Strategies for Treating and Dewatering Contaminated Soils and Sediments Simultaneously - 13389

Jody Bickford and Martin Foote, Ph.D.
MSE Technology Applications, Inc.
200 Technology Way, Butte, MT 59701
jody.bickford@mse-ta.com

ABSTRACT

MSE Technology Applications, Inc. (MSE) was asked to perform a series of treatability studies by Global Technologies, Inc. (Global) and M^2 Polymer Technologies, Inc. (M^2 Polymer) using Global's metal treatment agent, Molecular Bonding System (MBS) and M^2 Polymer's superabsorbent polymer, Waste Lock 770 (WL-770). The primary objective of the study was to determine if the two products could be used as a one-step treatment process to reduce the leachability of metals and dewater soils and/or sediments simultaneously.

Three phases of work were performed during the treatability study. The first phase consisted of generating four bench-scale samples: two treated using only MBS and two treated using only WL-770, each at variable concentrations. The second phase consisted of generating nine bench-scale samples that were treated using MBS and WL-770 in combination with three different addition techniques. The third phase consisted of generating four intermediate-scale samples that were treated using MBS and WL-770 simultaneously.

The soils used in the treatability study were collected at the Mike Mansfield Advanced Technology Center in Butte, Montana. The collected soils were screened at 4 mesh (4.75 millimeters (mm)) to remove the coarse fraction of the soil and spiked with metallic contaminants of lead, cadmium, nickel, mercury, uranium, chromium, and zinc.

INTRODUCTION

MSE conducted the treatability testing on a contaminated (spiked) soil sample using the metal treatment agent MBS and the superabsorbent polymer WL-770, both separately and in combination to treat and dewater soil samples simultaneously.

MBS has been tested by the U.S. Environmental Protection Agency (EPA) for a Superfund Innovative Technology Evaluation (SITE) demonstration at the Midvale Slag Superfund Site in Midvale, Utah (Ref. 1). Some 500 tons of three waste streams contaminated with arsenic, cadmium, and lead were treated with MBS. The treated soils passed the EPA's Multiple Extraction Procedure (MEP) test (Ref. 2). The MEP was designed to simulate initial and subsequent leaching of a waste placed into an improperly designed landfill, where the waste would be exposed to prolonged exposure of acidic precipitation.

In addition to the SITE demonstration, MBS has undergone extensive bench and pilot-scale testing and has been used in several full-scale remediation projects. In each of these projects, MBS treatment has been able to reduce the leachability of hazardous contaminants from sediments and soils to levels below the regulatory limits for the Toxicity Characteristic Leaching procedure (TCLP) (Ref. 3). The TCLP was developed to simulate the mobility of contaminants from solids and multiphase wastes placed into a landfill. MBS was previously used at the Brookhaven National Laboratory (BNL) for treatment of Brookhaven soils spiked with cadmium and lead (Ref. 4). The analyses of TCLP leachates from the treated soils were well below both the TCLP standards and the Universal Treatment Standards. MSE also performed treatability tests using MBS for mercury contaminated soil at the Y-12 Plant Building 81-10 area with success. (Ref. 5 & 6)

The superabsorbent WL-770 has also undergone extensive bench-scale and pilot-scale testing by MSE for the U. S. Department of Energy (DOE) and others and has been used in several full-scale remediation projects. Included in these bench-scale and pilot-scale projects was the treatment of dredge material from the West Branch of the Grand Calumet River in Hammond, Indiana (Ref. 7). During this project the superabsorbent was able to dewater the river sediments to achieve compliance with the Paint Filter Test (PFT) (Ref. 8) using less than 1% WL-770 by weight. Also, MSE tested WL-770 as an additive to a grout formulation for the solidification of Melton Valley Storage Tank Waste located at the Oak Ridge National Laboratory (Ref. 9). The superabsorbent WL-770 was able to eliminate bleed water from all the grouted wastes tested during the project. Lastly, WL-770 was used by the Savannah River Site at full-scale to solidify radioactive rainwater waste to produce a wasteform that passed both the PFT and the more stringent Liquid Release Test (Ref. 10) for disposal onsite at SRS.

TEST OBJECTIVES

The project described in this document was conducted to determine if the two products, MBS and WL-770, could be used as a one-step treatment process to reduce the leachability of metals and dewater soils and/or sediments simultaneously. Specifically, the project was conducted to determine the interactive effects of the two materials on the performance for the simultaneous application of the materials.

