where:

A = total gain in weight of tared flask (mg) and
B = calculated residue from solvent blank of the same volume as that used in the test (mg).

6. Precision and Bias

There is no standard against which bias of this test can be determined. Variability of replicates is influenced by sample heterogeneity. If large grease particles are present, the element of chance in sampling may be a major factor. One municipal wastewater discharge and 23 meat-packing plant discharges, both containing noticeable particles of grease, were analyzed in triplicate. Averages for the 3 wastewaters were 48, 57, and 25 mg/L; standard deviations averaged 11%. An oil refinery made duplicate determinations of its separator effluent on 15 consecutive days, obtaining results ranging from 5.1 to 11.2 mg/L. The average difference between pairs of samples was 0.37 mg/L.

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2540

SOLIDS

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2540 A. INTRODUCTION

Solids refer to matter suspended or dissolved in potable, surface, and saline waters, as well as domestic and industrial wastewaters. Solids may adversely affect water or effluent quality in a number of ways. Waters with high dissolved solids generally are of inferior palatability and may induce an unfavorable physiological reaction in the transient consumer, so a limit of 500 mg/L dissolved solids is desirable for drinking waters. Highly mineralized waters also are unsuitable for many industrial applications. Waters high in suspended solids may be aesthetically unsatisfactory for bathing and other purposes.

Solids analyses are important for controlling water and wastewater treatment processes and assessing compliance with regulatory requirements.

1. Terminology

Fixed solids—the total, suspended, or dissolved solids remaining in a sample after ignition for a specified time at a specified temperature. [Determinations of fixed and volatile solids do not distinguish precisely between inorganic and organic matter because some inorganic compounds can be lost during ignition. Organic matter can be better characterized via total organic carbon (Section 5310), biochemical oxygen demand (Section 5210), and chemical oxygen demand (Section 5220) methods.]

Settleable solids—the material in a sample that settles out of suspension within a defined period. This may include floating material, depending on the technique used (e.g., 2540 F.3*b*).

Total dissolved solids (TDS)—the portion of total solids in a water sample that passes through a filter with a nominal pore size of 2.0 μm (or smaller) under specified conditions.

Total solids—the material left in a sample vessel after evaporation and subsequent oven drying at a defined temperature. *Total solids* includes both total suspended and total dissolved solids, which are physically separated via filtration. Whether a solids particle is filtered into the suspended or dissolved portion principally depends on a filter's thickness, area, pore size, porosity, and type of holder, as well as the physical nature, particle size, and amount of solids being filtered.

Total suspended solids (TSS)—the portion of total solids in an aqueous sample retained on the filter. Note: Some clays and colloids will pass through a 2.0-μm filter.

Volatile solids—the total, suspended, or dissolved solids lost from a sample after ignition for a specified time at a specified temperature. [Determinations of fixed and volatile solids do not distinguish precisely between inorganic and organic matter because some inorganic compounds can be lost during ignition. Organic matter can be better characterized via total organic carbon (Section 5310), biochemical oxygen demand (Section 5210), and chemical oxygen demand (Section 5220) methods.]

2. Sources of Error and Variability

Sampling, subsampling, and measuring 2- or 3-phase samples may introduce significant errors. Maintain sample homogeneity during transfer, and handle carefully to ensure sample integrity. If part of a sample adheres to the container, consider this in evaluating and reporting results. During drying, some samples form a crust that prevents water evaporation; special handling is required to deal with this issue (see below).

Take special care with viscous samples, which might entrain air during mixing and can be difficult to transfer in accurate volumes.

Also, take special care with samples with high dissolved solids levels, which are difficult to rinse completely during filtration.

Because a sample being analyzed will contain both total dissolved solids (TDS) and total suspended solids (TSS), the apparatus and rinsing techniques used may adversely affect either one or both of the TSS and TDS results. To avoid this, take care to keep the TDS fraction from migrating into the unrinsed edge of the filter beneath the funnel and being retained as TSS weight. If this is suspected, then investigate the filter apparatus seal's effectiveness and consider additional rinses.

