Designation: D5907 – 10

Standard Test Methods for Filterable Matter (Total Dissolved Solids) and Nonfilterable Matter (Total Suspended Solids) in Water[1]

This standard is issued under the fixed designation D5907; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 These test methods cover the determination of filterable matter, total dissolved solids (TDS), and nonfilterable matter, total suspended solids (TSS), in drinking, surface, and saline waters, domestic and industrial wastes. The practical range of the determination of nonfilterable particulate matter (TSS) is 4 to 20 000 mg/L. The practical range of the determination of filterable matter (TDS) is 10 mg/L to 150 000 µg/g.

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 The test methods appear in the following order:

Filterable Matter (TDS) and Nonfilterable Matter (TSS), mg/L	Sections 10-14
Total Dissolved Solids High Precision Method, µg/g	Sections 15-19

1.4 *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 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D596 Guide for Reporting Results of Analysis of Water
D1129 Terminology Relating to Water
D1192 Guide for Equipment for Sampling Water and Steam in Closed Conduits[3]
D1193 Specification for Reagent Water
D1429 Test Methods for Specific Gravity of Water and Brine
D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
D3370 Practices for Sampling Water from Closed Conduits
D3856 Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water
D3977 Test Methods for Determining Sediment Concentration in Water Samples
D4411 Guide for Sampling Fluvial Sediment in Motion
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
D5905 Practice for the Preparation of Substitute Wastewater
E319 Practice for the Evaluation of Single-Pan Mechanical Balances
E898 Test Method of Testing Top-Loading, Direct-Reading Laboratory Scales and Balances
E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

3. Terminology

3.1 *Definitions*:

For definitions of other terms used in this test method, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 *filterable matter*—also commonly referred to as total dissolved solids (TDS). 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 (TSS). 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 theise test methods.

4. Significance and Use

4.1 Solids, both as filterable matter (TDS) and nonfilterable matter (TSS), are important in the treating of raw water and wastewater, and in monitoring of streams.

4.2 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

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[1] These test methods are under the jurisdiction of ASTM Committee D19 on Water and are 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.

[3] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

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

nuisance slime beds and odors while imposing a long-term biological oxidation load over limited receiving water areas.

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

4.4 Stream monitoring is important for environmental reasons, such as compliance with discharge permits. Stream improvements, water pollution monitoring, mass wasting, algal studies, and sediment loads are but a few of the many reasons streams are monitored.

5. Interferences

5.1 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.

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

5.3 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 (TDS) or nonfilterable (TSS) portion.

5.4 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.

5.5 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 (TSS) and low values to the filterable matter (TDS). Biological material, such as algae, may also prolong filtration time or plug the filter.

5.6 Some samples may be hygroscopic, requiring prolonged drying, extra careful desiccation, and rapid weighing. For filterable matter (TDS), 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.

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

5.8 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 (TSS). 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.

6. Apparatus

6.1 *Analytical Balance*, capable of measuring to the nearest 0.1 mg.[4] For the high precision total dissolved solids method, the recommended balance should have a capacity of 200 g and be capable of measuring to the nearest 0.01 mg.

6.2 *Drying Oven*, capable of maintaining a temperature between 103 and 105°C and between 178 and 182°C .

NOTE 1—To prevent dust and sample from being blown around, it is preferred that the oven for the particulate matter (TSS) be of a gravity convection type. If this is not possible, samples should be shielded from the forced air of mechanical convection ovens.

6.3 *Evaporating Dishes*, 70-mL to 250-mL capacity, not to exceed 200 g in weight.

6.4 *Glass Fiber Filters*, without organic binder.[5] 47-mm diameter filters are needed for the high precision TDS method when field filtration is not utilized.

NOTE 2—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.

6.5 *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. A 47-mm filter assembly is needed for the high precision TDS method when field filtration is not used.

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

6.7 *Vacuum Source*.

7. Reagents and Materials

7.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type I or II of Specification D1193. Type III or IV may be used if they effect no measurable change in the blank or sample.