MATERIAL DESCRIPTIONS AND SOIL PREPARATION

The metal treatment agent MBS is a light grey, finely powdered solid and is used to treat soils, slag, sediments, paint chips, and flue gas in scrubber and bag house applications. A sample of MBS is shown in Figure 1.

The superabsorbent polymer WL-770 is a white solid comprised of particles ranging from less than 1 mm in diameter to approximately 3 mm in diameter. A sample of WL-770 is shown in Figure 2. WL-770 is routinely used to sorb aqueous liquid wastes and has an affinity for some of the heavy metals.

Figure 1. MBS Sample.

The contaminated soil used in the treatability testing was prepared by collecting soil from a site at the Mike Mansfield Advanced Technology Center in Butte, Montana. The collection site is shown in Figure 3. The soils were screened at 4 mesh (4.75 mm) to remove the coarse fraction of the soil. A sample of the screened soil is shown in Figure 4. The soil was then spiked with the metallic contaminants lead, cadmium, nickel, mercury, uranium, chromium, and zinc. The spiking process consisted of preparing a solution of soluble metallic salts by dissolving the quantities of salts shown in Table 1 into deionized (DI) water.

This solution was then added to and thoroughly mixed with the screened soil. The wet soils were then dried for a period of 36 hours, after which the soil was again thoroughly mixed to assist in dispersing the metals throughout the soil. A sample of the dried spiked soil is shown in Figure 5.

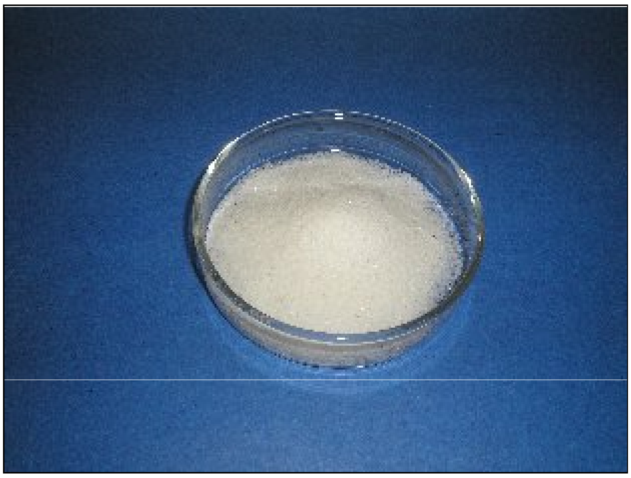

Figure 2. Waste Lock 770 Sample.

Figure 3. Soil Collection Site.

Table 1. Spiked Soil Preparation Information.

Metal	Metal Salt Added	Chemical Formula	Quantity Added, g/kg of Soil
Lead	Lead Nitrate	$PbNO_3$	3.90
Mercury	Mercury Chloride	$HgCl$	0.47
Cadmium	Cadmium Nitrate	$Cd(NO_3)_2$	1.65
Chromium	Sodium Dichromate	$Na_2Cr_2O_7 \cdot H_2O$	28.65
Zinc	Zinc Sulfate	$ZnSO_4 \cdot 7H_2O$	11.32
Nickel	Nickel Sulfate	$NiSO_4 \cdot 6H_2O$	29.60
Uranium	Uranyl Nitrate	$UO_2(NO_3)_2 \cdot 6H_2O$	0.05

Figure 4. Sample of Uncontaminated Soil.

Figure 5. Sample of the Dried Spiked Soil.

Samples of both the uncontaminated soil and the spiked soil were submitted to the MSE Analytical Laboratory for TCLP analysis. The solution developed from the TCLP test was analyzed to determine the leachable quantities of the contaminant metals in both the spiked and the uncontaminated soil samples. The results of the TCLP analyses are presented in Table 2.

As shown in the data presented in Table 2, all the metal spikes resulted in metals concentrations larger than the regulatory limit except for lead. The leachable lead value in the spiked soil was lower than the regulatory limit, and this is probably due to interactions between the metal salts used for the spiking process. The large quantity of sulfate added to the soil from the zinc and nickel salts could react with lead to form the fairly insoluble solid compound of lead sulfate. Due to the lack of soluble lead in the spiked soil, a second, smaller batch of lead-spiked soil was prepared. The second lead-contaminated soil sample was prepared by collecting soil from the same site as the previous larger sample. This soil sample was also screened at 4 mesh (4.75 millimeters) to remove the coarse fraction of the soil. The soil was then spiked with lead nitrate at the same level as shown in Table 1 using the same process as for the large sample.