If using a pipet to measure and transfer sample, place the pipet tip in the center (depth and width) of the well-mixed container. If using a magnetic stir plate and stir bar, set the speed to shear larger particles and blend sample to a more uniform particle size. Then pipet the sample from mid-depth midway between the container's wall and vortex. Centrifugal force may separate particles of different sizes and densities, resulting in poor precision when the sample-withdrawal point varies. Avoid using a magnetic stirrer with samples containing magnetic particles. When using a graduated cylinder, transfer samples to the cylinder immediately after shaking or stirring to avoid any settling of the sample's solids.

Drying temperature, heating duration, and sample matrix can affect weight loss due to volatilization of organic matter, mechanically occluded water, water of crystallization, and gases from heat-induced chemical decomposition. They also can affect weight gains due to oxidation. When liquid samples are put directly into any oven heated above boiling temperature, they may splatter and lose some of the parameter of interest. Therefore, samples can be evaporated to dryness or near dryness at a temperature below boiling using a steam bath, hot plate, or oven before being dried to a constant weight per the method's specifications.

Pay close attention to all samples during postdrying desiccation. Open the desiccator as few times as possible to minimize the entry of moist air. If samples are stronger desiccants than the desiccants used in the desiccator, they may take on water. In general, weigh samples as soon as possible after removal from the desiccator to minimize water absorption from the atmosphere.

Residues dried between 103 and 105 °C may retain both water of crystallization and some mechanically occluded water. Occluded water, organic matter, and carbonate loss [as carbon dioxide (CO_2)] may slow drying time significantly. Residues dried at 180 ± 2 °C should lose mechanically occluded water, but may also lose organic matter and some salts.

Dry samples to constant weight if possible; this entails multiple cycles of drying, cooling, and weighing for each sample. Results for residues with high oil or grease content may be questionable because such samples are difficult to dry to constant weight in a reasonable timeframe. If samples cannot be analyzed or any samples fail to reach a constant weight, they must be qualified by indicating the number of drying cycles and the final weight change.

When weighing dried samples, make sure samples are cooled to ambient temperature before weighing. Also, it is important to remove excess water from glass-fiber filters before placing them on weighing dishes or pans. Excess water will cause filters to adhere to a weighing dish or pan during drying, leading to tearing or loss of material when filter is lifted for weighing, thereby skewing results. Be alert to changes in weight due to air exposure and sample degradation. This is especially critical for the low-level TSS method. See each method for further discussion of specific interferences.

Use reagent water to rinse filters and filtered solids and to clean labware. Special samples may require higher-quality water (see Section 1080).

When an analysis deviates from the stated procedures for any reason, record the variations and present them with the results so the reasons for not following the method are documented.

3. Sample Handling and Preservation

Use borosilicate glass, plastic, or fluoropolymer [e.g., polytetrafluoroethylene (PTFE)] bottles, so long as suspended solids in sample do not adhere to container walls. Begin analysis as soon as possible, but in no case hold samples for more than 7 d from the time of collection (168 hrs). Settleable solids must be analyzed within 48 h. Between collection and analysis, refrigerate at 6 °C or less without freezing to minimize microbiological decomposition of solids. Bring samples to ambient temperature before beginning the analysis.

4. Selection of Method

Methods 2540 B–F are suitable for determining solids in potable, surface, and saline waters, as well as domestic and industrial wastewaters. The analytical range for 2540 B–D is 2.5 to 200 mg/L for a 1000-mL sample, but may be extended by using a small sample volume for analysis. Method 2540 G is suitable for determining solids in soils and sediments, as well as solid and semisolid materials produced during water and wastewater treatment.

5. Quality Control (QC)

The QC practices that are integral to each method are summarized in Table 2020:1 and Table 2020:2.

Analyze 5% or more of all samples in duplicate or at least one duplicate sample with each batch of 20 or fewer samples. The laboratory may plot duplicate determinations on a control chart for evaluation. Typically, the relative percent difference (RPD) of duplicates should not exceed 10%, but RPDs may vary considerably because of sample matrix and concentration.

Analyze one method blank (MB) each batch of 20 or fewer samples for each method except settleable solids (2540 F). Blank analysis includes all container- and filter-preparation steps and procedures except sample addition. If any MB measurements are at or above the reporting level, take immediate corrective action (see Section 1020 B.5). This may include re-analyzing the sample batch.

