Analysis of Soils and Sediments for Loss on Ignition

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Method Summary

The following standard operating procedure (SOP) describes the analysis of soils and sediments for loss on ignition (LOI) by the U.S. Geological Survey's Mercury Research Laboratory (MRL). Sample is weighed into aluminum boats and heated to 550°C for two hours. The percent of sample mass lost following heating is reported as LOI. One sample out of every 14 analyzed is weighed in triplicate to assess method precision.

Laboratory Safety

Analysts who use the MRL must have read, understood, and signed the Chemical Hygiene Plan for the MRL prior to potential exposure to any chemicals. The analyst must have a thorough understanding of the required safety protocols for the lab chemicals prior to their use of the lab. Adequate personal protection equipment such as safety glasses, gloves, and chemical resistant clothing must be worn when exposure to hazardous chemicals are possible. Caution should always be exercised; chemicals are present in the laboratory and often in use by other analysts. Hazardous chemicals should only be handled by adequately trained personnel under a high volume fume hood with extreme caution.

The analysis of LOI involves extremely high temperatures, and due caution should be exercised. Adequate personal protection equipment such as safety glasses, heat shielding gloves, and chemical resistant clothing must be worn when required.

Sample Analysis

Unless otherwise specified, solid samples are lyophilized ("freeze-dried" under vacuum while frozen) to a consistent weight and homogenized (via ball mill, coffee grinder/food processor, or mortar/pestle) prior to analysis. The samples should appear to be well pulverized and mixed to a consistent composition before weighing. A typical analysis contains up to 64 samples, with one sample out of every 14 weighed in triplicate

1. Arrange an adequate number of aluminum boats onto a tray. The maximum number of boats the furnace can accommodate is 64.

2. Using a ball point pen, number each aluminum boat on the tag. Make sure that you press hard enough to leave an imprint; the ink will not be visible following combustion.

3. Weigh 100 – 500 mg of sample into each aluminum boat. Make sure to record into the appropriate data sheet the vial ID, aluminum tray number, empty aluminum boat mass, and the mass of the aluminum boat with the added sample.

4. Carefully transfer the boats to the furnace, place in the bottom, and heat at 550°C for two hours. During the combustion, open the adjacent lab door several inches and turn on the fume hood to provide ventilation out of the building.

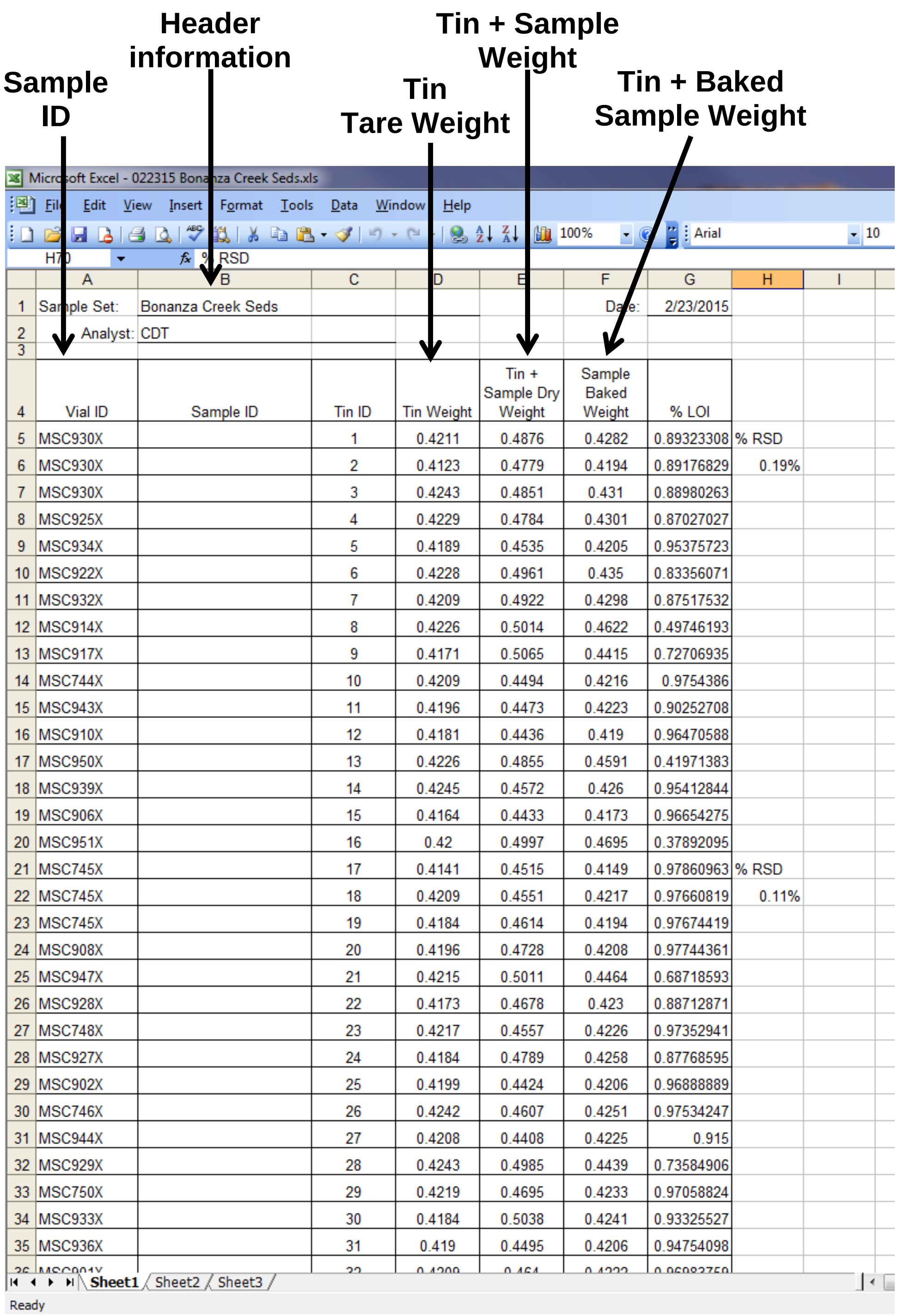
5. After two hours, turn off the furnace and allow the samples to cool. Carefully remove the samples.

