



Designation: D 4517 – 85 (Reapproved 1999)

An American National Standard

Standard Test Method for Low-Level Total Silica in High-Purity Water by Flameless Atomic Absorption Spectroscopy¹

This standard is issued under the fixed designation D 4517; 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 This test method covers the determination of total silica in water.

1.2 This test method is applicable in the range from 25 to 250 µg/L of silica as SiO₂. Higher concentrations may be determined by decreasing the aliquot volume (see Note 6). Concentration range should not be extended by dilution.

1.3 This test method determines total silica, and does not distinguish between soluble and insoluble forms.

1.4 This test method was tested on reagent water only. It is the user's responsibility to assure the validity of the test method for waters of other matrices.

1.5 *This standard does not purport to address 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.*

2. Referenced Documents

2.1 ASTM Standards:

D 859 Test Method for Silica in Water²

D 1066 Practice for Sampling Steam²

D 1129 Terminology Relating to Water²

D 1192 Specification for Equipment for Sampling Water and Steam in Closed Conduits²

D 1193 Specification for Reagent Water²

D 3370 Practices for Sampling Water from Closed Conduits²

D 3919 Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry²

D 4453 Practice for Handling of Ultra-Pure Water Samples²

3. Terminology

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

¹ This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.03 on Sampling of Water and Water-Formed Deposits, Surveillance of Water, and Flow Measurement of Water. Current edition approved Aug. 30, 1985. Published October 1985.

² *Annual Book of ASTM Standards*, Vol 11.01.

4. Summary of Test Method

4.1 Total silica is determined using an atomic absorption spectrophotometer in conjunction with a graphite furnace. A sample is placed in a graphite tube, evaporated to dryness, charred, and atomized. Since the graphite furnace uses the sample much more efficiently than flame atomization, the detection of low concentrations of elements in small sample volumes is possible. Finally, the absorption signal during atomization is recorded and compared to standards. A general guide for the application of the graphite furnace is given in Practice D 3919. Pretreatment of the graphite tube may be used to enhance the sensitivity and repeatability, or both, of the test.³

4.2 Total silica is determined using a freshly ultrasonically treated and shaken aliquot of sample.

4.3 This test method determines low-level total silica in high purity water. Refer to Test Method D 859, Method B, for determination of molybdate-reactive silica.

5. Significance and Use

5.1 Control of silica in boiler feedwater and boiler water is necessary to minimize the formation of scale-forming silicates which decrease heat transfer in the boiler. Volatilization and carryover of silica with the steam may cause hard, glassy siliceous deposits to form on turbine blades which reduce turbine efficiency.

5.2 Colloidal silica which is not removed by boiler water pretreatment processes may be solubilized in the boiler and thus contribute to the dissolved silica concentration in the boiler. Both dissolved and total silica are of interest.

6. Interferences

6.1 For a complete discussion of general interferences with furnace procedures, the analyst is referred to Practice D 3919.

6.2 Relatively pure water such as demineralizer effluent, condensate, or high-pressure boiler feedwater are of sufficient purity to minimize potential interferences such as sodium, orthophosphate, or sulfate.

³ Rawa, Judith A. and Earl L. Henn, "Determination of Trace Silica in Industrial Process Waters by Flameless Atomic Absorption Spectrometry," *Analytical Chemistry*, Vol 51, March 1979.