DETERMINATION OF PERCENT DRY SOLIDS

1. SCOPE AND APPLICATION

This method is used to determine the percent (%) dry solid matter in a variety of sample matrices including soil and sediment. The % solid results can be used to determine both a suitable wet mass of sample to be prepared for certain analyses as well as report the final results on a dry weight basis. This procedure was adapted from Standard Methods, 20th edition method 2540G. This SOP covers the tests PERC-SOLID and PCT-SOLID. The PCT-SOLID test is associated with the virtual ID of the sample and the PERC-SOLID test is used with samples ID's specific for each chemistry group. The component name for both tests is '% Solid'.

2. SUMMARY OF THE METHOD

The sample is homogenized and a 10 g portion is thoroughly dried between 103 ºC and 105 ºC and weighed. If 10 g of sample is not available please see your supervisor before proceeding.

3. APPARATUS AND EQUIPMENT

3.1 Stainless steel spatulas.

3.2 Aluminum weighing boats.

3.3 Denver S-403 top loading balance or equivalent.

3.4 Fisher Scientific Isotemp oven or equivalent.

3.5 Stainless steel tray.

3.6 Computer with network connection.

4. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Soil and sediment samples are stored at 4 °C in room C301 'Chemistry Receiving and Storage room' refrigerators. Samples with a solid matrix requesting metals analysis do not require preservation, however many samples are logged in for multiple laboratory tests that may require refrigeration.

Note: The Metals section will perform the % solid determination on all samples including those requiring VOC analyses only and laboratory proficiency samples. Samples requiring only VOC analyses (S-VOC-MS or S-8260B-AA) must be opened and handled first by VOC personnel. Therefore, VOC personnel will notify the Metals section of when to perform the % solid determinations for these samples.

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5. SAMPLE PREPARATION & ANALYSIS

5.1 Sample Homogenization:

5.1.1 If the sample cannot be thoroughly mixed within its container without spilling, empty the entire sample into a clean stainless steel tray. Mix each sample thoroughly to achieve homogeneity. A mortar and pestle may be used to break up large particles or clumps. Return the sample to the jar.

5.1.2 If multiple sample containers are received with the same field ID, see your supervisor before proceeding. If necessary, empty all containers onto a stainless steel tray, homogenize as above and return to the original containers; this procedure helps to ensure that samples with the same field ID assigned to different tests are as similar as possible.

Note: A sample may have several laboratory IDs for different tests within the laboratory. All containers of the same sample will have the same field ID and virtual ID.

5.2 Percent solids determination:

5.2.1 Visually inspect the sample containers to verify that the integrity of the sample has not been compromised. Create a non-conformance report (NCR) if a sample container or container lid is damaged (see DEP SOP LB-002). Determine if the non-conformance will affect the quality of reported results. For example, if a sample container is found with a crack in the glass and liquid from the sample has leaked out of the container, the quality of the results will be affected and the results will be qualified appropriately.

5.2.2 **Label** the tag or the bottom of the aluminum weigh boat with the sample number using a pen. The ink will not be visible but the indentation in the weigh boat should be legible.

5.2.3 **Verify** the balance's calibration prior to use each day and record the calibration results in the calibration logbook for the balance. Also record the balance's serial number in the Excel Percent Solid spreadsheet. Data is entered in to the Percent Solid spreadsheet electronically. Handwritten data is not acceptable. The Percent Solid spreadsheets are located on the network at \\FLDEP1\DEAR\Labs\Lab5\Chemistry\InorganicPrep\METALS\% SOLID LOG BOOK\.

5.2.4 **Record** the mass of the empty weigh boat in the Percent Solid spreadsheet as the 'Mass of empty vessel in grams.'

5.2.5 Tare the balance and **weigh** approximately 10 g of the sample into its corresponding weigh boat. **Record** this value as the 'Wet Mass of sample in grams.' **Repeat** for each sample. A duplicate must be analyzed for every ten samples. A blank (BLK) must also be analyzed by taking an empty aluminum weigh boat through the entire process. Only one BLK is needed per day.

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5.2.6 **Dry** the samples in a temperature controlled and monitored oven at 103-105 °C. If the procedure was performed over the weekend, the temperature control monitors for the ovens must be checked and documented to ensure the temperature was maintained at 103-105 °C. Notify the supervisor if the oven temperature was out of the acceptable range before proceeding.

5.2.7 **Record** the oven's serial number and oven temperature (in degrees Celsius) in the Percent Solid spreadsheet (**DAY 1)**. This temperature should be the same daily temperature recorded on the sheet attached to the oven.

Note: On each day the oven is used for the percent solid procedure, the temperature of the oven shall be checked and documented on the temperature log attached to the front of each oven.

5.2.8 **Remove** and weigh each sample and **record** the weight in the Percent Solid spreadsheet under the heading, 'First dry weight of vessel plus sample mass in grams.'

5.2.9 **Return** the sample to the oven at 103 - 105 °C for an additional hour, re-weigh and record this weight under the heading, 'Second dry weight of vessel plus sample mass in grams.' **Record** the oven temperature (in degrees Celsius) in the Percent Solid spreadsheet (**DAY 2)**. This temperature should be the same daily temperature recorded on the sheet attached to the oven.