8. Hazards

8.1 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.

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[4] The balance prescribed in this test method should be tested periodically according to Practice E319 or Test Method E898.

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

9. Sampling

9.1 Collect the sample in accordance with the applicable ASTM standard as follows: Specification D1192 and Practices D3370.

9.1.1 If sampling is required from an open channel flow, use Guide D4411 and Test Methods D3977 on a separate sample bottle to determine the sediment concentrations instead of the TSS.

9.1.2 The user of the method is advised to use a slurry rotary sample divider to obtain reproducible aliquots of suspended solids for the TSS determination.[6]

9.2 If the TDS concentration is shown to change as a function of time more than the repeatability measured in the interlaboratory testing of the method for a particular sample matrix, it will be necessary to filter the sample in the field and transfer the sample volume needed for the high precision TDS method to refrigerated sample collection bottles.

Filterable Matter (TDS) and Nonfilterable Matter (TSS)

10. Summary of Test Method

10.1 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 (TSS).

10.2 The filtrate from 10.1 may be used to determine the filterable matter (TDS). The filtered sample (liquid phase) is evaporated to dryness and heated to 180°C in a tared vessel to a constant weight.

11. Procedure

11.1 Prepare the glass fiber filters before use.

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 (TDS) 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 (TDS) is to be determined, heat a clean dish to 178 to 182°C in an oven for 1 h. After removing from the oven, cool in desiccator and weigh to the nearest 0.1 mg and record weight.

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 (TSS).

11.3.1.1 Start with a volume of sample equal to about 10 mL/cm^2 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 cm^2 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 (TDS).

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*:

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[6] The sole source of supply of the apparatus (Quinn Model SS-001, for wet slurry division) known to the committee at this time is Quinn Process Equipment Co., 3400 Brighton Blvd., Denver, CO 80216. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

TABLE 1 Nonfilterable Matter (TSS)[A]

Number of Laboratories	Expected Amount in mg/L	Measured Amount in mg/L	S_T, in mg/L	S_O, in mg/L	Bias, in mg/L	Bias, %	Statistically Significant
6	5	4.75	0.23	NA	−0.25	−5	yes
11	10	9.4	0.69	0.94	−0.6	−6	yes
11	15	14.8	1.41	0.94	−0.2	−1	no
11	30	28.9	1.50	0.56	−1.1	−4	yes
10	36	34.6	0.98	0.56	−1.4	−4	yes
11	50	49.2	2.05	1.79	−0.8	−2	no
11	67.4	65.3	2.53	1.79	−2.1	−3	yes
11	70	68.7	2.82	1.63	−1.3	−2	no
10	80	78.7	2.97	1.63	−1.3	−2	no

[A] NA = not available. There is no acceptable Youden pair for this sample set.

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/cm^2 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.Table 1

NOTE 11—For nonfilterable matter (TSS) samples with high dissolved solids contents, such as seawater and brine solutions, small increments of extra wash water may be required. Tests such as conductivity, chloride, dissolved solids, etc. can be used to determine when there are no significant dissolved solids in the wash water. For filterable matter (TDS), this generally is not a significant problem.

11.7 After the filter has been sucked dry, release the vacuum and carefully remove the filter from the filtering apparatus and place on the planchet, or remove the Gooch crucible from the crucible holder.

11.8 If filterable matter is being determined, carefully transfer the contents from the filtering flask into the evaporating dish (see 11.2). Rinse the filtering flask three times with a small portion of water and add the rinse to the evaporating dish.

11.9 If nonfilterable matter (TSS) is being determined, dry the filter at least 1 h at 103 to 105°C. The drying time should be long enough to ensure a constant weight. Place in a desiccator, cool, and weigh to the nearest 0.1 mg as in 11.1.3.

NOTE 12—The drying time should be checked on new types of samples and periodically on familiar samples to be sure that it is sufficient for the mass to be constant; that is, the difference is less than 0.5 mg, or 4 % of the previous weighing, whichever is greater.

