Sediment Percent Organic Determination Using Ash-Free Dry Weight

1. SCOPE AND APPLICATION

   1.1. This method is used to measure the percentage of organic material in sediment collected from freshwater and marine environments. Percent organic is determined using ash-free dry weight (AFDW) which is a measurement of the weight of organic material.

   1.2. This procedure is used for samples with LIMS Test ID SDMNT-AFDW.

   1.3. Method Detection Limits (MDL)

      1.3.1. No MDL has been established for sediment results, but the minimum reporting limit for percent organics is 1.0% dry weight. Results less than 1.0% dry weight are reported as 1.0 with a "K" qualifier.

2. SUMMARY OF METHOD

   2.1. Sediment samples are placed in a drying oven to remove water. The resulting dry weight is the weight of both the organic and inorganic contents of the sample. The dried sample is then heated in a muffle furnace where combustion eliminates the organic content, leaving only inorganic ash. The AFDW is the dry weight (inorganic + organic contents) minus the weight of the ash (inorganic contents only), which equals the weight of the organic content of the sample.

   2.2. Interferences

      2.2.1. Samples must be stored at ≤ 6°C until analyzed. Holding time is 28 days.

      2.2.2. The muffle furnace must remain at 550 °C during analysis to ensure that all organic matter in the sample goes through the combustion process.

3. EQUIPMENT AND SUPPLIES – Record any equipment repairs or maintenance in LIMS **Laboratory Logbook**, *Maintenance Log*

   3.1. Mettler PB403-S balance (SN 1128150043)

      3.1.1. Check calibration each day before use. See SOP BB-037-1.0

   3.2. Beaker, 250 mL, one per sample

   3.3. Rinse bottle containing deionized water

   3.4. Enamel pan, or other large vessel for mixing sediment

   3.5. Metal spatula

   3.6. Crucible, 100 mL, oven-dried, one per sample

   3.7. Crucible tongs

   3.8. Crucible cooling pad

   3.9. Furnace gloves

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3.10. Drying oven, 105 °C ± 2° (Isotemp180 L Gravity Oven SN 420737712)

3.11 Muffle Furnace preheated to 550°C (See Section 7.1) located in Room B338 or B425.

4. REAGENTS AND STANDARDS

4.1. Deionized (DI) water

4.2. Aqueous sodium hexametaphosphate

4.2.1. Add 6.2 g of $(NaPO_3)_6$ to 1 L of deionized water. Record preparation in LIMS standard prep tracker.

5. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1. See SOP FS 1000 for sample collection and preservation. Samples must be stored at ≤ 6°C until analyzed. Holding time is 28 days.

6. SAMPLE PREPARATION

6.1. See SOP BB-030 for sample custody and label instructions.

6.2. Homogenize the sediment sample by gently but thoroughly mixing with a metal spatula.

6.2.1. For sediment that is sticky or heavy with clay, add up to 10 mL of aqueous sodium hexametaphosphate. This surfactant aids in separating particles.

6.2.2. The entire sample may be emptied into an enamel pan or other large vessel if more room is needed for mixing.

6.3. Write the **date**, **time** and **analyst** in the AFDW section of the *Sediment Run Log*.

6.4. Weigh a 250 mL beaker. Record the **beaker #** and **beaker weight** in the *Sediment Run Log*.

6.5. Add sediment to the beaker using the spatula and record the **beaker + wet sediment weight**.

6.5.1. Weigh out enough sediment to have at least 25 g when dry: typically ~50 g for coarse grain (sand) and ~100 g for fine silt or clay samples.

6.6. Repeat steps 6.2 through 6.5 for each sample so they can be dried together in the oven.

6.7. Place the beaker in the sediment oven at 105 °C to completely dry the sample. After 24 hours, cool and weigh the beaker and record the **beaker + dry sediment weight**.

6.7.1. Depending on the sediment matrix, drying may take from a few hours to a few days. Examine beaker and sediment for any moisture. Allow more time for the sediment to dry if any moisture is visible.

