

**Keywords:** *MST, ISDP*

**Retention:** *Permanent*

## **Analysis of Harrell Monosodium Titanate Lot #120111**

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February 2012

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



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**Printed in the United States of America**

**Prepared for  
U.S. Department of Energy**

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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 #120111 qualification and the first 12 verification samples met all the requirements in the specification indicating the material is acceptable for use in the process. Analyses of Pail 125 verification sample fails the criteria for solids content and has measurably lower pH, density, and total bottle weight. The verification sample for Pail 125 was retested for weight percent solids after checking that all of the solids had been suspended. The sample again failed to meet acceptance criteria. SRNL recommends accepting Pails 1 through 120. Pails 121 through 125 should be rejected and returned to the vendor.

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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 to provide MST for use in the Actinide Removal Process (ARP). A 1000-mL qualification sample for Lot #120111 was sent to the Savannah River National Laboratory (SRNL) on January 13, 2012 to confirm the material meets the 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 (distributed roughly evenly through the entire lot of pails). For the verification of this lot, Harrell Industries sent 13 samples, one each from pails #1, 20, 30, 40, 50, 60, 70, 80, 90, 100, 110, 120 and 125 of 125 total pails.

## **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: strontium (Sr) decontamination factor (DF), volatile organic analysis (VOA), total inorganic carbon-total organic carbon (TIC-TOC), ion chromatography (IC) for fluoride, chloride, and bromide, 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	16.15 (±0.22) %	12	1.126 (0.02%)
Pail #1	16.24 (±0.13) %	12	1.122 (0.07%)
Pail #20	16.10 (±0.03) %	12	1.123 (0.10%)
Pail #30	16.18 (±0.03) %	12	1.126 (0.03%)
Pail #40	16.16 (±0.05) %	12	1.132 (0.42%)
Pail #50	16.12 (±0.08) %	12	1.120 (0.09%)
Pail #60	16.14 (±0.05) %	12	1.132 (0.28%)
Pail #70	15.48 (±0.08) %	11.5	1.122 (0.07%)
Pail #80	15.36 (±0.04) %	11.5	1.123 (0.06%)
Pail #90	15.48 (±0.16) %	11.5	1.122 (0.11%)
Pail #100	15.51 (±0.08) %	11.5	1.123 (0.19%)
Pail #110	15.45 (±0.05) %	11.5	1.121 (0.19%)
Pail #120	15.35 (±0.05) %	11.5	1.126 (0.42%)
Pail #125	10.82 (±0.02) %	11	1.045 (0.38%)
Average	15.47 (±0.38) %	11.5	1.119 (1.93%)
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?
Sr DF	Sr test	2.60 (±0.27)	>1.79	YES
Alcohol content <sup>i</sup>	VOA	98.4 mg/L	≤500 mg/L max	YES
Total Inorganic Carbon	TIC	10.1 mg/L	≤100 mg/L max	YES
Total Organic Carbon	TOC	37.0 mg/L	≤300 mg/L max	YES
Total Halides (F+Cl+Br)	IC	31 mg/L	≤100 mg/L max	YES
Particle Size, <0.8 μm	Microtrac <sup>®</sup>	3.54 vol %	<10 vol %	YES
Particle Size, >37 μm	Microtrac <sup>®</sup>	0 vol %	<1 vol %	YES
Particle Size, geometric standard deviation (absorbance mode)	Microtrac <sup>®</sup>	2.91	≤3.5	YES

<sup>i</sup> The alcohol content is derived from the TOC and VOA data. The VOA result gave 0.45 mg/L isopropanol. Using the conservative case that all of the remaining carbon from the TOC result was from methanol (and knowing methanol is 37.5 wt % carbon), we calculate the MeOH as 97.97 ppm. This gives a total alcohol content of 98.4 ppm.

The “Particle Size, geometric standard deviation” is defined as the 50th percentile result divided by the 16th percentile result. Microtrac<sup>®</sup>, TIC-TOC, and IC results have a 10% analytical uncertainty. VOA results have a 20% analytical uncertainty. The inductively coupled plasma – emission spectroscopy (ICP-ES) results used for measuring the Sr DF have an average analytical uncertainty of 10.2%. Results in parentheses are derived from the standard deviation.

The TIC and TOC results are in terms of mg/L of carbon. If we assume that the entire TIC result is carbonate, this translates to a carbonate concentration of  $\leq 0.0008$  M.

#### **4.0 Conclusions**

Analyses of the Harrell Lot #120111 MST material indicate the qualification sample and all but Pail 125 of the verification samples fall within the specifications required for use at ARP. The verification sample for Pail 125 was retested for weight percent solids after checking that all of the solids had been suspended. The sample again failed to meet acceptance criteria. A visual inspection of the sample shows a much less dense solid than the samples that pass. SRNL recommends accepting Pails 1 through 120. Pails 121 through 125 should be rejected and returned to the vendor.

#### **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.



**Distribution:**

A. B. Barnes, 999-W  
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