



Designation: D3373 – 17

Standard Test Method for Vanadium in Water¹

This standard is issued under the fixed designation D3373; 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 test method covers the determination of dissolved and total recoverable vanadium in most waters and wastewater by graphite furnace atomic absorption spectrophotometry.

1.2 The optimum range of this test method is 10 to 200 $\mu\text{g/L}$ of vanadium based on a 20- μL sample size. Concentrations higher than 200 $\mu\text{g/L}$ may be determined by proper dilution of sample. A detection level as low as 4 $\mu\text{g/L}$ of vanadium has been reported.

1.3 This test method has been used successfully with reagent water, lake water, tap water, river water, condensate from a medium Btu coal gasification process, and well water. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

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 and health practices and determine the applicability or regulatory limitations prior to use.*

1.6 Former Test Method A (Catalytic) was discontinued. Refer to **Appendix X1** for historical information.

1.7 *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.*

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. Referenced Documents

2.1 *ASTM Standards:*²

D1066 Practice for Sampling Steam

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D1976 Test Method for Elements in Water by Inductively-Coupled Argon Plasma Atomic Emission Spectroscopy

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

D3919 Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry

D4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents

D5673 Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry

D5810 Guide for Spiking into Aqueous Samples

D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

3. Terminology

3.1 *Definitions:*

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *continuing calibration blank, n*—a solution containing no analytes (of interest) which is used to verify blank response and freedom from carryover.

3.2.2 *continuing calibration verification, n*—a solution (or set of solutions) of known concentration used to verify freedom from excessive instrumental drift; the concentration is to cover the range of calibration curve.

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

3.2.3 *total recoverable vanadium, n*—a descriptive term relating to the metal forms of vanadium recovered in the acid-digestion procedure specified in this test standard.

4. Summary of Test Method

4.1 Vanadium is determined by an atomic absorption spectrophotometer utilizing a graphite furnace for sample atomization.

4.2 A sample volume of several microlitres, depending upon the concentration of the analyte, is transferred to a graphite tube housed within an electrical furnace and the system is heated in an inert or reducing atmosphere. The sample is evaporated to dryness, charred (pyrolyzed or ashed) and atomized.

4.3 Ground state atoms, produced in atomization, absorb the light of their spectrum emanating from a source and passing through the sample vapor. The amount of light absorbed is proportional to the concentration of the analyte in the sample.

4.4 Since the graphite furnace uses the sample much more efficiently than does flame atomization, the detection of low concentrations of the elements of interest in small volumes is possible.

4.5 Finally, the absorption signal produced during atomization is recorded and compared to those of standards, taken through the same process, by means of an analytical curve.

4.6 A general guide for graphite furnace applications is given in Practice D3919.

4.7 Dissolved vanadium is determined after filtration and addition of HNO₃ to a pH of ≤ 2.

4.8 Total recoverable vanadium is determined following acid digestion and filtration.

5. Significance and Use

5.1 Vanadium can be found in waste that result from chemical cleaning of components in which the metal is alloyed.

5.2 National Pollutant Discharge Elimination Systems permits or other standards, or both, require monitoring pollutants in waste discharged onto the water shed of, or into, navigable waters, and those disposed of in such a manner that eventual contamination of underground water could result.

5.3 This test method affords an accurate and sensitive means of determining compliance or noncompliance, or both, with those permits.

5.4 ICP-MS or ICP-AES may also be appropriate but at a higher instrument cost. See Test Methods D5673 and D1976.

6. Interferences

6.1 For a complete discussion on general interferences with furnace procedures, the analyst is referred to Practice D3919.

7. Apparatus

7.1 *Atomic Absorption Spectrophotometer*, for use at 318.4 nm with background correction.

NOTE 1—The manufacturer's instructions should be followed for all instrumental parameters.

7.2 *Vanadium Hollow Cathode Lamp*.

7.3 *Graphite Furnace* capable of reaching temperature sufficient to atomize the element of interest.

7.4 *Graphite Tubes*, pyrolytically coated, compatible with furnace device are recommended.

7.5 *Pipettes*, microlitre with disposable tips, ranging in size from 1 to 100 µL.

7.6 *Data Storage and Reduction Devices, Computer- and Microprocessor-Controlled Devices, or Strip Chart Recorders* shall be utilized for collection, storage, reduction, and problem recognition (such as drift, incomplete atomization, changes in sensitivity, etc.). Strip chart recorders shall have a full scale deflection time of 0.2 s or less to ensure accuracy.

7.7 *Automatic Sampling Accessory* is recommended, if available.

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 Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.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, references 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.

8.3 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO₃).

8.4 *Vanadium Solution, Stock* (1.0 mL = 100 µg vanadium). Dissolve 0.2296 g of ammonium metavanadate (NH₄VO₃) in water and dilute to 1000 mL. A purchased vanadium stock solution of appropriate known purity is also acceptable.

8.5 *Vanadium Solution, Intermediate* (1.0 mL = 1.0 µg vanadium)—Dilute 5 mL of vanadium stock solution to 500 mL with water.

8.6 *Vanadium Solution, Standard* (1.0 mL = 0.100 µg vanadium)—Dilute 10.0 mL of vanadium intermediate solution to 100 mL with water.

8.7 *Nitrogen Gas*—standard, welders grade, commercially available.

NOTE 2—It is probable that argon or some other inert gas can be used in place of nitrogen. It is the analyst's responsibility to verify suitability.

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual 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.