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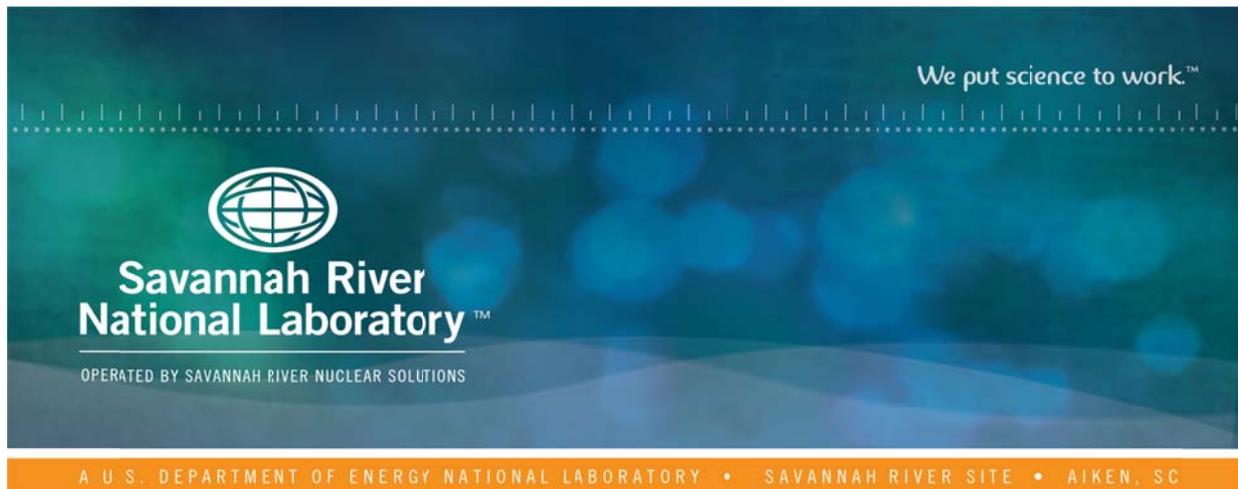
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# Technical Basis for the Reduction of the Maximum Temperature TGA-MS Analysis of Oxide Samples from the 3013 Destructive Examination Program

J. H. Scogin

March 24, 2016

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## **PREFACE OR ACKNOWLEDGEMENTS**

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The author would also like to thank the Materials Identification and Selection Working Group for their comment during the preparation of this report.

## **EXECUTIVE SUMMARY**

Thermogravimetric analysis with mass spectroscopy of the evolved gas (TGA-MS) is used to quantify the moisture content of materials in the 3013 destructive examination (3013 DE) surveillance program. Salts frequently present in the 3013 DE materials volatilize in the TGA and condense in the gas lines just outside the TGA furnace. The buildup of condensate can restrict the flow of purge gas and affect both the TGA operations and the mass spectrometer calibration. Removal of the condensed salts requires frequent maintenance and subsequent calibration runs to keep the moisture measurements by mass spectroscopy within acceptable limits, creating delays in processing samples.

In this report, the feasibility of determining the total moisture from TGA-MS measurements at a lower temperature is investigated. A temperature of the TGA-MS analysis which reduces the complications caused by the condensation of volatile materials is determined. Analysis shows that an excellent prediction of the presently measured total moisture value can be made using only the data generated up to 700 °C and there is a sound physical basis for this estimate.

It is recommended that the maximum temperature of the TGA-MS determination of total moisture for the 3013 DE program be reduced from 1000 °C to 700 °C. It is also suggested that cumulative moisture measurements at 550 °C and 700°C be substituted for the measured value of total moisture in the 3013 DE database. Using these raw values, any of predictions of the total moisture discussed in this report can be made.

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## LIST OF ABBREVIATIONS

3013	DOE-STD-3013-2012
CI	Confidence Interval
DE	Destructive examination
MS	Mass Spectroscopy
SS	sum of squares
Stdev	Standard Deviation
TGA	Thermogravimetric Analysis

## 1.0 Introduction

Thermogravimetric analysis with mass spectroscopy of the evolved gas (TGA-MS) is used to quantify the moisture content of materials in the 3013 destructive examination (3013 DE) surveillance program. The TGA measures the amount of mass lost from a sample while being heated, and the mass spectrometer (MS) can quantify the amount of moisture released from the sample. The moisture content of a sample of material is estimated by the total moisture, defined as the cumulative MS moisture measured at 85 minutes, which is the time required for the TGA to reach 1000 °C plus a short time after the end of the run.

The inventory of 3013 containers is divided into several bins or classes based on the anticipated ability to challenge the 3013 storage container. Most of the recent 3013 DE samples are from the pressure and corrosion bin and consequently have high halide salt concentrations. These halide salts have relatively high vapor pressures at elevated temperatures and readily volatilize at high end of the TGA temperature range, 1000 °C.<sup>1,2,3</sup> As the volatilized salts condense in the gas lines just outside the TGA furnace, they can restrict the flow of purge gas which can affect both the TGA operations and the mass spectrometer calibration.

Removal of the condensed salts requires frequent maintenance and calibration runs to keep the moisture measurements by mass spectroscopy within acceptable limits. Unfortunately, the frequent maintenance and calibration runs create delays in processing samples. During the delays in running samples, the samples can absorb moisture from the atmosphere, biasing the moisture measurement when it is finally made.

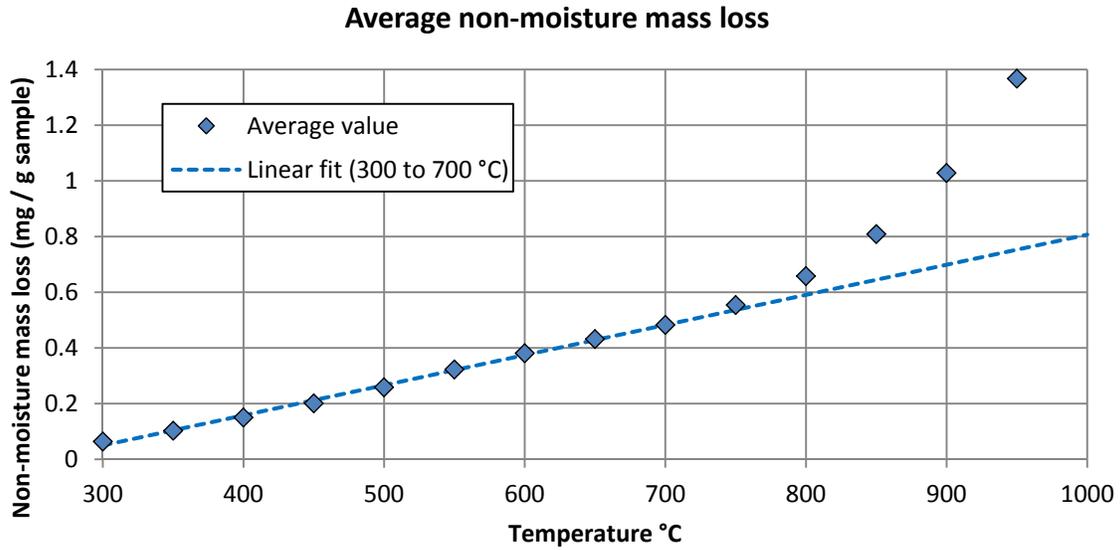
As operational delays in the TGA-MS analysis are largely a consequence of maintenance downtime and recalibration due to the volatilization of salts at high temperature, it would seem reasonable to try to reduce the downtime by operating under less severe conditions if reasonable estimates of the total moisture could be made from TGA-MS measurements at lower temperatures. This task is easily divided into two separate questions: what is the highest temperature that will minimize volatilization of salts or other condensable materials, and can the estimation of the moisture content of a sample currently made by the total moisture measurement be made using only data collected to a lower temperature?<sup>2</sup>

