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SM2540C – Total Dissolved Solids Dried at 180º 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 10 mg/L to 20,000 mg/L. A well-mixed sample is filtered through standard glass fiber filter. The filtrate is retained in a Pyrex dish, evaporated, and dried to constant weight at 180º C. The weight of residue in the evaporating dish in milligrams is divided by the volume of sample filtered in liters to determine the concentration of total dissolved solids in the sample.

1.2. This procedure is restricted to use by an analyst experienced in the operation of an oven and analytical balance. 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.

2.2 Primary Source (PS) – A standard that is used to make up the calibration points of a curve.

2.3 Second Source (SS) – A standard made from a manufacturer other than that of the primary source.

2.4 Initial Calibration Verification (ICV) – An ICV is a second source standard that is used to verify the correctness of the primary source calibration curve. The

ICV is run at a level equal to that of a Laboratory Control Sample (LCS) or the midpoint on the calibration curve.

2.5 Continuing Calibration Check (CCC) or Continuing Calibration Verification (CCV) – A standard used to verify that the response of the instrument has not changed since initial calibration.

2.6 Calibration Blank (CB), Initial Calibration Verification Blank (ICB), Method Blank (MBLK) or Continuing Calibration Blank (CCB) – A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes.

2.7 LCS (Laboratory Control Sample) and LCSD (Laboratory Control Sample Duplicate) are prepared by spiking laboratory reagent water, Ottawa sand or air sampling device with the target analyte or compound. They are used to validate the analytical batch with respect to accuracy and precision.

3. Interferences

3.1. Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and /or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.

3.2. Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180º C to ensure that all the bicarbonate is converted to carbonate.

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

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. Pyrex dishes

5.3. Drying oven set at 103 - 105º C

5.4. Oven set at 180º C ± 2º C

5.5. Suction flask

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

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

5.8. Porcelain or Magnetic filter funnel

5.9. Rubber stopper

5.10. Analytical balance capable of weighing to 0.1 mg
5.11. Laboratory vacuum system
5.12. Tweezers for handling filters
5.13. Tongs for handling Pyrex dishes
5.14. Dessicator, provided with desiccant containing a color indicator for moisture
5.15. Pan or tray, covered in aluminum to hold and carry Pyrex dishes
5.16. Class A Graduated cylinder
5.17. Class A Volumetric flasks

6. **Reagents**

6.1. Reagent Water – Purified water which does not contain any measureable 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. Laboratory Control Sample Stock Solution - KHP (300 mg/L) – Dry a thin layer of $KHC_8H_40_4$ (KHP) crystals in beaker for 1 hour at 103° C - 110° C. After cooling in dessicator, weigh 600 mg of the dried KHP and dissolve in 2000 ml of reagent water. The solution is stable for up to one month and should be refrigerated at 6° C or less. A commercially available reference sample may also be used.

6.3. RBS - A low-foaming, liquid detergent for use in automatic washers and ultrasonic baths to clean laboratory glassware, stainless steel, plastic and other hard materials.

6.4. 1:1 HCL Solution – Add 250 ml of reagent water to a clean 1L beaker. Add 250 ml hydrochloric acid. Stir well using a stir bar and allow the solution to cool. Transfer to appropriate acid wash bottle.

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 standards 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 SM2540C 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 80% - 120% 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 – 25% 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 2540C (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 2540C

Analyte	QC Type	Accuracy (%R) LCL	Accuracy (%R) UCL	Precision (%RPD)
TDS	LCS/LCSD	80	120	25
	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 2540C requires LCSs to be analyzed at a frequency rate of one LCS per batch of 20 samples.

9.7. 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.

10. Procedure

10.1. Always wear gloves and use tweezers when handling filters and tongs when handling Pyrex dishes.

10.2. Preparation of Glass Fiber Filters:

10.2.1. 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.

10.2.2. 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.

10.2.2.1. Place bottom of 47 mm magnetic filter funnel on top of the 125 ml side arm suction flask using a rubber stopper to create a seal. Connect the flask to the laboratory vacuum system using rubber tubing.

10.2.2.2 Using tweezers, place a 47 mm glass fiber filter, wrinkled side up, on the bottom of the 47 mm magnetic filter funnel. Then place the lid of the funnel on securely.

10.2.2.3 Apply vacuum using a reagent water squirt bottle, wash the filter with at least 60 ml of reagent water. 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.

10.2.2.4 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.

10.2.2.5 Remove from oven, cool, and place in desiccator until needed. Make sure that the desiccant is dark blue. Replace or recharge desiccant when color changes to light blue or pink.

10.3. Preparation of Evaporating Dishes:

10.3.1. To clean Pyrex dishes, soak them for about an hour in a solution made up of 20 ml of RBS solution (see 6.3.) and 1 L of hot tap water. Use a scrub brush if needed to remove solids. Rinse with hot tap water followed by a final rinse with reagent water.