11.10 Evaporate the liquid for the filterable matter on a steam bath or in an oven at 103 to 105°C. After the liquid is gone, dry the evaporating dish at 178 to 182°C for at least 1 h. The drying time should be long enough to ensure a constant weight. Place in a desiccator, cool, and weigh to the nearest 0.1 mg as in 11.1.3.

NOTE 13—The drying time should be checked on new types of samples and periodically on familiar samples to be sure that it is sufficient for the mass to be constant; that is, the difference is less than 0.5 mg, or 4 % of the previous weighing, whichever is greater.

11.11 With each batch of samples that are run, a blank shall be run. The blank shall be taken through the process without the addition of a sample in 11.4. If a blank filter shows any increase in mass or a loss of greater than 0.4 mg, rerun the samples associated with it. If the mass of a blank evaporating dish varies by more than ±0.5 mg from the initial mass, rerun the samples associated with it. The blank result is not subtracted from the sample.

NOTE 14—A blank filter carried through the process generally loses a mass of about 0.2 mg. So, blank requirements represent the range of -0.2 ± 0.2 mg.

12. Calculation

12.1 Calculate the amount of nonfilterable matter (TSS) as follows:

$$total\ nonfilterable\ matter\ (TSS),\ in\ mg/L =$$

TABLE 2 Filterable Matter (TDS)

Number of Laboratories	Expected Amount in mg/L	Measured Amount in mg/L	S_T, in mg/L	S_O, in mg/L	Bias, in mg/L	Bias, %	Statistically Significant
6	37.5	36.7	2.8	2.1	–0.8	–2	no
6	56.2	54.8	3.1	2.1	–1.4	–2	no
6	112	101	12	15	–11	–10	no
6	135	126	21	15	–11	–8	no
6	188	173	16	9.3	–15	–8	no
6	253	228	11	9.3	–25	–10	yes
6	262	243	18	2.9	–19	–7	no
6	300	279	21	2.9	–21	–7	no

$$\frac{(mg\ of\ residue + filter) - (mg\ of\ filter)}{mL\ of\ sample\ filtered/1000} \qquad (1)$$

12.2 Calculate the amount of filterable matter as follows:

$$total\ filterable\ matter\ (TDS),\ in\ mg/L =$$

$$\frac{(mg\ of\ residue + dish) - (mg\ of\ dish)}{mL\ of\ sample\ filtered/1000} \qquad (2)$$

13. Report

13.1 Do not report results smaller than the nearest milligram per litre. The precision and bias data from the round-robin suggest the method is good to two significant figures at most. There should be supporting data available in the laboratory before reporting more significant figures.

14. Precision and Bias

14.1 The single-operator precision and overall precision and bias of this test method are given in Table 1 for nonfilterable matter (TSS) and Table 2 for filterable matter (TDS). The material tested was a purchased commercial suspended solids material in an unspecified mixture of salt.[7] The material is only available at the maximum concentration tested. Other concentrations were created for testing by diluting the original solution. The precision and bias statement reflects only the results for this specified matrix and may not reflect other matrices. The material tested was the only material known to the committee to be available in a liquid form that can test all aspects of the test method. The limit of available known material in a form that can test all aspects of this test method prohibits testing the full range of the method.

14.2 Six independent laboratories and operators successfully completed the round robin study for filterable matter (TDS). Six to eleven independent laboratories successfully completed the round robin study for nonfilterable matter (TSS). The precision and bias evaluation for this test method was conducted using a Youden pair design and conforms to Practice D2777–86. Under the allowances made in 1.4 of D2777–98, these precision and bias data do meet existing requirements for interlaboratory studies of Committee D19 test methods. Information on low-level results from laboratories that survived the ranking tests, but not meeting full requirements of the test method, is given in Appendix X2.

14.3 A duplicate and known control sample should be run each day that a sample is analyzed. The duplicate and control sample shall meet satisfactory limits as established by the control chart before an analysis is considered satisfactory.

