Applied Microbiology
&
Biotechnology Laboratory

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Standard Operating Procedure

AMBL-105-D

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Total Suspended Solids by Gravimetric Determination

METHOD SUMMARY

This SOP describes the procedure for measuring total suspended solids in water and wastewater. This method is based on Method 2540 D of *Standard Methods for the Examination of Water and Wastewater*, 24th Edition, 2023.

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. 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 on the surface of a glass-fiber filter.	N	A solid wastewater solids residue is considered desiccated and to have heat-killed (> 71°C) bacteria. The filter and solid residue may be disposed in the laboratory trash.

METHOD DESCRIPTION

1.0 Introduction and Applicability

Total suspended solids or TSS is the measure of the undissolved solid matter in a water that remains on the surface of a filter after all the water has been evaporated. Suspended solids affect water quality by making it unfit or unsafe to drink, aesthetically unacceptable for recreational use and aquatic habitats, and unsuitable for use in many industrial or other applications. A known volume of a well-mixed sample is filtered through a standard glass-fiber filter, collecting the solid residue on the surface of the filter. The filter and residue are evaporated to a constant weight condition in an oven maintained at a temperature of 103-105°C. The mass of the dried residue is determined and used to calculate the concentration of total suspended solids in the sample.

This method is applicable for measurement of total suspended solids in all natural waters, in raw, process and treated agricultural, municipal and industrial wastewaters. This method is not considered applicable to wastewater slurries behaving as a Newtonian fluid, non-Newtonian fluids or treated drinking water.

2.0 Apparatus

a. Glass-fiber filter, with a 47 mm diameter, nominal pore size $\leq$ 2.0 µm and $\geq$ 1.0 µm, and no binders.
b. Graduated cylinder, Class A
c. Wide-bore pipet, Class B
d. Forceps capable of lifting and holding a filter without tearing or puncturing it.
e. Filter pans, aluminum or other inert material, to hold filters.
f. Convection oven operated at 103-105°C for drying samples to a constant weight condition.

g. Muffle furnace operated at 550 ± 50°C (if volatile solids will be determined).
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. Filtration funnel assembly for a 47 mm size diameter filter.
n. Vacuum suction flask, 1000 mL capacity.
o. Vacuum hose (thick-walled)
p. Vacuum trap

3.0 Reagents

a. Reagent water, deionized/reverse osmosis (DI/RO) or distilled water (DW)

4.0 Procedure

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

b. 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 reagent water. Leave the vacuum on until all traces of water have been removed from the filter. Wet filters can adhere to the filter pan during the drying process and cause filter fibers to be torn from the filter when lifted for weighing. Remove the hose and turn off the vacuum. Use forceps to carefully remove the filter from the filtration apparatus support screen by holding and slowly lifting the filter only by the outer edge of the filter. Transfer the filter to a filter pan. Place the filter and pan into a drying oven operated at a temperature of 103-105°C. Dry the filter at this temperature for no less than 60 minutes if measuring only total suspended solids or, if volatile suspended solids will be determined (see SOP 105E) place the filter in a muffle furnace at a temperature of 550 ± 50°C for no less than 15 minutes. In a desiccator, cool the dried filter to room temperature. Remove the filter from the filter pan, weigh and record its weight - this is the tared weight of the filter. Replace the filter in the filter pan and store it in a desiccator until used.

c. 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 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 reagent 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. Remove the hose and turn off the vacuum. Carefully remove the filter from the filtration apparatus support screen using forceps by lifting and holding the filter only by the clean outer edge without solid residue. Transfer the filter to a filter pan.

d. Dry the filter with solid residue 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.

e. Remove the filter pan containing the filter from the oven, place it in a desiccator and cool it to room temperature. Carefully remove the filter from the filter pan using the forceps without touching the dried solid residue and weigh it. Record this as the first 103°C weight.

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

g. 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 103°C weight.

4.0 Calculation and Reporting

a. Calculate the concentration of total suspended solids

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

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

F = tared filter weight, mg, and

S = mL of sample volume.

b. 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.

c. Report as follows:

Calculated Range (mg/L)	Reported to nearest (mg/L)
< 50 mg/L	1 mg/L
50 – 99 mg/L	5 mg/L
100 – 4999 mg/L	10 mg/L
> 5000 mg/L	50 mg/L

d. Once the calculated TSS value exceeds 10,000 mg/L, it is recommended to measure solids using SOP 105 F.

5.0 Quality Assurance and Quality Control

a. Sample and Filter Handling

The suspended solid material in a liquid medium sample is considered relatively non-homogeneous. The sub-sampling of a non-homogenous material prior to the filtration step as well as how the filter is handled throughout the procedure can introduce variability and are thus considered part of this method's quality assurance and quality control practices.