This report will discuss reducing the maximum temperature of TGA-MS analysis to reduce the complications caused by the condensation of materials which are volatile at high temperatures. The data used to develop this discussion will be the entire set of FY2014 and FY2015 TGA-MS analyses performed for the 3013 DE program. For each 3013 DE there is both initial moisture and final moisture samples run in duplicate. One initial moisture sample was run in triplicate, giving a total of 65 TGA-MS runs (Appendix B). Analysis will be presented to demonstrate that at 700 °C only a small amount of condensable material is volatilized. It will be shown that an excellent prediction of the measured total moisture value can be made using only the data generated up to 700 °C and there is a sound physical basis for this estimate.

As part of this investigation, the use of TGA measurements only was evaluated for use as predictor of total moisture. While these measurements showed some utility, the confidence interval of these predictions was six times larger than the predictions using TGA-MS data. The TGA only predictions will not be presented in this report.

### 1.1 Determination of a lower temperature for TGA-MS moisture measurement

For the first estimate of determining a lower temperature, the non-moisture mass loss was examined. This is calculated by subtracting the measured moisture by the MS from the total weight loss measured by the TGA at each temperature. The averaged results of this calculation for all the samples are shown in Figure 1. The dashed line is a linear fit of the averaged data from 300 °C to 700 °C. Most of the non-moisture mass loss up to 700 °C is CO<sub>2</sub> and NO and not condensable species. Since CO<sub>2</sub> and NO continue to

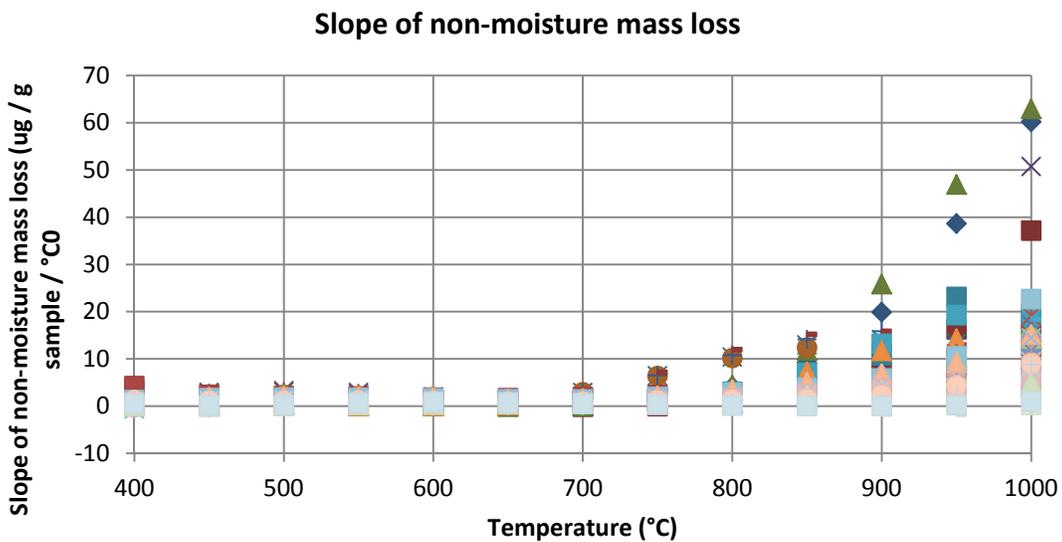


**Figure 1: Average non-moisture mass loss**

evolve throughout the entire temperature range, the amount of volatile salts is more likely to be approximated by the difference between the data points and the dashed line. It can be seen that by 800 °C, the deviation from the dashed line is clear, and there is a slight increase in generation by 750 °C.

Examining the slope of the non-moisture mass loss provides a clearer picture. As seen in Figure 2, beginning at a temperature of 700 °C, a few samples have an increase in the rate of non-moisture mass loss. By 750 °C, several samples have an increased generation rate of non-moisture mass loss.

It is important to account for the fact that after the TGA-MS has reached the target temperature; the sample temperature will continue to increase for some period of time. If the analysis were to go to 750 °C, the sample temperature might reach 775 °C before it started to cool. To avoid significant volatilization



**Figure 2: Slope of non-moisture mass loss**

and subsequent condensation of non-moisture material, the TGA-MS analysis should only run to 700 °C. While most of the materials which are volatile above 700 °C are salts, any material which will condense in the gas transfer line has the potential to create instrument problems.

### 1.2 Prediction of total moisture from moisture data up to 700 °C

The term moisture at temperature is used to refer to the cumulative moisture (mass 18) measured by the mass spectrometer up to that temperature; e.g. the moisture at 400 °C. It should be noted that the moisture at 1000 °C is less than the total moisture, as it does not include the small amount of moisture detected by the MS between the time 1000 °C is reached and 85 minutes, and the total moisture itself is only an estimate of the moisture content of the sample.

After examining a number of potential indicators up to 700 °C, the best indicator of the total moisture was found to be cumulative moisture measured up to 700 °C. In Figure 3, the moisture at 700 °C for all measured FY2014 and FY2015 samples is plotted as a function of total moisture. (A larger version of Figure 3 is provided in Figure 11 in Appendix A.) It can be seen that moisture at 700 °C is a close approximation of the total moisture. To more clearly show the variation between the two measured values, the residuals are shown in Figure 4. The 95% confidence interval of the residuals is shown by the dashed lines.

Using the value of moisture at 700 °C with no adjustment provides a reasonable estimate of total moisture. But even with such good agreement between the moisture at 700 °C and the total moisture, it is possible to get a better prediction, which has physical meaning, of the total moisture using only the data collected up to 700 °C.

