Standard Operating Procedure for:

Organic Matter in Sediment, Loss on Ignition Method (2030R02 Org Matter LOI.doc)

Missouri State University

and

Ozarks Environmental and Water Resources Institute (OEWRI)

Prepared by: _____ Date: _____
OEWRI Quality Assurance Coordinator

Approved by: _____ Date: _____
OEWRI Director

Table of Contents

1 Identification of the test method

The determination of organic matter in sediment using the Loss on Ignition method.

2 Applicable matrix or matrices

This method is suitable for use with sediment and soil samples.

3 Detection Limit

The detection limit for this procedure is approximately 1%.

4 Scope of the test method

This standard operating procedure provides the Missouri State University (MSU) laboratory personnel with guidance on the procedure for determining organic matter in sediment samples using the Loss on Ignition (LOI) method.

5 Summary of test method

Sediment samples are dried in a 105ºC oven to remove moisture. Pre-washed crucibles are placed in 105ºC oven for 4 hours to remove all moisture. 5-grams of samples are placed in the porcelain crucibles and the pre-burn total weight is recorded. The loaded crucibles are placed in 105ºC oven for 2 hours to remove all moisture. These samples are then placed in a 600ºC muffle furnace for 8 hours to incinerate the organic matter in the sediment. After 8 hours the samples are re-weighed and the difference is recorded. If measuring for calcium in the sample, the samples are then placed inside of the muffle furnace for 1 hour at 1000°C. Samples are then cooled and weighed for loss on ignition percentages.

6 Definitions

6.1 Analytical batch: The set of samples processed at the same time

6.2 Field duplicate (FD): Two samples taken at the same time and place under identical circumstances and that are treated identically throughout field and laboratory procedures. Analysis of field duplicates indicates the precision associated with sample collection, preservation, and storage as well as laboratory procedures.

6.3 Laboratory duplicate (LD): Two aliquots of the same environmental sample treated identically throughout a laboratory analytical procedure. Analysis of laboratory duplicates indicates precision associated with laboratory procedures but not with sample collection, preservation or storage procedures.

6.4 Method detection limit (MDL) -- The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero.

7 Interferences

Insufficient drying will cause the retention of water in the sediment sample with a result of an incorrect mass being recorded for the sample mass.

8 Health and safety

This analysis involves handling sediment samples that may contain live microorganisms and therefore pose some threat of infection. Laboratory personnel who are routinely exposed to such sediment samples are encouraged to protect themselves from sediment borne illnesses by wearing clean disposable gloves and washing their hands frequently. A dust mask can be worn to protect against dust from the dried sediment.

9 Personnel qualifications

Laboratory and field personnel shall have a working knowledge of this analytical procedure and will have received training from an MSU employee knowledgeable of the proper sample analysis procedures.

10 Equipment and supplies

10.1 Oven: set to 104 ± 1ºC

10.2 Furnace: set to 600ºC and 1000° C

10.3 Analytical balance: Capable of weighing to the nearest 0.01g.

10.4 Porcelain crucibles: to contain a 5g sample and 1g standards. Caution: do not touch crucibles with fingers, use tongs.

11 Reagents and standards

Calcite and limestone standards are located in the bottom drawer of the cabinet underneath the muffle furnaces. These standards should be used with all samples being examined over 950°C with the Organic Matter Loss on Ignition method. Anything under 950°C does not require a standard. Standards are used to check the procedures and data collected from this method.

12 Sample collection, preservation, shipment and storage

Samples can be collected by a variety of approved methods (Methods for sample collection are provided in the Sediment Collection SOP). Samples will be placed into plastic bags and labeled. There are no special provisions for shipment. Sample bags should be opened and placed into 60º C oven immediately upon return to lab.

13 Quality control

13.1 Field Duplicate (FD): Samples are collected in duplicate bags and processed the same way. Analysis of field duplicates indicates the precision associated with sample collection, preservation, and storage as well as laboratory procedures.

13.2 Laboratory Duplicate (LD): Two 5g aliquots of the same sample are processed in the same way. Analysis of laboratory duplicates indicates precision associated with laboratory procedures but not with sample collection, preservation or storage procedures.

13.3 Standards (ST): There are two standards used for this process. Calcite and limestone samples are used as standards in this procedure. 1 gram of each sample should be run when burning the samples at 1000°C with association to the field samples being examined. Each rack should have 12 samples, 1 Field

Duplicate (FD), 1 Laboratory Duplicate (LD), 1 Standard of calcite, and 1 Standard of limestone.

14 Calibration and standardization

The scale should be checked for accuracy by using the weights located in Temple 125, in the fourth cabinet on the top self. When using these weights, personnel must use the tongs or glove provided in the weight set.

15 Procedure

15.1 Samples are dried at 60°C in the drying ovens located in Temple 125 or Temple 103. These samples are dried in the original bags in which the sample is collected Standards (both calcite and limestone) should be placed in a separate bag and dried with the samples being examined.

15.2 Porcelain crucibles should be pre-dried before samples are added. Put crucibles in the oven at 104 ± 1°C for four hours. Take out and place the crucibles in the desiccator for 30 minutes to allow them to cool. Weigh the crucibles before adding any samples.