Include 1 laboratory-fortified blank (LFB) each batch of 20 or fewer samples for all tests except settleable solids (2540 F) and total, fixed, and volatile solids in solid and semisolid samples (2540 G). Plot the percent recoveries on a control chart for laboratory evaluation. Laboratories may purchase known standards or prepare in-house working controls for use.

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2540 B. TOTAL SOLIDS DRIED FROM 103 TO 105 °C

1. General Discussion

a. Principle: Evaporate a well-mixed sample in a preweighed dish and dry it to constant weight in a 103 to 105 °C oven. The increase compared to the empty preweighed dish weight represents total solids. This result may not be the actual weight of dissolved and suspended solids in wastewater samples.

To meet the LFB requirement (2540 A.5), a total solids standard can be created as follows: Dry, grind, and sieve a soil for use as a working control. This control may or may not be mixed with other reagents (e.g., Celite 545 or Sigmacell Cellulose Type 20) and may have water added according to the laboratory's procedures.

b. Interferences: Highly mineralized water with a significant concentration of calcium, magnesium, chloride, or sulfate may be hygroscopic and require prolonged drying, proper desiccation, and rapid weighing. Exclude large, floating particles or submerged agglomerates of nonhomogeneous materials from sample if they are not desired in the final result.

Optionally, disperse visible floating oil and grease with a blender or homogenizer before withdrawing a sample portion for analysis. If oil and grease sticks to blender sides and blades, thus potentially affecting sample composition, note this in the lab report.

Residues dried at temperatures from 103 to 105 °C may retain both water of crystallization and some mechanically occluded water. A loss of CO_2 occurs when bicarbonate converts to carbonate during drying. Usually, very little organic matter will volatilize. It may take a long time to attain constant weight because occluded-water removal is marginal at this temperature.

Because excessive residue in the dish may form a water-trapping crust, limit sample to 200 mg or less of residue.

2. Apparatus

a. Sample dishes: Dishes of approximately 90-mm diameter and 100-mL capacity made of one of the following materials:

1) porcelain.
2) platinum.
3) high-silica glass (may react with highly alkaline samples)
4) other material shown to be resistant to the sample matrix and weight stable at the required evaporation and drying temperatures. Aluminum is NOT appropriate for this purpose.

b. Wide-bore pipets, Class B in glass, mechanical, or electronic.

c. Graduated cylinders, Class A.

d. Steam bath (optional) for sample evaporation.

e. Hot plate or block (optional) for sample evaporation. Must be capable of maintaining a temperature greater than 100 °C without boiling samples.

f. Predrying oven (optional) for sample evaporation that operates at temperatures approximately 2 °C below boiling to prevent splattering.

g. Drying oven that operates at 103 to 105 °C.

h. Muffle furnace that operates at 550 ± 50 °C.

i. Desiccator, which includes either a desiccant whose color changes in response to moisture concentration or an instrument for measuring moisture (e.g., a hygrometer).

j. Analytical balance, capable of weighing to 0.1 mg.

k. Magnetic stirrer with TFE stirring bar (optional).

l. Blender or homogenizer (optional).

m. Low-form beaker, Class B or better.

3. Procedure

a. Preparation of evaporating dish: If measuring volatile solids, then ignite clean evaporating dish at 550 ± 50 °C for 15 min or longer in a muffle furnace. If only measuring total solids, then heat the clean dish between 103 to 105 °C for 1 h or longer. Cool dishes to ambient temperature and weigh. Store weighed dishes in a desiccator or oven until needed.

b. Selection of sample size: Choose sample volume to yield between 2.5 and 200 mg dried residue. If necessary, successive sample portions may be added to the same dish after evaporation. Identify any sample that yields less than 2.5 mg or more than 200 mg of residue, and report the value as described in Sections 1020 and 2020.

c. Sample analysis: Stir or mix the sample and quantitatively transfer with a pipet or graduated cylinder to a preweighed dish. Evaporate samples to dryness using a drying oven, steam bath, or hot plate. Monitor and adjust temperature to avoid splattering. Dry the evaporated sample for 1 h or longer in an oven at temperatures between 103 to 105 °C. Cool the dish in a desiccator to ambient temperature, and weigh. Repeat cycle (drying for ≥1 h, cooling, desiccating, and weighing) until weight change is less than 0.5 mg.

