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Total Suspended Solids

a) Scope and Application

i) This method is applicable to the determination of non-filterable matter in drinking, surface, and saline waters, domestic and industrial wastes. The practical range of the determination is 2 to 20,000 mg/L..5 mg/L (based on the requirements for 2.5 mg of residue on the filter and a maximum sample volume of 1000 ml) to 20,000 mg/L (based on the requirement for no more than 200 mg of residue on the filter and a sample volume of 10 ml).

b) Summary of Method

i) A well-mixed sample is filtered through a glass-fiber filter, and the residue retained on the filter is dried to constant weight at 103 - 105°C. The increase in weight of the filter represents the total suspended solids.

ii) If the suspended material clogs the filter and prolongs filtration, it may be necessary to increase the diameter of the filter or decrease the sample volume.

c) Interferences

i) Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.

ii) Samples high in filterable residue (dissolved solids), such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter minimizes this potential interference.

iii) Certain biological materials, such as algae, slimes, insects, or other small crustaceans, may be considered to be positive interferences for nonfilterable matter. Modifications or adjustments may be needed to generate a better value.

d) Apparatus and Materials

i) Glass fiber filter discs, without organic binder: Whatman 47 mm diameter, 1.5 µm pore size , 0.70 µm pore size, or equivalent can be used. Pore size should be recorded.

ii) Filter support: Filtering apparatus with reservoir and a coarse (40-60 microns) fritted disc as a filter support.

NOTE: Many funnel designs are available in glass or porcelain. Some of the most common are Hirsch or Büchner funnels, membrane filter holders and Gooch crucibles. All are available with coarse fitted disc.

iii) Suction flask.

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iv) Drying oven: Capable of maintaining a temperature of 103-105°C.

v) Desiccator.

vi) Analytical balance: Capable of weighing to 0.1 mg.

vii) Graduated cylinder.

viii) Wash bottle.

e) Sample Handling

i) Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.

ii) Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration 4 ± 2°C, or freezing -20 ± 2°C to minimize microbiological decomposition of solids, is recommended. Holding time for TSS samples is 7 days. Sample should be stored in a plastic or resistant- glass container.

f) Procedure

i) Preparation of ~~filters~~filter:

~~Number glass fiber filters with a fine-point indelible ink pen. Allow ink to dry for 24 hours.~~

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(1) Place the ~~numbered~~ glass fiber filter on the membrane filter apparatus.

(2) While vacuum is applied, wash the filter with three successive ~~30~~20 ml volumes of deionized, distilled water. Allow the vacuum pump to run until the filters are "dry".

(3) Remove the filter from membrane filter apparatus and dry in an oven at 103 - 105°C for ~~four hours~~1 hour.

(4) Cool in a desiccator~~, weigh each filter,~~ and record the ~~filter number and~~ weight ~~in a notebook set aside for this purpose, and return~~of the filter ~~to the drying tray~~using an analytical balance.

(5) Return the ~~tray~~filter to the oven, 103 - 105°C, for one hour. Repeat the drying cycle until a constant weight is obtained (weight verification 0.5 mg or less is obtained).

(6) Record the second filter ~~weight in the notebook~~weigh and store the ~~filters in aluminum cups in an oven at 60 ± 5°C, or~~filter in a desiccator until needed.

(7) If fixed suspended solids are to be measured, ignite prepped filter at 550° C for 15

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minutes in a muffle furnace. Cool in a desiccator to balance temperature, weigh and record the filter weight. Repeat cycle of drying or igniting, cooling, desiccating and weighing until a constant weight is obtained (weight verification 0.5 mg or less is obtained). Record the second filter weigh and store the filter in a desiccator until needed.

ii) Sample analysis:

List filter numbers and sample indefications on Suspended Solids sheet.

(1) Choose a sample volume to yield between 2.5 and 200 mg of residue. If the volume filtered fails to meet the minimum yield, increase sample volume up to 1 L. If the filtration time exceeds 10 minutes discard the measured sample volume and filter and use another filter with a smaller volume size.

(1)(2) Assemble the filtering apparatus, place a pre-weighed numbered filter wrinkle side up on it,the filtering apparatus, wet the filter and begin suction.

(2)(3) Shake the sample vigorously and with a small portion of sample. Quantitatively measure 500 mL of sample into the quantitatively transfer the estimated volume size selected in step 1 to the filter using a graduated cylinder. Remove all traces of water by continuing to apply vacuum after sample has passed through. Record the volume of sample used beside the corresponding filter and sample on the Suspended Solids sheet.

(3)(4) **NOTE**: A smaller volume may be used if sample contains a large amount of suspended matter.

Pour the measured sample volume onto the filter. Remove all traces of water by continuing to apply vacuum after sample has passed through.

(4)(5) With suction on, wash the filter, non-filterable residue and filter funnel wall with three portions of deionized, distilled water allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum after water has passed through.

(5)(6) Carefully remove the filter from the filter support. Alternatively, remove crucible and filter from crucible adapter. Dry at least one hour at 103-105°C. Cool in a desiccator and weigh using an analytical balance. Repeat the drying cycle until a constant weight is obtained (weight loss of 0.5 mg or less is obtained). Record both weights on the data sheet and determine the concentration of the Suspended Solids in mg/L.

(7) Determine the concentration of the Suspended Solids in mg/L by calculating the amount of nonfilterable residue as follows:

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

A = weight of filter and dried residue (mg).
B = weight of filter (mg).

