Bench-Scale Grout Testing for the Solidification of Melton Valley Storage Tank Waste Stored at the Oak Ridge National Laboratory

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Introduction

The U.S. Department of Energy (DOE) tasked MSE Technology Applications, Inc. (MSE) to evaluate grouts using various mixing and addition methods to solidify radioactive aqueous liquid wastestreams using surrogates. Oak Ridge National Laboratory (ORNL) identified a low-level legacy sludge for grout testing. The ORNL legacy sludge is stored in the Melton Valley storage tanks (MVST) and has been collected for the past 20 years from all operations at ORNL that produce low-level waste (LLW), including the Radiochemical Engineering Development Center. This legacy waste is a priority and must be disposed before ORNL can close the older tanks. This waste was originally going to be concentrated and dried by evaporation and disposed of at the Waste Isolation Pilot Plant in New Mexico as a transuranic waste. However, if the waste sludge can be solidified without concentration by using a grout material, the transuranic levels would be below the 100 nanocuries per gram level, and the wasteform could be disposed at the Nevada Test Site (NTS) as a LLW, saving ORNL approximately $100M.

The Oak Ridge Transuranic Waste Processing Center, operated by Wastern Advantage, Inc. (formerly operated by EnergX TN, LLC), must process approximately 350,000 gallons (gal) of remote-handled sludge from the MVST to solidify the waste to meet the NTS Waste Acceptance Criteria.

Project Objectives

- Mixed with Solidification Agents
- Flowable during Mixing
- Self-Leveling in Waste Container
- Non-RCRA Wasteform
- No Free Liquid

MVST Surrogate Waste Formulation

Chemical Name	Compound	Target Weight (g)
Sodium Sulfate	Na_2SO_4	0.41
Calcium Nitrate Tetrahydrate	$Ca(NO_3)_2{\cdot}4H_2O$	29.52
Ferric Nitrate Nonahydrate	$Fe(NO_3)_3{\cdot}9H_2O$	26.26
Ferric Chloride	$FeCl_3$	3.24
Potassium Nitrate	KNO_3	29.62
Magnesium Nitrate Hexahydrate	$Mg(NO_3)_2{\cdot}6H_2O$	145.90
Thorium Nitrate Tetrahydrate	$Th(NO_3)_4{\cdot}4H_2O$	28.67
Uranyl Nitrate Hexahydrate	$UO_2(NO_3)_2{\cdot}6H_2O$	103.55
Sodium Aluminate	$NaAlO_2$	33.85
Calcium Hydroxide	$Ca(OH)_2$	140.55
Sodium Carbonate Anahydrous	Na_2CO_3	12.72
Sodium Silicate Pentahydrate	$Na_2SiO_3{\cdot}5H_2O$	82.72
Sodium Nitrite	$NaNO_2$	7.11
Organic (SRS PUREX surrogate)	NA	8.1
	Total	**1,276.65**

NA=not applicable since SRS PUREX is a chemical mixture.

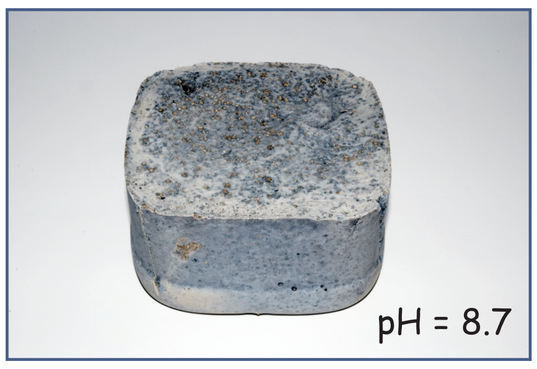
TCLP Data for the MVST Grouted Wasteforms