4. Calculation

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

where:

A = final weight of dried residue + dish (mg) and
B = weight of dish (mg).

5. Precision

Single-laboratory duplicate analyses of 41 samples of water and wastewater were made with a standard deviation of differences of 6.0 mg/L.

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2540 C. TOTAL DISSOLVED SOLIDS DRIED AT 180 °C

1. General Discussion

a. Principle: Filter a well-mixed sample through a standard glass-fiber filter. Then, transfer the filtrate to a pre-weighed dish, evaporate to dryness, and transfer to an oven at 180 ± 2 °C to achieve a constant weight. The increase compared to the empty preweighed dish weight represents TDS.

These results may differ from the theoretical value for solids calculated from a chemical analysis of a sample. Approximation methods for correlating chemical and physical analyses are available.[1] The filtrate collected from the TSS determination (2540 D) may be used to determine TDS.

To meet the LFB requirement (2540 A.5), analysts can prepare a TDS standard. For example: Dry 70 to 80 mg of NaCl at 103 to 105 °C for 1 h or longer, weigh 50 mg, and dissolve in reagent water to a final volume of 1 L. This results in a 50-mg/L TDS standard. Similarly, other appropriate TDS standard concentrations may be prepared.

b. Interferences: See 2540 A.2. Highly mineralized waters (e.g., calcium, chloride, magnesium sulfate) may be hygroscopic and require prolonged drying, proper desiccation, and rapid weighing. Samples with high bicarbonate concentrations require careful, possibly prolonged drying at 180 ± 2 °C to ensure that bicarbonate completely converts to carbonate.

Residues dried at 180 ± 2 °C will lose almost all mechanically occluded water, but some water of crystallization may remain, especially if sulfates are present. Organic matter may volatilize and be lost, but not completely removed. Carbon dioxide loss occurs when bicarbonates convert to carbonates, and carbonates may be decomposed partially to oxides or basic salts. Some chloride and nitrate salts may be lost. In general, evaporating and drying water samples at 180 ± 2 °C yields TDS values closer to those obtained by adding individually determined mineral species than the values obtained when drying between 103 and 105 °C.

Because excessive residue in the dish may form a water-trapping crust, limit samples to 200 mg or less of residue.

2. Apparatus

Apparatus listed in 2540 B.2*a–l*, and in addition:

a. Glass-fiber filter disks, 22 to 125 mm diameter, ≤2-µm nominal pore size without organic binder.

b. Filtration apparatus: One of the following, suitable for the filter selected:

1) Membrane filter funnel—various capacities, to fit selected filter.

2) Gooch crucible—25- to 40-mL capacity, with Gooch crucible adapter.

3) Filtration apparatus with reservoir and coarse (40- to 60-µm) fritted disk as filter support.

c. Suction flask with sufficient capacity for sample size selected.

d. Oven that operates at 180 ± 2 °C.

e. Forceps.

3. Procedure

a. Preparation of glass-fiber filter disk: Insert disk with wrinkled side up into filtration apparatus. Apply vacuum and wash disk with at least 3 successive volumes of 20 mL or more of reagent-grade water. Continue suction to remove all traces of water. If using commercially prepared glass-fiber filter disks, the washing step may be skipped if the manufacturer certifies that the filters meet this method's requirements.

b. Preparation of evaporating dish: If measuring volatile solids, ignite cleaned evaporating dish at 550 ± 50 °C for 15 min or more in a muffle furnace. If only measuring TDS, then heat cleaned dish to 180 ± 2 °C for 1 or more hours in an oven. Cool dishes to ambient temperature and weigh. Store in a desiccator or oven until needed.

c. Selection of filter and sample sizes: Choose sample volume to yield between 2.5 and 200 mg dried residue. If filtration will take longer than 10 min to complete, then increase filter size or decrease sample volume. Identify any sample that yields residue less than 2.5 mg or more than 200 mg, and report the value as described in Sections 1020 and 2020.

d. Sample analysis: Stir or mix sample and use a pipet or graduated cylinder to transfer a measured volume onto a glass-fiber filter with applied vacuum. Wash the entire exposed surface of filter with at least 3 successive volumes of 10 mL or more of reagent-grade water. Allow complete drainage between washings, and continue suction until all traces of water are removed. Transfer total filtrate (with washings) to a preweighed evaporating dish and evaporate to dryness on a steam bath, hot plate, or block, or in a drying oven. If necessary, add successive portions to the same dish after evaporation. Dry evaporated sample for 1 h or more in an oven at 180 ± 2 °C, cool in a desiccator to ambient temperature, and weigh. Repeat cycle (drying, cooling, desiccating, and weighing) until weight change is less than 0.5 mg.

