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SM2540D – Total Suspended Solids Dried at 103ºC - 105° C

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1. Scope and Application

1.1. This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes. The practical range of the determination is 1.0 mg/L to 20,000 mg/L. A well-mixed sample is filtered through a weighed standard glass fiber filter. The residue retained on the filter is dried to constant weight at 103ºC - 105°C. The increase in the weight of the filter represents the total suspended solids.

1.2. This procedure is restricted to use by an analyst experienced in the operation of an oven, analytical balance and manifold system. Additionally, the analyst must complete the requirements of the GA EPD 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 GA EPD Chemical Hygiene Plan for additional information regarding chemicals required by this method.

2. Definitions

2.1 Refer to Section 3 and Section 4 of the Georgia EPD Laboratory Quality Assurance Plan (see SOP reference 13.5) for Quality Control Definitions.

3. Interferences

3.1. Filtration apparatus, filter material, pre-washing, post-washing and drying temperatures are specified as these variables have been shown to affect the results.

3.2. Exclude large floating particles or submerged agglomerates of nonhomogeneous materials from the sample if it is determined that their inclusion is not representative.

3.3. For samples high in dissolved solids, thoroughly wash the filter to ensure removal of dissolved material.

3.4. Too much residue in the filter will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.

3.5. Prolonged filtration times resulting from filter clogging may produce high results owing to increased colloidal materials captured on the clogged filter.

4. Safety

4.1. Refer to the Laboratory Safety/Chemical Hygiene Plan & Fire Safety Plan, online revision (Reference 13.7).

5. Apparatus and Equipment

5.1. Sample Container: half gallon plastic container or equivalent

5.2. Aluminum weighing dishes

5.3. Glass fiber filters 47 mm (Whatman grade 934AH, Gelman type A/E, or equivalent)

5.3.1 Pre-washed and dried glass fiber filters may also be used. Environmental Express PN# F92447MM, 47 MM, Washed and Dried Only or equivalent.

5.4. Three position manifold system

5.5. Drying oven set at 103 - 105º C

5.6. Filtration apparatus with filter funnel, reservoir and coarse fritted disk

5.7. Analytical balance capable of weighing to 0.1 mg

5.8. Desiccator, provided with desiccant containing a color indicator for moisture

5.9. Laboratory vacuum system

5.10. Graduated cylinder – Class A

5.11. Magnetic stirrer with TFE stirring bar

5.12. Class A Volumetric flask

6. **Reagents**

6.1. Reagent Water – Purified water which does not contain any measurable quantities of target analytes or interfering compounds for each compound of interest. (Deionized, HPLC, Milli-Q water or equivalent. Milli-Q water has a resistivity of 18.2 [MΩ·cm] @ 25° C and a TOC of 50 µg/L or less).

6.2. Celite 545 Analytical Filter Aid – Fisher Scientific Co. Catalog No. C212-500.

6.3. Laboratory Control Sample Stock Solution (LCSSS) - (100 mg/L) – 200 mg of Celite 545 Analytical Filter Aid (See 6.2) is dissolved and brought to volume in a 2000 ml volumetric flask with reagent water (see 6.1). Mix

well before each use using a magnetic stirrer. The solution is stable for up to one month.

6.3.1. A commercially available reference sample may also be used.

7. Sample Collection

7.1. Sample is collected in plastic ½ gallon container.

7.2. Samples should be cooled in ice as soon as possible and stored at 0 - 6°C (not frozen).

7.3. No chemical preservation is required.

7.4. Holding time is 7 days.

8. Calibration

8.1. 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.

9. Quality Control

9.1. Refer to Table 14. 1 for the Reporting Limits (RL), Table 14.2 for Quality Assurance Criteria and Table 14.3 for Quality Control (QC) procedures associated with this method.

9.2. Refer to reference 13.2. for training and certification procedures.

9.3. Refer to reference 13.3. for control charting procedures.

9.3.1. Note: All QC criteria for SM2540D are static, however control charts must still be generated on a semiannual basis for trend monitoring purposes.

9.4. Control Limits

9.4.1. Because the LCS is essentially the same as a continuing calibration confirmation standard (CCC), the EPD Laboratory sets the default LCS control limits as static and a range of 90% - 110% recovery.

