Analytical Results for MOX Colemanite Concrete Samples Received on January 15, 2013

M.M. Reigel D.R. Best

February 2013

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

Keywords: *MOX Colemanite Concrete*

Retention: Permanent

Analytical Results for MOX Colemanite Concrete Samples Received on January 15, 2013

M.M. Reigel D.R. Best

February 2013

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.



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	Date
APPROVAL:	
E.N. Hoffman, Manager Engineering Process Development	Date
S.L. Marra, Manager Environmental & Chemical Process Technology Research Programs	Date

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 total density, partial hydrogen density, and partial boron density of the colemanite concrete.

SRNL received twelve samples of colemanite concrete for analysis on January 15, 2013. The average total density of each of the samples measured by the ASTM method C 642, the average partial hydrogen density was measured using method ASTM E 1311, and the average partial boron density of each sample was measured according to ASTM C 1301. The lower limits and measured values for the total density, hydrogen partial density, and boron partial density are presented in the following table. For all the samples tested, the total density and the hydrogen partial density met or exceeded the specified limit. All of the samples met or exceeded the boron partial density lower bound with the exception of samples G3-M11-2000-H, G3-M11-3000-M, and G5-M1-3000-H which are below the limit of 1.65E-01 g/cm³.

Sample ID	Вс	nsity Lower ound 'cm³]	Hydroge Density Lo [g/c	wer Bound	Boron Partial Density Lower Bound [g/cm³]		
	Limit Measured		Limit	Measured	Limit	Measured	
G3-M11-2000-B		2.03		7.12E-02		1.70E-01	
G3-M11-2000-M	1.00	2.04		7.25E-02	1.65E-01	1.79E-01	
G3-M11-2000-H		2.02	6.04E-02	7.73E-02		1.61E-01	
G3-M11-3000-B		2.00		6.65E-02		1.69E-01	
G3-M11-3000-M		2.02		7.33E-02		1.64E-01	
G3-M11-3000-H		2.06		7.26E-02		1.70E-01	
G4-M1-2000-B	1.88	2.03		6.81E-02		1.72E-01	
G4-M1-2000-M		2.03		6.85E-02		1.67E-01	
G4-M1-2000-H		2.05		6.88E-02		1.72E-01	
G5-M1-3000-B		2.03		7.47E-02		1.74E-01	
G5-M1-3000-M		2.03		7.42E-02		1.66E-01	
G5-M1-3000-H		2.03		7.24E-02		1.59E-01	

TABLE OF CONTENTS

LIST OF TABLES	vii
LIST OF ABBREVIATIONS	viii
1.0 Introduction	1
2.0 Experimental Results	1
2.1 Total Density	1
2.2 Partial Hydrogen Density	4
2.3 Boron Partial Density	6
3.0 Conclusions	7
4.0 References	8

LIST OF TABLES

Table 1-1. Acceptable Material Neutron Absorber Characteristics
Table 2-1. Mass measurements of duplicate samples after each treatment
Table 2-2. Calculated results using equations 1 – 7 for colemanite concrete samples received January 15, 2013
Table 2-3. Mass loss from 25 – 650 °C for samples received on January 15, 2013 5
Table 2-4. Boron results for colemanite concrete samples received January 15, 2013 6
Table 3-1. Lower Bounds and Average Measured Values for the Total Density, Hydrogen Partial Density, and Boron Partial Density for the Colemanite Concrete Samples Received January 15, 2013

LIST OF ABBREVIATIONS

ASTM American Society for Testing and Materials

DSC Differential Scanning Calorimetry

ICP Inductively Coupled Plasma

MFFF Mixed Oxide Fuel Fabrication Facility

OES Optical Emission Spectrometer

PSAL Process Science Analytical Laboratory
SRNL Savannah River National Laboratory

TGA Thermal Gravimetric Analysis

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. The properties listed in Table 1-1 are from Table 2.1.2.3 in Reference 1. Savannah River National Laboratory (SRNL) is tasked with measuring the properties of colemanite concrete identified in Table 1-1.

Material Type	Total Density Lower	Hydrogen Partial Density	Boron Partial Density		
	Bound (g/cm³)	Lower Bound (g/cm³)	Lower Bound (g/cm³)		
Borated Concrete (Colemanite)	1.88	6.04E-02	1.65E-01		

2.0 Experimental Results

Twelve samples of colemanite concrete were delivered to SRNL on January 15, 2013. All samples were cuboids with approximate dimensions of 160 mm x 40 mm x 40 mm. The samples arrived in zip top bags and each sample was wrapped in a moist towel.

