Laboratory Manager Approval: Kristy E. Hrehor / 08/19/2021

QA Manager Approval: Jeffrey Moore / 08/19/2021

SM 2540 G – Percent Solids

1 Scope and Application

1.1 This method is applicable to the determination of total solids and its fixed and volatile fractions in such solid and semisolid samples as river and lake sediments, sludges separated from water and wastewater treatment processes, and sludge cakes from vacuum filtration, centrifugation, or other sludge dewatering processes.

1.2 Restricted Procedure
This procedure is restricted to use by an analyst experienced in the operation of an Oven. Additionally, the analyst must complete the requirements of the GAEPD Initial Demonstration of Analyst Proficiency prior to the analysis of actual samples. Analysts are further warned that performance of this analysis involves the use of potentially hazardous chemicals; refer to the GAEPD Chemical Hygiene Plan for additional information regarding chemicals required by this method.

1.3 Percent solids requested by the Hazardous Waste program are analyzed by the Organics Laboratory. This data is shared with the Inorganic lab and used for the calculation of results for EPA SW846 Method 9010C/9012B (Refer to SOP 3-039).

2 Definitions

2.1 Refer to the Section 3 and Section 4 of the GA EPD Laboratory Quality Assurance Plan, online revision. (See SOP reference 13.2)

3 Interferences

3.1 The determination of both total and volatile solids in these materials is subject to negative error due to loss of ammonium carbonate and volatile organic matter during drying.

3.2 The mass of organic matter recovered from sludge and sediment requires a longer ignition time than that specified for wastewaters, effluents, or polluted waters.

3.3 Make all weighings quickly because wet samples tend to lose weight by evaporation. After drying or ignition, residues often are very hygroscopic and rapidly absorb moisture from the air.

4 Safety

4.1 Refer to the EPD Laboratory Safety / Chemical Hygiene Plan & Fire Safety Plan, online revision (See SOP reference 13.6)

5 Apparatus and Equipment

5.1 Sample Container: wide mouth glass or plastic container
5.2 Aluminum weighing dishes
5.3 Oven set at 103 -105ºC
5.4 Desiccator, provided with desiccant containing a color indicator for moisture
5.5 Analytical balance capable of weighing to 0.1 mg
5.6 Spatulas

6 Reagents

6.1 Not applicable.

7 Sample Collection

7.1 Samples are collected in wide mouth plastic or glass containers.
7.2 No chemical preservation is necessary.
7.2 Samples are stored between 0- 6° C (not frozen) for preservation.
7.3 Sample holding time is 7 days

8 Calibration

8.1 Analytical balances are serviced and calibrated once per year by an independent technician.
8.1.2 The balance used for this analysis must have the calibration verified each day of use with certified weights that bracket the expected weight range of the analysis.
8.1.3 All precision oven temperatures are measured with NIST approved thermometers and these measurements are recorded every morning in the daily temperature log.

8.2 Calibration Curve
Not applicable.
8.3 Calibration Verification
Not applicable. A sample duplicate is analyzed once per batch.

9 Quality Control

9.1 Refer to Table 14.1 for Reporting Limits (RLs), Table 14.2 for Quality Control Acceptance Criteria, and Table 14.3 for Quality Control Procedures associated with this method.

10 Procedure

10.1 Remove sample bottles, standards, and reagents from cold storage and allow them to equilibrate to room temperature prior to sample preparation and/or analysis.
10.2 Perform backlog of pending samples. Batch samples in groups of 20. At least 10% of samples must be analyzed in duplicate.
10.3 Weigh a pre-dried aluminum dish using a calibrated balance. Record weight on % Solid Printout.docx bench sheet.
10.4 Place 25 grams of sample in the dish and weigh. Record weight % Solid Printout.docx bench sheet as wet weight of sample + dish, in grams.
10.5 Place in oven set at 103 -105ºC overnight.
10.5.1 Remove from oven and place dried sample into desiccator for at least one hour and then weigh on analytical balance. If dried overnight, multiple weighing is not required.

10.5.2 Record weight on % Solid Printout.docx bench sheet as weight of dried residue + dish, in grams.

10.5.3 Enter data into the % solids overnight.xls worksheet to perform % Solids Calculations.

10.6 If % solids need to be determined on same day, then place sample in oven at 103-105°C for 2 hours.

10.6.1 After 2 hours, remove sample from oven and place in desiccator for one hour.

10.6.2 Remove sample from desiccator and weigh on analytical balance. Record weight on % solids sameday bench sheet.

10.6.3 Place sample back in oven for one hour. Remove sample and place in desiccator for one hour.

10.6.4 Remove dish from desiccator and weigh on analytical balance. Record weight on bench sheet as second drying.

10.6.5 Weight change must be less than 4% of the previous weighing or 0.0005 g from the previous weight, whichever is less.

10.6.6 If weight change is greater than 4% of the previous weighing or greater than 0.0005 g from the previous weight, repeat cycle of drying, cooling, desiccating, and weighing until constant weight is obtained.