Table 2. Uncontaminated and Spiked Soil Analytical Results.

Metal	Regulatory Limit, mg/L	Analytical Results Uncontaminated Soil, mg/L	Analytical Results Spiked Soil, mg/L
Lead	5	0.0907	0.035
Mercury	0.2	0.00005	0.479
Cadmium	1	0.0132	22.5
Chromium	5	0.0378	102
Zinc	4.3	0.614	129
Nickel	11	0.0275	37.9
Uranium	0.044	0.00506	0.447
Lead *	5	0.0907	87.4
* Second soil sample spiked with only lead			

A sample of the lead-spiked soil was submitted to the MSE Analytical Laboratory for TCLP analysis. The solution developed from the TCLP was analyzed to determine the leachable quantity of lead in the spiked soil. The result of that analysis is shown at the bottom of Table 2.

PHASE I – BENCH-SCALE TESTING USING INDIVIDUAL TREATMENTS

The first phase of the treatability testing consisted of treating four soil samples. Two of the samples were treated with only WL-770 at 2.5 and 3 weight percent (wt %) and two samples were treated with only MBS at 5 and 10 wt %. Initially, DI water was added to the soil matrix and thoroughly mixed to increase the soil moisture to the appropriate percentage: 30 percent moisture more than saturated the soils; 40 percent moisture caused the samples to be exceedingly wet. Subsequent to the moisture addition, the appropriate treatment agent was introduced to the soil and moderately mixed to constitute treatment. Table 3 contains information related to the water and treatment agent quantities for each sample. Samples of the treated soils are shown in Figure 6 through Figure 9. Both of the WL-770 samples passed the PFT for liquid release after the superabsorbent polymer was added to the samples.

Table 3. Phase 1 – Individual Treatment Sample Description.

Sample Number	DI Water, Weight Percent	WL-770, Weight Percent	MBS, Weight Percent
1	30	2.5	---
2	30	---	5
3	40	3	---
4	40	---	10

Figure 6. Soil With 30% Water and 2.5% WL-770.

Figure 7. Soil With 40% Water and 3% WL-770.

Figure 8. Soil With 30% Water and 5% MBS.

Figure 9. Soil With 40% Water and 10% MBS.

The four treated samples were submitted to the MSE Analytical Laboratory for TCLP. The solution developed from the TCLP was analyzed to determine the leachable quantities of the contaminant metals in the treated soils. The TCLP results for the initial set of samples are shown in Table 4.

Table 4. Phase I- Bench-Scale Individual Treatment Analytical Results.

Metal	Regulatory Limit, mg/L	Treated with 2.5 wt % WL-770, mg/L	Treated with 3 wt % WL-770, mg/L	Treated with 5 wt % MBS, mg/L	Treated with 10 wt % MBS, mg/L	Spiked Soil Concentration, mg/L
Lead	5	0.295	0.258	0.981	0.003	0.035
Mercury	0.2	0.180	0.193	**0.213**	0.002	0.479
Cadmium	1	**1.57**	**1.50**	0.447	N.D.	22.5
Chromium	5	**70.1**	**57.5**	**6.55**	**15.1**	102
Zinc	4.3	**28.3**	**23.9**	3.99	0.067	129
Nickel	11	**22.3**	**19.3**	**15.5**	0.121	37.9
Uranium	0.044	0.012	0.018	0.030	0.002	0.447

The bolded values shown in Table 4 denote those analyses that are greater in concentration than the regulatory limit. Because several of the metal concentrations were above the regulatory limit, additional samples were treated using larger quantities of MBS during Phases II and III testing to determine a range of MBS dosing rates. Notice that WL-770 was able to tie up mercury and uranium to levels below the regulatory limits. MBS at 5 wt % treated all the metals below the regulatory limits except mercury, chromium, and zinc, and MBS at 10 wt % was able to achieve compliance for all the metals except chromium, which was spiked with 20 times the regulatory limit for that metal.