14.4 Until such time as other quality assurance/quality control (QA/QC) procedures are established, it is recommended that the user use Guide D3856 as a guide for establishing QA/QC.

14.5 Before this test method is applied to the analysis of samples, the analyst shall establish his/her own precision and bias data.

Total Dissolved Solids High Precision Method

15. Summary of Test Method

15.1 A well-mixed sample is filtered through a weighed standard glass fiber filter. The suspended solids are retained on the filter and the filtrate is be used to determine the filterable matter (TDS) in duplicate. The filtered sample (liquid phase) is evaporated to dryness and heated to 180°C in a tared vessel to a constant weight.

16. Procedure

16.1 Prepare the 47-mm glass fiber filters before use.

16.1.1 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 centimeter 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.

16.1.2 Commercially available pre-washed filters meeting these specifications may also be used.

16.2 *Field Filtration* —Where it is necessary to utilize field filtration due to significant changes in TDS concentration with time (see 9.2) filters may be discarded after use without washing and solution transferred to refrigerated sample collection bottles in the field and transported and stored at 4 °C until analysis can be initiated.

16.2.1 *Preparation of the Field Collection Bottles*—Cool the clean sample collection bottle to 4°C, prior to sampling.

16.3 *Laboratory Filtration*:

16.3.1 Assemble the 47-mm filter apparatus with the prepared filter (see 16.1) attached to a clean suction flask and start the suction. If the filter is not sealed around the edges by the

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[7] Alpha-trol solution available from Alpha-trol, Inc., P.O. Box 867, Levittown, PA 19058. Another source of solution has been identified. Should satisfactory data from other sources be made available, such data will be included in the precision and bias statement.

TABLE 3 Total Dissolved Solids High Precision Method[A]

Youden Pair	1	2	3	4	5	6
Actual Amount in µg/g	335	410	630	770	900	1210
Measured Amount in µg/g	313	423	670	809	1015	1207
S_T in µg/g	40	52	20	28	28	54
RSD, %	13	12	3	3	3	4
S_O in µg/g	63		10		40	
RSD, %	17		1		4	
Bias in µg/g	–22	13	40	39	115	–3
Bias, %	–6.5	3.1	6.3	5.1	13	–0.3
Within Certified Range	No	Yes	Yes	Yes	No	Yes

[A] Six participating laboratories.

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.

16.3.2 Mix the sample thoroughly, and quickly transfer sample onto the filter and continue to apply suction until all traces of water have passed through.

16.3.3 After the filter has been sucked dry, release the vacuum and carefully remove the filter from the filtering apparatus and discard.

16.4 *Determine the Proper Sample Volume*:

16.4.1 Perform a preliminary total dissolved solids determination starting at Section 11 if the approximate total dissolved solids concentration of the test sample is not known.

16.4.2 Choose a sample volume to yield approximately 25-200 mg of solids. Calculate the approximate volume of solution for the test sample from the preliminary total dissolved solids determined in 16.4.1 by dividing the minimum and maximum milligrams of solids by the total dissolved solids in milligrams per liter and select a volume that is between the minimum calculated and the maximum.

16.5 *Preparation of the Evaporating Dishes or Beakers*—Heat duplicate clean 50-mL to 250-mL capacity evaporation dishes or beakers, depending on the concentration of totals dissolved solids to be determined, to 178 °C to 182 °C in an oven for 1 h. After removing from the oven, cool in desiccator and weigh to the nearest 0.1mg and record weights.

16.6 *Procedure*:

16.6.1 Weigh a pre-cleaned and dried graduated cylinder to the nearest 0.1 g.

16.6.2 Transfer the volume of sample as determined in 16.4.2 to the graduated cylinder, weigh and record the sample weight.

16.6.3 Carefully transfer the proper sample volume of filtrate into the evaporating dish or beaker, reserving any excess solution for stepwise evaporation. Repeat the procedure with a duplicate sample portion into a second evaporating dish or beaker.

16.6.3.1 Class A pipettes, and direct transfer and weighing of solution after zeroing the balance were also used in place of the graduated cylinders during interlaboratory testing and these practices are permissible.

