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, SM 2540 G-2020. This SOP covers the tests PERC-SOLID and PCT-SOLID. The PCT-SOLID test, the most common, is associated with the virtual ID of the sample and is recorded as a parameter of the sample, whereas the PERC-SOLID test is used with sample IDs specific for each chemistry group and the result is included on the final report. 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 Mettler PM460 top loading balance or equivalent.

3.4 Fisher Scientific Heratherm 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 on the MT-105C-PS form. Data is entered in to the MT-105C-PS form electronically. Handwritten data is not acceptable. The MT-105C-PS form is located on the Division of Environmental Assessment and Restoration (DEAR) website. An Excel version can be found on the network at \\floridadep\data\DEAR\Labs\Lab5\Chemistry\InorganicPrep\METALS\% SOLID LOG BOOK. Always make sure the most up-to-date form is used by checking the version number on the DEAR website.

5.2.4 **Record** the mass of the empty weigh boat on the MT-105C-PS form as the 'Mass of Empty Vessel (g)' 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 (g)' in grams. **Repeat** for each sample. A duplicate must be analyzed for every ten samples. A blank (D-BLK) must also be analyzed by taking an empty aluminum weigh boat through the entire process. Only one blank is

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needed per day unless the PERC-SOLID test is used, and in this case, a blank is prepared for every 10 samples.

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) on the MT-105C-PS form (**DAY 1)**. This temperature should be the same daily temperature recorded on the MT-TEMPLOG form 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 MT-TEMPLOG form attached to the front of each oven.

5.2.8 **Remove** and weigh each sample and **record** the weight on the MT-105C-PS form under 'First Dry Weight of Vessel + Sample Mass (g)' 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 'Second Dry Weight of Vessel + Sample Mass (g)' in grams. **Record** the oven temperature (in degrees Celsius) on the MT-105C-PS form (**DAY 2)**. This temperature should be the same daily temperature recorded on the MT-TEMPLOG form 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 and 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 containers to indicate that this analysis has been performed.

6 ENTRY OF PERCENT SOLID DATA INTO THE MT-105C-PS FORM AND LIMS

6.1 Open the MT-105C-PS form found on the DEAR website. An Excel version of this form can be found in: \\floridadep\data\DEAR\Labs\Lab5\Chemistry\InorganicPrep\METALS\%

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SOLID LOG BOOK. Always make sure the most up-to-date form is used by comparing the version number on the Excel version of the form to the version number on the DEAR website.

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

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

6.1.3 After all the data is entered and results calculated, save the file again using Text (Tab delimited) as the file type. Save into the folder \\floridadep\data \Labs\Lab3\Chem_Data\Inorganic\Metals\Percent_Solid on the network. Select '**Yes**' on the prompt to save.

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

6.2 Import the text file (from Section 6.1.3) into QC Manager (QCM) in the Laboratory Information Management System (LIMS).

6.2.1 In LIMS, navigate to Laboratory>QC>QC Manager. Select the instrument **Metals: Percent Solid**.

6.2.2. Click on **File,** then **Convert Instrument File** to open the text file needed from \\floridadep\data\Labs\Lab3\Chem_Data\Inorganic\Metals\Percent_Solid. A 'Save To LIMS' dialog box will appear with the name of the file (the format should be as described in Section 6.1.2).

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

6.2.4 Highlight all samples. Assign the **Test ID** as **PCT-SOLID** (not PERC-SOLID) by selecting '**No**' on the prompt. Assign the **Matrix** as **S-SOIL**. Assign a new **Prep Batch** and **Analysis Batch**. **Each batch of 10 samples must be assigned a new Prep Batch ID. All samples that went through the procedure together, even as multiple batches, should be assigned the same Analysis Batch ID in QCM. If Test ID PERC-SOLID is required, assign the Matrix as SEDIMENT.**

6.2.5 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 must have a unique Prep Batch ID and one sample analyzed in duplicate.**

6.2.6 Navigate to the Component Table to verify that the Relative Percent Difference (RPD) is passing (Section 7). If the RPD is passing, proceed. If the RPD is not passing, contact your supervisor and do not upload the associated sample results. Highlight all samples **(but only highlight the AVG and RPD for the replicate sample sets, not the A & B replicates)**.

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Change '**Upload?**' to '**Y**'. **Double check the % solid results to make sure that they are 3 digits.** Save the data.

6.2.7 Select **Tools>Upload (Save first)>By Highlighted Component Rows**. Select **Upload Data** (first option).

6.2.8 When QCM prompts, click '**Complete**'.

7. QUALITY CONTROL AND CALCULATIONS

Replicate Samples – Verify that one 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 of 5 % or less. If the RPD is outside of the control limit, the AVG sample must be qualified with a 'J' qualifier code and have the comment 'The % solid RPD for this sample was outside the control limit.'

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 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 in the trash after analysis.

9. REFERENCES

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

9.2 SM 2540 G-2020

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.

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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.

02/14/2024 Reference updated from SM 2540 G-2015 to SM 2540 G-2020.

01/30/2026 Significant reorganizing and formatting changes to Section 6. Minor formatting changes and phrasing revisions throughout the document. Updated all network file paths.

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