Sample ID	Sludge Fraction	Metals Concentration (mg/L)			
		Cd	Cr	Pb	Hg
RCRA Regulatory Limits		1	5	5	0.2
W25-1	0.064	<0.03	0.558	<0.04	0.0084
W25-2	0.068	<0.03	0.682	<0.04	0.0127
W25-3	0.122	<0.03	0.629	<0.04	0.0284
W25-4	0.162	<0.03	0.584	<0.04	0.0289
W25-5	0.111	<0.03	0.570	<0.04	0.0165
W25-6	0.122	<0.03	0.529	<0.04	0.0176
W25-7	0.122	<0.03	0.567	<0.04	0.0231
W25-8	0.244	<0.03	0.516	0.0922	0.0352
W25-9	0.122	<0.03	0.630	<0.04	0.0193
W28-1	0.131	<0.03	0.422	<0.04	0.0083
W28-2	0.149	<0.03	0.573	<0.04	0.0103
W28-3	0.119	<0.03	0.393	<0.04	0.0072
W28-4	0.131	<0.03	0.699	0.0709	0.0078
W28-5	0.130	<0.03	0.437	0.0976	0.0053
W28-6	0.130	<0.03	0.316	<0.04	0.0056
W28-7	0.262	<0.03	0.507	<0.04	0.0106
W28-8	0.132	<0.03	0.639	0.0634	0.0063
W35-1	0.169	<0.03	0.790	<0.04	0.0040
W35-2	0.193	<0.03	1.140	<0.04	0.0078
W35-3	0.153	<0.03	0.557	<0.04	0.0040
W35-4	0.169	<0.03	0.615	<0.04	0.0040
W35-5	0.169	<0.03	0.643	<0.04	0.0040
W35-6	0.338	<0.03	0.956	<0.04	0.0061
W35-7	0.169	<0.03	0.634	<0.04	0.0040
W35-8	0.169	<0.03	0.563	<0.04	0.0054
W23-1	0.133	<0.03	0.721	<0.04	0.0040
W23-2	0.152	<0.03	0.745	<0.04	0.0040
W23-3	0.121	<0.03	0.462	<0.04	0.0040
W23-4	0.132	<0.03	0.596	<0.04	0.0040
W23-5	0.133	<0.03	0.601	<0.04	0.0040
W23-6	0.266	<0.03	0.386	<0.04	0.0040
W23-7	0.135	<0.03	0.734	<0.04	0.0040
W23-8	0.134	<0.03	0.532	0.171	0.0040

The sample identification (ID) = Tank number (W-xx) and sequential number.

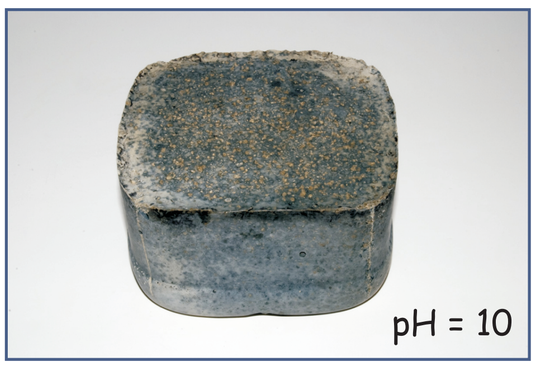
Bench-Scale Grout Formulations

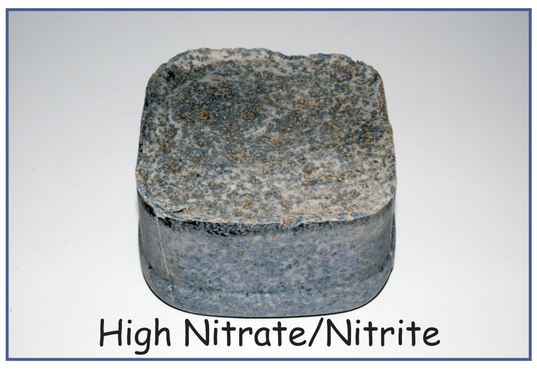
The bench-scale samples were generated at the 1:1 weight-based grout loading ratio of dry cement mixture to MVST surrogate. The dry cement mixture consisted of 30 weight percent (wt%) fly ash, blast furnace slag, Portland cement, and 10 wt% Metso Beads (sodium metasilicate). A superabsorbent polymer, Waste Lock 770, was also added to the grout formulation to help control bleed water. Samples were generated at pH values of 8.7, 10, and 12 with a high nitrate/nitrite version and a high total organic carbon (TOC) version.