6.8. Return beaker containing dried sample to oven until ready to begin analysis.

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7. SAMPLE ANALYSIS

7.1. To preheat the muffle furnace to 550 °C, set the furnace control to 4 until it is near the desired temperature (about 2.5 hours), and then turn to 2.

***Note*: The muffle furnace is not thermostatically controlled. The controls adjust the percentage of time the heating elements are on. This setting may vary slightly depending on outside temperature and changes in voltage levels and heating-element conditions.**

7.2. Remove sediment beaker from drying oven to cool once again before weighing.

7.3. Weigh one crucible per sample. Record the **crucible #** and **crucible weight** in the *Sediment Run Log.*

7.4. Weigh approximately 25 g of the dry sediment into the crucible. Record the **crucible + dry sediment weight**.

7.5. Use tongs to place the crucible into the preheated muffle furnace. Leave samples in the furnace at 550 °C for 4 hours.

7.6. Remove the crucible using the crucible tongs and place it on the cooling pad. Label the area as "DANGER! HOT!" Allow the crucible to cool down to room temperature.

7.7. Once cool, re-wet the sediment with DI water by completely covering the sediment remains. This water addition allows molecules to rehydrate after the ashing process.

7.8. Place the crucible in the sediment oven at 105 °C for another 24 hours, or until completely dry.

7.9. Record the **1st crucible + ashed sediment weight** in the *Sediment Run Log.*

7.10. Return the crucible to the sediment oven for another 24 hours.

7.11. Weigh the crucible and record the **2nd crucible + ashed sediment weight**. If the difference between the 1st weight and the 2nd weight is <0.010 g, then the analysis is done. If >0.010 g, then continue to the next step.

7.12. Return the crucible to the sediment oven for another 24 hours.

7.13. Weigh the crucible and record the **3rd crucible + ashed sediment weight**. Continue drying the sediment until the difference between the last two measurements is < 0.010g.

8. DATA ARCHIVAL

8.1. In LIMS, select **Laboratory/Biology/Sediment Manager**.

8.2. Click on the AFDW button. Press [Ctrl] + [Ins], or click row/add, to add a row for each sediment sample to be uploaded.

8.3. Enter the Analyst and Analysis Date from the *Sediment Run Log.* Only one date/time and analyst can be selected. If there are more than one, adjustments can be made in Step 8.10.

8.4. Using the *Sediment Run Log,* enter each Sample ID into the Sample ID column.

8.4.1. For laboratory duplicates, enter same ID (no suffix) for both samples.

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8.5. Enter each corresponding **crucible weight** into the Crucible Weight column. Enter each corresponding **crucible + dry sediment weight** into the % Dry Weight column.

8.6. Enter the last **crucible + ashed sediment weight** recorded in the logbook into the % Ashed Weight column. Verify that the difference from the previous weight is <0.010g.

8.7. Select **File/Calculate % Organics** from the menu or click the calculator icon.

8.8. Print the spreadsheet. To print, press [Print Screen] key, then Paste into a blank Word document. Crop and adjust size for easy visibility. Print.

8.8.1. On the printout, highlight any results in the **% Organics** column that are < 1.0 and write "1.0 K" next to the result.

8.8.2. Calculate averages for any laboratory duplicates and write the result in the margin. Do not average if either result is "1.0 K". Calculate the RPD to 2 significant figures and record it on the printout.

8.9. In the **Sediment Manager**, change any results in the **% Organics** column that were highlighted in the printout to "1.0".

8.10. Select **File/Upload Data** from the menu. Using the *Sediment Run Log*, corrections for analyst, analysis date or time for each sample can be made in this screen.

8.11. Add a "K" in the **Remark** column for any samples that had a result changed to "1.0".

8.12. For lab duplicates, verify the calculated average and add an "A" qualifier in the **Remark** column.

8.12.1. If either duplicate result is "1.0 K", do not average or add "A". Upload the duplicate with the higher result and report the other duplicate result in the comment: "Laboratory duplicate result is _. Precision data is unavailable due to the small amount of analyte in the QC sample."