PHASE II – BENCH-SCALE TESTS USING ALTERNATIVE ADDITION METHODS

The second phase of the testing consisted of treating nine bench-scale samples of spiked soil with both treatment agents. This portion of the testing was divided into three segments. During the first segment, three samples of spiked soil were treated by adding the treatment agents to the samples using different addition methods. All three samples were generated using 30 wt % DI water, 2.5 wt % WL-770, and 5 wt % MBS. The first of these samples was treated with WL-770 initially, allowed to stand for a period of approximately three minutes, and then treated with MBS and moderately mixed. The second sample was treated in the opposite order with MBS initially followed by WL-770 and moderately mixed. The third sample was treated with a previously prepared mixture of MBS and WL-770 so that both reagents were added to the treatment sample simultaneously and moderately stirred. The three treated bench-scale samples were submitted to the MSE Analytical Laboratory for TCLP. The solution developed from the TCLP was analyzed to determine the leachable quantities of the contaminant metals in the treated soils. The analytical results for the alternative addition method treated soil samples are shown in Table 5. The bolded values in the table denote samples that failed the regulatory limits for that specific metal. Notice that only mercury, chromium and nickel failed the regulatory limit for the 30 wt % DI water, 2.5 wt % WL-770, and 5 wt % MBS samples. All of the samples also passed the PFT for liquid release.

Table 5. Phase II – Bench-Scale Alternative Addition Method Analytical Results.

Metal	Regulatory Limit, mg/L	2.5 wt % WL-770 then 5 wt % MBS Results, mg/L	5 wt % MBS - then 2.5 wt % WL-770 Results, mg/L	2.5 wt % WL-770 and 5 wt % MBS Added Simultaneously Results, mg/L	Spiked Soil Concentration, mg/L
Lead	5	1.31	0.0236	0.0340	0.035
Mercury	0.2	**0.218**	0.0021	0.0044	0.479
Cadmium	1	0.428	0.0016	0.0059	22.5
Chromium	5	**10.5**	4.58	**6.65**	102
Zinc	4.3	3.85	1.30	1.05	129
Nickel	11	**15.8**	**13.7**	**14.5**	37.9
Uranium	0.044	0.0228	0.0024	0.0031	0.447

The second segment consisted of treating three samples of the spiked soil with varying percentages of MBS, 2.5 wt % WL-770, and 30 wt % DI water. The three samples were treated with larger percentages of MBS in an attempt to reduce the leachable quantities of chromium and nickel in the soil. The first sample was treated with 10 wt % MBS, the second sample was treated with 15 wt % MBS, and the third sample was treated with 20 wt % MBS. All three samples were treated with a previously prepared mixture of MBS and WL-770 so that both reagents were added to the samples simultaneously. The three treated samples were submitted to the MSE Analytical Laboratory for TCLP. The solution developed from the TCLP was analyzed to determine the leachable quantities of the contaminant metals in the treated soils. The analytical results for the samples generated by adding MBS and WL-770 simultaneously are presented in Table 6 along with the duplicate lead sample generated because of suspected chemical spike interference with lead. Notice that the only regulatory limits that were not achieved were the chromium and nickel limits for the 5 wt % MBS sample. All of the samples passed the regulatory limits for each of the metals for the 10, 15 and 20 wt % MBS samples that were generated with 30 wt % DI waster and treated simultaneously with 2.5 wt % WL-770. All of the samples also passed the PFT for liquid release.

Chromium does not readily form a sulfide precipitate. Therefore, the treatment of the chromium is largely based a chemical reaction process where hexavalent chromium (Cr^{+6}) is reduced to trivalent chromium (Cr^{+3}), which is distinctly less soluble than Cr^{+6}. MBS is able to produce to conditions necessary to chemically reduce Cr^{+6}. The variations seen in the analytical results for chromium presented in Table 6 are probably a result of variations produced from mixing the small samples and the very large chromium spike.

Table 6. Phase II – Bench-Scale Simultaneous Addition Method Analytical Results.

