Analytical Results for MOX Colemanite Samples Received on July 22, 2013

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Savannah River National Laboratory™
OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

REVIEWS AND APPROVALS

AUTHORS:

M. M. Reigel, Engineering Process Development Date

D. R. Best, Engineering Process Development Date

TECHNICAL REVIEW:

A. D. Cozzi, Engineering Process Development, Reviewed per E7 2.60 Date

APPROVAL:

E. N. Hoffman, Manager Date
Engineering Process Development

S. L. Marra, Manager Date
Environmental & Chemical Process Technology Research Programs

LIST OF REVISIONS		
Revision Number	**Summary of Changes**	**Date**
0	**Initial Issue**	**8/15/13**
1	**Corrected administrative errors and typos**	**5/20/14**

EXECUTIVE SUMMARY

The Mixed Oxide Fuel Fabrication Facility (MFFF) will use colemanite bearing concrete neutron absorber panels credited with attenuating neutron flux in the criticality design analyses and shielding operators from radiation. The Savannah River National Laboratory (SRNL) is tasked with measuring the boron oxide content of the colemanite raw aggregate material prior to it being mixed into the concrete. SRNL received ten samples of colemanite for analysis on July 22, 2013. The elemental boron content of each sample was measured according to ASTM C 1301. The boron oxide content was calculated using the oxide conversion factor for boron.

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
ICP	Inductively Coupled Plasma
MFFF	Mixed Oxide Fuel Fabrication Facility
OES	Optical Emission Spectrometer
PSAL	Process Science Analytical Laboratory
SRNL	Savannah River National Laboratory

1.0 Introduction

The Mixed Oxide Fuel Fabrication Facility (MFFF) will use colemanite bearing concrete neutron absorber panels credited with attenuating neutron flux in the criticality design analyses and shielding the operator from radiation.[1] MOX services requires the boron oxide (B_2O_3) content of the colemanite raw aggregate material to be determined prior to mixing and pouring of the sample concrete.

2.0 Experimental Procedure and Results

Ten samples of colemanite raw aggregate were delivered to Savannah River National Laboratory on July 22, 2013. Each sample weighed approximately one kilogram. Subsamples of the samples received on July 22, 2013 were ball milled, dried in an oven to remove moisture, and digested in triplicate using the ASTM method for trace metals analysis in limestone.[2] Aliquots of each sample were weighed in separate beakers and then 10 ml of HCl and 4 ml of HNO_3 were added. The acid mixture was heated at 95 °C for two hours in a beaker, with the sample covered with a watch glass. After heating was complete, the sample cooled for an additional 60 minutes to ensure complete boron dissolution. The sample was then diluted up to a final volume of 100 ml with deionized water. The samples were analyzed on the Agilent 730 Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES). Boron was calibrated using a High Purity NIST traceable standard (Lot 1204016), Appendix A: Boron Certificate of Analysis. An internal standard (yttrium) was used to compensate for matrix effects. The dissolution method prescribed in the ASTM method resulted in complete dissolution of the samples.

2.1 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

3.0 Results and Discussion

The digestion and analysis described in this report provides the elemental concentration of boron in the colemanite raw aggregate. An oxide conversion factor has to be applied to the raw data to calculate the concentration of boron oxide (B_2O_3) in the colemanite. The oxide conversion factor for boron to B_2O_3 is 3.2199. The elemental boron and boron oxide content for the ten colemanite samples are listed in Table 1. The average boron oxide content of the triplicate samples are also provided in Table 1.

Table 1. Boron and Boron Oxide Content for MOX Colemanite Samples.

Sample	Elemental Boron (wt%)	Boron Oxide (wt %)	Average B_2O_3 (wt %)
Colemanite #1 (A)	13.1	42.2	41.8
Colemanite #1 (B)	13.1	42.2	
Colemanite #1 (C)	12.7	40.9	
Colemanite #2 (A)	12.8	41.2	41.1
Colemanite #2 (B)	12.8	41.2	
Colemanite #2 (C)	12.7	40.9	
Colemanite #3 (A)	12.2	39.3	39.7
Colemanite #3 (B)	12.1	39.0	
Colemanite #3 (C)	12.7	40.9	
Colemanite #4 (A)	12.3	39.6	39.8
Colemanite #4 (B)	12.2	39.3	
Colemanite #4 (C)	12.6	40.6	
Colemanite #5 (A)	13.4	43.1	41.9
Colemanite #5 (B)	12.7	40.9	
Colemanite #5 (C)	12.9	41.5	
Colemanite #6 (A)	12.2	39.3	39.4
Colemanite #6 (B)	12.3	39.6	
Colemanite #6 (C)	12.2	39.3	
Colemanite #7 (A)	14.1	45.4	43.6
Colemanite #7 (B)	13.4	43.1	
Colemanite #7 (C)	13.1	42.2	
Colemanite #8 (A)	13.2	42.5	42.6
Colemanite #8 (B)	13.5	43.5	
Colemanite #8 (C)	13.0	41.9	
Colemanite #9 (A)	13.2	42.5	40.6
Colemanite #9 (B)	12.5	40.2	
Colemanite #9 (C)	12.1	39.0	
Colemanite #10 (A)	13.3	42.8	43.3
Colemanite #10 (B)	13.6	43.8	
Colemanite #10 (C)	13.4	43.1	

4.0 References

1. Wead, R., "Radiation Shielding and Fixed Neutron Absorber Panel Material and Inspection Requirements," DCS01-ZMJ-DS-SPE-M-19109-2, Revision 2, 2007.