10.3.2. Next, rinse the Pyrex dishes with a 1:1 HCl solution followed by a rinse with reagent water.

10.3.3. Clean dishes are heated to 180 +/- 2º C overnight in oven. Store dishes in the desiccator until needed. Allow dishes to cool in desiccator for at least 2 hours before using for sample and QC analysis. Make sure that the desiccant is dark blue. Replace or recharge desiccant when color changes to light blue or pink.

10.4. Sample and QC 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.

10.4.3 Analyze an LCS and LCSD consisting of 100 ml each of the Laboratory Control Sample Stock Solution (see section 6.2.) with each batch.

10.4.4 Analyze a Method Blank consisting of 100 ml of reagent water with each batch.

10.4.5 Analyze duplicate samples as directed in section 9.7.

10.4.6 Always wear gloves and use tweezers when handling filters and tongs when handling Pyrex dishes.

10.4.7 Using a batch sheet as a reference, fill in the Sample ID# column on the TDS result worksheet.

10.4.8 Check the balance log to determine if the balance has been calibrated on each day of analysis. If it has not, calibrate the balance. Remove the prepared filters from the desiccator.

10.4.9 Weigh the clean Pyrex dish immediately before use. Record the weight on the worksheet.

10.4.10 Place bottom of 47 mm magnetic filter funnel on top of the 125 ml side arm suction flask using a rubber stopper to create a seal. Connect the flask to the laboratory vacuum system using rubber tubing.

10.4.11 Using tweezers, place a prepared 47 mm glass fiber filter, prepared as described in section 10.7., wrinkled side up, on the bottom of the 47 mm magnetic filter funnel. Place the lid of the funnel on securely and apply vacuum. Wet the filter paper with a few squirts of reagent water to help funnel seal properly (10 -15 ml max).

10.4.12 Mix sample well and immediately measure and filter 100 ml of sample with vacuum through the prepared filter.

10.4.13 Rinse filter, bottom spout and wall of funnel with 30 ml of reagent water and allow complete drainage.

10.4.14 Pour all of filtrate and washings into the pre-weighed Pyrex dish prepared per section 10.3.

10.4.15 Thoroughly rinse the graduated cylinder, magnetic filter funnel, bottom spout and flask with approximately 30 ml of reagent water using a squirt bottle between samples.

10.4.16 Repeat steps 10.4.10 – 10.4.15 until all samples and QC have been filtered. Place the Pyrex dishes on an aluminum covered pan and place in oven set at 103-105^{o} C to evaporate samples to dryness, usually overnight.

10.4.17 Once samples have completely evaporated, place them in an oven set at 180° C ± 2° C for at least two hours. Oven temperature and time is recorded on TDS worksheet.

10.4.18 Remove dishes from oven and place in desiccator for at least two hours to cool completely. Record oven #, oven temperature, and the time the samples were removed from oven on TDS worksheet. After samples have cooled, weigh dish and record weight on TDS worksheet.

10.4.19 Repeat cycle of drying, cooling, desiccating, and weighing until constant weight is obtained. Weight stability may be determined by a weight change of less than 4% of previous weight or 0.5 mg, whichever is less for a minimum of 3 samples.

10.4.20 If the sample results do not yield a dried residue of between 2.5 and 200 mg, J flag result and comment on sample.

11 Calculations

11.1 Percent Recovery:

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

11.1.1 Where:

R_{spike} = Calculated spike concentration

11.2 Duplicate Precision:

11.2.1 SM 2540C (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*:

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

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):

$\text{Diff}_{\text{replicates}}$ = 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:

$$\text{Filterable Residue}\left(\frac{\text{mg}}{\text{L}}\right) = \frac{(\text{A} - \text{B}) * 1000}{C}$$

11.3.2 Where:

A= weight of dried residue + clean and dried 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. See SOP reference 13.4.

13 References

13.1 Standard Methods 2540C, Total Dissolved Solids Dried at 180ºC, SM Online 2015.

13.2 GA EPD Laboratory SOP's- 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, 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 2540C

Parameter/Method	Analyte	Matrix (aqueous)	
		RL	Unit
SM 2540C	Total Dissolved Solids	10	mg/L

Table 14.2 Acceptance Criteria for Method SM 2540C

QC Type	Analyte	Accuracy Water (%R)	Precision Water (RPD)
CCC(LCS)[1]	Total Dissolved Solids	80-120	25
Sample Duplicate	Total Dissolved 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 2540C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
SM 2540C	Total Dissolved 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	Recalculate results: locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Continuing Demonstration of Capability Continuing Demonstration: Demonstrate ability to generate acceptable accuracy and precision using a variety of analysis options of a QC sample(s)	Required every Six Months once IDF has been approved.	QC Acceptance Criteria Table 14.2 and Continuing Demonstration SOP	Recalculate results: locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
SM 2540C	Total Dissolved Solids	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 reanalyze, if possible	
		Sample Residue	Calculate for all samples	Choose a sample that will yield between 2.5 mg and 200 mg of dried residue	Initiate a corrective action, "J" flag and comment if volume fails to meet the minimum 2.5 mg yield or is above the 200 mg maximum.	If unable to reanalyze, flag with a "J"

Updates to Previous Version:

Updated for online Revision and updated Section 2.