Applied Microbiology
&
Biotechnology Laboratory

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Standard Operating Procedure

AMBL-105-B

Prepared:	12/27/2017
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Prepared by:	Terry E. Baxter
Reviewed by:	

Total Dissolved Solids by Gravimetric Determination

METHOD SUMMARY

This SOP describes the procedure for measuring total dissolved solids in water and wastewater. This method is based on Method 2540 C 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, the handling of natural waters or untreated wastewaters that potentially contain pathogenic organisms, and optionally handling concentrated hydrochloric acid. The specific hazards associated with this method are as follows.

Skin Contact by Acid: Can cause severe burns to the skin upon contact.

Eye Contact by Acid: Can cause extreme pain and eye damage with possible cataracts and glaucoma occurring after exposure.

Ingestion of Acid: May cause corrosion of the lips, mouth, throat and stomach, and cause vomiting.

Inhalation of Acid Fumes: May cause irritation to the nose and upper respiratory tract.

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 a shower and eye wash station be located nearby. When using hydrochloride acid (optional) to dissolve hard-to-remove residue from the evaporating dish, work must be conducted in the fume hood.

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

Gloves (nitrile, PVC or neoprene)

Safety goggles or glasses

Laboratory coat

Acid resistant apron (when handling acid)

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 (> 71°C) bacteria. Soak the residue in deionized water for approximately 30 minutes, and then use a brush and tap water to clean the dish. The rinse water and removed dissolved solids may be then rinsed down the laboratory sink.
Rinsing evaporating dishes with hydrochloric acid generates in from 3 to 5 mL of a strong acid (low pH) waste liquid.	Y	If acid rinsing is required to dissolve a hard-to-remove residual, the acid rinse waste liquid is considered hazardous because of the low pH. It must be collected and saved in a glass container until either it is used to neutralize high pH solutions, or is picked up for hazardous waste disposal.

METHOD DESCRIPTION

1.0 Introduction and Applicability

Total dissolved solids is a measure of the dissolved matter in a water that remains after all the water has been evaporated. A higher level of dissolved solids is typically accompanied with a higher level of hardness. Dissolved solids affect water quality by making it unfit or unpalatable to drink, unsuitable for use in many industrial applications, and unsuitable for cooking or other applications where water is heated. A known volume of a well-mixed sample is filtered through a standard glass-fiber filter and the

filtrate collected. The filtrate is evaporated to a constant weight condition in an oven maintained at a temperature of 180°C to remove mechanically occluded water. The mass of the dried sample's dissolved solids is determined and used to calculate the concentration of total dissolved solids in the sample.

This method is applicable for measurement of total dissolved solids in all natural waters, in raw, process and treated agricultural, municipal and industrial wastewaters and in 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. Volumetric pipet, Class A or Wide-bore pipet (optionally), 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 180 ± 2°C for drying samples to a constant weight condition.
g. Muffle furnace operated at 550 ± 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.
m. Glass-fiber filter, with a 47 mm diameter, nominal pore size ≤ 2.0 µm and ≥ 1.0 µm, and no binders.
n. Filtration funnel assembly for a 47 mm size diameter filter.
o. Vacuum suction flask, 1000 mL capacity.

3.0 Reagents

a. Hydrochloric acid, approximately 37% (optional), used during cleaning for dissolving hard-to-remove residue from the surface of the evaporating dish.
b. Distilled or deionized water.

4.0 Procedure

a. Read Method 2540C Total Dissolved Solids Dried at 180°C (*Standard Methods*).

b. Prepare a sample evaporating dish by ensuring that it is cleaned and does not contain residue from a previous use. If necessary, perform an acid rinse of the inside surface of the evaporating dish with approximately 3-5 mL of concentrated hydrochloric acid. Carefully and slowly tilt and rotate the dish so that the acid contacts any part of the inside surface having a hard-to-remove residue. When done, transfer the acid into the next dish being cleaned or into the acid rinse disposal container. Rinse all surfaces of the evaporating dish using first tap water and then using deionized or distilled water for the final cleansing rinse. Dry the clean evaporating dish either in a convention oven at a temperature of 180 ± 2°C for no less than 60 minutes if measuring only total solids or alternately, ignite the evaporating dish in a muffle furnace at a temperature of 550 ± 50°C for no less than 15 minutes if volatile dissolved solids will be determined (see SOP 105E). 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. Prepare a glass-fiber filter by placing and centering a filter disk onto the filter support screen of the filtration apparatus and attach the funnel. Apply a low to moderate vacuum and rinse the filter with three successive volumes of ≥30 mL deionized or distilled water. Leave the vacuum on until all traces of water have been removed from the filter. If this filter will be used for determining total suspended solids and total dissolved solids from the same sample volume, prepare the filter as described for total suspended solids in SOP AMBL 105D.

d. Equilibrate the sample's temperature to that of the room's temperature and use a pipet or graduated cylinder to transfer a volume of well-mixed sample onto the filter with the vacuum applied. Use a volumetric pipet for samples having little or no suspended matter to clog the narrow opening of the pipet or optionally use the wide bore pipet to avoid clogging the tip. Use a graduated cylinder for samples having solids that clog the wide bore pipet tip. Select a sample volume that will result in a dried residue ranging from 2.5 to 200 mg. Avoid filtration times exceeding 10 minutes. Rinse the entire surface area of the exposed filter with three successive volumes of ≥10 mL deionized or distilled water. Allow the water to completely drain between each rinsing and leave the vacuum on until all traces of water have been removed from the filter. Transfer the entire volume of sample filtrate and rinse water to a pre-weighed dish. If necessary, filter and add additional sample portions to the same dish after the previous portion has been evaporated. Record the total volume of sample added.

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

f. Dry the sample in a convection oven at a temperature of 180°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.

g. Remove the dish containing the sample from the oven, cooling it to room temperature and then weigh it. Record this as the first 180°C weight.

h. Repeat the drying cycle for no less than 60 minutes, and again cool, weigh and record the second 180°C weight.

i. Calculate the weight change between the first and second weights, and if the change is >0.5 mg, repeat 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 180°C weight.

4.0 Calculation and Reporting

a. Calculate the concentration of total dissolved solids

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

where A = final 180°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 Dissolved Solids (TDS) = ____ mg/L"

or as "____ mg/L TDS"

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

Although the dissolved material in the sample's liquid medium is considered relatively homogeneous, the filtration step and inconsistent or incomplete sample drying can lead to 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. Prepare a LFB control sample for total dissolved solids by drying approximately 200 mg NaCl at a temperature of 103-105°C for a period not less than 60 minutes. Weigh 50 mg of the dried NaCl and record the actual weight to the nearest 0.1 mg. Dissolve in distilled water to a volume of 1 liter. Measure the total dissolved solids of this standard 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.

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.