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Analysis of Harrell Monosodium Titanate Lot #s 46000706120, 46000722120, and 46000808120

K. M. L. Taylor-Pashow

October 2012

Savannah River National Laboratory
Savannah River Nuclear Solutions, LLC
Aiken, SC 29808

Prepared for the U.S. Department of Energy under contract number DE-AC09-08SR22470.

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EXECUTIVE SUMMARY

Monosodium titanate (MST) for use in the Actinide Removal Process (ARP) must be qualified and verified in advance. A single qualification sample for each batch of material is sent to SRNL for analysis, as well as a statistical sampling of verification samples. The Harrell Industries Lot #s 46000706120, 46000722120, and 460008081120 qualification and verification samples met each of the selected specification requirements that were tested with the exception of a few pails being marginally below the lower weight percent solids limit. These deviations from the specifications are viewed as negligible since the corresponding density of the slurries indicates no appreciable shortage of MST solids. Therefore, SRNL recommends acceptance and use of these pails.

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LIST OF ABBREVIATIONS

ARP	Actinide Removal Process
CSSX	Caustic Side Solvent Extraction
DF	decontamination factor
IC	ion chromatography
ICP-ES	inductively coupled plasma – emission spectroscopy
MCU	Modular CSSX Unit
MST	monosodium titanate
SRNL	Savannah River National Laboratory
TIC-TOC	total inorganic carbon – total organic carbon
VOA	volatile organic analysis

1.0 Introduction

Harrell Industries is under contract with Savannah River Remediation (SRR) to provide MST for use in the Actinide Removal Process (ARP). Qualification samples from Lot #s 46000706120, 46000722120, and 46000808120 were sent to the Savannah River National Laboratory (SRNL) to confirm the material meets certain requirements specified in the purchase specification.[1]

The vendor is also obligated to send verification samples from ~10% or more of the pails of MST product for each lot. The verification samples are selected from the entire inventory of pails so that the set of verification samples represents pails filled from the beginning to the end of the pail-filling operation for the entire lot of MST. For the verification of Lot # 46000706120, Harrell Industries sent 13 samples, one each from pails #10, 20, 30, 40, 50, 60, 70, 80, 90, 100, 110, 120, and 130 of 130 total pails. For the verification of Lot # 46000722120, Harrell Industries sent 15 samples, one each from pails #1, 10, 20, 30, 40, 50, 60, 70, 80, 90, 100, 110, 120, 130, and 140 of 149 total pails. For the verification of Lot # 46000808120, Harrell Industries sent 15 samples, one each from pails #10, 20, 30, 40, 50, 60, 70, 80, 90, 100, 110, 120, 130, 140, and 150 of 153 total pails.

SRR requested analysis of the qualification samples for weight percent solids, density, pH, volatile organics, and particle size. They also requested analysis of the verification samples for weight percent solids, density, and pH.[2] The work was controlled by a Task Technical and Quality Assurance Plan (TTQAP).[3]

2.0 Experimental Procedure

SRNL analyzed the qualification and verification samples for density, pH, and weight percent solids. Density was measured using an electronic pipette in triplicate. The pH was measured by colorimetric pH strips, and the weight percent solids were measured in triplicate using a Mettler-Toledo Halogen Moisture Analyzer HG63 instrument.

Aliquots of the qualification samples were removed under well mixed conditions to provide sub-samples for each of the analyses. SRNL performed the following analyses: volatile organic analysis (VOA) and particle size using a Microtrac® S3500 analyzer.

3.0 Results and Discussion

The results of the weight percent, pH, and density measurements are reported in Tables 3-1 through 3-3, while the results of the additional qualification sample analyses are reported in Tables 3-4 through 3-6.

Table 3-1. Weight Percent, pH, and Density Results for All Samples from Lot # 46000706120