Table 1 shows the best possible fits for several different methods of predicting the total moisture from the moisture data collected up to 700 °C. Adding a constant (0.151 mg / g sample) to the moisture at 700 °C simply centers the residuals around zero and keeps the same width of the 95% confidence interval as the unadjusted data. Incremental improvements of the 95% confidence interval of the residuals are obtained by using a proportional fit and a linear fit. However, a significant reduction in the 95% confidence

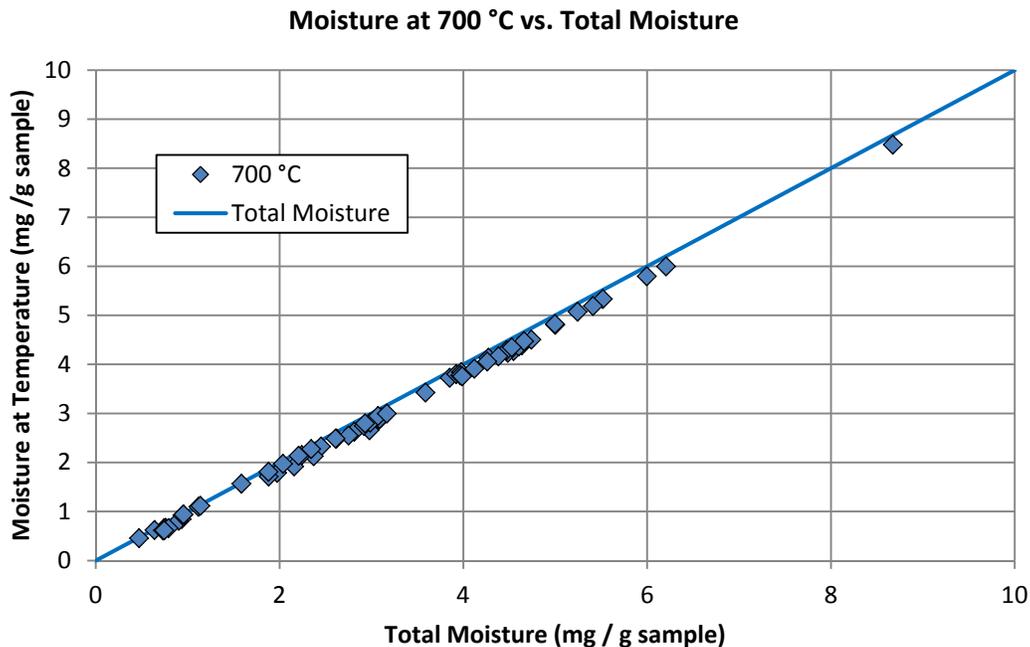
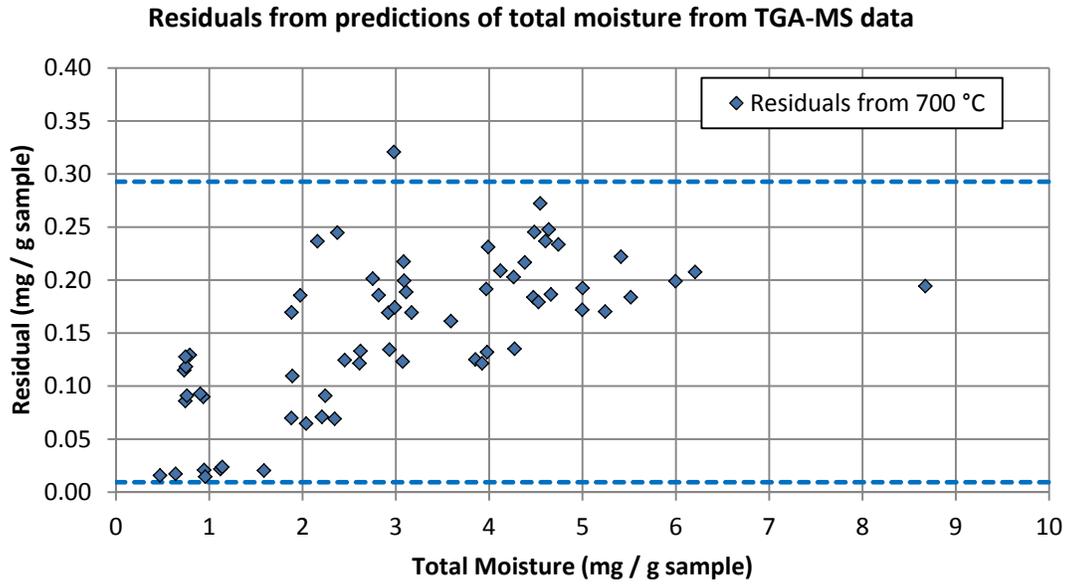


Figure 3: Moisture at 700 °C against Total Moisture



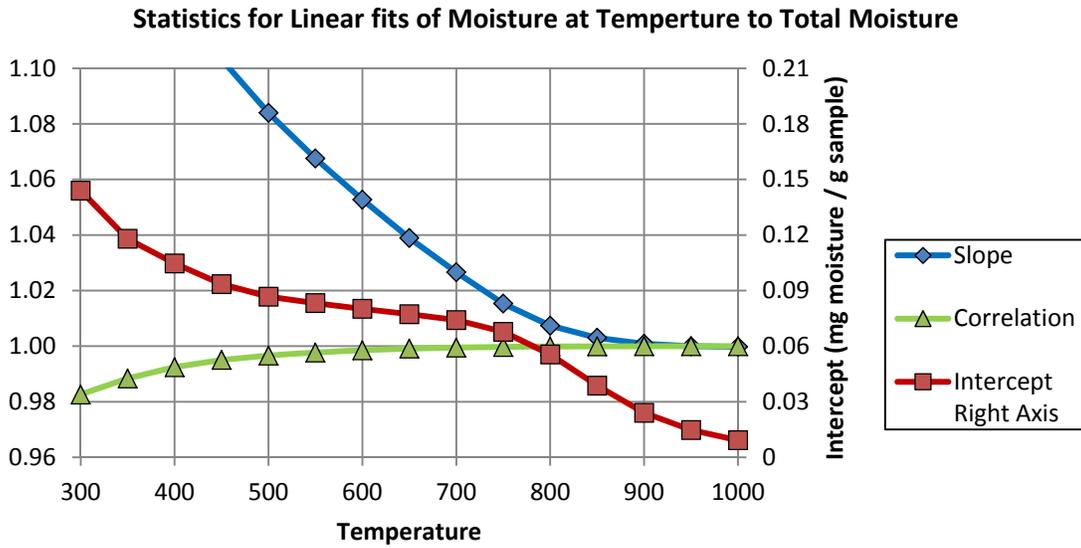
**Figure 4: Residuals of moisture at 700 °C from total moisture**

interval is found with a least squares fit using segmented moisture of two temperatures ranges, from start (around 25 °C) to 550 °C and from 550 °C to 700 °C. To determine if such additional complexity is warranted, the fit using the segmented moisture must first be shown to have a physical meaning.