2. "Major and Trace Elements in Limestone and Lime by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP) and Atomic Absorption (AA)," ASTM International, ASTM C 1301-95 (reapproved 2009).

Appendix A. Boron Certificate of Analysis

HIGH-PURITY STANDARDS
www.highpuritystandards.com
P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

Certificate of Analysis

Product Description:

Name:	Boron	Source Material:	Boric Acid
Part Number:	**1000 7-4**	Material Purity:	99.999%
Lot Number:	**1204016**	Matrix:	H_2O

Certified Value: 1000 µg/mL ± 3 µg/mL

The Certified value is based on gravimetric and volumetric preparation, and confirmed against SRM 3107 (lot number 070514) by inductively coupled plasma optical emission spectrometry (ICP-OES) using an internal laboratory-developed method. The uncertainty in the certified value is calculated for a 95% confidence interval and coverage factor *k* is about 2.

Density: 1.000 g/mL ± 0.002 g/mL @ 22.8°C

Uncertified Values:

Titration Value: 994.97 µg/mL

Trace Metal Impurity Scan: The data reported are based upon a scan of this specific lot at 1000 µg/mL via ICP analysis. The values are reported in µg/L.

Ag	<	0.02	Cu	<	0.1	Li	<	1	Rb	<	0.02	Th	<	0.02
Al	<	0.1	Dy	<	0.02	Lu	<	0.02	Re	<	0.02	Ti	<	0.02
As	<	0.05	Er	<	0.02	Mg	<	0.5	Rh	<	0.02	Tl	<	0.02
Au	<	0.02	Eu	<	0.02	Mn	<	0.5	Ru	<	0.02	Tm	<	0.02
B		M	Fe	<	5	Mo	<	0.02	Sb	<	0.02	U	<	0.1
Ba	<	0.1	Ga	<	0.02	Na	<	10	Sc	<	0.02	V	<	0.05
Be	<	0.02	Gd	<	0.02	Nb	<	0.02	Se	<	0.1	W	<	0.02
Bi	<	0.02	Ge	<	0.02	Nd	<	0.02	Si	<	5	Y	<	0.02
Ca	<	5	Hf	<	0.02	Ni	<	0.02	Sm	<	0.02	Yb	<	0.02
Cd	<	0.02	Ho	<	0.02	Os	<	0.02	Sn	<	1	Zn	<	0.1
Ce	<	0.02	In	<	0.02	Pb	<	0.05	Sr	<	0.02	Zr	<	0.02
Co	<	0.05	Ir	<	0.02	Pd	<	0.02	Ta	<	0.02			
Cr	<	0.1	K	<	1	Pr	<	0.02	Tb	<	0.02			
Cs	<	0.02	La	<	0.02	Pt	<	0.02	Te	<	0.02			

Preparation Information:

The standard solution is prepared using high purity materials and assayed by analytical methods for conformity prior to use. This standard was prepared using the methods developed at NIST for SRM Spectrometric Standard Solutions under appropriate laboratory conditions.

The matrix is 18 megaohm deionized water.

Stability of this product is based upon rigorous short term and long term testing of the solution for the certified value. This testing includes, but is not limited to, the effect of temperature and packaging on the product.

Intended Use:

This Certified Reference Material (CRM) is intended for use as a calibration standard for the quantitative determination of boron, calibration of instruments such as ICPOES, ICPMS, AAS and XRF, and validation of analytical methods. It also can be used in EPA, ASTM and other methods.

Lot No.: **1204016**
Rev. No.: 5.0.0
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Traceability Information:

The traceability of this standard is maintained through an unbroken chain of comparisons to appropriate standards with suitable procedure and measurement uncertainties. The maintenance of the base and derived units of International System of Units (SI) with traceability of measurement results (contemporary metrology) to SI ensures their comparability over time as follows.

a. **Standard Weight and Analytical Balance**
The standard weights (NBS weights Inventory No 20231A) are calibrated every two years by South Carolina Metrology Laboratory that is a participant in "NIST Weights and Measures Measurement Assurance Program" with a certificate of measurement traceability to NIST primary standards.
The balances are calibrated yearly by the ISO 17025 accredited metrology service, and are verified weekly by an in-house method using standard weights.

b. **Volumetric Device**
The calibration of volumetric vessels is checked annually using the NBS 602 method.

c. **Thermometer**
The standard thermometers are calibrated every year by the ISO 17025 accredited metrology service. The thermometers used in-house are verified against the standard thermometers yearly.

d. **Calibration Standards:**
The Calibration Standard is directly traceable to SRM 3100 Series Spectrometric Standard Solutions.

Packaging and Storage Conditions:

The standard is packaged in a pre-cleaned polyethylene bottle. To maintain the integrity of this product, the solution should be kept tightly capped and stored under normal laboratory conditions.

Refer to Material Safety Datasheet (MSDS) for hazardous information.

Expiration Information:

The expiry date is guaranteed to be valid for eighteen months from the shipping date provided. For this reason, standards from the same lot may have different expiration dates.

Preparation Date:	**February 9, 2012**
Shipped Date:	MAR 0 9 2012
Expiration Date:	SEP 0 9 2013
Certificate Issue Date:	**February 29, 2012**

Quality Information:

ACLASS Testing

ISO/IEC 17025:2005 Accreditation
Certificate Number AT-1529

ACLASS Calibration

ISO Guide 34:2009 (RMP) Accreditation
Certificate Number AR-1436

Vanny T. Yib,	**Kim-Phuong Tran**	**Theodore Rains, PhD**
Inorganic Laboratory Manager	**Quality Manager**	**President**

Lot No.: 1204016
Rev. No.: 5.0.0
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