6. Weigh the combusted sample and record the combusted weight into the data sheet.

Quality Assurance and Control Objectives

A successful run must meet the following criteria. The relative standard deviation of the triplicate analysis must be < 10%, and all values for LOI must be positive. If the analysis fails either of these criteria, check that the combusted sample mass was entered into the correct position in the data sheet and/or reweigh the samples (if possible). If this does not correct the failure, repeat the entire analytical batch as that the data is likely compromised.

APPENDIX 1. Example of a completed LOI sample setup sheet.



	A	B	C	D	E	F	G	H
1	Sample Set:	Bonanza Creek Seds				Date:	2/23/2015	
2	Analyst:	CDT						
3								
4	Vial ID	Sample ID	Tin ID	Tin Weight	Tin + Sample Dry Weight	Sample Baked Weight	% LOI	
5	MSC930X		1	0.4211	0.4876	0.4282	0.89323308	% RSD
6	MSC930X		2	0.4123	0.4779	0.4194	0.89176829	0.19%
7	MSC930X		3	0.4243	0.4851	0.431	0.88980263	
8	MSC925X		4	0.4229	0.4784	0.4301	0.87027027	
9	MSC934X		5	0.4189	0.4535	0.4205	0.95375723	
10	MSC922X		6	0.4228	0.4961	0.435	0.83356071	
11	MSC932X		7	0.4209	0.4922	0.4298	0.87517532	
12	MSC914X		8	0.4226	0.5014	0.4622	0.49746193	
13	MSC917X		9	0.4171	0.5065	0.4415	0.72706935	
14	MSC744X		10	0.4209	0.4494	0.4216	0.9754386	
15	MSC943X		11	0.4196	0.4473	0.4223	0.90252708	
16	MSC910X		12	0.4181	0.4436	0.419	0.96470588	
17	MSC950X		13	0.4226	0.4855	0.4591	0.41971383	
18	MSC939X		14	0.4245	0.4572	0.426	0.95412844	
19	MSC906X		15	0.4164	0.4433	0.4173	0.96654275	
20	MSC951X		16	0.42	0.4997	0.4695	0.37892095	
21	MSC745X		17	0.4141	0.4515	0.4149	0.97860963	% RSD
22	MSC745X		18	0.4209	0.4551	0.4217	0.97660819	0.11%
23	MSC745X		19	0.4184	0.4614	0.4194	0.97674419	
24	MSC908X		20	0.4196	0.4728	0.4208	0.97744361	
25	MSC947X		21	0.4215	0.5011	0.4464	0.68718593	
26	MSC928X		22	0.4173	0.4678	0.423	0.88712871	
27	MSC748X		23	0.4217	0.4557	0.4226	0.97352941	
28	MSC927X		24	0.4184	0.4789	0.4258	0.87768595	
29	MSC902X		25	0.4199	0.4424	0.4206	0.96888889	
30	MSC746X		26	0.4242	0.4607	0.4251	0.97534247	
31	MSC944X		27	0.4208	0.4408	0.4225	0.915	
32	MSC929X		28	0.4243	0.4985	0.4439	0.73584906	
33	MSC750X		29	0.4219	0.4695	0.4233	0.97058824	
34	MSC933X		30	0.4184	0.5038	0.4241	0.93325527	
35	MSC936X		31	0.419	0.4495	0.4206	0.94754098	
36	MSC901X		32	0.4209	0.464	0.4222	0.96983759	

Appendix 2. Definition of equations.

$$\text{Loss on Ignition} = \frac{\left(\begin{pmatrix}\text{Mass of Tin}\\ \text{with Sample}\end{pmatrix} - \begin{pmatrix}\text{Tin Tare}\\ \text{Weight}\end{pmatrix}\right) - \left(\begin{pmatrix}\text{Mass of Tin}\\ \text{with Baked Sample}\end{pmatrix} - \begin{pmatrix}\text{Tin Tare}\\ \text{Weight}\end{pmatrix}\right)}{\begin{pmatrix}\text{Mass of Tin}\\ \text{with Sample}\end{pmatrix} - \begin{pmatrix}\text{Tin Tare}\\ \text{Weight}\end{pmatrix}}$$

$$\begin{matrix}\text{Percent Relative}\\ \text{Standard Deviation}\end{matrix} = \frac{\begin{pmatrix}\text{Standard Deviation of}\\ \text{Triplicate Loss on Ignition}\\ \text{In Sample}\end{pmatrix}}{\begin{pmatrix}\text{Mean of Triplicate}\\ \text{Loss on Ignition}\\ \text{In Sample}\end{pmatrix}} \times 100$$