A study of the intercepts of the linear fit by temperature provides insight to the physical nature of the system. Figure 5 shows the slope, intercept, and correlation coefficient for the linear fits of cumulative moisture at various temperatures to the total moisture. As expected, the slopes of the fits are greater than one, and decrease to unity as the temperature is raised and a higher percentage of the total moisture is collected. As the temperature increases, the intercepts of the linear fits at first decrease, level out between

Best Fits	No adjustment	Constant	Proportional	Linear	Segmented
SS residual (mg / g sample) <sup>2</sup>	1.8042	0.3221	0.2784	0.1951	0.05760
Stdev of the residual (mg / g sample)	0.1679	0.07095	0.06595	0.05565	0.03048
95% CI of the residual (mg / g sample)	+0.2928, +0.0093	+0.1417, -0.1417	+0.1494, -0.1142	+0.1112, -0.1112	+0.0609, -0.0609
correlation	-	-	-	0.9989	0.9997
regression constant c (mg / g sample)	0	0.1510	0	0.07276	0.07158
temp 1 (°C)	700	700	700	700	Start to 550
regression coefficient a	1	1	1.0459	1.0269	0.9958
temp 2 (°C)					550 to 700
regression coefficient b					1.7581
Equation for $\hat{m}$	$m_{700}$	$m_{700} + c$	$a m_{700}$	$a m_{700} + c$	$a m_{25 \text{ to } 550} + b m_{550 \text{ to } 700} + c$

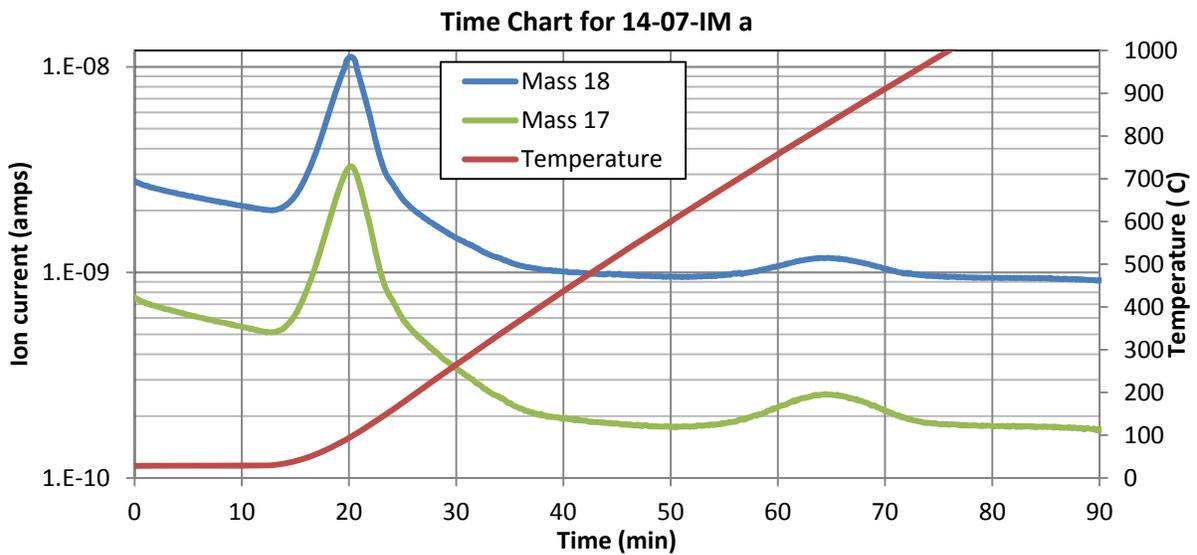
**Table 1 Fit statistics for moisture at temperature vs. total moisture**



**Figure 5: Statistics from linear fit of moisture at temperature to total moisture**

500 °C and 700 °C, and then decrease again after 700 °C. The behavior of the intercepts suggests that there are two types of moisture, loosely bound and strongly bound.<sup>4,5,6,7</sup> The loosely bound water is mostly removed by 550 °C, the strongly bound water comes off between 700 °C and 950 °C.

This behavior is clearly shown by 14-07-IM a in Figure 6, where loosely bound moisture comes off in a large peak at 90 °C which returns to the baseline by 400 °C, and the strongly bound moisture is released in a much smaller peak between 700 ° and 950 °C. A logarithmic scale has been used so that the tightly bound moisture peak is easily seen. In this example, the tightly bound moisture is only 3.2% of the total moisture. While many other samples show similar behavior, not all samples have such clear distinction between the loosely bound and strongly bound moisture.



**Figure 6: TGA-MS showing loosely and strongly bound moisture**

The concept of tightly and loosely bound moisture on plutonium dioxide is widely supported in the literature. Haschke and Ricketts have proposed that the first monolayer is chemisorbed on the surface of the oxide and that succeeding monolayers are physisorbed.<sup>8</sup> Stakebake has shown that high purity oxide similar to 3013 material has a second region of mass loss between 700 and 1000 °C.<sup>9</sup> Other analyses of TGA data of Pu oxide show that small amounts of water are detected above 600 °C.<sup>5-6</sup> While salts are known to absorb significant amounts of moisture, the literature indicates that NaCl and KCl are largely anhydrous and that MgCl<sub>2</sub> and CaCl<sub>2</sub> are converted to oxide by 500 °C.<sup>4, 10-13</sup> The observed loosely bound moisture corresponds to the physisorbed moisture and the waters of hydration on the salt materials, and the strongly bound moisture corresponds to the chemisorbed moisture on the PuO<sub>2</sub> surfaces.

Understanding the two moisture types helps us interpret the regression coefficients of the segmented fit. Let us look at the fit in terms of the loosely and strongly bound moisture. The coefficient for the moisture released in the lower temperature range up to 550 °C, which corresponds to the loosely bound moisture, is almost unity (0.996), while the regression constant (0.0716 mg/g) and the datum from 550 °C to 700 °C ( $1.76 * (m_{700} - m_{550})$ ) provide an estimate of the strongly bound moisture. The best split for the segmented data is between 550 °C and 600 °C, which is the point at which all the loosely bound moisture has been released, and the strongly bound moisture has not yet begun to come off. A graphical description of how the segmented fit generates the prediction of the total moisture is shown in Figure 7.

A graphical comparison of the unadjusted moisture at 700 °C and the segmented fit as predictors of the total moisture is shown in Figure 8. (A larger version of this plot is available as Figure 12 in Appendix A.) The residuals from the two predictions are shown in Figure 9. It is clear that the segmented fit has removed much of the scatter present in the 700 °C data. The dashed lines represent the 95% confidence interval of the residuals for each prediction. For comparison, the residuals of the linear and the segmented fit are shown in Figure 10.