Metal	Regulatory Limit, mg/L	5 wt % MBS Results, mg/L	10 wt % MBS Results, mg/L	15 wt % MBS Results, mg/L	20 wt % MBS Results, mg/L	Spiked Soil Concentration, mg/L
Lead	5	0.0236	0.00046	0.00092	0.00108	0.035
Mercury	0.2	0.0021	0.00035	0.00129	0.00169	0.479
Cadmium	1	0.0016	ND	ND	0.00108	22.5
Chromium	5	4.58	0.301	0.128	3.42	102
Zinc	4.3	1.30	0.217	0.0177	0.0162	129
Nickel	11	**13.7**	0.141	0.169	0.174	37.9
Uranium	0.044	0.0024	0.00387	0.00558	0.00042	0.447
Lead *	5	0.155	0.00709	0.0120	NT	87.4

* Second spiked sample generated for lead only
ND – non detect
NT – not tested

In the third segment, three samples of lead-spiked soil were treated with varying percentages of MBS with 2.5 wt % of WL-770 and 30 wt % DI water. The first sample was treated with 5 wt % MBS, the second sample was treated with 10 wt % MBS, and the third sample was treated with 15 wt % MBS. Previously prepared mixtures of MBS and WL-770 were added to all three samples so that both reagents were added to each sample simultaneously. The three treated samples were submitted to the MSE Analytical Laboratory for TCLP. The solution developed from the TCLP was analyzed to determine the leachable quantity of lead in the treated soils. The analytical results for the lead spiked soil samples are shown at the bottom of Table 6. All three of the lead-spiked samples treated with 2.5 wt % WL-770 and MBS ranging from 5 to 15 wt % passed the regulatory limit for lead and the PFT for liquid release.

PHASE III – INTERMEDIATE-SCALE TESTING

The third phase of the treatability testing consisted of treating four larger scale samples consisting of 12 Kg of contaminated soil each. Initially, DI water was added to the soil matrix and thoroughly mixed to increase the soil moisture to a value of 30 wt %. As previously explained, this value was chosen over 40 wt % due to the excess water in the 40 wt % samples. Subsequently, a previously prepared mixture of MBS and WL-770 was introduced to the soil of each sample and moderately mixed to constitute treatment. All four of the intermediate-scale samples were treated with 2.5 wt % WL-770, because that quantity of material was able to dewater the contaminated soil and pass PFT. The first sample was treated with 5 wt % MBS, the second sample was treated with 10 wt % MBS, the third sample was treated with 15 wt % MBS, and the fourth sample was treated with 20 wt % MBS. A photograph showing a sample of the dry, spiked soil is presented in Figure 10. A sample of the wet spiked soil is shown in Figure 11, and a sample of the wet, spiked soil being mixed with MBS and WL-770 is shown in Figure 12.

Figure 10. Dry, Spiked Soil Used for the Intermediate-Scale Treatment.

Figure 11. Wet, Spiked Soil Used for the Intermediate-Scale Testing.

Figure 12. Intermediate-Scale Soil Sample during Simultaneous Treatment.

The four, intermediate-scale treated samples were submitted to the MSE Analytical Laboratory for TCLP. The solution developed from the TCLP was analyzed to determine the leachable quantities of contaminant metals in the treated soils. The analytical results for the Phase III intermediate-scale soil samples are shown in Table 7. The bolded values in the table are those values that were above the regulatory limit for that specific metal. The 15 and 20 wt % MBS samples were able to pass the regulatory limits for all the metals tested, the 10 wt % MBS sample passed all the limits except for nickel and the 5 wt % MBS sample passed all the regulatory limits for the metals tested except for chromium and nickel. All of the samples treated during the intermediate-scale testing also passed the PFT for liquid release. This testing provides a range for MBS application when using WL-770 in combination to treat contaminated wet soils. The analytical results also trend well; with decreasing metals concentrations with increasing MBS dosing.

Table 7. Phase III - Intermediate-Scale Analytical Results.

Metal	Regulatory Limit, mg/L	5 wt % MBS Results, mg/L	10 wt % MBS Results, mg/L	15 wt % MBS Results, mg/L	20 wt % MBS Results, mg/L
Lead	5	0.0237	0.0205	0.0124	0.0078
Mercury	0.2	0.00168	0.0039	0.0026	0.0033
Cadmium	1	0.00279	0.0072	0.0033	0.0023
Chromium	5	**5.53**	3.91	2.22	1.39
Zinc	4.3	1.30	0.124	0.0931	0.0295
Nickel	11	**14.4**	**17.9**	0.164	0.124
Uranium	0.044	0.00295	0.00395	0.00087	0.00046

CONCLUSIONS

During Phase I bench-scale testing, WL-770 (without MBS) applied at 2.5 and 3 wt % was able to dry the contaminated soil samples to pass a PFT for samples with 30 and 40 wt % DI water. Also, WL-770 was able to reduce the leachability of mercury and uranium to concentrations below the regulatory limits as a stand alone product as shown in Table 4. The metals treatment agent MBS, without the addition of the dewatering agent, WL-770, was able to reduce the leachability of the contaminant metals cadmium, zinc, and uranium below the regulatory limits for those metals at a 5 wt % MBS sample and for all the metals tested except chromium in the 10 wt % MBS sample.