9.4.2. The EPD Laboratory sets the LCSD recovery to the same limits as the LCS recovery.

9.4.3. The EPD Laboratory sets the default precision limits for the LCS/LCSD to be 0 – 15% RPD.

9.4.4. 10% of samples must be analyzed in duplicate. Precision of duplicates must agree within 5% of the average net result of the duplicates per SM 2540D (see reference 13.1.). This is the same as 10% RPD (see section 11.2. for justification).

9.4.5. The default control limits are presented in Table 9.4.5.1.

Table 9.4.5.1 Default QC Limits for SM 2540D

Analyte	QC Type	Accuracy (%R) LCL		UCL	Precision (%RPD)
	LCS/LCSD	90	-	110	15

Total Suspended Solids	Sample Duplicates	NA	10

9.5. Because the limits for this method are static, no Appendix A will be created for this SOP.

9.6. SM 2540D requires LCS's to be analyzed at a frequency rate of one LCS per batch of 20 samples.

9.7. Sample Duplicates must be analyzed at a frequency of 10% of all samples.

9.7.1. For batches of 1 – 10 samples, a minimum of one duplicate analysis is required.

9.7.2. For batches of 11 – 20 samples, a minimum of two duplicate analyses are required.

9.8. Filter as much volume as possible to yield between 2.5 mg and 200 mg of dried residue. If the volume filtered fails to meet the minimum 2.5 mg yield or exceeds the maximum 200 mg yield, initiate a corrective action and comment on the sample (See 10.4.14 and 10.4.15).

10. Procedure

10.1. **Always wear gloves and use tweezers when handling the aluminum dishes and filters**.

10.2. Make sure the manifold is connected to the 20L plastic carboy located under the counter. Note: When disposing of sample waste from the carboy, make sure to turn off vacuum before attempting to unscrew lid of waste container or the lid may be too hard to get off.

10.3. <u>Preparation of Filters and Aluminum Dishes:</u>

10.3.1. If using pre-weighed/pre-dried filters made by Environmental Express PN# F92447MM or equivalent, store filters in desiccator if not using immediately after opening. These filters are already clean and dry and ready for analysis.

10.3.2. Non pre-weighed/pre-dried filters may be prepared in advance and stored in a desiccator. Place filters, wrinkled side up, in the bottom of a thoroughly cleaned filter funnels.

10.3.2.1. Apply vacuum and wash filters with at least 60 ml of reagent water (see 6.1.) using a clean Class A graduated cylinder. Continue suction to remove all traces of water. Turn off vacuum and discard washings. Prepare enough clean filters to be used for samples expected each week.

10.3.2.2. Using tweezers remove filters from filtration apparatus and transfer to aluminum weighing dishes. Dry in oven set to 103ºC - 105°C for at least one hour, but overnight is preferable to ensure thorough drying.

10.3.2.3. Remove dishes from oven and immediately place in desiccator labeled for clean filters. Cool in desiccator for at least one hour before weighing. Weigh the filters and record their weights.

10.3.2.4. Repeat drying in the 103ºC - 105°C oven for one more hour, then cool in the desiccator for one hour and weigh again. Continue drying and weighing each filter until a constant weight is obtained or until weight change is less than 4% of the previous weighing or 0.5 mg from the previous weight, whichever is less.

10.3.2.5. Alternately, if you prepare clean filters and allow them to dry in the oven at 103ºC - 105°C overnight, you do not have to obtain a constant weight by drying and weighing. A study was performed to show that drying overnight is as effective as drying to constant weight with repeated drying/weighing.

10.3.3. Store filters in desiccator until needed. Minimize opening and closing of the desiccator. Moist air can enter and affect filter weight. Make sure that the desiccant is dark blue. Replace or recharge desiccant when color changes to light blue or pink.

10.4. Sample Analysis:

10.4.1. Remove sample bottles, standards, and reagents from cold storage and allow equilibration to room temperature prior to sample preparation and/or analysis.

10.4.2. Perform backlog of pending samples. Batch samples in groups of 20. At least 10% of samples must be analyzed in duplicate.

10.4.2.1. Using the batch sheet as a reference, fill in the Sample ID# column on the Solids result worksheet. Note: If drying samples overnight in the oven, use the SUSSOL Overnight.xlsx worksheet. If drying/weighing samples the same day, use the SUSSOL Sameday.xls worksheet.

10.4.3. Check the balance log to determine if the balance has been calibrated on each day of analysis. If it has not, calibrate the balance.