If determining volatile solids, follow procedure in 2540 E.

4. Calculation

$$\text{total dissolved solids mg/L} = \frac{(A-B)\times 1000}{\text{sample volume, mL}}$$

where:

A = final weight of dried residue + dish (mg)
B = weight of dish (mg)

5. Precision

Single-laboratory analyses of 77 samples of a known of 293 mg/L were made with a standard deviation of differences of 21.20 mg/L.

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2540 D. TOTAL SUSPENDED SOLIDS DRIED FROM 103 TO 105 °C

1. General Discussion

a. Principle: Filter a well-mixed sample through a preweighed standard glass-fiber filter, and then dry the filter and the retained residue to a constant weight in a 103 to 105 °C oven. The increase in filter weight represents TSS. To estimate an unknown sample matrix TSS concentration, calculate the difference between TDS and total solids.

To meet the LFB requirement (2540 A.5), obtain a TSS QC standard from a reputable supplier or it can be prepared as follows: weigh 100 mg of Sigmacell Cellulose Type 20 or Celite 545, dilute to 1 liter with reagent water, and stir for 15 min or more to mix well. This results in a 100 mg/L TSS standard.

b. Interferences: See 2540 A.2 and B.1*b*. Unless representative of source, exclude large floating particles or submerged agglomerates of nonhomogeneous materials from sample. Limit sample size so it yields 200 mg or less of residue, because excessive filter residue may form a water-entrapping crust.

If suspended solids clog the filter, prolonging filtration, consider increasing filter diameter or decreasing sample volume.

When samples contain high concentrations of dissolved solids, thoroughly wash all exposed filter surfaces to ensure that dissolved material is removed (see also 2540 A.2). Prolonged filtration due to filter clogging may capture more colloidal materials, resulting in artificially high results.

Handle all filters carefully, with the wrinkled side up. Transfer filters using forceps to grip the residue-free edge. Weigh only the filters, not the support pans or dishes, unless a Gooch crucible is used.

2. Apparatus

Apparatus listed in 2540 B.2 and C.2, except for evaporating dishes, steam bath, hot plate or block, and 180 ± 2 °C drying oven. In addition:

a. Weighing dishes or pans, constructed of aluminum or other inert material, to hold filters.

b. Forceps.

3. Procedure

a. Preparation of glass-fiber filter disk: Insert filter with wrinkled side up in filtration apparatus. Apply vacuum and wash disk with 3 successive volumes of 20 mL or more of reagent-grade water. Continue suction to remove all traces of water. Remove filter from filtration apparatus and transfer to an inert weighing dish. If a Gooch crucible is used, remove crucible and filter combination. Dry in a 103 to 105 °C oven for 1 h or more. Cool in desiccator to ambient temperature and weigh. Store filters (on inert dishes or pans) in desiccator or 103 to 105 °C oven until needed. Adequate filter preparation is demonstrated by negligible weight loss or gain for method blanks.

If measuring volatile solids, ignite at 550 ± 50 °C for 15 min or more in a muffle furnace. Cool to room temperature before proceeding. (Alternatively, the ignition step may be performed after washing and drying at 103–105 °C for ≥1 h, but before weighing.)

If using commercially prepared glass-fiber filters, the ignition, washing, and weighing steps may be eliminated if the manufacturer certifies that the prepared filters meet this method's requirements. Verify filters using method blanks. Filters are verified if the measured weight differs from the manufacturer's weight by less than ±0.5 mg.

b. Selection of filter and sample sizes: Choose sample volumes to yield between 2.5 and 200 mg dried residue. If filtration takes more than 10 min to complete, increase filter size or decrease sample volume. Identify any sample that yields residue less than 2.5 mg or more than 200 mg, and report the value as described in Sections 1020 and 2020.

c. Sample analysis: Stir or mix the sample and use a pipet or graduated cylinder to transfer a measured volume onto a glass-fiber filter with applied vacuum. Wash the filter with at least 3 successive volumes of 10 mL or more of reagent-grade water. Allow complete drainage between washings, and continue suction until all traces of water are removed. When filtering samples with high dissolved solids concentrations, additional washings may be required to ensure that dissolved material is removed from all exposed filter surfaces.