2.1 Total Density

The total density of each sample was determined according to ASTM standard C 642-06 for determining the density of hardened concrete.³ The ASTM method was followed with the exception of sample size. Section 4.1 of the ASTM procedure specifies a sample size of approximately 800 grams. As the samples themselves weighed less than 550 grams, a reduced sample size was used. Duplicate samples for total density were obtained by cleaving pieces from the as-received sample. Mass measurements were taken after each treatment (Table 2-1) as outlined in the ASTM standard with the masses designated as A through D, where:

A = mass of oven-dried sample in air between 100 to 110 $^{\circ}$ C, g

B = mass of surface-dry sample in air after immersion, g

C = mass of surface-dry sample in air after immersion and boiling, g

D = apparent mass of sample in water after immersion and boiling, g

Using the calculations in the ASTM method, the following properties were calculated (Table 2-2):

Absorption after immersion,
$$\% = \frac{B-A}{A} \times 100$$
, (1)

Absorption after immersion and boiling,
$$\% = \frac{C-A}{A} \times 100$$
, (2)

Bulk density,
$$dry = \frac{A}{C-D} \times \rho$$
, (3)

Bulk density after immersion =
$$\frac{B}{C-D} \times \rho$$
, (4)

Bulk density after immersion and boiling =
$$\frac{c}{c-p} \times \rho$$
, (5)

Apparent density =
$$\frac{A}{A-D} \times \rho$$
, (6)

Volume of permeable pore space =
$$\frac{C-A}{C-D} \times 100$$
 (7)

Table 2-1. Mass measurements of duplicate samples after each treatment

Sample ID Run Number		Dry (A) [g]	Saturated (B) [g]	Boiled (C) [g]	Suspended (D) [g]		
Date	•	1/22/2013	1/24/2012	1/29/2013	1/29/2013		
G2 M11 2000 P	1	4.894	5.819	5.767	2.892		
G3-M11-2000-B	2	11.754	14.041	13.911	6.991		
C2 M11 2000 M	1	11.338	13.439	13.456	6.842		
G3-M11-2000-M	2	10.592	12.411	12.228	6.196		
G3-M11-2000-H	1	12.292	14.413	14.438	7.356		
G3-WH1-2000-П	2	9.482	11.454	11.320	5.634		
G3-M11-3000-B	1	9.549	11.653	11.627	5.759		
ОЗ-МП1-3000-Б	2	16.267	19.685	19.620	9.842		
G3-M11-3000-M	1	6.203	7.529	7.435	3.678		
G3-M11-3000-M	2	5.690	6.738	6.614	3.318		
G3-M11-3000-H	1	13.829	16.604	16.529	8.367		
G3-W11-3000-H	2	10.857	12.733	12.640	6.523		
G4-M1-2000-B	1	6.626	8.008	7.948	3.962		
G4-M1-2000-D	2	11.303	13.318	13.360	6.856		
G4-M1-2000-M	1	7.953	8.734	8.770	4.449		
G4-WH-2000-WI	2	10.181	12.088 12.102		6.170		
G4-M1-2000-H	1	9.856	11.503	11.500	5.924		
U4-M1-2000-H	2	7.538	8.994	8.927	4.496		
G5-M1-3000-B	1	8.570	10.315	10.238	5.169		
G3-1411-3000-D	2	14.021	16.766	16.740	8.457		
G5-M1-3000-M	1	15.022	17.814	17.888	9.161		
G3-M11-3000-M1	2	8.560	10.337	10.352	5.221		
G5-M1-3000-H	1	13.480	15.856	15.772	8.057		
G3-M1-2000-H	2	10.980	13.356	13.396	6.705		

The density, ρ , used in these calculations is that of water, (1 g/cm³). The results of the calculations performed with Equations 1-7 are tabulated and averaged in Table 2-2.