10.4.4. Only remove a few prepared filters out of the desiccator at a time when weighing to avoid prolonged exposure to ambient moisture prior to weighing.

10.4.5. Weigh aluminum dish and filter combination, on a calibrated balance. Record the weight and dish # on the Solids Result worksheet. Be sure that

the same weighing dish and filter are kept together from this point forward until analysis has been completed and the filter discarded.

10.4.6. Once all prepared filters needed for the batch have been weighed, make sure the filter funnels on the manifold have been thoroughly cleaned with reagent water.

10.4.6.1. Transfer the filter from the aluminum weighing dish to the cleaned filter funnel on the manifold, using tweezers. Record manifold # on worksheet.

10.4.6.2. Make sure the valves are closed on all three manifolds. The red dot on the valve handle should be facing up.

10.4.6.3. Turn on the vacuum to begin suction.

10.4.6.4. Wet the filter with small volume of reagent water to seat it. Do this prior to every field and QC sample analyzed.

10.4.7. Prepare a Method Blank by measuring 1000 ml of reagent water (See 6.1) and pour onto the assigned Method Blank filter. Continue to apply vacuum until all traces of water have been pulled through filter.

10.4.7.1. Turn off vacuum and carefully remove filter from filtration apparatus and transfer to aluminum weighing dish. Make sure to analyze one Method Blank for every batch of samples.

10.4.8. Analyze a LCS and LCSD with each batch.

10.4.8.1. Prior the analyzing the LCS and LCSD, the Laboratory Control Sample Stock Solution (LCSSS) (see section 6.3.) must be stirred for at least 30 minutes with a magnetic stir bar on a stir plate. Stir at a steady speed.

10.4.8.2. Prepare and analyze the LCS and LCSD. Immediately prior to the analysis, stop stirring and quickly measure 100 ml of the LCSSS in a Class A graduated cylinder.

10.4.8.3. Next, pour the 100 ml of LCSSS onto the filter. Rinse the cylinder with reagent water in a squirt bottle and add the rinsate to the filter.

10.4.8.4. Record this volume on the worksheet. Thoroughly clean the filter funnels on the manifold with reagent water.

10.4.8.5. Transfer the filter assigned for the LCS from the aluminum weighing dish to cleaned filter funnel on the manifold using tweezers.

10.4.8.6. Make sure the valves are closed on all three manifolds. The red dot on the valve handle should be facing up.

10.4.8.7. Turn on the vacuum to begin suction.

10.4.8.8. With vacuum applied to the filter, wet the filter with small volume of reagent water to seat it

10.4.8.9. Next, pour the 100 ml of LCSSS onto the filter. Rinse the cylinder with reagent water in a squirt bottle and add the rinsate to the filter.

10.4.8.10. Rinse the filter funnel with approximately 30 ml of reagent water to remove all remnants of the LCSSS from the sides of the funnel and onto the filter.

10.4.8.11. Continue to apply vacuum until all traces of water have been pulled through the filter.

10.4.8.12. Turn off vacuum and carefully remove filter from the weighing dish and transfer to the appropriate aluminum weighing dish.

10.4.8.13. ***Rinse the graduated cylinder and filter funnel thoroughly between each sample (QC or field).***

10.4.8.14. Repeat steps 10.4.8.2. through 10.4.8.13. for the LCSD.

10.4.9. Field Samples:

10.4.9.1. Filter as much volume as possible to yield between 2.5 mg and 200 mg of dried residue. If the volume filtered is 1000 ml then no comment is needed. If volume filtered fails to meet the minimum 2.5 mg yield or exceeds the maximum 200 mg yield and is less than 1000 ml, initiate a corrective action and "J" flag and comment on the sample.

10.4.9.2. For each batch, choose a QC sample and analyze a sample duplicate for a minimum of 10% of the samples.

10.4.9.3. One sample is analyzed in duplicate for batches of up to10 samples.

10.4.9.4. Two samples analyzed in duplicate for batches of 11 – 20 samples.

10.4.9.5. Rinse the graduated cylinder and filter funnel thoroughly.

10.4.9.6. Shake appropriate sample vigorously, then immediately measure an appropriate volume of sample (see 10.4.9.1) using a clean Class A graduated cylinder.

10.4.9.7. Place filter funnel on manifold position #3 and make sure the filter funnel has been thoroughly cleaned with reagent water.

