

# **Analysis of Harrell Monosodium Titanate Lot #46000619120**

K. M. L. Taylor-Pashow

September 2012

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

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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 #46000619120 qualification and the 13 verification samples met each of the selected specification requirements that were tested and, consequently, the material is acceptable for use in the ARP process.

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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). A 1-L qualification sample from Lot #46000619120 was sent to the Savannah River National Laboratory (SRNL) to confirm the material meets certain requirements specified in the purchase specification.<sup>1</sup>

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 this lot, Harrell Industries sent 13 samples, one each from pails #1, 10, 20, 30, 40, 50, 60, 70, 80, 90, 100, 110, and 120 of 121 total pails.

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

## **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 sample 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<sup>®</sup> S3500 analyzer.

## **3.0 Results and Discussion**

The results of the weight percent, pH, and density measurements are reported in Table 3-1, while the results of the additional qualification sample analyses are reported in Table 3-2.

**Table 3-1. Weight percent, pH, and Density Results for All Samples**

Sample ID	Weight % Solids (Standard Deviation)	pH <sup>a</sup>	Density <sup>b</sup> (g/mL) (%RSD)
Qualification	15.59 (±0.096) %	12.0	1.127 (0.01%)
Pail #1	15.73 (±0.205) %	12.0	1.127 (0.07%)
Pail #10	15.78 (±0.155) %	12.0	1.128 (0.03%)
Pail #20	15.64 (±0.046) %	12.0	1.118 (0.30%)
Pail #30	15.65 (±0.121) %	12.0	1.128 (0.03%)
Pail #40	15.77 (±0.098) %	12.0	1.127 (0.12%)
Pail #50	15.82 (±0.032) %	12.0	1.124 (0.38%)
Pail #60	15.75 (±0.078) %	12.0	1.125 (0.05%)
Pail #70	15.62 (±0.066) %	12.0	1.125 (0.12%)
Pail #80	15.65 (±0.030) %	12.0	1.127 (0.11%)
Pail #90	15.64 (±0.116) %	12.0	1.122 (0.15%)
Pail #100	15.49 (±0.040) %	12.0	1.127 (0.05%)
Pail #110	15.51 (±0.076) %	12.0	1.123 (0.10%)
Pail #120	15.66 (±0.169) %	12.0	1.123 (0.05%)
Average	15.66 (±0.097) %	12.0	1.125 (0.24%)
Acceptable Range <sup>1</sup>	15-17 %	> 10	no requirement

a) The uncertainty of the pH measurement is 0.5 pH units.

b) Density measurements taken at 23 °C.

**Table 3-2. Results of the Qualification Sample Analyses**

Property	Method	Result	Specification	Pass ?
Volatile Organics	VOA	33 ppm <sup>i</sup>	n/a <sup>ii</sup>	n/a
Particle Size, < 0.8 µm	Microtrac <sup>®</sup>	5.33 vol %	<10 vol %	YES
Particle Size, > 37 µm	Microtrac <sup>®</sup>	0.15 vol %	<1 vol %	YES
Particle Size, geometric standard deviation (absorbance mode)	Microtrac <sup>®</sup>	3.13	≤3.5	YES

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

#### 4.0 Conclusions

Analyses of the Harrell Lot #46000619120 MST material indicate the material falls within the specifications required for use at ARP.

<sup>i</sup> Isopropanol = 33 ppm, all other analytes = < 0.25 ppm

<sup>ii</sup> Purchase specification does not include a specification for volatile organics, only total alcohol content of < 500 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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