0.466	9-17-07	Several Locations	0.389

\* Bucket lid cracked

\*\* Bucket lid broken

There was not enough sample volume from the top gummy layer of the clay samples to gather a second full sample for LRT; therefore, samples were collected from different locations within the bucket and included some of the top gummy layer. Observing the LRT values in Table VIII, the clay samples at the lower ratios that resulted in a top gummy layer have LRT values for the second data set that run between the values taken for the top layer and halfway down the sample bucket for the samples with uncompromised bucket lids. The clay samples at the 2:1 ratios with cracked lids also had second LRT values that were bracketed by the two original LRT values; however, the Organoclay and composite sample with a broken lid had LRT values below the middle section LRT number. The clay samples that were generated using the higher ratios showed little change between LRT data for each of the sampling events; the LRT values were either comparable or slightly lower for the samples that had compromised lids. The Nochar samples show little change with the exception of the Cleanex and the composite Nochar samples. The Cleanex lid was broken, exposing the sample to the ambient laboratory conditions, while the composite sample lid at the 1:2.5 ratio was only cracked; consequently, there was much less exposure. Looking at the second set of LRT data for those samples, it seems reasonable that their LRT values decreased over time while the others remained somewhat constant. Even though the lids were compromised for some of the samples, the LRT data sets still correlate fairly well when compared to historical LRT data for volatile organic surrogates (Refs. 5, 6).

### **208-Liter Drum Testing**

Almost 9 months after the 208-L (55-gal) drum samples were generated, they were physically destroyed to check for liquid release characteristics, void spaces, and wasteform physical consistency. Samples were collected from the top and bottom sections of the large-scale samples after determining that no voids were present in the samples and checking sample consistency. Figures 18, 19, and 20 show the 208-L (55-gal) drum samples after cutting away the front section of the drums. The sample consistency for both of the clay sorbents continued to be a hard paste with the Petroset II-G sample being the harder of the two. The sample consistency for the Nochar sample was very rubbery (note that not all of the sorbent was incorporated into the liquid organic surrogate waste). The Nochar sample was cut open at the part of the drum where the mixing was not adequate enough to incorporate the sorbent into the surrogate waste, which resulted in a section approximately 3 to 5 inches thick where the sorbent was not incorporated fully. Approximately half of the outside surface of the sample and part of the bottom had an unmixed sorbent layer, while the other half of the drum and bottom surface did not. Figure 21 shows the Nochar sample after cutting sections out of the sample while checking for voids. The second set of LRT data for all three 208-L (55-gal) drum samples show how consistent the LRT data is for the first and second data sets, which demonstrates the ability of all three sorbents tested to tie up the liquid organic waste over the short term. The 208-L (55-gal) drum LRT data sets are presented in Table IX.



**Fig. 18. Organoclay sample.**



**Fig. 19. Petroset II-G sample.**



**Fig. 20. Nochar sample.**



**Fig. 21. Nochar sample during physical destruction of the sample.**

Table IX. 208-L (55-Gal)-Scale Results for the Composite Organic Surrogate and Sorbent Combinations

Sorbent Name	Surrogate Name	Weight-Based, Waste-Loading Ratio (Sorbent to Surrogate)	Original Sample Date	Sample Location Within Bucket	LRT Percent Liquid Release by Volume (WIPP WAC – 1% Release)	Second Sample Date	Sample Location Within Bucket	LRT, Percent Liquid Release by Volume (WIPP WAC 1% Release)
Petroset II-G	Composite	3.44:1	12-27-06	Top	0.005	9-12-07	Top	0.004
				Middle	0.004		Middle	NA
				Bottom	0.009		Bottom	0.005
Organoclay	Composite	2.53:1	12-27-06	Top	0.020	9-12-07	Top	0.003
				Middle	0.006		Middle	NA
				Bottom	0.008		Bottom	0.011
Nochar	Composite	1:2.5	12-27-06	Top	0.143	9-12-07	Top	0.142
				Middle	0.136		Middle	NA
				Bottom	0.149		Bottom	0.142
NA - not applicable since samples were not taken from the middle section								

### Organic Surrogate Sorbent Cost Projections

Cost projections were determined based on the solidification of 379 L (100 gal) of the composite organic surrogate at the optimum waste-loading ratios and deployment strategies determined during the 208-L (55-gal) drum tests.