Sample ID	Weight % Solids (Standard Deviation)	pH[a]	Density[b] (g/mL) (%RSD)
Qualification	14.99 (±0.105) %	11.5	1.124 (0.06%)
Pail #10	15.04 (±0.207) %	11.5	1.120 (0.05%)
Pail #20	14.93 (±0.046) %	11.5	1.120 (0.07%)
Pail #30	14.98 (±0.197) %	11.5	1.120 (0.03%)
Pail #40	14.90 (±0.095) %	11.5	1.120 (0.02%)
Pail #50	14.96 (±0.095) %	11.5	1.121 (0.03%)
Pail #60	14.94 (±0.065) %	11.5	1.123 (0.05%)
Pail #70	14.96 (±0.029) %	11.5	1.122 (0.02%)
Pail #80	14.98 (±0.156) %	11.5	1.123 (0.10%)
Pail #90	14.98 (±0.031) %	11.5	1.123 (0.04%)
Pail #100	14.99 (±0.047) %	11.5	1.121 (0.05%)
Pail #110	14.89 (±0.101) %	11.5	1.121 (0.02%)
Pail #120	15.03 (±0.058) %	11.5	1.120 (0.03%)
Pail #130	14.84 (±0.067) %	11.5	1.122 (0.11%)
Average	14.96 (±0.054) %	11.5	1.121 (0.13%)
Acceptable Range[1]	15-17 %	> 10	no requirement

a) The uncertainty of the pH measurement is 0.5 pH units.
b) Density measurements taken at 21 °C.

The weight percent solids measurements for the qualification sample and several of the pails in Lot# 46000706120 fell below the lower limit of the acceptable range of 15 wt %. However, taking into account the standard deviation, all but pail #130 fall within 2 standard deviations of the 15 wt % limit. Since there is no apparent trend in the wt % solids measurements, indicating poor suspension of the solids at the beginning or end of the pail filling, and the overall wt % solids measurement for the lot falls within the acceptable range (taking into account the standard deviation), it is recommended this pail also be accepted. The density measurement for this pail is comparable to the rest of the lot, indicating there is not a significantly lower amount of MST in this pail.

Table 3-2. Weight Percent, pH, and Density Results for All Samples from Lot # 46000722120

Sample ID	Weight % Solids (Standard Deviation)	pH[a]	Density[b] (g/mL) (%RSD)
Qualification	15.02 (±0.087) %	11.5	1.122 (0.04%)
Pail #1	15.20 (±0.047) %	11.5	1.123 (0.10%)
Pail #10	15.11 (±0.047) %	11.5	1.123 (0.11%)
Pail #20	15.16 (±0.056) %	11.5	1.118 (0.13%)
Pail #30	15.04 (±0.036) %	11.5	1.121 (0.04%)
Pail #40	15.00 (±0.191) %	11.5	1.121 (0.04%)
Pail #50	15.17 (±0.067) %	11.5	1.122 (0.07%)
Pail #60	15.00 (±0.147) %	11.5	1.121 (0.05%)
Pail #70	15.02 (±0.092) %	11.5	1.122 (0.02%)
Pail #80	14.96 (±0.215) %	12.0	1.123 (0.09%)
Pail #90	15.12 (±0.061) %	11.5	1.120 (0.02%)
Pail #100	15.02 (±0.060) %	11.5	1.122 (0.04%)
Pail #110	14.96 (±0.165) %	12.0	1.120 (0.05%)
Pail #120	14.93 (±0.100) %	12.0	1.123 (0.02%)
Pail #130	15.00 (±0.085) %	12.0	1.120 (0.05%)
Pail #140	14.89 (±0.126) %	12.0	1.120 (0.07%)
Average	15.04 (±0.091) %	11.5	1.121 (0.12%)
Acceptable Range[1]	15-17 %	> 10	no requirement

a) The uncertainty of the pH measurement is 0.5 pH units.
b) Density measurements taken at 21 °C.

The weight percent solids measurements for four of the pails in Lot# 46000722120 fell below the lower limit of the acceptable range of 15 wt %. However, taking into account the standard deviation, these four pails fall within 1 standard deviation of the 15 wt % limit, and should therefore be accepted.

Table 3-3. Weight Percent, pH, and Density Results for All Samples from Lot # 46000808120

Sample ID	Weight % Solids (Standard Deviation)	pH[a]	Density[b] (g/mL) (%RSD)
Qualification	15.02 (±0.176) %	12.0	1.118 (0.04%)
Pail #10	14.92 (±0.114) %	11.5	1.122 (0.06%)
Pail #20	15.01 (±0.080) %	11.5	1.119 (0.02%)
Pail #30	15.01 (±0.091) %	12.0	1.120 (0.13%)
Pail #40	14.95 (±0.091) %	11.5	1.116 (0.19%)
Pail #50	14.84 (±0.030) %	11.5	1.119 (0.04%)
Pail #60	14.97 (±0.065) %	12.0	1.103 (0.33%)
Pail #70	14.95 (±0.190) %	12.0	1.119 (0.01%)
Pail #80	15.00 (±0.159) %	11.5	1.121 (0.07%)
Pail #90	14.91 (±0.015) %	11.5	1.118 (0.04%)
Pail #100	14.96 (±0.114) %	12.0	1.125 (0.05%)
Pail #110	14.90 (±0.070) %	11.5	1.119 (0.04%)
Pail #120	14.97 (±0.112) %	12.0	1.116 (0.20%)
Pail #130	14.88 (±0.080) %	12.0	1.120 (0.04%)
Pail #140	14.91 (±0.055) %	12.0	1.116 (0.06%)
Pail #150	14.97 (±0.117) %	12.0	1.117 (0.01%)
Average	14.95 (±0.051) %	12.0	1.118 (0.42%)
Acceptable Range[1]	15-17 %	> 10	no requirement