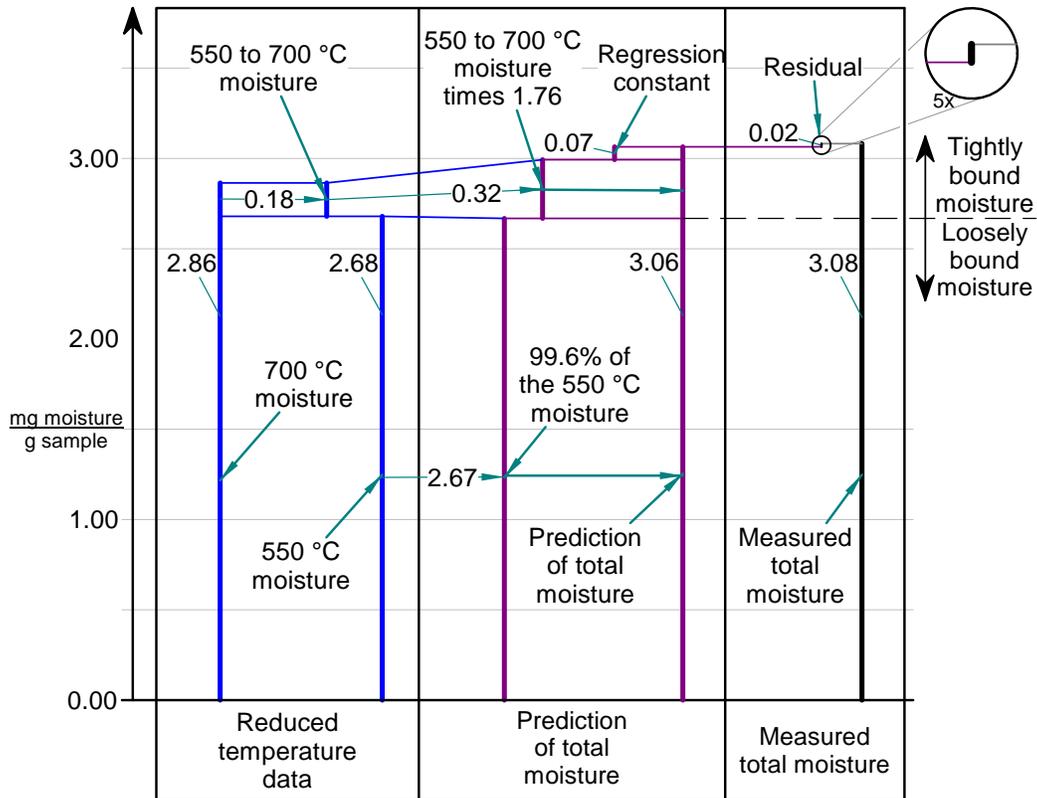


Figure 7: Graphical description of segmented fit using data from sample 15-06 IM a

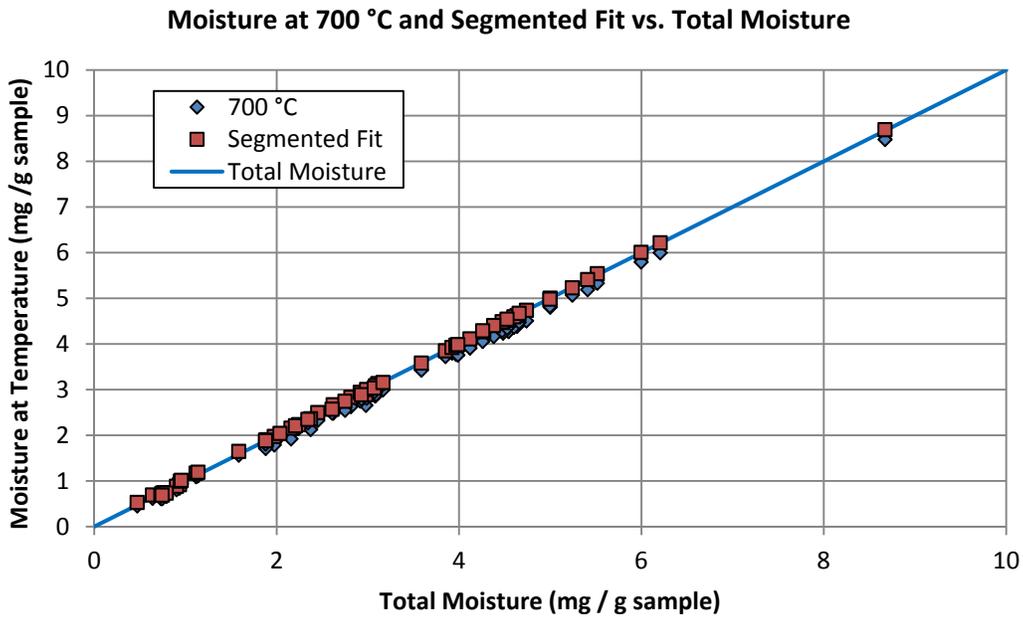


Figure 8: Comparison of predictions of total moisture

Using the segmented prediction, the error in the prediction of total moisture is now small when compared to the other sources of error in the measurement. It would be reasonable to assert that operating the TGA-MS up to 700 °C rather than 1000 °C has negligible effect on the ability to measure the total moisture of the sample.

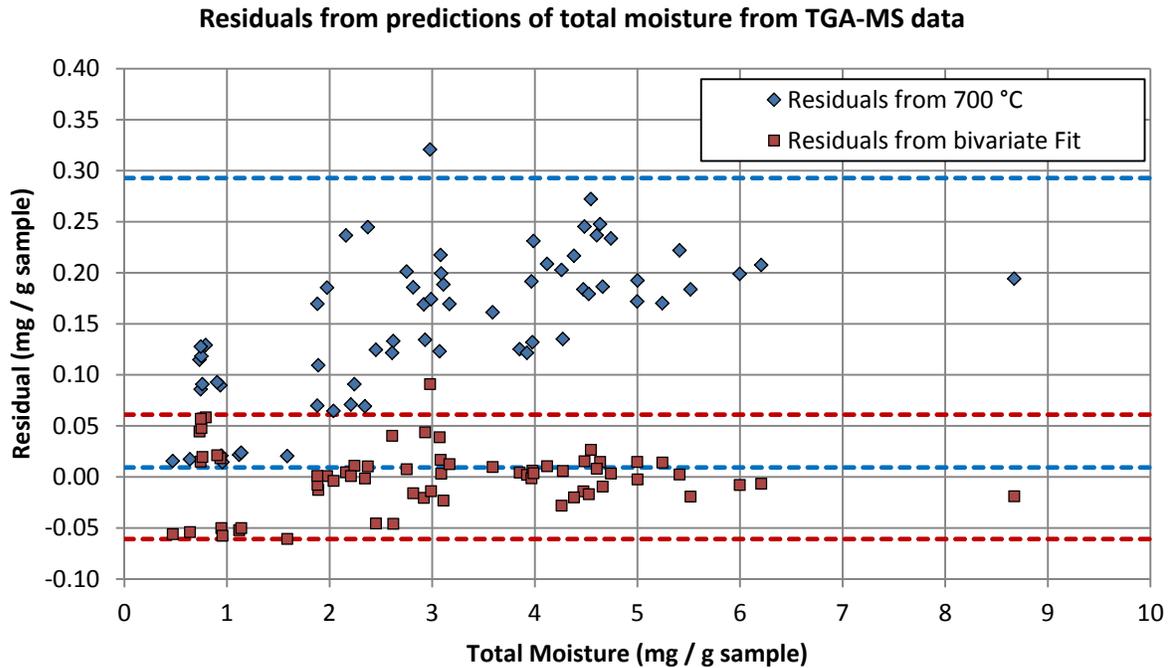
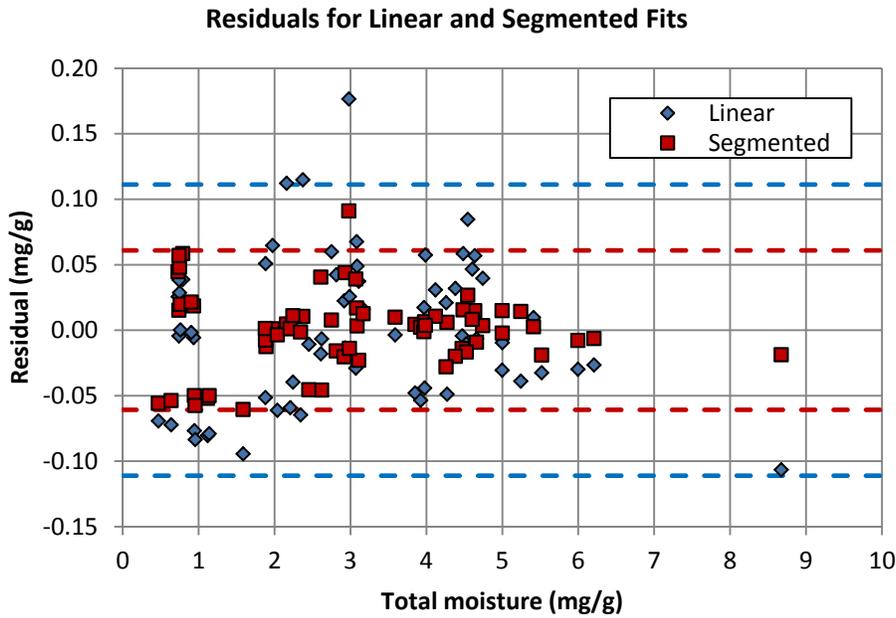