Table 2-2. Calculated results using equations 1 – 7 for colemanite concrete samples received January 15, 2013

Sample ID	Run Number	Eq. 1 [%]	Eq. 2 [%]	Eq. 3 [g/cm ³]	Eq. 4 [g/cm ³]	Eq. 5 [g/cm ³]	Eq. 6 [g/cm ³]	Eq. 7 [%]	Average (Eq. 4) [g/cm ³]
C2 M11 2000 D	1	18.901	17.840	1.702	2.024	2.006	2.444	30.362	2.03
G3-M11-2000-B	2	19.457	18.349	1.699	2.029	2.010	2.468	31.166	2.03
G3-M11-2000-M	1	18.531	18.681	1.714	2.032	2.035	2.522	32.026	2.04
G3-WH11-2000-WI	2	17.173	15.446	1.756	2.057	2.027	2.409	27.120	2.04
G3-M11-2000-H	1	17.255	17.460	1.736	2.035	2.039	2.490	30.306	2.02
G5-M11-2000-П	2	20.797	19.382	1.668	2.015	1.991	2.464	32.323	2.02
G3-M11-3000-B	1	22.034	21.756	1.627	1.986	1.981	2.519	35.405	2.00
ОЗ-М11-3000-Б	2	21.012	20.609	1.664	2.013	2.007	2.532	34.289	2.00
G3-M11-3000-M	1	21.377	19.860	1.651	2.004	1.979	2.456	32.788	2.02
G3-WH1-3000-WI	2	18.418	16.243	1.726	2.044	2.006	2.398	28.035	2.02
G3-M11-3000-H	1	20.067	19.526	1.694	2.034	2.025	2.532	33.081	2.06
ОЗ-М11-3000-П	2	17.279	16.422	1.775	2.081	2.066	2.505	29.145	2.00
G4-M1-2000-B	1	20.857	19.946	1.663	2.009	1.994	2.488	33.163	2.03
G4-W11-2000-D	2	17.827	18.194	1.738	2.048	2.054	2.542	31.622	2.03
G4-M1-2000-M	1	9.820	10.273	1.840	2.021	2.030	2.270	18.907	2.03
G4-M1-2000-M	2	18.731	18.871	1.716	2.037	2.040	2.538	32.384	2.03
G4-M1-2000-H	1	16.711	16.679	1.768	2.063	2.062	2.507	29.483	2.05
G4-W11-2000-H	2	19.315	18.428	1.701	2.030	2.014	2.478	31.346	2.03
G5-M1-3000-B	1	20.362	19.464	1.691	2.035	2.020	2.520	32.907	2.03
ОЗ-М11-3000-В	2	19.578	19.392	1.693	2.024	2.021	2.520	32.827	2.03
G5-M1-3000-M	1	18.586	19.079	1.721	2.041	2.050	2.563	32.840	2.03
G2-1411-2000-141	2	20.759	20.930	1.668	2.015	2.018	2.564	34.920	2.03
G5-M1-3000-H	1	17.626	17.001	1.747	2.055	2.044	2.486	29.707	2.03
ОЭ-МП-3000-П	2	21.639	21.999	1.641	1.996	2.002	2.568	36.103	2.03

2.2 Partial Hydrogen Density

The hydrogen partial density of the colemanite concrete was determined using the ASTM method for determining volatile content using thermogravimetric analysis. ⁴ The ASTM E 1131-08 defines highly volatile matter as components that will volatilize <200°C and medium volatile matter as components that will degrade or volatilize in the range 200 - 750 °C. In an evaluation of the thermal decomposition of colemanite, Waclawska et al, determined that the release of water from colemanite was complete at 600 °C and that melting and crystallization of a calcium borate phase occurred above 650 °C.5 A Netzsch STA 409 Luxx, which couples Differential Scanning Calorimetry (DSC) with Thermal Gravimetric Analysis (TGA), was used for determining the partial hydrogen density of the colemanite concrete samples. After loading the sample, the chamber was purged with nitrogen at 60 ml/min prior to heating. Triplicate samples were heated at 5 °C/min up to 650 °C in a flowing nitrogen atmosphere of 60 ml/min. The results for each of the twelve samples received January 15, 2013 are shown in Table 2-3. The initial mass loss is associated with the free water from the mix. The second mass loss beginning at approximately 400 °C is due to the thermal decomposition of the colemanite. Table 2-3 summarizes the mass change and hydrogen partial density for all twelve samples. The mass loss graphs for all twelve samples are in Appendix A.