(8) Report solids, residue at 103 – 105 °C, suspended, concentrations as follows:
a. Less than 1,000 mg/L, whole numbers
b. 1,000 mg/L and above, three significant figures

g) Quality Control

i) Method detection limits (MDL): Method detection limits should be established using the guidelines in Chapter ~~H~~VI, Section ~~E~~8.

ii) Method blank: see Chapter ~~H~~VI, Section ~~D~~6.

iii) Laboratory duplicate: see Chapter ~~H~~VI, Section ~~D~~6.

iv) Reference materials: The laboratory must analyze a standard reference material once a year, as available.

h) References

U.S. EPA, 1979, "Methods for Chemical Analysis of Water and Wastes", Method 160.2.

U.S. Geological Survey, Techniques of Water-Resources Investigations of the United States Geological Survey. Chapter A1, Methods for the Determination of Inorganic Substances in Water and Fluvial Sediments. Book 5, Laboratory Analysis, 3rd Ed.; Method I-3765-85, p. 443, (1989).

U.S. Geological Survey, Techniques of Water-Resources Investigations of the United States Geological Survey. Chapter A1, Methods for the Determination of Inorganic Substances in Water and Fluvial Sediments. Book 5, Laboratory Analysis, 3rd Ed.; Total Fixed Suspended Solids Method I-3766-85, p. 457, (1989)

Standard Methods for the Examination of Water and Wastewater, Method 2540 D, "Total Suspended Solids Dried at 103 – 105 ° C Solids Ignited at 550°C" 22nd Edition, pp 2-66, 2012.

Code of Federal Regulations 40, Ch. 1, Pt. 136, Appendix B.

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Fixed Suspended Solids

a) Scope and Application

i) This methodprocedure is used to obtain the amount of fixed suspended solidsmatter present in the solid fraction of sewage, activated sludge,total suspended solids. This procedure is applicable to the determination of fixed matter in drinking, ground, surface, and saline waters, domestic and industrial wastes, or bottom sediments.

b) Summary of Method

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i) The residue obtained from the determination of total suspended solids is ignited at 550 ± 250°C in a muffle furnace. The remaining solids after ignition is reported as mg fixed suspended solids/L.

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c) Interferences

i) The principal source of error in the determination is failure to obtain a representative sample.

ii) The test is subject to many errors due to loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics, and decomposition of mineral salts during combustion.

d) Apparatus and Materials

i) FiltrationGlass fiber filter discs, without organic binder: Whatman 47 mm diameter, 1.5 µm pore size, 0.70 µm pore size, or equivalent can be used. Pore size should be recorded.

ii) Filter support: Filtering apparatus with reservoir and a coarse (40-60 microns) fritted disc as a filter support.

NOTE: Many funnel designs are available in glass or porcelain. Some of the most common are Hirsch or Büchner funnels, membrane filter holders and Gooch crucibles. All are available with coarse fitted disc.

iii) Suction flask.

iv) Drying ovenMuffle Furnace: Capable of maintaining a temperature of 103-105 ± 2550°C.

14.4.3 Muffle furnace.

14.4.4v) Desiccator.

14.4.5vi) Analytical balance: Capable of weighing to 0.1 mg.

vii) Graduated cylinder.

viii) Wash bottle.

e) Sample Handling

i) Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration

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4 ± 2°C, or freezing -20 ± 2°C to minimize microbiological decomposition of solids is recommended. Holding time for FSS samples is 7 days. Sample should be stored in a plastic or resistant- glass container

f) Procedure

i) Ignite the residue from the suspended solids procedure at 550 ± 50°C for approximately 15 to 20 minutes in a muffle furnace to a constant weight.

ii) Let the dish or filter disk partially cool in air until most of the heat has dissipated. Transfer to a desiccator and, cool to room temperature and record the filter weight using an analytical balance. Repeat the drying cycle until a constant weight is obtained (weight verification of 0.5 mg or less is obtained). Record the second filter weight on the data sheet and determine.

iii) Determine the concentration of the fixed suspended solids by using the following equation in mg/L by subtracting the weight of the residue plus the dishfilter after ignition from the weight of the dishfilter times 1000 over the volume of sample used.

$$Concentration\ (mg/L) = \frac{(W_{RD} - W_D) \times 1000}{V}$$

where,

W_{RD} = Weight of residue and dishplus filter (mg)
W_D = Weight of dish filter (mg)
V = Volume of sample filtered (mL)

g) Quality Control

14.7.1i) Method detection limits (MDL): Method detection limits should be established using the guidelines in Chapter IVI, Section E8.

14.7.2ii) Method blank: see Chapter IVI, Section C6.

14.7.3iii) Laboratory duplicate: see Chapter IVI, Section C6.

14.7.4iv) Reference materials: The laboratory must analyze a standard reference material once a year, as available.

h) References

U.S. EPA, 1979, Methods for Chemical Analysis of Water and Wastes, Method 160.2.

Annual Book of ASTM Standards, "Fixed and Volatile Solids Ignited at 550°C", Standard D 2540, Method E, p 2-77 (1980).

Standard Methods for the Examination of Water and Wastewater, Method 2540 E, "Fixed and Volatile Solids Ignited at 550^{0}C" 22nd Edition, pp 2-67, 2012.

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U.S. Geological Survey, Techniques of Water-Resources Investigations of the United States Geological Survey. Chapter A1, Methods for the Determination of Inorganic Substances in Water and Fluvial Sediments. Book 5, Laboratory Analysis, 3rd Ed.; Total Fixed Suspended Solids Method I-3766-85, p. 457, (1989)

U.S. Geological Survey, Techniques of Water-Resources Investigations of the United States Geological Survey. Chapter A1, Methods for the Determination of Inorganic Substances in Water and Fluvial Sediments. Book 5, Laboratory Analysis, 3rd Ed.; Method I-3765-85, p. 443, (1989).

Code of Federal Regulations 40, Ch. 1, Pt. 136, Appendix B.

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