Figure 9: Residuals from predictions of total moisture



**Figure 10: Residuals from Linear and Segmented Fits**

## 2.0 Conclusions and Recommendations

Reducing the final temperature of the TGA-MS moisture determination from 1000 °C to 700 °C will reduce the volatilization of salts from the 3013 DE sample and their subsequent condensation outside the heated zone of the furnace. This in turn will reduce the time spent in maintenance and recalibration of the instruments, which will allow for more timely measurements. The reduction in temperature will result in almost no loss of precision in the total moisture measurement. The error introduced by predicting the total moisture from moisture values up to 700 °C is small compared with other uncertainties in the measurement. The 95% confidence interval of the moisture at 700 °C used as a predictor of total moisture is [+0.00093 to +0.029] weight percent. The 95% confidence interval of the segmented fit is [-0.0061 to +0.0061] weight percent, which is smaller by a factor of 2.3 and centered around zero. By comparison, the 95% confidence interval of the linear fit is [-0.011 to +0.011] weight percent.

It is recommended that the maximum temperature of the TGA-MS determination of total moisture for 3013 DE program be reduced from 1000 °C to 700 °C. It is also suggested that cumulative moisture measurements at 550 °C and 700°C be substituted for the measured value of total moisture in the 3013 DE database. Using these raw values, any of predictions of the total moisture discussed in this report can be made. After further analysis of historical data, it is possible improved predictions of the total moisture can be made using these raw values.