1) A well-mixed sample is essential for minimizing the non-homogeneous nature of the suspended material. Mixing the sample in a beaker using a magnetic stirrer is generally preferred as long as the mixing regime provided consists of a vertical mixing component as well as the horizontal rotation of the sample. A sample can also be well-mixed by hand, either by inverting a closed sample container multiple times or stirring the sampling with a stirring rod or the pipet in a way that can fully agitate the sample. When using a pipet, it is best to use a pipet bulb that does not leak air so that sample does not enter the pipet while agitating the sample. However, mixing by hand requires that the sample be agitated prior to each time a sample aliquot is taken.

2) Improper alignment and placement of the filter on the support grid can cause a tear or crease in the filter when the filtration funnel is placed over the filter, or in the worst case, leave a portion of the porous support grid exposed. A tear or crease weakens the edge of the filter and can result in the loss of filter material when lifting or moving the filter. Realigning a filter that has a tear, will allow some sample and solids to pass through the tear and thus bypass the filter. An exposed portion of the support grid would also allow some sample and solids to bypass the filter. Proper alignment of the filter on the support grid should be visually verified before placing the filtration funnel over the filter.

3) A filter is picked up using forceps that will not puncture or tear the filter, and should always be held by placing the forceps near the edge of the filter that remains free of solid material residue. Forceps that touch the solids residue on the filter can pick up some of the residue and transfer it to the next filter that is handled. Make sure that the forceps are clean prior to handling any filter.

4) Extended filtration times can occur when the sample solids concentration is high and too much sample volume has been used. A solids particle-size distribution that is predominantly and only just somewhat larger than the filter's effective pore size can cause also extend filtration time because the filter's pore will become clogged more rapidly. Regardless, extended filtration times can lead to the filter adhering to the support grid and causing the filter to tear or lose fibers from the bottom of the filter when the filter is lifted. One effect that occurs during extended filtration times is the formation of ice within the support grid along the bottom of the filter since the air temperature beneath the filter will decrease because of the vacuum. Always lift the filter from the support screen after the vacuum has been turn off and the vacuum line removed and lift slow enough so a filter adhered to the support grid can be detected and measures to prevent tearing fibers from the bottom of the filter may be taken. If freezing is expected, wait a few minutes to allow the ice to thaw before removing the filter.

5) A damp filter, with or without solid residue, that is placed into the weighing pan tends to adhere to the pan's flat surface that results in a visible loss of filter fibers on the pan when the filter is lifted. Ensuring that the rinse water is removed from the solids and filter to the extent possible before the vacuum is turned off and the vacuum hose removed can prevent this from happening. Following this practice is also beneficial for potentially reducing the number of drying cycles.

6) Verifying that the filter and solid material residue have achieved a constant weight is critical. Constant weight is defined as having been achieved if the mass change between two subsequent drying cycles is less than 0.5 mg. Although many samples can achieve a constant weight condition after one drying cycle, it requires no less than one additional drying cycle to verify that this has been achieved. The exception to this is when a drying time study is conducted for the specific sample and suspended solids type that demonstrates overnight drying alone can achieve constant weight. An AMBL study conducted in 2016 demonstrated that overnight drying achieved a constant weight for mixed liquor suspended solids.

b. Data Quality Assessment and Corrective Actions

Assessing data quality and method performance is done by preparing and analyzing various quality control samples with some defined frequency. The results of these QC samples are then evaluated against preferably lab-specific performance criteria or against criteria considered acceptable performance indicators across multiple labs.

1) Analyze a method blank (a clean, dried, and tared filter) throughout the entire process with each batch of 20 or fewer samples. If a single sample is being analyzed, a method blank must also be analyzed. Evaluate the method blank result against the AMBL-specific criteria of <0.2 mg mass difference between two subsequent drying cycles.

2). 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. Evaluate sample duplicates by calculating relative percent difference (RPD) as follows.

$$RPD, \% = \frac{|Sample - Duplicate\ Sample|}{(Sample + Duplicate\ Sample)/2} \times 100$$

Evaluate the RPD value against the AMBL-specific criteria of < 5%.

3) Analyze one laboratory-fortified blank and laboratory-fortified blank duplicate sample set (LFB/LFBD) for each 20 samples analyzed, not including method blanks or duplicate samples. Prepare a LFB sample for total suspended solids by weighing 100 mg Celite 545 (record the actual weight) to the nearest 0.1 mg. Suspend in distilled water to a volume of 1 liter. Measure the total suspended solids of this standard in duplicate. The RPD of the LFB/LFBD analyses should not exceed an absolute value of 10%. An AMBL-specific criteria has not yet been established.

6.0 References

1. American Public Health Association, American Water Works Association, Water Environment Federation. Lipps WC, Braun-Howland EB, Baxter TE, eds. *Standard Methods for the Examination of Water and Wastewater*. 24th ed. Washington DC: APHA Press; 2023.