The projected costs presented in Table X are the costs associated with only purchasing the sorbent materials to solidify 379 L (100 gal) of the composite organic surrogate. No labor costs have been considered for this cost comparison; however, the labor costs to generate wasteforms using Nochar would be considerably more expensive than to generate wasteforms using the Petroset II-G and Organoclay granular clay sorbents. The Nochar sorbent also has a very limited mixing timeframe when being mixed with volatile organic liquids, which would make field applications difficult. No mixing equipment would be necessary for generating the clay wasteforms since they are designed to eliminate the mixing process, which would reduce the solidification costs and worker exposure.

Table X. Summary of Sorbent Cost Evaluations for the Optimum Waste-Loading Ratios at the 208-L (55-Gal) Scale

Sorbent Name	Surrogate Name	Optimum Weight-Based, Waste-Loading Ratio (Sorbent to Surrogate)	Optimum Deployment Strategy	Sorbent Cost (\$/379 L (100 Gal) of Composite Organic Surrogate)
Petroset II-G*	Composite	3.44:1	Sorbent into surrogate with no mixing	\$7,412.54
Organoclay*	Composite	2.53:1	Sorbent into surrogate with no mixing	\$3,361.86
Nochar**	Composite	1:2.5	Surrogate into sorbent with extreme mixing	\$2,370.29

\* No mixing required  
\*\* Mixing costs not included

## CONCLUSIONS

The sorbent testing sequence proved that Petroset II-G, Organoclay, and Nochar have the ability to solidify the PUREX, Pubex, Cleanex, and the composite organic wastestream, which is comprised of the three specific wastestreams. The sorbents were tested at bench-, 19-L (5-gal), and 208-L (55-gal) scale, and optimized weight-based, waste-loading ratios were determined for each of the sorbents with the composite organic waste at the 208-L (55-gal) scale-up level. The sorbents were able to pass the PFT and LRT at the optimized waste-loading ratios for the 208-L (55-gal) drum scale-up samples.

The two clay sorbents, Petroset II-G and Organoclay, proved to be very easy to work with since the sorbents were designed to solidify liquid waste without the need for mixing. The Petroset II-G sorbent testing resulted in an optimized waste-loading ratio of 3.44:1 (sorbent to surrogate), and the optimized ratio for the Organoclay sorbent was 2.53:1. After the two samples were physically inspected for voids and sample consistency, it was determined that no voids existed within the samples and that their consistency was still a hard paste, with the Petroset II-G sample being the harder of the two.

The Nochar sorbent material was much more difficult to work with when generating the large-scale samples. A 20-hp drum mixer was necessary to get a mix with the Nochar and the composite organic surrogate waste at the 208-L (55-gal) scale; however, there was only a 6-s mixing window after the surrogate was added to the sorbent. The Nochar sorbent testing resulted in an optimized waste-loading ratio of 1:2.5 sorbent to surrogate, which would be difficult to achieve in applications outside the laboratory setting. However, after cutting the 208-L (55-gal) sample, it was determined that not all of the sorbent material had been incorporated into the surrogate waste even with extreme mixing.

## RECOMMENDATIONS

MSE recommends that Organoclay or Petroset II-G be used to solidify the composite organic waste generated from the three specific wastestreams at the REDC at ORNL. Based on the weight-based, waste-loading ratios, less of the Organoclay would be necessary to solidify the same volume of liquid organic waste, resulting in a lower cost and a smaller volume of solidified waste generated for disposal.

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