Using forceps, carefully remove filter from filtration apparatus and transfer to an inert weighing dish or pan as a support. If using a Gooch crucible, remove crucible and filter combination from the crucible adapter. Dry for 1 h or more in a 103 to 105 °C oven, cool in a desiccator to ambient temperature, and weigh. Repeat successive cycles of drying, cooling, desiccating, and weighing until the weight change is less than 0.5 mg. If determining volatile solids, treat the residue according to 2540 E.

4. Calculation

$$\text{total suspended solids mg/L} = \frac{(A-B)\times 1000}{\text{sample volume, mL}}$$

where:

A = final weight of filter + dried residue (mg)
B = weight of filter (mg)

5. Precision

The standard deviation was 5.2 mg/L (coefficient of variation 33%) at 15 mg/L, 24 mg/L (10%) at 242 mg/L, and 13 mg/L (0.76%) at 1707 mg/L in studies by 2 analysts of 4 sets of 10 determinations each.

Single-laboratory duplicate analyses of 50 samples of water and wastewater were made with a standard deviation of differences of 2.8 mg/L.

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2540 E. Fixed and Volatile Solids Ignited at 550 °C

1. General Discussion

a. Principle: The residue from 2540 B, C, or D is ignited to constant weight at 550 ± 50 °C. The remaining solids are *fixed* total, dissolved, or suspended solids, while those lost to ignition are *volatile* total, dissolved, or suspended solids. This determination is useful in controlling wastewater treatment plant operations because it offers a rough approximation of the amount of organic matter present in wastewater solids, activated sludge, and industrial wastes.

b. Interferences: There may be negative errors in volatile solids calculations if volatile matter was lost during the drying step of 2540 D.3*c*. If a sample contains far more fixed than volatile solids, then volatile solids determined by this procedure may be subject to considerable error; try estimating volatile solids via another test [e.g., total organic carbon (Section 5310)].

Highly alkaline residues may react with any silica in sample or crucibles.

Usually, about 15 min of ignition is required for up to 200 mg residue. However, longer ignition times may be needed if drying heavier residues or more than one sample.

To avoid breakage due to drastic temperature change, dishes and filters may be partially cooled in any oven, steam bath, or hot plate listed in 2540 or else in ambient air until most of the heat dissipates. Transfer to a desiccator for final cooling in a dry atmosphere. Do not overload desiccator. Weigh dish or filter as soon as it has cooled to ambient temperature.

2. Apparatus

See 2540 B.2, C.2, and D.2.

3. Procedure

Bring a muffle furnace to 550 ± 50 °C. Insert a dish or filter containing residue produced by 2540 B, C, or D into furnace. Ignite for at least 15 min at 550 ± 50 °C, cool in a desiccator to ambient temperature, and weigh. Repeat successive cycles of drying, cooling, desiccating, and weighing until the weight change is less than 0.5 mg.

4. Calculation

$$\text{volatile solids mg/L} = \frac{(A-B)\times 1000}{\text{sample volume, mL}}$$

$$\text{fixed solids mg/L} = \frac{(B-C)\times 1000}{\text{sample volume, mL}}$$

where:

A = final weight of residue + dish or filter before ignition (mg),
B = final weight of residue + dish or filter after ignition (mg), and
C = weight of dish or filter (mg).

5. Precision

The standard deviation was 11 mg/L at 170 mg/L volatile total solids in studies by 3 laboratories on 4 samples and 10 replicates. Bias data on actual samples cannot be obtained.