15.2 A 5 gram sample is placed into a porcelain crucible and the weight is recorded. The standards used should have 1 gram sample placed in a porcelain crucible and the weight is recorded. There should be 1 Field Duplicate and 1 Laboratory Duplicate per 12 samples.

15.3 Samples are then re-dried in a 104 ± 1ºC oven for 2 hours to remove moisture. The samples are weighed and placed in the desiccator to cool. After the samples are cooled, the weight is recorded.

15.4 These samples are placed in a 600ºC muffle furnace for 8 hours to incinerate the organic matter in the sediment.

15.5 After 8 hours of burning the samples are allowed to cool for a minimum of 45 minutes before opening the door. The cooling process is achieved by putting the samples in a desiccator for a minimum of 30 minutes before re-weighing.

15.6 The samples are then re-weighed and the weight is recorded. If organic matter is the only thing that is being investigated than stop at this step.

15.7 Samples are placed back into the muffle furnace for 1 hour at 1000°C and allowed to cool for a minimum of 1 hour with the door closed. Place the samples in the desiccator to continue the cooling process.

15.8 The samples are then re-weighed and the weight is recorded.

15.9 After the weights from the samples are recorded the examiner will dispose of the sample by using tongs to dump the sample in an appropriate receptor.

15.10 The difference is used to calculate organic matter content in percentage by weight.

16 Data acquisition, calculations, and reporting

16.1 For each sample analyzed, including quality control samples, record; the sediment pre-burn mass, the furnace-dry mass (post-burn), and the calculated percent organic matter in the appropriate places on the bench sheet (see below).

16.2 Calculate % organic matter using equation 2.

Equation 2. % Organic Matter = 100*(9/5)

Where: 9 = Post-burn 600°C weight and
5 = weight of Pre-burn (105°C) sediment (g).

16.3 Results should be reported to 0.01% precision.

17 Computer hardware and software

17.1 Word: This document and attached bench sheet are prepared using Microsoft Word. The Word document file name for this SOP is: 2030R01 Org Matter LOI.doc

17.2 Excel: Quality control charts are created using Excel.

18 Method performance

18.1 The desired performance criteria for this measurement are:

a. Detection limit: 1%
b. Precision: ± 20%
c. Minimum Quantification Interval: 0.01%

19 Pollution prevention

All wastes from these procedures shall be collected and disposed of according to existing waste policies within the MSU Geography, Geology, and Planning Department.

20 Data assessment and acceptable criteria for quality control measures

20.1 The analyst should review all data for correctness (e.g., calculations).

20.2 Precision values are calculated for pairs of duplicate analyses.

20.3 Record the precision values as a percent on the bench sheet.

20.4 The desired precision is ± 20%

20.5 The completed bench sheet is reviewed by the analyst's supervisor or the OEWRI QA coordinator.

21 Corrective actions for out-of-control or unacceptable data

21.1 The results for precision data are compared to the acceptable values for this analysis; ± 20%.

21.2 If a precision value exceeds 20% then the analyst should write in the comments section of the bench sheet: “These data are associated with an out-of-control duplicate analysis. The UCL = 20%.” Note: “UCL” is the Upper Control Limit (i.e., 20%).

21.3 The samples can be reanalyzed if necessary.

21.4 If data are unacceptable for any reason, the analyst should review their analytical technique prior to conducting this analysis again.

22 Waste management

The wastes generated in this method are not hazardous. They can be discarded in the following manner: the sediment can be discarded in a proper receptacle.

23 References

23.1 Pavlowsky, Robert T. 1995. Spatial Variability of Mining-Related Zinc and Lead Dispersal in Fluvial Sediments, Galena Watershed, Wisconsin-Illinois. University of Wisconsin – Madison, PhD Thesis.

24 Tables, diagrams, flowcharts and validation data

24.1 There are no tables, diagrams, flowcharts or validation data for this method.

24.2 See page 8 for the bench sheet. The analyst should make a copy of this form for each batch of samples analyzed.

Results Report
Geomorphology Research Lab OEWRI-MSU

Soil Organic Matter by Percent Loss-on-Ignition Test

Sample Description:
Date of Analysis:
Analyst:
Procedure: *OEWRI: Organic Matter in Sediment, Loss on Ignition Method (2030R01 Org Matter LOI)*
Checked by:

ID #	Crucible #	*1* Crucible Tare Weight (g)	*2* Air dry Total 60 C Weight (g)	*3* Air dry Sample 60 C Weight (g)	*4* Preburn Total 105 C Weight (g)	*5* Preburn Sample 105 C Weight (g)	*6* Preburn 105 C Weight Loss (g)	*7* Postburn Total 600 C Weight (g)	*8* Postburn Sample 600 C Weight (g)	*9* Postburn 600 C Weight Loss (g)	*10* Hotburn Total 1000 C Weight (g)	*11* Hotburn Sample 1000 C Weight (g)	*12* Hotburn 600 C Weight Loss (g)	**Water** Weight Loss in Oven (%)	**OM** Weight Loss on Ignition (LOI-%)	**Carb** Weight Loss on Ignition (LOI-%)
		(balance output)	(balance output)	(2-1)	(balance output)	(4-1)	(3-5)	(balance output)	(7-1)	(5-8)	(balance output)	(10-1)	(8-11)	(100 x 6/3)	(100 x 9/5)	(100 x 12/5)

25 Bench Sheet