

STANDARD OPERATING PROCEDURE

For
SM 2540B

THE DETERMINATION OF TOTAL SOLIDS TOTAL SOLIDS DRIED AT 103-105⁰C

SOP #: SM 2540 B

REVISION #: 0

DATE: October 2000

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LIST OF REVISIONS

Rev. #	Date	Description of Revision	Page #
0	October 2000	None	
0	October 2000	Replaced old DEP Logo with state seal + MassDEP (December 2006)	Title page & header



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METHOD # SM 2540 B
Approved for NPDES

TITLE: Total Solids Dried at 103-105⁰C

1.0 SCOPE AND APPLICATION

- 1.1 Total solids is the term applied to the material residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature. Total solids include total suspended solids, the portion of total solids retained by a filter, and total dissolved solids, the portion that passes through the filter.
- 1.2 Residues dried at 103 to 105⁰C may retain not only water of crystallization but also some mechanically occluded water. Loss of CO₂ will result in conversion of bicarbonate to carbonate. Loss of organic matter by volatilization usually will be very slight. Because removal of occluded water is marginal at this temperature, attainment of constant weight may be very slow.
- 1.3 Results for residues high in oil and grease may be questionable because of the difficulty of drying to constant weight in a reasonable time.

2.0 SUMMARY OF METHOD

- 2.1 A well-mixed sample is evaporated in a weighed dish and dried to constant weight in an oven at 103 to 105⁰C. The increase in weight over that of the empty dish represents the total solids. The results may not represent the weight of actual dissolved and suspended solids in wastewater samples.

3.0 DEFINITIONS

- 3.1 Occluded Water - liquid water that is entrapped in microscopic pockets spaced irregularly throughout solid crystals.
- 3.2 Water of Crystallization - water that forms an integral part of the molecule or crystal structure of a component of the solid. It exists in stoichiometric quantities.
- 3.3 Nonessential Water - It does not occur in any sort of stoichiometric proportion. The solid as a consequence of physical forces retains it.

4.0 INTERFERENCES

- 4.1 Highly mineralized water with a significant concentration of calcium, magnesium, chloride, and or sulfate may be hygroscopic and require prolonged drying, proper desiccation, and rapid weighing.
- 4.2 Because excessive residue in the dish may form a water trapping crust, limit sample to no more than 200mg residue.



5.0 SAFETY

- 5.1 Standard laboratory protective clothing is required. A facemask is worn for hazardous samples.

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Analytical Balance - capable of weighing to 0.1 mg.
- 6.2 Drying Oven - for operation at 103 to 105⁰C.
- 6.3 Evaporating Dishes – Porcelain.
- 6.4 Desiccator - provided with a desiccant containing a color indicator or moisture concentration.

7.0 REAGENTS AND STANDARDS

- 7.1 Sodium Chloride Standard- 0.824 g/L of NaCl.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

- 8.1 Begin analysis as soon as possible because of the impracticality of preserving the sample. Refrigerate sample at 4⁰C up to time of analysis to minimize microbiological decomposition of solids. Preferably do not hold samples more than 24 hours. In no case hold sample more than 7 days. Bring samples to room temperature before analysis.

9.0 QUALITY CONTROL

- 9.1 An LFB of NaCl is analyzed with every batch.
- 9.2 An LRB and Duplicates (10% frequency) are analyzed with every batch.
- 9.3 A QCS is analyzed quarterly.
- 9.4 The analytical balance is calibrated every day.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Calibration of Analytical Balance
- 10.1.1 Press and hold Cal/Menu button until 'Cal' appears in display.
- 10.1.2 When blinking "100.0000" appears in display, place 100 g weight on pan.
- 10.1.3 Remove when blinking '0.0000' appears in display. The calibration is complete when 'Cal done' appears in display.



11.0 PROCEDURE

- 11.1 Preparation of evaporating dish - If only total solids are to be measured, heat clean dish to 103 to 105°C for 1 h. Store and cool dish in desiccator until needed. Weigh immediately before use.
- 11.2 Choose a sample volume that will yield a residue between 10 and 200 mg. Shake sample vigorously and transfer appropriate volume (usually 50 mL) to graduated cylinder.
- 11.3 Add sample to porcelain dish and place in drying oven at 97 to 98°C. Evaporate to dryness.
- 11.4 Increase oven temperature to 103 to 105°C and dry for an additional (at least) 1 hour.
- 11.5 Cool dish in desiccator to balance temperature, and weigh. Repeat cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained, or until weight change is less than 4% of previous weight or 0.5 mg, whichever is less.

12.0 DATA ANALYSIS AND CALCULATIONS

$$mg \text{ total solids} / L = \frac{(A - B \times 1000)}{\text{Sample vol., mL}}$$

A = weight of dried residue + dish, mg

B = weight of dish, mg

13.0 METHOD PERFORMANCE

- 13.1 SM: Single laboratory duplicate analysis of 41 samples of water and wastewater was made with a standard deviation of differences of 6.0 mg/L.
- 13.2 LFB of NaCl - True Value = 824 mg/L; Avg. Rec. = 814 mg/L or 98.8% Standard Deviation = 23 mg/L.
- 13.3 Duplicates - The RPD = 4.6 mg/L. The Sd of the RPD = 4.4 mg/L.

14.0 POLLUTION PREVENTION

- 14.1 There are no hazardous chemicals used in this method.

15.0 WASTE MANAGEMENT

- 15.1 The U.S. Environmental Protection Agency requires that laboratory waste management practices are consistent with all applicable rules and regulations.



16.0 REFERENCES

- 16.1 Standard Methods for the Examination of Water and Wastewater, 19th Edition, 1995, American Public Health Association.

17.0 TABLES AND VALIDATION DATA

TABLE 1. Validation Data

Sample # Value	Reported Value	True Value	Acceptance

**TABLE 2. Precision of Total Solids in Reagent Water
by Standard Method 2540 B**

Date	Based on Sample Numbers	No. of Samples (n)	Precision (RPD) ^b					
			Mean	SD ^a	Warning Limits (± 2 SD)		Control Limits (± 3 SD)	
					Upper (UWL)	Lower (LWL)	Upper (UCL)	Lower (LCL)
^a SD = Standard deviation								
^b RPD = Relative percent difference								



TABLE 3. Method Detection Limit (MDL) for Total Solids Analysis in Reagent Water by Standard Method 2540 B

Date of Study	No. of Samples Spiked (n)	Spiked Concentration (mg/L)	Accuracy (Mean % Recovery ^a)	Precision (SD ^b in mg/L)	MDL (mg/L)
<p>^a Recovery of spiked concentration</p> <p>^b SD = standard deviation of mean concentration measured</p> <p>^c Method Detection Level (MDL) is 0.02 mg/L F. WES' Reporting Detection Level (RDL) is 0.10 mg/L F.</p>					