2540 F. Settleable Solids

1. General Discussion

a. Principle: The settleable solids in surface and saline waters, and in domestic and industrial wastes, may be determined and reported based on either volume (mL/L) or weight (mg/L).

b. Interferences: The volumetric procedure (Imhoff cone) generally has a practical lower measurement limit between 0.1 and 1.0 mL/L, depending on sample composition. The settled-sample-level reading may be affected by sample foaming, sample separation, or pockets of liquid between large settled particles. When measuring heavy sludges whose measured settleable solids may be more than 100 mL/L, analysts may use a 1000-mL, Class A graduated cylinder instead of a cone. (Imhoff cones typically either lack graduation marks at that level or have marks scaled per 100 mL; a graduated cylinder may offer better resolution.)

If the settled matter contains pockets of liquid between large settled particles, then estimate the liquid volume, subtract it from settled-solids volume, and note in the lab report. If settleable and floating solids separate, do not estimate the floating material as settleable matter. If biological or chemical floc is present, the gravimetric method (2540 F.3*b*) is preferred.

2. Apparatus

a. Volumetric:
1) Imhoff cone.
2) Graduated cylinder, Class A.
3) Stir-rod, made of glass or other inert material.

b. Gravimetric:
1) Apparatus listed in 2540 D.2.
2) Glass vessel, minimum 9-cm-dia. A standpipe, graduated cylinder, or other vessel may be used as long as it satisfies the 9-cm-diam requirement and can hold the required sample volume.

3. Procedure

a. Volumetric: Fill an Imhoff cone or graduated cylinder to the 1-L mark with a well-mixed sample. Settle for 45 min, then gently agitate sample near the cone sides with a rod or by spinning. Allow sample to settle for another 15 min, and record volume of settleable solids in the cone as mL/L. When applicable, correct the recorded volume for interference from pockets of liquid volume as described in 2540 F.1b.

b. Gravimetric:
1) Determine TSS as in 2540 D.
2) Stir or mix sample and quantitatively transfer at least 1 L of well-mixed sample into a 9-mm-diameter glass vessel to fill vessel to at least 20 cm deep. Use a vessel with a larger diameter and larger sample volume if necessary. Let stand quiescent for 1 h. Then without disturbing the settled or floating material, carefully siphon 250 mL from the center of container at a point halfway between the liquid surface and the surface of the settled material. Determine TSS (mg/L) of supernatant (2540 D); these are the nonsettleable solids.

4. Calculation

settleable solids mg/L = TSS mg/L – nonsettleable solids mg/L

5. Precision and Bias

Precision and bias data are currently unavailable.

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2540 G. TOTAL, FIXED, AND VOLATILE SOLIDS IN SOLID AND SEMISOLID SAMPLES

1. General Discussion

a. Applicability: This method can be used to determine total solids and its fixed and volatile fractions in such solid and semisolid samples as river and lake sediments; sludges separated from water and wastewater treatment processes; and sludge cakes from vacuum filtration, centrifugation, or other sludge-dewatering processes.

b. Interferences: The determination of both total and volatile solids in these materials is subject to negative error due to loss of ammonium carbonate and volatile organic matter during drying. Although this is also true for wastewater, the effect tends to be more pronounced with sediments—especially sludges and sludge cakes. The mass of organic matter recovered from sludge and sediment requires a longer ignition time than that specified for wastewaters, effluents, or polluted waters. Carefully observe specified ignition time and temperature to control losses of volatile inorganic salts if these are a problem. Take all weight measurements quickly because wet samples tend to lose weight via evaporation. After drying or ignition, residues often are hygroscopic, rapidly absorbing moisture from the air. Highly alkaline residues may react with silica in samples or silica-containing crucibles.

2. Apparatus

All of the apparatus listed in 2540 B.2 except the magnetic stirrer and pipets. A balance capable of weighing to nearest 10 mg may be used.

3. Procedure

a. Total solids:
1) Preparation of evaporating dish—If measuring volatile solids, ignite a clean evaporating dish at 550 ± 50 °C for 15 min or longer in a muffle furnace. If only measuring total solids, heat dish for 1 h or longer in a 103 to 105 °C oven. Cool in desiccator to ambient temperature and weigh. Store in desiccator or 103 to 105 °C oven until needed.
2) Sample analysis
a) Fluid samples—If sample contains enough moisture to flow readily, then stir or shake to homogenize, transfer approximately 25 to 50 g to a prepared evaporating dish, and weigh (dish plus sample). Evaporate to dryness on a water bath, on a hot plate or block, or in a drying oven, then dry the evaporated sample at 103 to 105 °C for 1 h or longer, cool to ambient temperature in a desiccator, and weigh. Repeat successive cycles of drying, cooling, desiccating, and weighing until the weight change is less than 50 mg.
b) Solid samples—If sample consists of discrete pieces of solid material (e.g., dewatered sludge), then take care to obtain a representative sample whose particle size will not impede drying. One of the following manual-processing options may be used:

