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Standard Test Method for Filterable and Nonfilterable Matter in Water¹

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1. Scope*

1.1 This test method covers the determination of filterable and nonfilterable matter in drinking, surface, and saline waters, domestic and industrial wastes. The practical range of the determination of nonfilterable particulate matter is 4 to 20 000 mg/L. The practical range of the determination of filterable matter is 10 to 20 000 mg/L.

1.2 Since the results measured by this test are operationally defined, careful attention must be paid to following the procedure as specified.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see Section 9.

2. Referenced Documents

2.1 ASTM Standards:²

- D 596 Guide for Reporting Results of Analysis of Water
- D 1129 Terminology Relating to Water
- D 1192 Guide for Equipment for Sampling Water and Steam in Closed Conduits
- D 1193 Specification for Reagent Water
- D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water
- D 3370 Practices for Sampling Water from Closed Conduits
- D 3856 Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water
- D 5847 Practice for the Writing Quality Control Specifications for Standard Test Methods for Water Analysis
- E 319 Practice for the Evaluation of Single-Pan Mechanical Balances

¹ This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

E 898 Methods of Testing Top-Loading, Direct-Reading Laboratory Scales and Balances

3. Terminology

3.1 *Definitions:* For definitions of other terms used in this test method, refer to Terminology D 1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *filterable matter*—also commonly referred to as total dissolved solids. It is that dissolved matter that is capable of passing through a glass fiber filter and dried to constant weight at 180°C, as determined by following the procedures outlined in this test method.

3.2.2 *nonfilterable matter*—also commonly known as total suspended solids. It is that particulate matter that is retained on a glass fiber filter and dried to a constant weight at 103 to 105°C, as determined by following the procedures outlined in this test method.

4. Summary of Test Method

4.1 A well-mixed sample is filtered through a weighed standard glass fiber filter. The suspended solids are retained on the filter, which is dried at 105°C and weighed. The increased mass on the filter represents the nonfilterable matter.

4.2 The filtrate from 4.1 may be used to determine the filterable matter. The filtered sample (liquid phase) is evaporated to dryness and heated to 180°C in a tared vessel to a constant weight.

5. Significance and Use

5.1 Solids, both as filterable and nonfilterable matter, are important in the treating of raw water and wastewater, and in monitoring of streams.

5.2 Waste solids impose a suspended and settleable residue in receiving waters. Suspended and soluble materials provide a matrix for some biological slime and, in sufficient quantity, impair respiration of organisms. These solids may create nuisance slime beds and odors while imposing a long-term biological oxidation load over limited receiving water areas.

5.3 Knowledge of suspended and soluble materials is important in treating raw water supplies. Knowledge of solids loading can aid in determining the type or amount of treatment, or both, necessary to make the water acceptable for use. Such information may also be used to determine acceptability of

*A Summary of Changes section appears at the end of this standard.

water after treatment. Too little treatment may not be desirable and excess treatment costs money.

5.4 Stream monitoring is important for environmental reasons. Stream improvements, water pollution monitoring, mass wasting, algal studies, and sediment loads are but a few of the many reasons streams are monitored.

6. Interferences

6.1 For some samples, chemical reactions may cause some materials to change from one phase to another. For example, in some groundwaters, ferrous ions may form insoluble ferric hydroxides. Softened water high in carbonates may precipitate calcium carbonate. In such cases, holding time may have a critical impact upon both the filterable and nonfilterable matter. Such samples may have to be filtered in the field.

6.2 This test method is not meant to include nonrepresentative particulates such as leaves, sticks, insects, fish, etc. These should be removed before the analysis.

6.3 Certain materials may be measured poorly, or not at all. Some materials may decompose or volatilize at the required temperature. Other substances, such as glycerin or sulfuric acid, will remain liquid at the required temperature, giving variable results. Oils and greases may present similar problems and can end up in either the filterable or nonfilterable portion.

6.4 Suspended solids samples high in dissolved matter, such as saline waters, brines, and some wastes, may be subject to a positive interference by the retention of dissolved matter, such as salts and sugars, on the filter. Care must be taken in the final rinsing of the filter so as to minimize this potential interferent. Additional washing may be necessary.

6.5 Clogging of the filter with too fine or too much material will prolong the filtering time and retain smaller particles that would normally pass through the filter, thus giving elevated values to nonfilterable matter and low values to the filterable matter. Biological material, such as algae, may also prolong filtration time or plug the filter.