a) The uncertainty of the pH measurement is 0.5 pH units.
b) Density measurements taken at 22 °C.

The weight percent solids measurements for many of the pails in Lot# 46000808120 fell below the lower limit of the acceptable range of 15 wt %. However, taking into account the standard deviation, all but pails #50 and #90 fall within 2 standard deviations of the 15 wt % limit. Since there is no apparent trend in the wt % solids measurements, indicating poor suspension of the solids at the beginning or end of the pail filling, and the overall wt % solids measurement for the lot falls within the acceptable range (taking into account the standard deviation), it is recommended that these pails also be accepted. The density measurements for these two pails are comparable to the rest of the lot, indicating there is not a significantly lower amount of MST in these pails.

Table 3-4. Results of the Qualification Sample Analyses for Lot # 46000706120

Property	Method	Result	Specification	Pass ?
Volatile Organics	VOA	39 ppm[i]	n/a[ii]	n/a
Particle Size, < 0.8 μm	Microtrac®	7.61 vol %	<10 vol %	YES
Particle Size, > 37 μm	Microtrac®	0.13 vol %	<1 vol %	YES
Particle Size, geometric standard deviation (absorbance mode)	Microtrac®	3.42	≤3.5	YES

Table 3-5. Results of the Qualification Sample Analyses for Lot # 46000722120

Property	Method	Result	Specification	Pass ?
Volatile Organics	VOA	6.5 ppm[iii]	n/a[ii]	n/a
Particle Size, < 0.8 μm	Microtrac®	5.19 vol %	<10 vol %	YES
Particle Size, > 37 μm	Microtrac®	0.00 vol %	<1 vol %	YES
Particle Size, geometric standard deviation (absorbance mode)	Microtrac®	3.34	≤3.5	YES

Table 3-6. Results of the Qualification Sample Analyses for Lot # 46000808120

Property	Method	Result	Specification	Pass ?
Volatile Organics	VOA	81 ppm[iv]	n/a[ii]	n/a
Particle Size, < 0.8 μm	Microtrac®	5.53 vol %	<10 vol %	YES
Particle Size, > 37 μm	Microtrac®	0.00 vol %	<1 vol %	YES
Particle Size, geometric standard deviation (absorbance mode)	Microtrac®	3.45	≤3.5	YES

The "Particle Size, geometric standard deviation" is defined as the 50th percentile result divided by the 16th percentile result. Microtrac® results have a 10% analytical uncertainty. VOA results have a 20% analytical uncertainty.

4.0 Conclusions

Analyses of the Harrell Lot #s 46000706120, 46000722120, and 46000808120 MST materials indicate the materials fall within the specifications required for use at ARP with the exception of a few pails being marginally below the lower weight percent solids limit. These deviations from the specifications are viewed as negligible since the corresponding density of the slurries indicates no appreciable shortage of MST solids. Therefore, SRNL recommends acceptance and use of these pails.

[i] Isopropanol = 39 ppm, all other analytes = < 0.25 ppm
[ii] Purchase specification does not include a specification for volatile organics, only total alcohol content of < 500 ppm.
[iii] Isopropanol = 6.5 ppm, all other analytes = < 0.25 ppm
[iv] Isopropanol = 81 ppm, all other analytes = < 0.25 ppm

5.0 References

1. Specification for Purchase of 15 wt % Monosodium Titanate (MST) for 96-H ARP, Specification No. X-SPP-H-00012, Rev. 6, November 2010.
2. C. Duffey, "MST Qualification and Verification", X-TTR-H-00017, Rev. 0, February 2012.
3. K. M. L. Taylor-Pashow, "Task Technical and Quality Assurance Plan for Monosodium Titanate (MST) Qualification and Verification", SRNL-RP-2012-00094, Rev. 0, March 2012.

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