16.6.4 Evaporate the liquid in an oven at 103 to 105° C or a steam bath. After the liquid is gone, repeat with any reserved solution from 16.6.3 until all of the test solution has been dried. Rinse the filtrate container three times with a small portion of water and add the rinse to the evaporating dish.

16.6.5 Dry the evaporating dish or beaker at 178 to 182°C for at least 1 h. The drying time should be long enough to ensure a constant weight. Place in a desiccator, cool, and weigh to the nearest 0.1 mg.

16.7 With each batch of samples that are run, a blank shall be run. The blank shall be taken through the process without the addition of a sample. If a blank filter shows any increase in mass or a loss of greater than 0.4 mg, rerun the samples associated with it. If the mass of a blank evaporating dish varies by more than ±0.5 mg from the initial mass, rerun the samples associated with it. The blank result is not subtracted from the sample.

17. Calculation

17.1 Calculate the amount of total dissolved solids as follows:

$$\text{total dissolved solids, } \mu g/g = \frac{(mg\ of\ residue + dish) - (mg\ of\ dish)}{(g\ of\ sample + cylinder) - (g\ of\ cylinder)/1000} \quad (3)$$

Average duplicate results and round to the nearest µg/g.

NOTE 15—Reporting in mg/L using this method requires multiplication of the calculated result by the density, which can be determined in accordance with Test Method D1429.

18. Report

18.1 Do not report results smaller than the nearest microgram per gram. The precision and bias data from the round-robin suggest the method is good to three significant figures at most. There should be supporting data available in the laboratory before reporting more significant figures.

19. Precision and Bias[8]

19.1 The single-operator precision and overall precision and bias for the total dissolved solids high precision test method are given in Table 3 for the three Youden pairs representing the gold mining pit dewatering matrix, which were determined in

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[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1185.

TABLE 4 Summary of Interlaboratory Statistics For Duplicate Blind Samples

Description	Gold Mining Groundwater	Substitute Waste Water	Gold Leaching Pregnant Solution	Gold Mine Waste Rock Acid Drainage
Mean in µg/g	428	1467	6602	137 290
S_T in µg/g	32	114	243	11 261
RSD, %	7	8	4	8
S_O in µg/g	28	43	50	1102
RSD, %	7	3	1	1
R in µg/g[A]	90	320	681	31 531
R, %	21	22	10	23

[A] R = 2.6 S_T.

accordance with Practice D2777-03. The material tested was commercially prepared to simulate the major components of the pit dewatering matrix. Samples were distributed to eight laboratories and six of the laboratories provided test data. Results from this collaborative study may not be typical for matrices other than those studied.

19.1.1 *Bias*—The accuracy of this test method for total dissolved solids is deemed satisfactory based on the values in Table 3. Users are encouraged to employ these or similar reference materials to verify that this test method is performing accurately in their laboratory.

19.2 The single-operator precision and overall precision were determined in accordance with Practice D2777-03 for duplicate blind samples of gold mining groundwater, substitute waste water prepared in accordance with Practice D5905-98, gold leaching pregnant solution and gold mine waste rock acid drainage.

19.2.1 *Precision*—Six laboratories cooperated in evaluating this test method, providing six sets of data for total dissolved solids, resulting in the precision data summarized in Table 4.

19.3 The single-operator and minimum standard deviations were pooled and were related to concentration according to the following equation:

$$So = 0.00783\ TDS + 25.3$$

$$R^2 = 0.997$$

19.4 The overall and reproducibility standard deviations were pooled and were related to concentration according to the following equation:

$$St = 0.0823\ TDS - 49.2$$

$$R^2 = 0.999$$

20. Quality Control (QC)

20.1 In order to be certain that analytical values obtained using these test methods are valid and accurate within the confidence limits of the test, the following QC procedures must be followed when analyzing filterable matter (TDS) and nonfilterable matter (TSS).

