Ozarks Environmental and Water Resources Institute (OEWRI)
Missouri State University (MSU)

Standard Operating Procedure for:

Organic Matter and Total Carbon in Sediment - Loss on Ignition Method

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Identification of the test method

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

Applicable matrix or matrices

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

Detection limit

The detection limit for this procedure is approximately 1%.

Scope of the test method

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

Summary of test method

Sediment samples are dried in a 105ºC oven to remove moisture. A 5 g sample is placed in a porcelain crucible and the pre-burn weight is recorded. 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. The difference is used to calculate organic matter content in percentage by weight.

Definitions

1. Analytical batch: The set of samples processed at the same time
2. 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.
3. 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.

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.

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 the dust from dried sediment.

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.

Equipment and supplies

1. Oven: set to 105 ± 1ºC
2. Furnace: set to 600° C
3. Analytical balance: Capable of weighing to the nearest 0.01g.
4. Porcelain crucibles: to contain a 5g sample and 1g standards.
5. Crucible holder rack and remover
6. Tongs or tweezers

Sample collection, preservation, shipment, and storage

Samples will be collected in accordance with project-approved methods and procedures. Samples will be placed into plastic bags and labeled. There are no special provisions for shipment. Sample bags should be opened and placed into a 60ºC oven immediately upon return to lab.

Quality control

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.
2. Laboratory Duplicate (LD): Two 5 g 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.

Calibration and standardization

The scale should be checked for accuracy by using the weights located in the OEWRI laboratory (Roy Blunt Hall 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.

Procedure

A. For Organic Matter in sediment:

1. Samples are dried at 60°C in the drying ovens located in the OEWRI laboratory. These samples are dried in the original bags in which the sample is collected.
2. A 5-gram sample is placed into a porcelain crucible and the weight is recorded.
3. Samples are then re-dried in a 105 ± 1ºC oven for 2 hours to remove moisture.
4. Allow samples to cool in the desiccator for 30 minutes.
5. Re-weigh samples after cooled for pre-burn weight.
6. Place samples in 600ºC muffle furnace for 8 hours to incinerate the organic matter in the sediment.
7. After 8 hours of burning, the samples are allowed to cool for a minimum of 1 hour in the furnace with the door closed.
8. Then move samples to desiccator for another hour to cool before re-weighing.
9. Record sample weights for the post burn weight of the samples.

B. Additional step for Total Carbon in sediment:

1. Place samples back into furnace at 1000 ºC for an additional hour.
2. Allow the samples to cool for an hour in the oven with door closed, and then another hour with the door open.
3. Re-weigh and record weights.
4. Dispose of samples using tongs to dump the sample in an appropriate receptor.
5. The difference is used to calculate organic matter content in percentage by weight.

Data acquisition, calculations, and reporting

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.
2. Calculate % organic matter using the following equation.

Equation 1 $$\%\ \text{Organic Matter} = \frac{h}{d} \text{ X } 100$$

Where: h = weight of Post-burn (600°C) sediment (g)
d = weight of Pre-burn (105°C) sediment (g)

3. Results should be reported to 0.01% precision.

Method performance

1. The desired performance criteria for this measurement are:
    a. Detection limit: 1%
    b. Precision: ± 20%
    c. Minimum Quantification Interval: 0.01%

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.

Data assessment and acceptable criteria for quality control measures

1. The analyst should review all data for correctness (e.g., calculations).
2. Precision values are calculated for pairs of duplicate analyses.
3. Record the precision values as a percent on the bench sheet.
4. The desired precision is ± 20%
5. The completed bench sheet is reviewed by the analyst's supervisor.

Corrective actions for out-of-control or unacceptable data

1. The results for precision data are compared to the acceptable values for this analysis; ± 20%.
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%).
3. The samples can be reanalyzed if necessary.
4. If data are unacceptable for any reason, the analyst should review their analytical technique prior to conducting this analysis again.

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.

References

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

Tables, diagrams, flowcharts, and validation data

1. See page 8 for the Organic Matter bench sheet
2. See page 9 for the Total Carbon and Organic Matter bench sheet.
3. The analyst should make a copy of the form required for each batch of samples analyzed.

Bench Sheet: Soil Organic Matter Loss-on-Ignition Test

Date: ______________________ **Analyst:** __________________ **Project:** ____

Equation variables		***(a)***	***(b)***	***(c)***	***(d)***	***(e)***	***(f)***	***(g)***	***(h)***	**Water weight loss percent**	**OM weight loss percent**
Sample ID	**Crucible #**	**Crucible Tare weight**	**Soil Weight (g) (5.0000)**	**Pre-burn Weight 105 °C (2hrs) (g)**	**Pre-burn Sample**	**Pre-burn Weigh Loss**	**600 °C (8 hrs)**	**Post-burn Sample Weight**	**Post-burn weight Loss**		
Calculations		*(Balance output)*	*(Balance output)*	*(Balance output)*	*(c-a)*	*(b-d)*	*(Balance output)*	*(f-a)*	*(d-g)*	*((100*(e/b)*	*((100*(h/c)*

Bench Sheet: Soil Organic Matter and Total Carbon Loss-on-Ignition Test

Date: ______________

Analyst: __________________

Project: ___________________

Calculation variables		***(a)***	***(b)***	***(c)***	***(d)***	***(e)***	***(f)***	***(g)***	***(h)***	***(i)***	***(j)***	***(k)***			
Sample ID	**Crucible #**	**Crucible Tare weight**	**Soil Weight (5.0000)**	**Preburn total Weight**	**Preburn Sample**	**Preburn Weigh Loss**	**600 °C (8 hrs)**	**Postburn Sample Weight**	**Postburn weight Loss**	**Hotburn total 1000 °C**	**Hotburn sample weight**	**Hotburn weight loss**	**Water weight loss percent**	**OM weight loss percent**	**Total Carbon loss percent**
Calculations		*Balance output*	*Balance output*	*Balance output*	*(c-a)*	*(b-d)*	*Balance output*	*(f-a)*	*(d-g)*	*Balance output*	*(i-a)*	*(g-j)*	*((100*(e/b)*	*((100*(h/c)*	*(100* (k/d))*