Applied Microbiology
&
Biotechnology Laboratory

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Standard Operating Procedure

AMBL-105-A

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Total Solids by Gravimetric Determination

METHOD SUMMARY

This SOP describes the procedure for measuring total solids in water and wastewater. This method is based on Method 2540 B of *Standard Methods for the Examination of Water and Wastewater*, 23rd Edition.

ENVIRONMENTAL HEALTH AND SAFETY

Hazards Assessment: This method involves the use of a convection oven and optionally a muffle furnace, and the handling of natural waters or untreated wastewaters that potentially contain pathogenic organisms. The specific hazards associated with this method are as follows.

Burns: Burns to the hands or arm are possible if the sides of the convention oven or muffle furnace are touched when placing the sample into or removing it from the oven or furnace. Burns will also occur if the hot porcelain evaporating dish itself is touched.

Biological Hazard: The presence of pathogenic organisms must be assumed, regardless of the water sample source. Natural waters, sewage and wastewater all contain bacteria, funguses, parasites, and viruses that can lead to intestinal or other infections, including but not limited to diarrhea, fever, nausea, cramps, vomiting, headaches, conjunctivitis (pink eye) and Hepatitis A.

Safety Equipment and Engineering Controls: This method requires that you wash your hands with soap when finished handling samples and that an eye wash station be located nearby.

Personal Protective Equipment (PPE): This method requires the use of the following PPE.

Gloves (nitrile, PVC or neoprene)

Safety goggles or glasses

Laboratory coat

Analysis-derived Wastes and Disposal:

Waste Generated	Hazardous (Y / N)	Disposal
This procedure generates a dried solid residue.	N	The solid residue is considered desiccated and to have heat-killed bacteria (>71°C). Remove the dried residue, which may be disposed in the laboratory trash. Residue removed while washing the dish may be rinsed down the laboratory sink.

METHOD DESCRIPTION

1.0 Introduction and Applicability

Total solids is a measure of the suspended and dissolved matter in a water that remains after all the water has been evaporated. Solids affect water quality by making it unfit or unsafe to drink, unsuitable for use in many industrial applications, and aesthetically unsuitable for many other applications as well. A well-mixed sample of a known volume is evaporated to a constant weight condition in an oven maintained at a temperature of 103°C to 105°C. The mass of the dried sample's solids is determined and used to calculate the concentration of total solids in the sample.

This method is applicable for measurement of total solids in all natural waters, in raw, process and treated agricultural, municipal and industrial wastewaters, and generally in wastewater slurries behaving as a Newtonian fluid and where the sample volume may be reliable measured. This method is not applicable to non-Newtonian fluids or treated drinking water.

2.0 Apparatus

a. Dish, for sample evaporation, made of porcelain (optionally use platinum or borosilicate glass).
b. Graduated cylinder, Class A
c. Wide-bore pipet, Class B
d. Hot plate or heating block (optional) for evaporating samples in a pre-drying step and capable of maintaining a temperature <100°C (preferably at 80°C) to prevent sample boiling.
e. Convection oven operated at 80°C (optional) for evaporating samples in a pre-drying step.
f. Convection oven operated at 103-105°C for drying samples to a constant weight condition.
g. Muffle furnace operated at 550°C ± 50°C.

h. Desiccator containing a desiccant that responds (color change) to moisture or a hygrometer that measures moisture.
i. Analytical balance capable of weighing to the nearest 0.1 mg or less.
j. Magnetic stirrer and stir bar (optional).
k. Blender or homogenizer (optional)
l. Beaker, low-form Class B or Class A having a volume sufficient enough to fully contain the sample and prevent sample loss from spillage or splattering when mixing.