6.6 Some samples may be hygroscopic, requiring prolonged drying, extra careful desiccation, and rapid weighing. For filterable matter, samples highly mineralized or high in bicarbonate may require careful and possibly prolonged drying. For the bicarbonate, the extended drying may be needed to ensure complete conversion to carbonate.

6.7 Too much material retained on the filter may entrap water, and may also require extended drying time for the suspended solids. For filterable matter, excessive residue in the dish may cause the formation of a water-trapping crust, giving elevated values.

6.8 For some users, 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. An example is determining chlorophyll content to estimate the amount of algae present. Such modifications may be beyond the scope of this test method.

7. Apparatus

7.1 *Glass Fiber Filters*, without organic binder.³

NOTE 1—Although there is no organic binder in these filters, they may contain a wet strength resin that is partially soluble. It is therefore important to adequately prewash the filters as prescribed.

7.2 *Membrane Filter Assembly*—A borosilicate glass, stainless steel, or plastic funnel with a flat, fritted, or grid base so as to provide uniform support and filterable surface. The top section of the funnel shall fit over the edge of the filter to provide a seal. The top should be removable to allow easy access for removing the filter. A Gooch crucible with a fritted bottom may be used in lieu of the funnel.

7.3 *Planchet or Pan*, made of aluminum or stainless steel, capable of supporting the filter when it is not on the filter assembly.

7.4 *Drying Oven*, capable of maintaining a temperature between 103 and 105°C for nonfilterable matter and between 178 and 182°C for filterable matter.

NOTE 2—To prevent dust and sample from being blown around, it is preferred that the oven for the particulate matter be of a gravity convection type. If this is not possible, samples should be shielded from the forced air of mechanical convection ovens.

7.5 *Analytical Balance*, capable of measuring to the nearest 0.1 mg.⁴

7.6 *Vacuum Source*.

8. Reagents and Materials

8.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type I or II of Specification **D 1193**. Type III or IV may be used if they effect no measurable change in the blank or sample.

9. Hazards

9.1 Care must be taken to ensure filter funnels and filtering flasks are in a sound state. Any tiny nick, scratch, or weakness in glass flasks or other apparatus can create a potential for an implosion hazard. Wrapping a flask is not adequate protection in case of an implosion. It is recommended that a solid shield, such as a plexiglass cage, be placed around any filtering flask.

10. Sampling

10.1 Collect the sample in accordance with the applicable ASTM standard as follows: Specification **D 1192** and Practices **D 3370**.

11. Procedure

11.1 Prepare the glass fiber filters before use.

³ Millipore AP-40, Whatman 934-AH, Gelman type A/E, or equivalent, was specified for the round-robin.

⁴ The balance prescribed in this test method should be tested periodically according to Practice **E 319** or Test Method **E 898**.

11.1.1 Place the glass fiber filter on the membrane filter assembly, or insert into the bottom of a suitable Gooch crucible, with the wrinkled surface up. While a vacuum is applied, wash the disc with three successive volumes of water. Each volume of water should be equal to 3 mL for each square centimetre of filterable surface area. For standard 47 mm filter holders with 35 mm diameter funnels, this would be 30 mL for each wash for a total of 90 mL. Continue the vacuum until the free water has been removed. Discard the washings.

NOTE 3—Proper washing is important for removing loose fiber and wet strength resins. One 90-mL wash is not as effective as three 30-mL washes.

NOTE 4—On some filters it may be difficult to tell which is the wrinkled side. Usually the opposite side has faint markings of the wire mesh used to manufacture the filter mat.

11.1.2 Skip 11.1.3 and 11.1.4 if only filterable matter is being determined.

11.1.3 Release the vacuum and carefully remove the filter with forceps. Place the filter on a planchet, and dry in an oven at 103 to 105°C for 1 h. Gooch crucibles with filter may be handled without the planchet.

11.1.4 Remove from the oven and place in a desiccator until cool. If the desiccation time exceeds 12 h, reheat and desiccate again. Weigh the filter plus planchet to the nearest 0.1 mg just before using. After oven drying, the filter shall be handled only with forceps, and the planchet or crucible shall be handled only with forceps, tongs, or lint-free gloves.

11.2 Preparation of the Evaporating Dish:

11.2.1 If filterable matter is to be determined, heat a clean dish to 178 to 182°C in an oven for 1 h. After removing from the oven, treat as in 11.1.3.