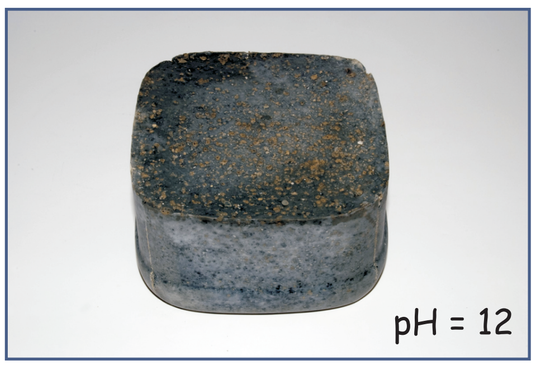


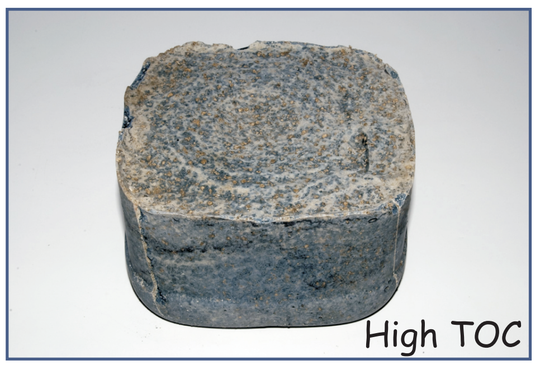
Test Results

- Flowable
- Self-Leveling
- Non-RCRA Wasteform
- No Bleed Water









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55-Gallon Grout Testing for the Solidification of Melton Valley Storage Tank Waste Stored at the Oak Ridge National Laboratory

Jody Bickford, MSE Technology Applications, Inc.

Project Objectives

- Mixed with Solidification Agents
- Flowable during Mixing
- Self-Leveling in Waste Container
- Non-RCRA Wasteform
- No Free Liquid

Grout Formulations

The grout formulation during the 55-gal drum testing varied the weight-based grout loading ratio of the dry cement mixture to MVST surrogate from 0.9:1 to 1.2:1 and the total suspended solids (TSS) from 5% to 10%. Two superabsorbent polymers were also tested. The dry cement mixture and the test matrix and results are presented below.

Dry Cement Mixture

- 30 wt% Portland Type I/II Cement
- 30 wt% Class F Fly Ash
- 30 wt% Grade 120 Blast Furnace Slag
- 10 wt% Metso Beads

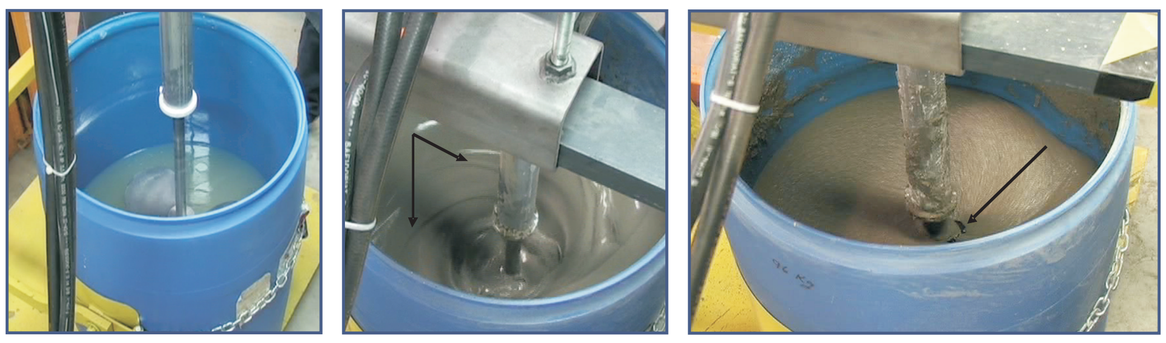
Test Equipment Used During 55-Gal Drum Testing

55-Gal Test Matrix and Results

Sample Number	TSS, wt%	Weight-Based Grout Loading Ratio, Dry Cement Mixture to MVST Surrogate	Superabsorbent Polymer, Type and wt%	Mixing Time, minutes	Final Viscosity Data, cP
1	5	1:1	Waste Lock 770 1.6	21	1,500
2	5	1:1	Waste Lock 770 1.6	21	500
3	5	0.9:1	Waste Lock 770 1.6	21	488
4	5	1.2:1	Waste Lock 770 1.6	37	*
5	10	1:1	Waste Lock 770 1.6	33	*
6	5	1:1	Waste Lock 770 1.6	45	2,130
7	5	1:1	Nochar Acid Bond 0.8	27	600
8	5	1:1	Waste Lock 770 3.2	18	784
9	5	1:1	Waste Lock 770 1.6	57	*

* Too thick to measure.