In order to calculate the hydrogen partial density, it is assumed the average total mass loss for each sample in Table 2-3 is due to water, both free and from the decomposition of colemanite. The hydrogen partial density is calculated from the average mass loss (Table 2-3) and density of the sample (Table 2-2) as shown in equations 8 - 12. Sample G3-M11-2000-B is used as the example calculation.

Moles
$$H_2O$$
 in 100 g concrete = $\frac{mass\ fraction\ H_2O}{MW\ H_2O} = \frac{31.41\ \frac{g\ H_2O}{g\ concrete}}{18.015\ g\ mol\ H_2O} = 1.74\ mol\ H_2O$, (8)

$$\frac{g H}{Mole \, H_2 O} = \ 2.016 \, g \, H, \tag{9}$$

$$g \ H \ in \ 100 \ g \ concrete = 1.74 \ mol \ H_2O \times 2.016 \ g \ H = 3.52 \ g \ H,$$
 (10)

Volume concrete =
$$\frac{mass\ concrete}{density\ concrete} = \frac{100\ g\ concrete}{2.03\frac{g}{cm^3}} = 49.35\ cm^3\ concrete,$$
 (11)

$$H \ density \ \frac{g}{ml} = \frac{mass \ H}{volume \ concrete} = \frac{3.52 \ g \ H}{49.35 \ ml \ concrete} = 7.12 E - 02 \ \frac{g \ H}{ml \ Concrete}. \tag{12}$$

Table 2-3. Mass loss from 25 – 650 $^{\circ} C$ for samples received on January 15, 2013

Sample ID	Mass Loss (%) 25 - 175 °C	Mass Loss (%) 300 - 600 °C	Total Mass Loss (%)	Average Total Mass Loss (%)	Total Hydrogen Partial Density (g/cm³)		
G3-M11-2000-B-1	15.43	11.90	28.38				
G3-M11-2000-B-2	18.95	11.32	32.34	31.41	7.12E-02		
G3-M11-2000-B-3	21.62	9.33	33.52				
G3-M11-2000-M-1	26.08	9.42	37.42				
G3-M11-2000-M-2	11.15	14.80	26.78	31.69	7.25E-02		
G3-M11-2000-M-3	17.62	11.30	30.86				
G3-M11-2000-H-1	19.64	8.67	29.97				
G3-M11-2000-H-2	24.79	8.17	34.95	34.13	7.73E-02		
G3-M11-2000-H-3	25.67	9.52	37.48				
G3-M11-3000-B-1	9.50	17.66	28.00				
G3-M11-3000-B-2	19.07	10.68	30.23	29.72	6.65E-02		
G3-M11-3000-B-3	18.64	10.14	30.92				
G3-M11-3000-M-1	18.05	11.40	31.03				
G3-M11-3000-M-2	18.80	11.89	32.64	32.36	7.33E-02		
G3-M11-3000-M-3	22.00	9.17	33.40				
G3-M11-3000-H-1	14.88	14.62	30.69				
G3-M11-3000-H-2	19.76	9.88	32.92	31.53	7.26E-02		
G3-M11-3000-H-3	19.15	9.53	30.97				
G4-M1-2000-B-1	13.42	10.90	25.75				
G4-M1-2000-B-2	16.09	19.19	34.21	29.98	6.81E-02		
G4-M1-2000-B-3	16.15	11.77	29.98				
G4-M1-2000-M-1	18.37	10.55	31.03				
G4-M1-2000-M-2	14.15	14.09	29.71	30.14	6.85E-02		
G4-M1-2000-M-3	15.17	13.08	29.69				
G4-M1-2000-H-1	17.96	10.47	30.39				
G4-M1-2000-H-2	15.82	11.26	29.13	30.05	6.88E-02		
G4-M1-2000-H-3	17.15	11.53	30.63				
G5-M1-3000-B-1	17.74	10.97	30.24				
G5-M1-3000-B-2	21.97	9.20	33.50	32.88	7.47E-02		
G5-M1-3000-B-3	22.72	9.94	34.89				
G5-M1-3000-M-1	21.11	9.92	32.92				
G5-M1-3000-M-2	16.55	12.58	30.82	32.68	7.42E-02		
G5-M1-3000-M-3	21.68	10.53	34.29				
G5-M1-3000-H-1	21.52	8.54	32.19				
G5-M1-3000-H-2	22.15	9.32	33.17	31.92	7.24E-02		
G5-M1-3000-H-3	18.42	10.11	30.39				

2.3 Boron Partial Density

Subsamples of the samples received on January 15, 2013 were crushed, dried in an oven to remove moisture, and digested in triplicate using the ASTM method for trace metals analysis in limestone. Aliquots of each sample were weighed in separate beakers and then 10 ml of HCl and 4 ml of HNO₃ were added. The acid mixture was heated at 85 °C for 60 minutes on a hotplate, 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 B: 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. Table 2-4 is the analytical results of the dissolution of colemanite concrete using the prescribed ASTM method.

