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 Wisconsin Mercury Research Laboratory (WMRL). Sample is weighed into aluminum boats and heated to 550º C for two hours. The percent of sample mass lost following heating is reported as LOI. One sample per analysis is weighed in triplicate to assess method precision.

Laboratory Safety

Persons involved in this method must have read, understood, and signed the Chemical Hygiene Plan for the WMRL prior to potential exposure to any chemicals present in the laboratory. Specific safety concerns for most chemicals are addressed in the Material Safety Data Sheets which are located within the laboratory. Although this method does not involve any hazardous chemicals, caution should still be exercised as that chemicals are always present in the laboratory and often in use by other analysts. 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 14 samples, of which one is weighed in triplicate. Multiple analyses may be completed in a single day, with each batch containing a triplicate analysis.

1. Arrange an adequate number of aluminum boats onto a tray. The maximum number of boats the furnace can accommodate is 16.

2. Using a ball point pen, number each aluminum boat on the tag. Make sure that you press hard enough to leave an imprint, as that 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 sample 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 550º C for two hours. During the combustion, open the adjacent lab door several inches and turn on the nearest fume hood to provide ventilation.

5. After two hours, turn off the furnace and allow the samples to cool. Carefully remove the samples as that the furnace may still be hot.

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.