NOTE 5—The dish should be as small as practical to contain the volume of the sample plus the rinses. The relative mass of the dish needs to be kept at a minimum in order to be able to measure small mass differences with any accuracy. This is because of the inherent difficulties of trying to control temperature and moisture on a large mass within the requirements of the test. For larger volumes, it may be more practical to evaporate smaller increments, refilling the dish when dry until all the sample is transferred.

NOTE 6—The dish should be made of a material that is inert to the sample. Materials such as aluminum will oxidize when heated with many liquids, increasing the mass of the pan. Glass or light weight ceramic material is generally preferred.

11.3 Determine the proper sample volume.

11.3.1 Sample volume determination for nonfilterable matter.

11.3.1.1 Start with a volume of sample equal to about 10 mL/cm² of filterable surface area. For the standard 47 mm filter holders with 35 mm diameter funnels, this would be about 100 mL. If this fails to yield at least 2.5 mg of dry solids on the filter, increase the sample volume until that mass is attained, a volume of 1 L is reached, or the “break point” in 11.3.1.3 is reached. Do not exceed 200 mg on the filter.

11.3.1.2 For other filter sizes, maintain at least 1 mg of dry solids per 4 cm² of filterable surface area, with a minimum of 2.5 mg.

11.3.1.3 If the filtration time exceeds 5 min, develop a “break-point” curve (see 11.3.3). This process needs to be done only when the character of a sample is unfamiliar or changes.

11.3.2 Sample volume determination for filterable matter.

11.3.2.1 Choose a sample volume to yield between 2.5 and 200 mg. If more than 5 min is needed for the filtration, perform the “break point” determination as per 11.3.3.

NOTE 7—If the solids are expected to be high, a known *proportion* of the total material, sample plus wash solution, that passed through the filter may be used for the determination. For example, if 200 mL of sample was filtered and only 190 mL of liquid passed through the filter (with all free filterable liquid passing through, leaving 10 mL of nonfilterable solids retained on the filter), the total volume of filtrate would be 250 mL, including the wash water. If a 100-mL portion of the filtrate could be used for the filterable solids test, the final mass of dried solids weighed would have to be divided by 0.4 to account for the 40 % proportion of the sample used.

11.3.3 Break-Point Determination:

11.3.3.1 Place filter in the filtering apparatus. For this procedure, the filter needs no preparation. Add a small, known volume of sample that will filter rapidly and time how long it takes to filter.

11.3.3.2 Repeat 11.3.3.1, increasing the volume until it can be determined at what point the filtration rate drops off rapidly.

11.3.3.3 Plot the time versus the volume filtered. Select the proper volume as that just short of the time that a significant change in filtration rate occurs. An example of a break point curve is shown in Appendix X1.

NOTE 8—If at least 2.5 mg of material cannot be retained on the filter because of plugging, a larger diameter filtration system is suggested. Fritted membrane style filter holders range in sizes up to 9 cm in diameter.

11.3.4 Analyze sample volumes of less than 20 mL by diluting 100 mL to 1 L and running the diluted sample. This is to assure that a representative sample is obtained. Pipetting is generally discouraged since the pipet tip can act as a filter.

11.4 Assemble the filter apparatus with the prepared filter (see 11.1) and start the suction. If the filter is not sealed around the edges by the funnel, such as in the case with a Gooch crucible, wet the filter with a small volume of water to seat it to the base or support. If filterable matter is to be determined, be sure the suction flask is clean.

NOTE 9—If the sample size is small, it may be convenient to place a smaller container, such as a large test tube, into the vacuum flask in order to catch the sample and rinses for filterable matter.

11.5 Mix the sample thoroughly, and quickly transfer a volume of sample as determined in 11.2 into a “to contain,” or TC, graduated cylinder. Pour this measured sample onto the filter and continue to apply suction until all traces of water have passed through.

NOTE 10—Because of the nature of TSS, it is important to thoroughly mix each sample immediately before every aliquot is taken. Many suspended solids settle rapidly, giving a distorted sample if not carefully mixed and quickly sampled.

11.6 With the suction still on, wash the graduated cylinder, the filter, and particulate matter, and the funnel wall with three portions of water, allowing complete drainage between washing. Each portion of wash water should be about 2 mL/cm² of filterable surface. For a 47 mm filter with a 35 mm diameter funnel, the volume of each portion should be 20 mL, for a total of 60 mL. If filterable matter is being run, save the wash water with the sample.