8.12.2. If the RPD from Step 8.8.2 is ≥ 25%, add a "J" in the **Remark** column and the comment "Result is J-qualified due to a laboratory duplicate RPD failure."

8.13. Be sure all samples are check marked before uploading. Select **File/Upload Data**. Close the window and exit **Sediment Manager**.

9. DATA UPLOAD AND AUTHORIZATION

9.1. In LIMS, select **Laboratory/Technical/Results Editor**.

9.2. In the "Sort By…" box, select Sample ID. Under "Analysis Group" checkmark test status C and select Bio-SedPrtSz/AFDW.

9.3. Select test SDMNT-AFDW. Highlight the sample IDs to be uploaded.

9.4. From the menu, select Result/Print. The *Result Report* window will open a preview. Click on the printer icon to print. Close the window and close Results Editor.

9.5. Stamp the printout with the Data Review stamp. Initial and date the *Data Entry* and *Uploaded* lines. Attach with paperclip to Sediment Manager printout from Step 8.8.

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9.6. Refer to SOP-BG-01 to perform a two-person verification of the data. Initial and date the *Verified* line of the Data Review stamp.

9.7. Once verified, make any necessary corrections in Results Editor. Data is authorized by clicking the Authorize Test (A) button.

10. QUALITY CONTROL

10.1. Analyze a laboratory duplicate for 5% of samples and a minimum of one replicate per batch. The acceptance limit is ≤ 25% RPD.

10.2. If duplicate results do not meet acceptance limit, re-analyze sample if possible. Report duplicate results that exceed the acceptance limit with a J-qualifier and comment "Result is J-qualified due to a lab duplicate RPD failure."

11. SAFETY/HAZARDOUS WASTE MANAGEMENT

11.1. Refer to the Laboratory Health and Safety Plan.

11.2. Read MSDS information for reagent handling and disposal.

12. REFERENCES

12.1. EPA Method 4805572010: Procedures for Handling and Chemical Analysis of Sediment and Water Samples

12.2. NELAC Quality Systems Manual

12.3. FDEP Laboratory Quality Manual https://floridadep.gov/dear/florida-dep-laboratory/content/dep-laboratory-quality-assurance-manual-and-sops

12.4. FDEP Laboratory Health and Safety Plan http://www.fldepnet.org/content/dear/bureau-laboratories-safety-documents

12.5. DEP SOP-001/01, *FS 1000 General Sampling Procedures*

12.6. DEP SOP BB-030-, *Sample Custody, Preparation Labels and Worksheet Instructions for Bench Biology Samples*

12.7. DEP SOP-BG-01 *Data-Entry Verification*

APPENDIX OF CHANGES

01/15/2009	Changed SOP Format. CF
01/11/2010	Revisions: Added Step 7.2 to cool sediment, edited Step 8.4 and 8.12 to clarify duplicate reporting procedure, edited Step 8.8 for printing instructions. CF Changed storage temperature to ≤ 6ºC. MF
01/06/2011	Updated reference to health and safety manual.
01/05/2011	Added corrective action for out of control duplicates.

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10/03/2013	Updated Step 7.11 to include requirement for 3rd weighing.
01/15/2015	Formatted for 508 compliance.
12/30/2016	Corrected format in Sections 3, 7, and 8; corrected balance SOP reference and added model number to drying oven in Section 3; made a note to sign the "Verified" line after 2-person verification in Section 9; removed Lab Health and Safety Plan link to "burlabs" from Section 12.
12/20/2017	Added new incubator in section 3; added reagent preparation procedure in section 4.
01/03/2019	Updated link on section 12.3 for FDEP Laboratory Quality Manual.
04/07/2020	Updated Muffle Furnace equipment information.

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