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Appendix A. Large Figures

Moisture at Temperature 700 °C vs. Total Moisture

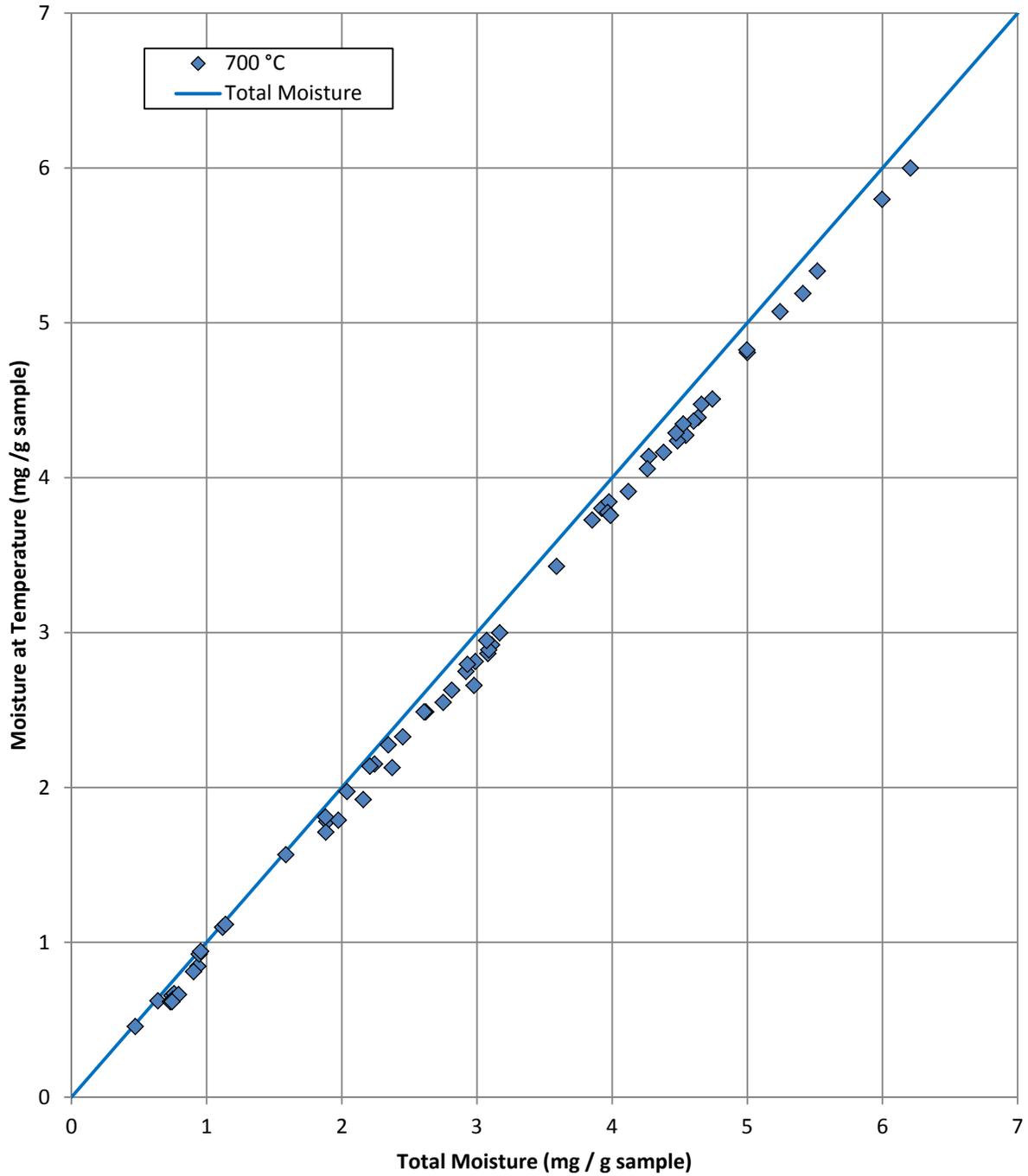
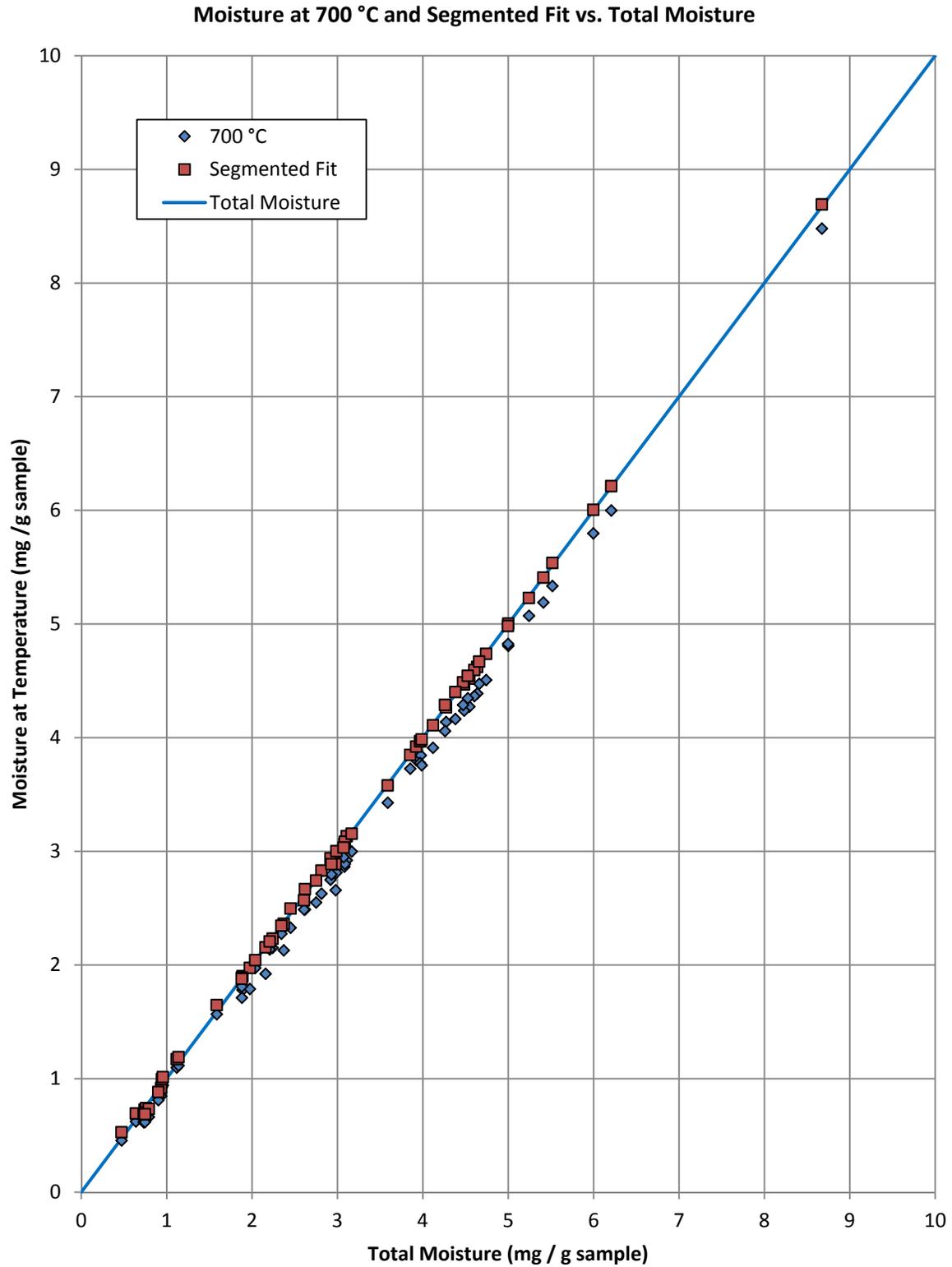


Figure 11: Moisture at 700 °C vs. Total Moisture (Large Version)



