

**Keywords:** *MST, ISDP*

**Retention:** *Permanent*

## **Analysis of Harrell Monosodium Titanate Lot #052511**

K. M. L. Taylor-Pashow

August 2011

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.



## **DISCLAIMER**

This work was prepared under an agreement with and funded by the U.S. Government. Neither the U.S. Government or its employees, nor any of its contractors, subcontractors or their employees, makes any express or implied:

1. warranty or assumes any legal liability for the accuracy, completeness, or for the use or results of such use of any information, product, or process disclosed; or
2. representation that such use or results of such use would not infringe privately owned rights; or
3. endorsement or recommendation of any specifically identified commercial product, process, or service.

Any views and opinions of authors expressed in this work do not necessarily state or reflect those of the United States Government, or its contractors, or subcontractors.

**Printed in the United States of America**

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



## **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 #052511 qualification and 14 verification samples met all the requirements in the specification indicating the material is acceptable for use in the process.

## TABLE OF CONTENTS

LIST OF TABLES .....	v
LIST OF ABBREVIATIONS .....	v
1.0 Introduction .....	1
2.0 Experimental Procedure .....	1
3.0 Results and Discussion .....	1
4.0 Conclusions .....	3
5.0 References .....	3

## LIST OF TABLES

Table 3-1. Weight percent, pH and Density Results for All Samples .....	2
Table 3-2. Results of the Qualification Sample Analyses .....	2

## 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 500-mL qualification sample for Lot #052511 was sent to the Savannah River National Laboratory (SRNL) 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 14 samples, one each from pails #1, 10, 20, 30, 40, 50, 60, 70, 80, 90, 110, 120, 130, and 140 of 143 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 mixing 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	15.72 ( $\pm 0.170$ ) %	11.5	1.119 (0.05%)
Pail #1	15.78 ( $\pm 0.108$ ) %	11	1.119 (0.31%)
Pail #10	15.79 ( $\pm 0.181$ ) %	11	1.126 (0.02%)
Pail #20	15.69 ( $\pm 0.154$ ) %	11.5	1.124 (0.01%)
Pail #30	15.90 ( $\pm 0.118$ ) %	11.5	1.126 (0.17%)
Pail #40	15.81 ( $\pm 0.076$ ) %	11.5	1.126 (0.08%)
Pail #50	15.77 ( $\pm 0.070$ ) %	11.5	1.125 (0.06%)
Pail #60	15.72 ( $\pm 0.045$ ) %	11	1.126 (0.06%)
Pail #70	15.79 ( $\pm 0.045$ ) %	11	1.121 (0.09%)
Pail #80	15.86 ( $\pm 0.127$ ) %	11	1.122 (0.05%)
Pail #90	15.75 ( $\pm 0.021$ ) %	11	1.123 (0.10%)
Pail #110	15.76 ( $\pm 0.116$ ) %	11	1.125 (0.17%)
Pail #120	15.73 ( $\pm 0.061$ ) %	11	1.123 (0.04%)
Pail #130	15.54 ( $\pm 0.101$ ) %	11	1.124 (0.08%)
Pail #140	15.80 ( $\pm 0.095$ ) %	11	1.123 (0.07%)
Average	15.76 ( $\pm 0.083$ ) %	11	1.124 (0.21%)
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 24 °C.

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

Property	Method	Result	Specification	Pass ?
Sr DF	Sr test	3.51 ( $\pm 0.243$ )	>1.79	YES
Alcohol content <sup>1</sup>	VOA	45 mg/L	$\leq 500$ mg/L max	YES
Total Inorganic Carbon	TIC	18 mg/L	$\leq 100$ mg/L max	YES
Total Organic Carbon	TOC	18 mg/L	$\leq 300$ mg/L max	YES
Total Halides (F+Cl+Br)	IC	<15 mg/L	$\leq 100$ mg/L max	YES
Particle Size, <0.8 $\mu\text{m}$	Microtrac <sup>®</sup>	6.04 vol %	<10 vol %	YES
Particle Size, >37 $\mu\text{m}$	Microtrac <sup>®</sup>	0 vol %	<1 vol %	YES
Particle Size, geometric standard deviation (absorbance mode)	Microtrac <sup>®</sup>	1.87	$\leq 3.5$	YES

<sup>1</sup> The alcohol content is derived from the TOC and VOA data. The VOA result gave 5.2 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 39.7 ppm. This gives a total alcohol content of 44.9 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 10.4%. 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  $\leq 0.0015$  M.

#### **4.0 Conclusions**

Analyses of the Harrell Lot #052511 MST material indicate the material falls within the specifications required for use at ARP.

#### **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  
D. A. Crowley, 773-43A  
S. D. Fink, 773-A  
B. J. Giddings, 786-5A  
C. C. Herman, 999-W  
S. L. Marra, 773-A  
A. M. Murray, 773-A  
F. M. Pennebaker, 773-42A  
W. R. Wilmarth, 773-A

K. M. L. Taylor-Pashow, 773-A  
T. B. Peters, 773-42A  
C. A. Nash, 773-42A  
M. R. Poirier, 773-42A  
F. F. Fondeur, 773-A

P. R. Jackson, 703-46A  
K. H. Subramanian, 766-H

E. J. Freed, 704-56H  
D. J. Martin, 241-152H  
M. W. Geeting, 241-152H  
T. A. Le, 766-H  
A. R. Shafer, 704-27S  
C. K. Chiu, 704-27S  
S. E. Campbell, 241-152H  
S. P. Mcleskey, 241-152H  
B. A. Gifford, 704-56H  
R. M. Wolfenden, 704-56 H  
K. L. Lang, 704-27S