55-Gal Sample During Sample Generation



55-Gal Sample During Physical Destruction

55-Gal Drum Density Data

Sample Number	TSS, wt%	Weight-Based Grout Loading Ratio, Dry Cement Mixture to MVST Surrogate	Left Half Bulk Density, g/mL	Right Half Bulk Density, g/mL	Volumetric Expansion
1	5	1:1	1.38	1.36	1.37
2	5	1:1	1.39	1.36	1.39
3	5	0.9:1	1.33	1.32	1.29
4	5	1.2:1	1.58	1.54	1.46
5	10	1:1	1.57	1.56	1.39
6	5	1:1	1.47	1.48	1.39
7	5	1:1	1.50	1.52	1.38
8	5	1:1	1.51	1.54	1.43
9	5	1:1	1.44	1.43	1.37

Test Results

- Homogeneous Sample Consistency
- Flowable and Self-Leveling Grout
- No Bleed Water
- Identified Organic-Phase Sorbent
- Uniform Densities

Conclusions

- TSS No Higher than 5 wt%
- Grout Loading Ratio No Higher than 1:1
- Use Waste Lock 770 for Superabsorbent Polymer not Nochar Acid Bond
- Use Petroset II Powder to Tie Up Organic Phase

80-Gallon Grout Testing for the Solidification of Melton Valley Storage Tank Waste Stored at the Oak Ridge National Laboratory

Jody Bickford, MSE Technology Applications, Inc.

Project Objectives

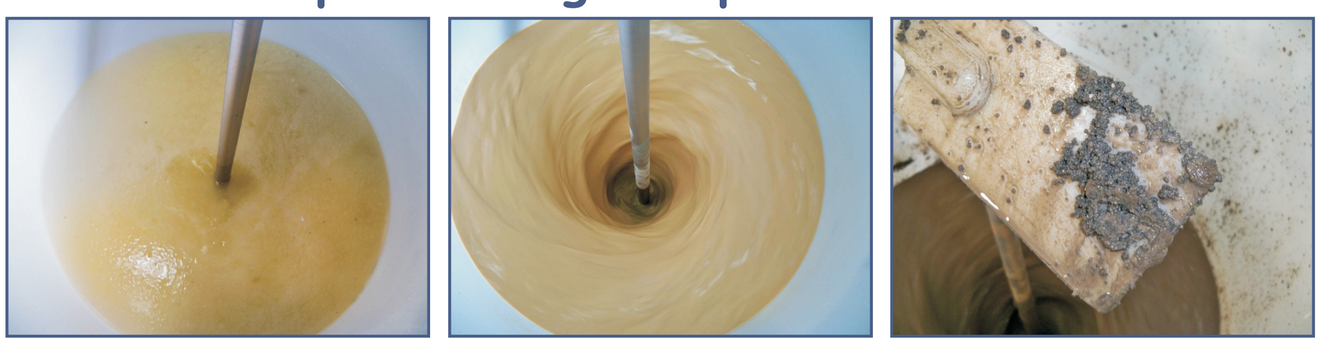
- Flowable and Self-Leveling Grout
- No Free Liquid
- Optimize Bleed Water Control Agents

80-Gal Samples During Sample Generation

80-Gal Test Matrix and Results

Sample Number	Major Dry Grout Components				Waste Lock 770, wt% of major dry components	Mixing Times, minutes	Final Viscosity Data, cP
	Fly Ash, wt%	Blast Furnace Slag, wt%	Portland Cement, wt%	Metso Beads, wt%			
1	30	30	30	10	1.6	22	2,000,000
2	35	30	30	5	0.8	60	2,183
3	30	35	30	5	0.8	93	2,110
4	30	30	35	5	0.8	63	2,362
5	30.83	30.83	30.83	7.5	1.2	60	12,920
6	31	31	31	7	1	78	3,083
7	31	31	31	7	1.5	120	14,080