**Figure 12: Comparison of predictions of total moisture (Large Version)**

**Appendix B: Data Summary**

Sample ID	Date	Sample Mass (g)	Total MS Moisture (mg/g)	Calibration Factor (Area/mg)	MS Mass 18 Area		
					550 °C	700 °C	Total (85 min)
14-01 IM B492 a	2015-01-22	2.647948	0.4718	5.09	6.1130	6.1463	6.3584
14-01 IM B492 b	2015-01-22	2.713118	0.6394	5.09	8.5523	8.5907	8.8300
14-01 FM B494 a	2015-02-18	2.662694	0.9447	5.09	12.4619	12.5194	12.8036
14-01 FM B494 b	2015-02-18	2.581992	0.9837	5.09	12.2928	12.3681	12.9280
14-02 IM B498 a	2015-01-15	3.027084	2.9789	5.09	37.5325	40.9536	45.8978
14-02 IM B498 b	2015-01-15	3.111736	2.7507	5.09	37.6204	40.3783	43.5680
14-02 FM B500 a	2015-01-16	2.68204	4.7412	5.09	58.3584	61.5349	64.7253
14-02 FM B500 b	2015-01-16	2.6597	4.6028	5.09	55.9930	59.1057	62.3126
14-03 IM B495 a	2015-03-10	2.648108	2.1587	5.09	22.9299	25.9068	29.0968
14-03 IM B495 b	2015-03-10	2.892204	2.3724	5.09	28.0068	31.3209	34.9253
14-03 FM B497 a	2015-03-12	2.593016	2.9185	7.35	49.1606	52.3965	55.6218
14-03 FM B497 b	2015-03-12	2.643336	2.9874	7.35	51.3882	54.6560	58.0407
14-04 IM B502 a	2015-04-10	2.770068	5.4110	5.66	77.8678	81.3549	84.8371
14-04 IM B502 b	2015-04-10	2.71032	4.6605	5.66	65.7584	68.6323	71.4942
14-04 FM B504 a	2015-03-18	2.629692	4.4725	5.66	61.0164	63.8317	66.5691
14-04 FM B504 b	2015-03-18	2.72012	4.5266	5.66	64.0489	66.9295	69.6910
14-05 IM B505 a	2015-04-20	2.618848	5.9967	5.66	82.8396	85.9373	88.8865
14-05 IM B505 b	2015-04-20	2.572358	6.2066	5.443	80.9181	83.9939	86.9014
14-05 IM B505 c	2015-06-18	1.465138	8.6728	5.443	65.7632	67.6132	69.1631
14-05 FM B507 a	2015-06-18	2.453594	4.3811	5.443	52.4188	55.6144	58.5089
14-05 FM B507 b	2015-06-23	2.475528	4.2606	5.443	51.5600	54.6746	57.4084
14-06 IM B508 a	2015-06-23	2.516562	2.6091	5.5125	34.1452	34.5074	36.1946
14-06 IM B508 b	2015-06-25	2.627426	2.2421	5.5125	30.8278	31.1561	32.4739
14-06 FM B510 a	2015-06-25	2.503766	2.9299	5.5125	38.0301	38.5835	40.4392
14-06 FM B510 b	2015-06-30	2.547144	3.0715	5.5125	40.9421	41.3981	43.1276
14-07 IM B 511 a	2015-07-07	2.881236	1.8794	5.5125	28.6417	28.7392	29.8505
14-07 IM B511 b	2015-07-08	2.797374	2.0381	5.5125	30.3291	30.4298	31.4279
14-07 FM B513 a	2015-07-08	2.766378	2.2075	5.5125	32.4365	32.5811	33.6639
14-07 FM B513 b	2014-02-11	2.8194	2.3437	5.5125	35.1774	35.3500	36.4260
14-08 IM B514 a	2014-02-11	2.513136	3.1677	5.5125	39.7601	41.5363	43.8840
14-08 IM B514 b	2014-02-24	2.603628	3.5886	5.5125	47.4165	49.1898	51.5058
14-08 FM B516 a	2014-02-24	2.612328	4.9972	5.5125	67.4888	69.4845	71.9620
14-08 FM B516 b	2014-03-05	2.614064	5.2425	5.5125	71.0930	73.0900	75.5449
14-09 IM B517 a	2014-03-05	2.985772	0.7310	5.5125	10.1122	10.1402	12.0319
14-09 IM B517 b	2014-03-12	2.918706	0.7507	5.5125	10.1413	10.1726	12.0784
14-09 FM B519 a	2014-03-12	2.444408	0.7915	5.5125	8.8868	8.9229	10.6658
14-09 FM B519 b	2014-03-25	2.539088	0.7445	5.5125	8.6021	8.6321	10.4204
15-01-IM B520 a	2014-03-25	2.853118	1.1180	5.3343	16.5504	16.6846	17.0158
15-01-IM B520 b	2014-04-07	2.956726	1.1391	5.3343	17.4503	17.5912	17.9654
15-01-FM B522 a	2014-04-07	2.037132	1.8891	5.3343	18.5114	19.3379	20.5281
15-01-FM B522 b	2014-05-08	2.069954	1.5864	5.3343	17.0613	17.2910	17.5167
15-02-IM B523 a	2014-05-08	2.92472	0.7423	5.3343	10.1996	10.2391	11.5812
15-02-IM B523 b	2014-05-09	2.81328	0.7595	5.3343	9.9879	10.0339	11.3982
15-02-FM B525 a	2014-05-09	2.98619	0.9352	5.3343	13.3978	13.4663	14.8977

Sample ID	Date	Sample Mass (g)	Total MS Moisture (mg/g)	Calibration Factor (Area/mg)	MS Mass 18 Area		
					550 °C	700 °C	Total (85 min)
15-02-FM B525 b	2014-06-05	2.89975	0.9034	5.3343	12.4732	12.5380	13.9742
15-03-IM B526 a	2014-06-05	2.565922	1.9743	5.3343	22.3212	24.4825	27.0227
15-03-IM B526 b	2014-06-11	2.67554	1.8814	5.3343	22.3182	24.4307	26.8519
15-03-FM B528 a	2014-06-09	2.529848	3.1092	5.3343	36.7143	39.4110	41.9583
15-03-FM B528 b	2014-06-09	2.586092	2.8139	5.3343	33.7028	36.2540	38.8174
15-04-IM B529 a	2014-06-30	2.427522	3.8518	5.3343	47.1579	48.2567	49.8778
15-04-IM B529 b	2014-06-30	2.416682	3.9234	5.3343	47.9343	49.0104	50.5780
15-04-FM B531 a	2014-07-02	2.20093	4.2723	5.3343	47.4192	48.5710	50.1581
15-04-FM B531 b	2014-07-02	2.218606	3.9764	5.3343	44.4084	45.4966	47.0594
15-05-IM B532 a	2014-07-08	2.360916	2.6207	5.3343	29.3844	31.3275	33.0041
15-05-IM B532 b	2014-07-08	2.463926	2.4513	5.3343	28.7146	30.5801	32.2182
15-05-FM B534 a	2014-07-14	2.435514	5.0004	4.2616	47.9501	49.9010	51.9001
15-05-FM B534 b	2014-07-14	2.530026	5.5179	4.2616	55.3415	57.5118	59.4934
15-06-IM B536 a	2014-07-16	2.537732	3.0815	4.2616	28.9755	30.9742	33.3260
15-06-IM B536 b	2014-07-16	2.604626	3.0879	4.2616	30.0720	32.0614	34.2753
15-06-FM B538 a	2014-07-21	2.519632	4.1198	4.2616	39.9822	41.9950	44.2375
15-06-FM B538 b	2014-07-21	2.612054	3.9676	4.2616	40.0310	42.0313	44.1653
15-07-IM B539 a	2014-07-30	2.47629	3.9877	4.2616	37.2663	39.6413	42.0819
15-07-IM B539 b	2014-07-30	2.67576	4.5457	4.2616	45.8586	48.7294	51.8351
15-07-FM B541 a	2014-08-05	2.381388	4.6366	4.2616	42.1510	44.5403	47.0544
15-07-FM B541 b	2014-08-05	2.382418	4.4833	4.2616	40.6847	43.0274	45.5185

**Distribution:**

T. B. Brown, 773-A  
D. A. Crowley, 773-43A  
D. E. Dooley, 773-A  
S. D. Fink, 773-A  
D. T. Hobbs, 773-A  
E. N. Hoffman, 999-W  
F. M. Pennebaker, 773-42A  
B. J. Wiedenman, 773-42A  
Records Administration (EDWS)

J. M. Duffey, 773-A  
M. L. Crowder, 773-A  
K. P. Crapse, 773-A  
F. F. Fondeur, 773-A  
P. M. Almond, 773-A  
D. R. Best, 772-F  
J. E. Laurinat, 703-41A  
N. M. Askew, 703-41A  
L. E. Roy, 703-41A

B. L. Garcia-Diaz, 999-2W  
K. A. Dunn, 773-41A  
G. T. Chandler, 773-A  
K. E. Zeigler, 773-41A

E. R. Hackney, 705-K  
S. J. Hensel, 705-K  
B. V. Nguyen, 705-K  
J. W. McClard, 703-H

L. A. Worl, LANL  
T. J. Venetz, Hanford  
D. C. Riley, LLNL