10.4.9.8. Transfer the filter assigned for the sample from the aluminum weighing dish to the cleaned filter funnel using tweezers and begin suction. Record manifold position # on worksheet.

10.4.9.9. With vacuum applied to the filter, wet the filter with a small volume of reagent water to seat it. Next, pour measured sample through the filter at a constant rate. Keep a swirling motion going between volume increments.

10.4.9.10. Using squirt bottle containing reagent water, thoroughly rinse the walls of the graduated cylinder and pour the rinseate onto the filter. Thoroughly rinse the filter funnel with reagent water. Continue to apply vacuum until all traces of sample water and rinsate have been pulled through filter.

10.4.9.11. If complete filtration of sample takes more than 10 minutes, start over with a clean filter and decrease sample volume. Note: filter as much sample as possible to make sure you meet minimum residue requirement of method.

10.4.9.12. Carefully remove filter from filtration apparatus and transfer to aluminum weighing dish.

10.4.10. Rinse the graduated cylinder and filter funnel thoroughly between each sample (QC or field).

10.4.10.1. If filtering of a sample takes longer than a minute or so, analyst can utilize manifold position #2 for next sample.

10.4.10.2. If filtering of sample in position #2 takes longer than a minute or so, analyst can utilize the manifold position #1 for next sample

10.4.11. After all QC and field samples and have been filtered, dry overnight at 103ºC - 105ºC in an oven, cool in desiccator for at least one hour before weighing. Record the weight on the Sussol Blank Overnight worksheet. Note: A study was performed to show that drying overnight was as effective as drying to constant weight with repeated drying/weighing.

10.4.12. Alternately, for same day drying, after all QC and field samples have been filtered, dry for a least one hour at 103ºC - 105ºC in an oven, cool in desiccator for at least one hour, and record weight on the Sussol Blank Same Day worksheet. Repeat drying in the 103° - 105°C oven for one more hour, then cool in the desiccator for at least one hour and weigh again. Continue drying and weighing each filter until a constant weight is obtained or until weight change is less than 4% of the previous weighing or 0.5 mg from the previous weight, whichever is less.

10.4.13. After all filters for the batch have been weighed and recorded, enter the volume and weight into the appropriate Excel Solids Result spreadsheet to calculate the residue and final results. Print completed spreadsheet and review results.

10.4.13.1. Verify calculation of one result from Excel Solids Result spreadsheet and write calculation on spreadsheet in noted section.

10.4.14. A corrective action will need to be filled out for all samples with residue less than 2.5 mg, unless 1000 ml is filtered. The following comment will need to be placed on all affected samples; “SUSSOL-SM-SM2540D – <J> Value Estimated. Insufficient sample residue due to insufficient sample filtration volume.” Then type corrective action number.

10.4.15. A corrective action will also need to be filled out for all samples with residues that exceed 200 mg. The following comment will need to be placed on all affected samples: “SUSSOL-SM-SM2540D – <J> Value Estimated. Sample residue exceeds the recommended maximum residue of 200 mg.” Then type corrective action number.

11. Calculations

11.1. Percent Recovery:

$$\text{LCS, LCSD, ICV \%Recovery} = \frac{R_{spike}}{\text{Expected Result}} * 100$$

11.1.1. Where:
R_{spike} = Calculated spike concentration

11.2. Duplicate Precision:

11.2.1. SM 2540D (see 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*:

$$\text{Average } (Avg_{replicates}) = \frac{R_{sample} - R_{duplicate}}{2}$$

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:

$$Diff_{replicates} = \frac{|Avg_{replicates} - R_{sample}|}{Avg_{replicates}} = \frac{|Avg_{replicates} - R_{duplicate}|}{Avg_{replicates}}$$

and

$$\%Diff_{replicates} = \frac{|Avg_{replicates} - R_{sample}|}{Avg_{replicates}} * 100 = \frac{|Avg_{replicates} - R_{duplicate}|}{Avg_{replicates}} * 100$$

11.2.4. *Relative Percent Difference (%RPD)*:

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

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

$$|R_{sample} - R_{duplicate}| = 2 * Diff_{replicates}$$

and

$$\frac{(2 * Diff_{replicates})}{Avg_{replicate}} * 100 = 2 * \frac{Diff_{replicates}}{Avg_{replicate}} * 100 = 2 * \%Diff_{replicates}$$