20.2 *Calibration and Calibration Verification*:

20.2.1 The balance used should be calibrated internally or with known weights prior to use.

20.2.2 Verify balance calibration with weights prior to use.

20.3 *Initial Demonstration of Laboratory Capability*:

20.3.1 If a laboratory has not performed the test before, or if there has been a major change in the measurement system, for example, new analyst, new instrument, etc., a precision and bias study must be performed to demonstrate laboratory capability.

20.3.2 Analyze seven replicates of a standard solution prepared from an Independent Reference Material containing a mid-range concentration of filterable matter (TDS) or nonfilterable matter (TSS). The matrix and chemistry of the solution should be equivalent to the solution used in the collaborative study. Each replicate must be taken through the complete analytical test method. The replicates may be interspersed with samples.

20.3.3 Calculate the mean and standard deviation of the seven values and compare to the acceptable ranges of bias in Table 1 for nonfilterable matter (TSS) or Table 2 for filterable matter (TDS). This study should be repeated until the recoveries are within the limits given in Table 1 for nonfilterable matter (TSS) or Table 2 for filterable matter (TDS). If a concentration other than the recommended concentration is used, refer to Practice D5847 for information on applying the F test and t test in evaluating the acceptability of the mean and standard deviation.

20.4 *Laboratory Control Sample (LCS)*:

20.4.1 To ensure that the test method is in control, analyze a LCS containing a mid-range concentration of filterable matter (TDS) or nonfilterable matter (TSS) with each batch or ten samples. If large numbers of samples are analyzed in the batch, analyze the LCS after every ten samples. The LCS must be taken through all of the steps of the analytical method including sample preservation and pretreatment. The result obtained for the LCS shall fall within ± 15 % of the known concentration.

20.4.2 If the result is not within these limits, analysis of samples is halted until the problem is corrected, and either all the samples in the batch must be reanalyzed, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

20.5 *Method Blank*:

20.5.1 Perform a blank as stipulated in 11.11. If those results cannot be attained, halt analysis of samples until the cause can be determined and eliminated. Either all the samples in the batch must be reanalyzed or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

20.6 *Matrix Spike (MS)*:

20.6.1 Filterable matter (TDS) and nonfilterable matter (TSS) cannot be feasibly spiked into samples.

20.7 *Duplicate*:

20.7.1 To check the precision of sample analyses, analyze a sample in duplicate with each batch. The value obtained must fall within the control limits established by the laboratory.

20.7.2 Calculate the standard deviation of the duplicate values and compare to the precision determined by the laboratory or in the collaborative study using an F test. Refer to 6.4.4 of Practice D5847 for information on applying the F test.

20.7.3 If the result exceeds the precision limit, the batch must be reanalyzed or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

20.8 *Independent Reference Material (IRM)*:

20.8.1 In order to verify the quantitative value produced by the test method, analyze an Independent Reference Material (IRM) submitted as a regular sample (if practical) to the laboratory at least once per quarter. The concentration of the IRM should be in the concentration mid-range for the method chosen. The value obtained must fall within the control limits established by the laboratory.