- take cores from each piece with a No. 7 cork borer and mix crumbled cores together well, or
- pulverize entire sample coarsely on a clean surface by hand (covered with clean gloves) or using a clean mortar and pestle.

Manually process samples as quickly as possible to prevent moisture loss. Processing via mechanical grinding is not recommended because moisture levels could drop during processing.

Transfer approximately 25 to 50 g to a prepared evaporating dish and weigh. Then, place in a 103 to 105 °C oven for 1 h or longer, cool to ambient temperature in a desiccator, and weigh. Repeat successive cycles of drying, cooling, desiccating, and weighing until the weight change is less than 50 mg.

b. Fixed and volatile solids: Transfer dried residue from 2540 G.3*a*2)a or b to a cool muffle furnace, heat furnace to 550 ± 50 °C, and then allow ignition to occur for 1 h or longer. If the residue contains large amounts of organic matter, consider first igniting it over a gas burner under an exhaust hood with enough air to lessen losses due to reducing conditions and to avoid odors in the laboratory. Alternatively, use a muffle furnace in a hood and open the door periodically to ensure air flow.

Cool in desiccator to ambient temperature, and weigh. Repeat successive cycles of igniting, cooling, desiccating, and weighing until the weight change is less than 50 mg.

4. Calculation

$$\%\ \text{total solids} = \frac{(A-B)\times 100}{C-B}$$

$$\%\ \text{volatile solids} = \frac{(A-D)\times 100}{A-B}$$

$$\%\ \text{fixed solids} = \frac{(D-B)\times 100}{A-B}$$

where:

A = final weight of dried residue + dish (mg),
B = weight of dish (mg),
C = weight of wet sample + dish (mg), and
D = final weight of residue + dish after ignition (mg).

5. Precision and Bias

Precision and bias data are currently unavailable.

Bibliography

Gratteau JC, Dick RI. Activated sludge suspended solids determinations. Water Sewage Works. 1968;115:468.

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TEMPERATURE

Approved by Standard Methods Committee, 2010. Editorial revisions, 2021.

2550 A. INTRODUCTION

Temperature readings are used in the calculation of various forms of alkalinity, in studies of saturation and stability with respect to calcium carbonate, in the calculation of salinity, in a number of colorimetric tests, and in general laboratory operations. In limnological studies, knowledge of water temperatures as a function of depth often are required. Elevated temperatures resulting from discharges of heated water may have significant ecological impact. The source of water supply, such as deep wells, often can be identified by temperature measurements alone. Industrial plants often require data on water temperature for process use or heat-transmission calculations.

2550 B. LABORATORY AND FIELD METHODS

1. Laboratory and Other Non-Depth Temperature Measurements

Normally, temperature can be measured using any standard liquid-in-glass or electronic thermometer with an analog or digital readout. The device should be able to distinguish temperature changes of 0.1 °C or less, and equilibrate rapidly (have a minimal thermal capacity). Abstain from using mercury-filled thermometers whenever possible to avoid the possibility of releasing mercury into the environment if the thermometer breaks. To prevent breakage in field operations, use a thermometer with a metal case.

Periodically check the device's bias (within the temperature range of use) against a reference thermometer certified by the National Institute of Standards and Technology (NIST, formerly National Bureau of Standards) and using tolerances suggested in NIST Handbook 105-6. The certified thermometer must be used with its certificate and correction chart. Note: Some commercial thermometers may be as much as 3 °C in error.

A total immersion thermometer is designed to indicate temperatures correctly when the bulb and the entire liquid column are exposed to the temperature being measured (except for a minimal emergent length for handling). A partial-immersion thermometer has a line around it at the immersion distance from the bottom. It indicates correctly when the bulb and the liquid column to that line are exposed to the temperature being measured and the emergent stem is at ambient temperature.