5.2.10 The difference between the two dry weights determined in 5.2.8 and 5.2.9 should be less than 0.05 g (assuming a 10 g wet weight). Subtract the tare weight from the Dry Mass measurement #2 of vessel & sample weight to determine the sample's dry weight. If the difference between the two dry weights is greater than 0.05 g, repeat steps 5.2.8 and 5.2.9. If the difference between the two dry weights is still greater than 0.05 g after repeating steps 5.2.8 and 5.2.9, contact the supervisor.

5.2.11 Determine the percent solids of the sample using the following formula:

% Solids = (dry weight/wet weight) * 100 %

Upon completion of the % solids determination, clearly label the sample bottles to indicate that this analysis has been performed.

6 ENTRY OF PERCENT SOLID DATA INTO PERCENT SOLID SPREADSHEET AND LIMS

6.1 Open the PS105%_ Template.xls file (or equivalent) found on \\FLDEP1\DEAR\LABS\LAB5\Chemistry\InorganicPrep\METALS\% SOLID LOG BOOK.

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6.2 Enter the virtual and sample IDs and masses as indicated. **Make sure the % solid results are 3 digits (when less than 10)**.

6.3 Save the Excel file as Metals_Perc-Solid_YYMMDD. Use the Start Date as the date.

6.4 After all the data is entered and results calculated, save the file again using Text (Tab delimited) as the Save as type. Save into the \\FLDEP1\DEAR\LABS\LAB3\Chem_Data\Inorganic\METALS\Percent_Solid folder on the network. Select '**Yes**' on the prompt to save.

6.5 Close the file. Select '**No**' on the prompt to save again.

6.6 Import the text file (6.4) into QC Manager (QCM).

6.6.1 In LIMS, go to Laboratory>QCM. Select the instrument **Metals: Percent Solid**.

6.6.2. Click on **File, then Convert Instrument File** and open the file needed in the \\FLDEP1\DEAR\LABS\LAB3\Chem_Data\Inorganic\METALS\Percent_Solid folder. A 'Save To LIMS' dialog box will appear with the name of the file (the format should be as described in 6.3).

6.6.3 Enter the **Analyst**, **Analysis Date/Time** (begin date and time) and **Analyte** (% Solid). **Convert** the file.

6.6.4 Highlight all samples. Assign the **Test ID** as **PCT-SOLID** (not PERC-SOLID) by **selecting 'No' on prompt.** Assign the **Matrix** as **S-SOIL**. Assign the **Prep Batch** and **Analysis Batch**. They both will be **New. Each batch of 10 samples should be assigned a new Prep Batch ID. All samples that went through the procedure together, even with multiple batches, should be assigned the same analysis batch ID in QCM.**

6.6.5 Highlight the duplicate samples and **Calculate** the average by selecting the calculator symbol at the top of the screen. A box will appear. Make sure '**Selected sample rows**' is selected for **Calculate By. Each batch of 10 samples should have a unique prep batch ID and a sample analyzed in duplicate.**

6.6.6 Go to the Component Table. Highlight all samples **(only highlight the AVG and RPD for the replicate sample sets, not the A & B replicates)**. Change **Upload?** to '**Y**'. **Double check the % solid results to make sure they are 3 digits.** Save the data.

6.6.7 Select **Tools**, **Upload** and **By Highlighted Component Rows**. Select **Upload Data** (first option) found at the top of the screen.

6.6.8 When QCM prompts 'Complete' exit out of program.

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7. QUALITY CONTROL AND CALCULATIONS

Replicate Samples – Verify that 1 duplicate was performed for every 10 samples (denoted 'A' and 'B'). Verify that each batch of 10 samples has a unique prep batch ID. Examine the precision value (Relative Percent Difference (RPD)) for each required duplicate. Ensure that it is within the control limit (5 % or less). RPDs outside the control limits must be qualified with a 'J' qualifier code and have the comment 'The % solid RPD for this sample was outside the control limit' (qualifier and comment on the AVG only).

The precision (RPD) is calculated as follows:

$$RPD = \frac{|A - B|}{(A + B)/2} \times 100$$

8. SAFETY/HAZARDOUS WASTE MANAGEMENT

8.1 Review the Bureau's Laboratory Health and Safety Plan

8.2 All samples should be handled with care, as they are potentially hazardous.

8.3 Always wear gloves, lab coat, and safety glasses when working in the laboratory.

8.4 Dispose of sample aliquots properly after analysis.

9. REFERENCES

9.1 Laboratory Health and Safety Plan, located on the Division of Environmental Assessment and Restoration internet page.

9.2 Standard Methods, 20th edition, Method 2540G

9.3 DEP SOP LB-002, Non-Conformance Reporting System.

Appendix of Significant Changes

Section 6 contains new % Solid Results data entry procedure.

Section 7 contains new quality control requirements.

08/24/2009 Section 6.3 and 7.1: Each batch of 10 samples should be assigned a unique prep batch ID.

07/17/2015 All network addresses were updated and a second temperature documentation requirement was added to section 5.2.9 and to the Excel spreadsheet.

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