21. Keywords

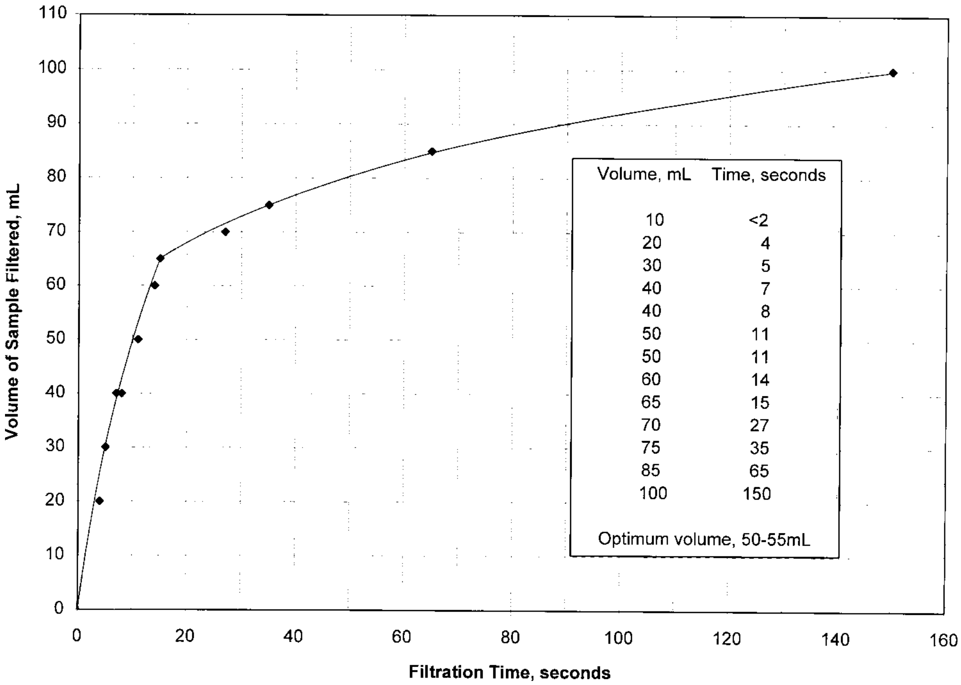
21.1 dissolved matter; filterable matter; nonfilterable matter; suspended matter; total dissolved solids; total suspended solids

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLE GRAPH ILLUSTRATING A BREAK-POINT DETERMINATION

X1.1 Fig. X1.1 illustrates a break-point determination.



Volume, mL	Time, seconds
10	<2
20	4
30	5
40	7
40	8
50	11
50	11
60	14
65	15
70	27
75	35
85	65
100	150

Optimum volume, 50-55mL

FIG. X1.1 Example of a Filtration Study for TSS Breakpoint

X2. INFORMATION ON LOW-LEVEL RESULTS

X2.1 Table X2.1 and Table X2.2 are for informational purposes only and are not meant to validate the test method at these low levels (the 2 and 5 mg/L TSS samples and the 7.5 and 18.8 mg/L TDS samples). Some of the information for these low-level sample values came from data that did not meet the requirements of the test method, mainly having insufficient mass at the end of the test.

TABLE X2.1 Nonfilterable Matter (TSS)

Number of Laboratories	Expected Amount in mg/L	Measured Amount in mg/L	S_T, in mg/L	S_O, in mg/L	Bias, in mg/L	Bias, %	Statistically Significant
10	2	1.5	0.36	0.16	–0.5	–25	yes
10	5	4.6	0.31	0.16	–0.4	–8	yes

TABLE X2.2 Filterable Matter (TDS)

Number of Laboratories	Expected Amount in mg/L	Measured Amount in mg/L	S_T, in mg/L	S_O, in mg/L	Bias, in mg/L	Bias, %	Statistically Significant
6	7.5	7.6	1.8	1.9	0.1	+ 1	no
6	18.8	17.9	2.7	1.9	–0.9	–5	no

SUMMARY OF CHANGES

Committee D19 has identified the location of selected changes to this standard since the last issue (D5907 – 09) that may impact the use of this standard. (Approved May 15, 2010.)

(*1*) Modified 1.3, 2.1, 4.4, Section 6, Section 9, 11.2.1, and Section 12.
(*2*) Sections 15-19, Table 3, and Table 4 were added to the test methods section.
(*3*) Section 4 was moved to Section 10.
(*4*) References to TSS for filterable materials and TDS for non filterable material were added throughout.

Committee D19 has identified the location of selected changes to this standard since the last issue (D5907 – 03) that may impact the use of this standard. (Approved May 1, 2009.)

(*1*) Section 1.3 was added to clarify that this method is not applicable to open channel flow.
(*2*) The SI statement was added in Section 1.4.
(*3*) Reference to D1192 was removed from Section 2 and Section 9.1.
(*4*) References relating to the new Sections 1.3 and 9.2 were added.
(*5*) Added Section 9.2 to discuss open channel flow sampling.

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