Average Boron **Average Partial Boron** Sample ID Content [wt %] Density [g/cm³] G3-M11-2000-B 8.40 1.70E-01 G3-M11-2000-M 8.74 1.79E-01 G3-M11-2000-H 7.96 1.61E-01 G3-M11-3000-B 1.69E-01 8.46 G3-M11-3000-M 8.10 1.64E-01 G3-M11-3000-H 8.27 1.70E-01 G4-M1-2000-B 8.49 1.72E-01 G4-M1-2000-M 8.24 1.67E-01 G4-M1-2000-H 8.40 1.72E-01 G5-M1-3000-B 8.59 1.74E-01 G5-M1-3000-M 8.16 1.66E-01 G5-M1-3000-H 7.86 1.59E-01

Table 2-4. Boron results for colemanite concrete samples received January 15, 2013

The average partial boron density for each sample is calculated using the calculated colemanite concrete density for each sample in Table 2-2. An example calculation for the partial density of sample G3-M11-2000-B is shown in equations 13 – 15:

Mass boron in 100 g concrete from Table
$$2-4 = 8.40 g$$
 (13)

Volume concrete =
$$\frac{mass\ concrete}{density\ concrete} = \frac{100\ g\ concrete}{2.03\frac{g}{cm^3}} = 49.35\ cm^3\ concrete$$
 (14)

Boron density
$$\frac{g}{ml} = \frac{mass B}{volume \ concrete} = \frac{8.40 \ g \ B}{49.35 \ cm^3} = 1.70E - 01 \ \frac{g \ boron}{cm^3 \ Concrete}$$
 (15)

Comparing the results in Table 2-4 to the acceptability limits in Table 1-1, samples G3-M11-2000-H, G3-M11-3000-M, and G5-M1-3000-H are below the limit (1.65E-01 g/cm³) for the average partial boron density.

3.0 Conclusions

The lower limits and measured values for the total density, hydrogen partial density, and boron partial density are presented in Table 3-1. For all the samples tested, the total density and the hydrogen partial density met or exceeded the lower bounds specified in Reference 1. All of the samples met or exceeded the boron partial density lower bound with the exception of samples G3-M11-2000-H, G3-M11-3000-M, and G5-M1-3000-H which are below the limit of 1.65E-01 g/cm³.

Table 3-1. Lower Bounds and Average Measured Values for the Total Density, Hydrogen Partial Density, and Boron Partial Density for the Colemanite Concrete Samples Received January 15, 2013

Sample ID	Bo	nsity Lower ound cm³]	Hydroge Density Lo [g/c	wer Bound	Boron Partial Density Lower Bound [g/cm³]		
	Limit	Measured	Limit	Measured	Limit	Measured	
G3-M11-2000-B		2.03		7.12E-02		1.70E-01	
G3-M11-2000-M		2.04		7.25E-02		1.79E-01	
G3-M11-2000-H	1.88	2.02	6.045.00	7.73E-02	1.65E-01	1.61E-01	
G3-M11-3000-B		2.00		6.65E-02		1.69E-01	
G3-M11-3000-M		2.02		7.33E-02		1.64E-01	
G3-M11-3000-H		2.06		7.26E-02		1.70E-01	
G4-M1-2000-B		2.03	6.04E-02	6.81E-02		1.72E-01	
G4-M1-2000-M		2.03		6.85E-02		1.67E-01	
G4-M1-2000-H		2.05		6.88E-02		1.72E-01	
G5-M1-3000-B		2.03		7.47E-02		1.74E-01	
G5-M1-3000-M		2.03		7.42E-02		1.66E-01	
G5-M1-3000-H		2.03		7.24E-02		1.59E-01	

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. "Equipment Calibration Services / Material Testing," WTA-040-8b, Section 9 (q), April 13, 2012.
- 3. "Standard Test Method for Density, Absorption, and Voids in Hardened Concrete," ASTM International, ASTM C 642-06.
- 4. "Standard Test Method for Compositional Analysis by Thermogravimetry," ASTM International, ASTM E 1131-08.
- 5. Waclawska, I., Stoch, L., Paulik, J., and Pauliik, F., "Thermal Decomposition of Colemanite," *Thermochimica Acta*, **126**, 307-18 (1988).
- 6. "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).