During the first part of Phase II testing, the combination of the WL-770 dewatering agent and the MBS treatment agent at 2.5 and 5 wt %, respectively, was able to reduce the leachability of the contaminant metals mercury, cadmium, zinc, and uranium to an order of magnitude below (and often to two orders of magnitude below) the regulatory limits in all the samples tested.

During second part of Phase II testing, it was shown that MBS and WL-770 could be added simultaneously to contaminated soil samples and still effectively treat and dewater the samples. Some of the metals were not bound sufficiently in the 5 wt % MBS sample however, all the metals tested passed their regulatory limits with 10, 15, and 20 wt % applications of MBS along with a simultaneous application of 2.5 wt % WL-770. The lead-spiked samples were also able to pass the regulatory limit for lead for all of the MBS applications tested. All of the samples tested during Phase II were able to pass the PFT for liquid release.

During the intermediate-scale testing, varying amounts of MBS with 2.5 wt % WL-770 were added simultaneously to the soil samples with 30 wt % DI water. Chromium and nickel were the only metals that did not meet regulatory requirements for the 5 wt % MBS sample and nickel was the only metal not to meet requirements for the 10 wt % MBS sample. The 15 and 20 wt % MBS samples met the regulatory limits for all the metals tested: lead, mercury, cadmium, chromium, zinc, nickel and uranium. All of the Phase III samples also passed the PFT for liquid release.

The treatment of contaminated soil using the two agents in concert was able to reduce both the leachable quantity of the metallic contaminants and the soil moisture levels in much the same manner as was observed when the two agents were applied separately. Also, the use of the two agents in concert did not exhibit significant interferences with the actions of either agent proving that MBS and WL-770 can be used simultaneously to bind metals and dewater soils and/or sediments.

REFERENCES

1. U.S. Environmental Protection Agency, Molecular Bonding System Innovative Technology Evaluation Report, U.S. EPA, National Risk Management Research Laboratory, Office of Research and Development, Cincinnati, Ohio, EPA/540/R-97/507, February 1998.

2. U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, *Multiple Extraction Procedure*, SW-846, Method 1320, Revision 0, September 1986.

3. U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, *Toxicity Characteristic Leaching Procedure*, SW-846, Method 1311, Revision 0, July 1992.

4. Adams, J.W., and Kalb, P.D., *Molecular Bonding System (MBS) Treatment of a BNL Soil Mixed Waste Surrogate*, Environmental & Waste Technology Center, Department of Advanced Technology, Brookhaven National Laboratory, May 1998.

5. MSE Technology Applications Inc., *Draft Report-Treatability Studies for Mercury Contaminated Soil at the Y-12 Plant, Building 81-10 Area, Oak Ridge, TN*, Report ECCP-A40, March 2003.

6. MSE Technology Applications Inc., *Draft Final Report-A Test Program for In Situ Stabilization of Mercury Contaminated Soil*, Report ECCP-43, November 2004.

7. MSE Technology Applications Inc., *Treatability Testing of Dredge Material for Reaches 3, 4 and 5 of the West Branch of the Grand Calumet River, Hammond, Indiana,* Letter Report, March, 2009.

8. U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 9095A, *Paint Filter Liquids Test* , U.S. EPA, Washington, D.C. Office of Solid Waste, web site: http:// www.epa.gov/epaoswer/hazwaste/test/main.htm

9. MSE Technology Applications Inc., *DOE Program Test Report for 55-Gallon Drum Grout Testing for the Solidification of Melton Valley Storage Tank Waste Stored at the Oak Ridge National Laboratory,* Report MSE-240, July 2009.

10. U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 9096, *Liquid Release Test*, U.S. EPA, Washington, D.C. Office of Solid Waste, web site: http:// www.epa.gov/epaoswer/hazwaste/test/main.htm