10.6.7 Record weight on bench sheet as weight of dried residue + dish, in grams.

10.6.8 Perform % Solids Calculation as defined in Section 11.1.

11 Calculations

11.1 Total Solids Determination

11.1.1 % Solids Equation:

$$\% \text{ Solids} = \frac{(A-B)x\ 100}{C-B}$$

11.1.2 Where:

A = weight of dried residue + dish, g
B = weight of dish
C = weight of wet sample + dish, g

11.2 Duplicate Precision:

11.2.1 SM 2540G (see SOP reference 13.1) specifies duplicate precision must be within 5% of the average of the replicates. This is the same as 10% RPD as shown below:

11.2.2 *Average of a Sample and Duplicate*:

$$\text{Average } (\text{Avg}_{\text{replicates}}) = \frac{\text{R}_{\text{sample}} - \text{R}_{\text{duplicate}}}{2}$$

11.2.3 *Difference from Average (Diff $_{replicates}$) and Percent Difference from Average (%Diff $_{replicates}$)*:

11.2.3.1 When an average is calculated from two values, the average is equally distant from each of those values (the absolute difference from the average is the same for both values). Therefore:

$$\text{Diff}_{\text{replicates}} = \frac{\left|\text{Avg}_{\text{replicates}} - \text{R}_{\text{sample}}\right|}{\text{Avg}_{\text{replicates}}} = \frac{\left|\text{Avg}_{\text{replicates}} - \text{R}_{\text{duplicate}}\right|}{\text{Avg}_{\text{replicates}}}$$

and

$$\%\text{Diff}_{\text{replicates}} = \frac{\left|\text{Avg}_{\text{replicates}} - \text{R}_{\text{sample}}\right|}{\text{Avg}_{\text{replicates}}} * 100 = \frac{\left|\text{Avg}_{\text{replicates}} - \text{R}_{\text{duplicate}}\right|}{\text{Avg}_{\text{replicates}}} * 100$$

11.2.4 *Relative Percent Difference (%RPD)*:

$$\%\text{RPD} = \frac{\left|\text{R}_{\text{sample}} - \text{R}_{\text{duplicate}}\right|}{\left(\frac{\text{R}_{\text{sample}} + \text{R}_{\text{duplicate}}}{2}\right)} * 100$$

11.2.5 However, the range of the results equals two times the difference of a replicate from the average:

$$\left|\text{R}_{\text{sample}} - \text{R}_{\text{duplicate}}\right| = 2 * \text{Diff}_{\text{replicates}}$$

and

$$\frac{\left(2 * \text{Diff}_{\text{replicates}}\right)}{\text{Avg}_{\text{replicate}}} * 100 = 2 * \frac{\text{Diff}_{\text{replicates}}}{\text{Avg}_{\text{replicate}}} * 100 = 2 * \%\text{Diff}_{\text{replicates}}$$

11.2.6 Therefore

$$2 * \%\text{Diff}_{\text{replicates}} = \%\text{RPD}$$

11.2.7 Where (calculations 11.2.2 through 11.2.6):

Diff $_{\text{replicates}}$	= Difference of replicate result from the average
%Diff $_{\text{replicates}}$	= Percent difference of the replicates from the average
%RPD	= Relative percent difference
Avg $_{\text{replicates}}$	= Average of the two replicates
R $_{\text{sample}}$	= Result of the sample replicate
R $_{\text{duplicate}}$	= Result of the duplicate replicate

12 Waste Management

12.1 See GA EPD Laboratory SOP-EPD Laboratory Waste Management Standard Operating procedures. (See SOP reference 13.5)

13 References

13.1 Standard Methods for the Examination of Water and Wastewater, 20th Edition, p. 2-60 and 2-61, Method 2540G, (1997).
13.2 EPD Laboratory Quality Assurance Plan, online revision.
13.3 GA EPD Laboratory SOP's – Initial Demonstration of Capability SOP 6-001, online revision or Continuing Demonstration of Capability SOP 6-002, online revision.
13.4 GA EPD Laboratory SOP-EPD Laboratory Procedures for Control Charts and Control Limits SOP, SOP 6-025, online revision.
13.5 GA EPD Laboratory SOP-EPD Laboratory Waste Management SOP, SOP 6-015, online revision.
13.6 GA EPD Laboratory Safety Plan – EPD Laboratory Safety / Chemical Hygiene Plan & Fire Safety Plan, online revision.

14 Reporting Limits (RLs), Precision and Accuracy Criteria, and Quality Control Approach

Table 14.1 RLs for Method SM 2540G

Parameter/Method	Analyte	Matrix (aqueous)	
		RL	Unit
SM 2540G	Percent solids	none	%

Table 14.2 Acceptance Criteria for Method EPA SM 2540G

Method	Analyte	Accuracy Water (%R)	Precision Water (RPD)
SM 2540G	Percent solids	NA	10%

Table 14.3 Summary of Calibration and QC Procedures for Method SM 2540G

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
SM 2540G	%Solids	Sample Duplicate	10% of samples	QC Acceptance Criteria Table 14.2	Evaluate out of control event, initiate a corrective action and comment on sample if unable to reanalyze	

Updates to Previous Version:
Updated for online revision