SRNL-STI-2013-0007	8
Revision	0

Appendix A. TGA Graphs for Samples Received January 15, 2013

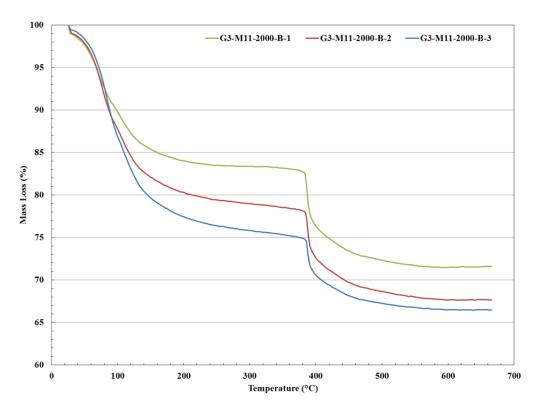


Figure A-1. TGA curve showing the mass loss for sample G3-M11-2000-B

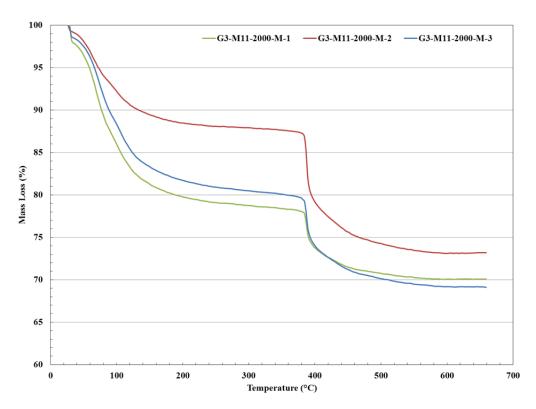


Figure A-2. TGA curve showing the mass loss for sample G3-M11-2000-M

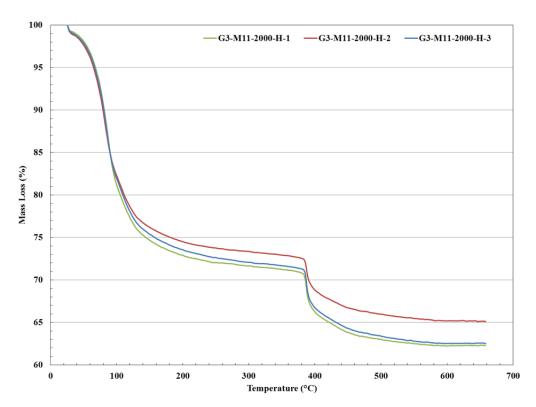


Figure A-3. TGA curve showing the mass loss for sample G3-M11-2000-H

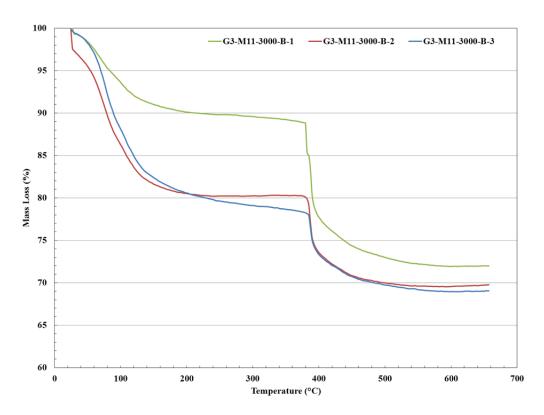


Figure A-4. TGA curve showing the mass loss for sample G3-M11-3000-B

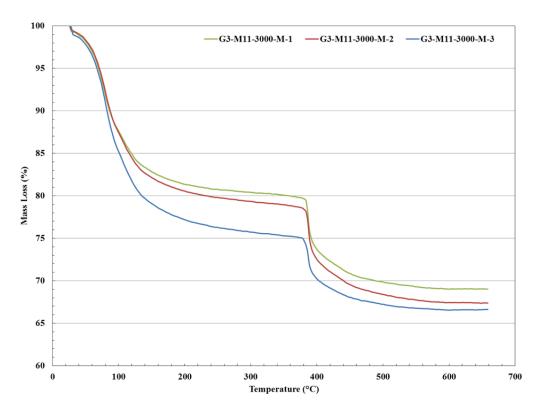


Figure A-5. TGA curve showing the mass loss for sample G3-M11-3000-M

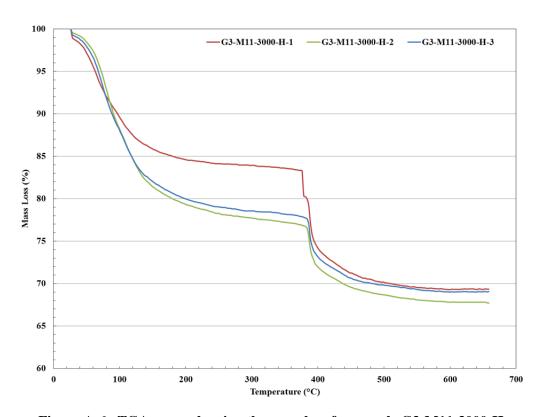


Figure A-6. TGA curve showing the mass loss for sample G3-M11-3000-H

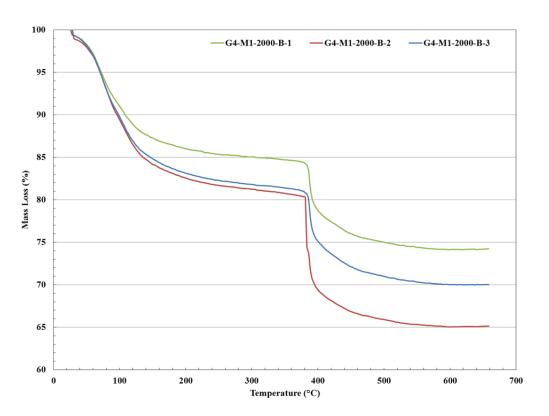


Figure A-7. TGA curve showing the mass loss for sample G4-M1-2000-B

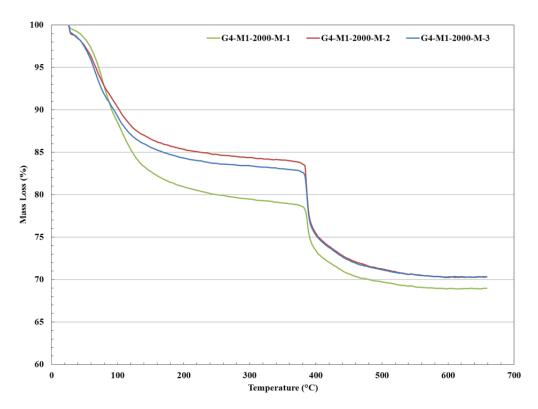


Figure A-8. TGA curve showing the mass loss for sample G4-M1-2000-M

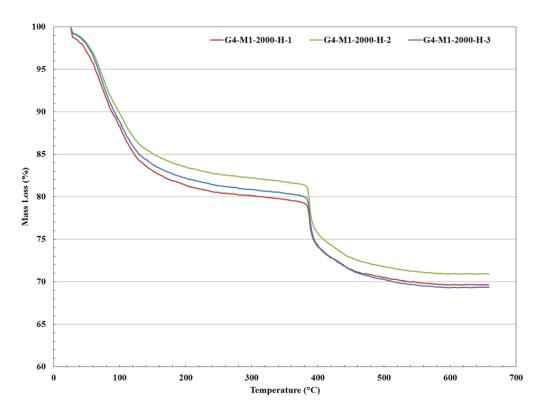


Figure A-9. TGA curve showing the mass loss for sample G4-M1-2000-H

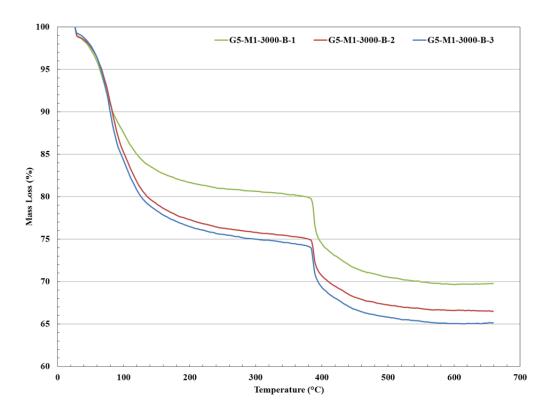


Figure A-10. TGA curve showing the mass loss for sample G5-M1-3000-B