11.2.6. therefore

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

11.2.7. Where (calculations 11.2.2. through 11.2.6.):

$\text{Diff}_{\text{replicates}}$ = Difference of replicate result from the average

$\%\text{Diff}_{\text{replicates}}$ = Percent difference of the replicates from the average

%RPD = Relative percent difference

$\text{Avg}_{\text{replicates}}$ = Average of the two replicates

R_{sample} = Result of the sample replicate

$R_{\text{duplicate}}$ = Result of the duplicate replicate

11.3. Sample Concentration Calculation:

11.3.1. Sample concentration is calculated as Filterable Residue in mg/L:

$$\text{Total Suspended Solids } \left(\frac{\text{mg}}{\text{L}}\right) = \frac{(\text{A} - \text{B}) * 1000}{C}$$

11.3.2. Where:

A= weight of dried residue + dish in mg

B= weight of dish in mg

C= volume of sample used in ml

12. Waste Management

12.1. See GA EPD Laboratory SOP-EPD Laboratory Waste Management Standard Operating procedures (Reference 13.4.).

13. References

13.1. Standard Methods 2540D, Total Suspended Solids Dried at 103° - 105° C, 1997, Editorial revisions, 2011.

13.2. GA EPD Laboratory SOPs- Initial Demonstration of Capability SOP 6-001, online revision and/or Continuing Demonstration of Capability SOP 6-002, online revision.

13.3. GA EPD Laboratory SOP- EPD Laboratory Procedures for Control Charting and Control and Control Limits SOP, SOP 6-025, online revision.

13.4. GA EPD Laboratory SOP- EPD Laboratory Waste Management SOP, SOP 6-015, online revision.

13.5. Manual for the Certification of Laboratories Analyzing Drinking Water, EPA/815-R-05-004, January 2005 or later.

13.6. EPD Laboratory - Quality Assurance Plan, Revision online revision.

13.7. GA EPD Laboratory Safety Plan – EPD Laboratory Safety/Chemical Hygiene Plan & Fire Safety Plan, online revision.

13.8. GA EPD Laboratory SOP – Balance Use, Maintenance and Training in the Inorganics Lab, SOP 3-013, online revision.

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

Table 14. 1 RLs for Method SM 2540D

Parameter/Method	Analyte	Matrix (aqueous)	
		RL	Unit
SM 2540D	Total Suspended Solids	2.5	mg/L

Table 14. 2 Acceptance Criteria for Method SM 2540D

QC Type	Analyte	Accuracy Water (%R)	Precision Water (RPD)
CCC(LCS)[1]	Total Suspended Solids	90-110	15
Sample Duplicate	Total Suspended Solids (Sample Duplicate)	--	10

[1]*LCS/LCSD for this analysis are interchangeable with CCCs. Therefore, control limits are static.*

Table 14. 3 Summary of Calibration and QC Procedures for Method SM 2540D

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
SM 2540D	Total Suspended Solids	Initial Demonstration: Demonstrate ability to generate acceptable accuracy and precision using four analysis of a QC check sample, a method blank and a blind sample. In addition, the analyst must prepare one standard.	Once per analyst	QC Acceptance Criteria Table 14.2 and Initial Demonstration SOP (Reference 13.2)	Recalculate results: locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Continuing Demonstration (CDC): Demonstrate ability to generate acceptable accuracy and precision using four analyses of a QC check sample (LCS) and a laboratory blank	Every 6 months once IDF has been approved	QC Acceptance Criteria Table 14.2 and Continuing Demonstration SOP	Recalculate results; Fix problem the rerun demonstration	
		Method Blank	One per batch	Value must be < RL	Correct problem then analyze blank and all samples processed with the contaminated blank	If unable to re-analyze, flag with a "B"
		Laboratory Control Sample (LCS/LCSD)	One LCS and LCSD each per analytical batch	QC Acceptance Criteria Table 14.2	Correct problem then reanalyze the LCS/LCSD and all samples in affected batch	If unable to reanalyze, flag with a "J"
		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	
SM 2540D	Total Suspended Solids	Sample Residue	Calculate for all samples	Choose a sample volume that will yield between 2.5 mg and 200 mg of dried residue	Initiate a corrective action and "J" flag comment if volume filtered fails to meet the minimum 2.5mg yield or is above the 200 mg maximum.	

Updates to Previous Version:
Section 6
Section 10
Section 13