3.0 Procedure

a. Read Method 2540B Total Solids Dried at 103-105°C (*Standard Methods*).

b. Prepare a sample evaporating dish by ensuring that it is cleaned and does not contain residue from a previous use, and use deionized or distilled water for the final cleansing rinse. Dry the clean evaporating dish either in a convention oven at a temperature of 103-105°C for no less than 60 minutes if measuring only total solids or alternately, or ignite the evaporating dish in a muffle furnace at a temperature of 550°C ± 50°C for no less than 15 minutes if volatile solids will be determined (see SOP 105F). Cool the cleaned and dry dish to room temperature, weigh and record its weight - this is the tared weight of the dish. Store the pre-weighed dish in a desiccator until used.

c. Equilibrate the sample's temperature to that of the room's temperature and use a wide-bore pipet or graduated cylinder to transfer a volume of well-mixed sample into a pre-weighed dish. Select a sample volume that will result in a dried residue ranging from 2.5 to 200 mg. If necessary, add additional sample portions to the same dish after the previous portion has been evaporated. Record the total volume of sample added.

d. Evaporate the sample on a hot plate or in a convection oven at a temperature of 80°C to remove the free-standing water.

e. Dry the sample in a convection oven at a temperature of 103-105°C for no less than 60 minutes. Drying samples overnight is acceptable and an appropriate procedural step for the AMBL. In most circumstances, this ensures that constant weight has been achieved.

f. Remove the dish containing he sample from the oven, cooling it to room temperature and then weigh it. Record this as the first 103°C weight.

g. Repeat the drying cycle for no less than 60 minutes, and again cool, weigh and record the second 103°C weight.

h. Calculate the weight change between the first and second weights, and if the change is >0.5 mg, continue repeating the drying cycle until the

change in weight between the final weight and the previous weight is ≤0.5 mg. Record and use this final 103°C weight.

4.0 Calculation and Reporting

a. Calculate the concentration of total solids

Total Solids, as mg TS/L $\frac{(A - D) \times 1{,}000}{S}$

where A = final 103°C weight of the dried residue + the tared dish, mg,

D = tared dish weight, mg, and

S = mL of sample volume.

b. Report as "Total Solids (TS) = _____ mg/L"

or as "_____ mg/L TS"

c. Identify any sample that yields a residue mass < 2.5 mg or > 200 mg and report the results as an "estimate" because the mass has exceeded the criteria of this analysis.

5.0 Quality Control

The typically non-homogeneous character of total solids and inconsistent or incomplete sample drying can lead to highly variable results and thus quality control is considered to be an important part of this method.

a. Analyze a method blank (a clean, dried, and tared evaporation dish) with each batch of 20 or fewer samples. If a single sample is being analyzed, a method blank must also be analyzed.

b. Analyze at least one sample in duplicate with each batch of 20 or fewer samples. If a single sample is being analyzed, this sample must be analyzed in duplicate.

c. Each analyst must analyze a laboratory-fortified blank and laboratory-fortified blank duplicate sample set (LFB/LFBD) to demonstrate initial capability and thereafter analyze a LFB/LFBD sample set for each 20 samples analyzed, not including method blanks, to demonstrate ongoing capability. The analyst may analyze their initial LFB/LFBD sample set at the same time they analyze their first sample, but then after measuring 20 samples, including duplicate samples, must analyze another LFB/LFBD sample set. Material to prepare a LFB control sample for total solids can be obtained by drying and grinding a soil, and using only the material that passes a No. 40 standard size sieve. Store this material in the oven at 103-105°C to achieve and maintain constant weight until ready to use. Prepare the actual LFB sample by equilibrating the LFB material's temperature in a desiccator to the room's

temperature, weigh from 10 to 100 mg of the material and add combine with distilled water to prepare a 1.0 liter suspension. Measure the total solids of this LFB sample.

d. Evaluate the results obtained from QC data as follows: The method blank results must demonstrate that the initial tared dish weight does not differ by more than ± 0.5 mg. The relative percent difference (RPD) of duplicate samples should not exceed an absolute value of 10%. The RPD of the LFB/LFBD analyses should not exceed an absolute value of 10%. Additionally, the percent recoveries for the LFB samples should be plotted on a control chart for an overall laboratory evaluation of capability associated with each new LFB material prepared or purchased.

e. Consider homogenizing samples that produce highly variable result. Take care that the sample is not heated while being homogenized; most homogenizers will heat the sample if operated for too long.

6.0 Bibliography

1. Rodger B. Baird, Andrew D. Eaton, and Eugene W. Rice (2017) *Standard Methods for the Examination of Water and Wastewater*. APHA, Washington, DC, 23nd Edition.