Designation: D516 – 22

Standard Test Method for Sulfate Ion in Water[1]

This standard is issued under the fixed designation D516; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 This turbidimetric test method covers the determination of sulfate in water in the range from 5 mg/L to 40 mg/L of sulfate ion (SO_4^{--}).

1.2 This test method was used successfully with drinking, ground, and surface waters. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

1.3 Former gravimetric and volumetric test methods have been discontinued. Refer to Appendix X1 for historical information.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1066 Practice for Sampling Steam
D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
D3370 Practices for Sampling Water from Flowing Process Streams
D4327 Test Method for Anions in Water by Suppressed Ion Chromatography
D5810 Guide for Spiking into Aqueous Samples
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry
E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129.

4. Summary of Test Method

4.1 Sulfate ion is converted to a barium sulfate suspension under controlled conditions. A solution containing glycerin and sodium chloride is added to stabilize the suspension and minimize interferences. The resulting turbidity is determined by a nephelometer, spectrophotometer, or photoelectric colorimeter and compared to a curve prepared from standard sulfate solutions.

5. Significance and Use

5.1 The determination of sulfate is important because it has been reported that when this ion is present in excess of about 250 mg/L in drinking water, it causes a cathartic action (especially in children) in the presence of sodium and magnesium, and gives a bad taste to the water.

5.2 Test Method D4327 ("Test Method of Anions in Water by Suppressed Ion Chromatography") may be used.

6. Interferences

6.1 Insoluble suspended matter in the sample must be removed. Dark colors that cannot be compensated for in the procedure interfere with the measurement of suspended barium sulfate ($BaSO_4$).

[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.

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

6.2 Polyphosphates as low as 1 mg/L will inhibit barium sulfate precipitation causing a negative interference. Phosphonates present in low concentrations, depending on the type of phosphonate, will also cause a negative interference.

6.3 Silica in excess of 500 mg/L may precipitate along with the barium sulfate causing a positive interference.

6.4 Chloride in excess of 5000 mg/L will cause a negative interference.

6.5 Aluminum, polymers, and large quantities of organic material present in the test sample may cause the barium sulfate to precipitate nonuniformly.

6.6 In the presence of organic matter certain bacteria may reduce sulfate to sulfide. To minimize the action of sulfate reducing bacteria, samples should be refrigerated at 4 °C when the presence of such bacteria is suspected.

6.7 Although other ions normally found in water do not appear to interfere, the formation of the barium sulfate suspension is very critical. Determinations that are in doubt may be checked by a gravimetric method in some cases, or by the procedure suggested in 11.2.1.

7. Apparatus

7.1 *Photometer*—One of the following which are given in order of preference.

7.1.1 Nephelometer or turbidimeter;

7.1.2 Spectrophotometer for use at 420 nm with light path of 4 cm to 5 cm;

7.1.3 Filter photometer with a violet filter having a maximum near 420 nm and a light path of 4 cm to 5 cm.

7.2 *Stopwatch,* if the magnetic stirrer is not equipped with an accurate timer.

7.3 *Measuring Spoon,* capacity 0.2 mL to 0.3 mL.

7.4 Filter photometers and photometric practices prescribed in this test method shall conform to Practice E60; spectrophotometer practices shall conform to Practice E275.

7.5 *Laboratory Glassware*—All glassware should be in good condition, clean, and free of contaminating substances.

7.5.1 If the testing is for regulatory purposes, volumetric flasks should be of Type A precision and serialized for quality control tracking.

7.6 *Stirring Apparatus*—A magnetic stirrer should be used along with a magnetic stir bar appropriately sized for the labware in use.

7.7 *Weighing Apparatus*—A scale or analytical balance of the appropriate accuracy and precision should be used. It should be calibrated and within its calibration interval.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D1193, Type I. Other reagent water types may be used provided it is first ascertained that the water is of sufficiently high purity to permit its use without adversely affecting the precision and bias of the test method. Type II water was specified at the time of round robin testing of this test method.

8.3 *Barium Chloride*—Crystals of barium chloride ($BaCl_2 \cdot 2H_2O$) screened to 20 to 30 mesh. To prepare in the laboratory, spread crystals over a large watch glass, desiccate for 24 h, screen to remove any crystals that are not 20 to 30 mesh, and store in a clean, dry jar.

8.4 *Conditioning Reagent*—Place 30 mL of concentrated hydrochloric acid (HCl, sp gr 1.19), 300 mL reagent water, 100 mL 95 % ethanol or isopropanol and 75 g sodium chloride (NaCl) in a container. Add 50 mL glycerol and mix.

8.5 *Sulfate Solution, Standard* (1 mL = 0.100 mg SO_4^{--})—Dissolve 0.1479 g of anhydrous sodium sulfate (Na_2SO_4) in water, and dilute with water to 1 L in a volumetric flask. A purchased stock solution of adequate purity is also acceptable.

8.6 *Filter Paper*—Purchase suitable filter paper. Typically the filter papers have a pore size of 0.45 µm membrane. Material such as fine-textured, acid-washed, ashless paper, or glass fiber paper are acceptable. The user must first ascertain that the filter paper is of sufficient purity to use without adversely affecting the bias and precision of the test method.

9. Sampling

9.1 Collect the sample in accordance with Practice D1066, and Practices D3370, as applicable.

10. Calibration

10.1 Follow the procedure given in Section 11, using appropriate amounts of the standard sulfate solution prepared in accordance with 8.5 and prepare a calibration curve showing sulfate ion content in milligrams per litre plotted against the corresponding photometer readings (10.2).

10.2 Prepare standards by diluting with water 0.0 mL, 5.0 mL, 10.0 mL, 15.0 mL, 20.0 mL, 30.0 mL, and 40.0 mL of standard sulfate solution to 100 mL volumes in volumetric flasks. These solutions will have sulfate ion concentrations of 0.0 mg/L, 5.0 mg/L, 10.0 mg/L, 15.0 mg/L, 20.0 mg/L, 30.0 mg/L, and 40.0 mg/L (ppm), respectively.

10.2.1 A separate calibration curve must be prepared for each photometer and a new curve must be prepared if it is

[3] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.