80-Gal Samples During Sample Generation



80-Gal Samples During Physical Destruction

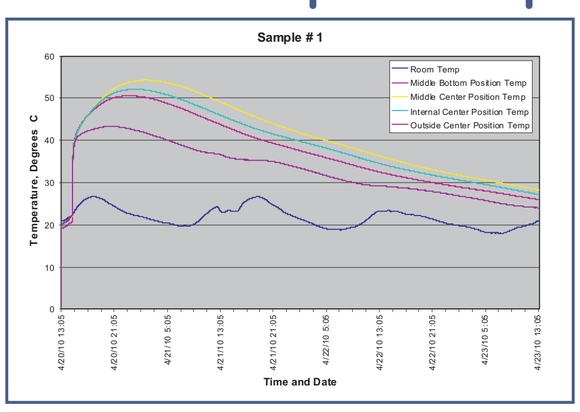
80-Gal Attenuation Data

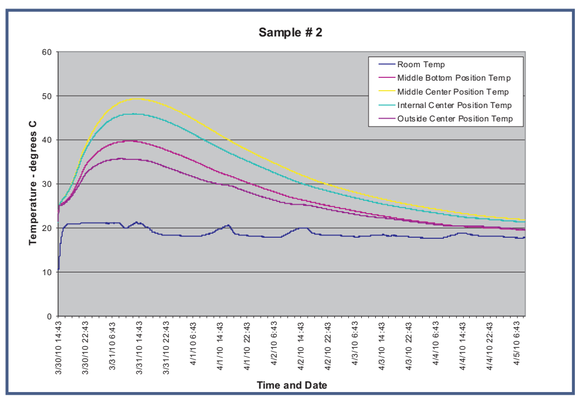
Sample Number and Location	Cs-137 Gamma Source - Radiation Reading, R/hr	Percent Gamma Attenuation	Cf-252 Neutron Source - Radiation Reading, mR/hr	Percent Neutron Attenuation
Empty box	170.5	NA	129.0	NA
1 - top	123.4	27.62	78.8	39.92
1 - bottom	122.9	27.92	80.7	38.47
2 - top	123.3	27.68	76.0	42.05
2 - bottom	120.6	29.27	76.9	41.36
3 - top	123.1	27.80	80.9	38.31
3 - bottom	123.1	27.80	79.0	39.76
4 - top	122.6	28.09	81.7	37.70
4 - bottom	123.1	27.80	75.4	42.51
Empty box	170.6	NA	133.3	NA

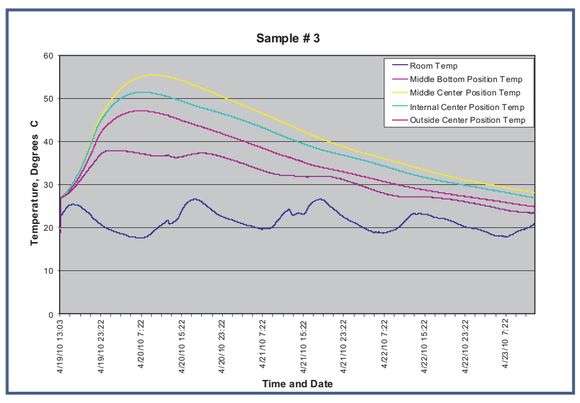
80-Gal Drum Density Data

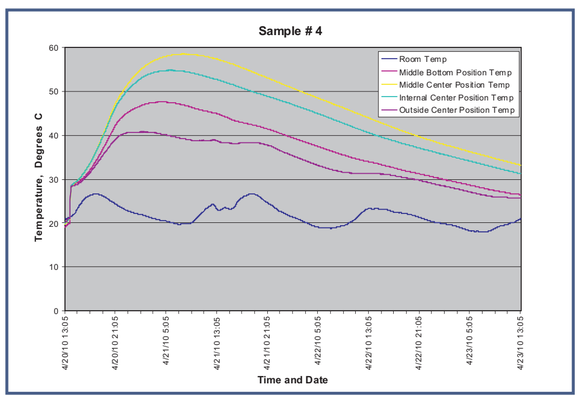
Sample Number	Whole Sample Density, g/mL	Left Half Density, g/mL	Right Half Density, g/mL	Top Half Density, g/mL	Bottom Half Density, g/mL
1	1.621	1.619	1.624	1.622	1.622
2	1.603	1.605	1.604	1.605	1.605
3	1.612	1.610	1.614	1.612	1.615
4	1.616	1.618	1.615	1.615	1.614

80-Gal Samples Temperature Data









Test Results

- Homogeneous Sample Consistency
- Flowable and Self-Leveling Grout
- Achieved No Bleed Water at Optimized Formulation
- Uniform Densities
- Optimized Formulation for Bleed Water Control Agents
- Provided Gamma and Neutron Attenuation

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