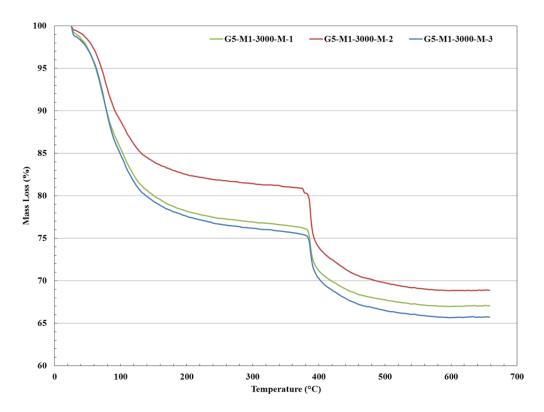


Figure A-11. TGA curve showing the mass loss for sample G5-M1-3000-M

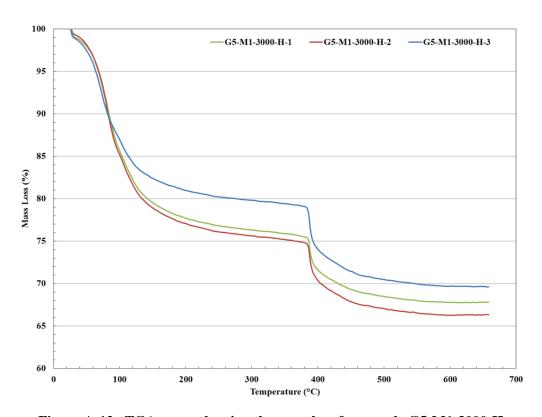


Figure A-12. TGA curve showing the mass loss for sample G5-M1-3000-H

Appendix B. Boron Certificate of Analysis



P.O. Bex 41727 Charleston, 9C 29423 Phore (040) 707-7900 Fax (843) 767-7906

Certificate of Analysis

Product Description:

 Name:
 Boron
 Source Material:

 Part Number:
 1000 7-4
 Material Purity:

 Lot Number:
 1204016
 Matrix:

Boric Acid 99.999% H₂O

Certified Value:

 $1000 \mu g/mL \pm 3 \mu g/mL$

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 $\mu g/mL$ via ICP analysis. The values are reported in $\mu g/L$.

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	Ti	<	0.02
Au	<	0.02	Eu	<	0.02	Mn	<	0.5	Ru	<	0.02	Tm	<	0.02
В		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 Page 1 of 2

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.

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.

Volumetric Device

The calibration of volumetric vessels is checked annually using the NBS 602 method.

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.

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

Quality Information:



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

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

Way I. y

Kim-Phuong Tran

Thomas C Prime

Vanny T. Yib, Inorganic Laboratory Manager

Kim-Phuong Tran Quality Manager

Theodore Rains, PhD

President

NOTICE: HPS products are intended for laboratory use only. All products should be handled and used by trained professional personnel. The responsibility for the safe handling and use of these products sents solely with the buyer and/or user. The data and information as stated was furnished by the manufacturer of the product. The information provided in this certificate penalise only to the lot number specified. None of the information provided in this certificate may be used, reproduced or transmitted in any form or by any means without written approval from High Parity Standards.

Lot No.: 1204016 Rev. No.: 5.0.0 Page 2 of 2

Distribution:

D.R. Best, 999-W

T.B. Brown, 773-A

D.R. Click, 773-A

A.D. Cozzi, 999-W

S. D. Fink, 773-A

K.M. Fox, 999-W

C. C. Herman, 999-W

E.N. Hoffman, 999-W

S. L. Marra, 773-A

F. M. Pennebaker, 773-42A

M.M. Reigel, 999-W

J.A. Spear, 235-11F

J. P. Vaughan, 773-41A