Designation: D 3867 – 99

An American National Standard

Standard Test Methods for Nitrite-Nitrate in Water[1]

This standard is issued under the fixed designation D 3867; 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 nitrite nitrogen, nitrate nitrogen, and combined nitrite-nitrate nitrogen in water and wastewater in the range from 0.05 to 1.0 mg/L nitrogen. Two test methods[2] are given as follows:

	Sections
Test Method A—Automated Cadmium Reduction	9 to 16
Test Method B—Manual Cadmium Reduction	17 to 24

1.2 These test methods are applicable to surface, saline, waste, and ground waters. It is the user's responsibility to ensure the validity of these test methods for waters of untested matrices.

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 specific hazard statements, see Note 1 and Note 2.

2. Referenced Documents

2.1 *ASTM Standards:*

D 992 Test Method for Nitrate Ion in Water[3]

D 1129 Terminology Relating to Water[4]

D 1141 Specification for Substitute Ocean Water[5]

D 1192 Specification for Equipment for Sampling Water and Steam in Closed Conduits[4]

D 1193 Specification for Reagent Water[4]

D 1254 Test Method for Nitrite Ion in Water[6]

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water[4]

D 3370 Practices for Sampling Water from Closed Conduits[4]

E 60 Practices for Photometric and Spectrometric Methods for Chemical Analysis of Metals[7]

E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers[8]

3. Terminology

3.1 *Definitions:* For definitions of terms used in these test methods, refer to Terminology D 1129.

4. Summary of Test Methods

4.1 A filtered sample is passed through a column containing copper-coated cadmium granules to reduce nitrate ion to nitrite ion. The combined nitrite-nitrate nitrogen is determined by diazotizing the total nitrite ion with sulfanilamide and coupling with *N*-(1-naphthyl)ethylenediamine dihydrochloride to form a highly colored azo dye that is measured spectrophotometrically.

4.2 The nitrite ion originally present in the sample can be determined separately by carrying out the procedure and omitting the cadmium reduction step.

4.3 The nitrate ion can be calculated as the difference between the combined nitrite-nitrate nitrogen and the nitrite nitrogen.

5. Significance and Use

5.1 Both test methods use identical reagents and sample processing. The only difference between the two methods is that one test method is automated and the other is manual. The ranges and interferences are identical.

5.2 The automated test method is preferred when large numbers of samples are to be analyzed. The manual test method is used for fewer samples or when automated instrumentation is not available.

5.3 These test methods replace Test Methods D 1254 (Nitrite) and D 992 (Nitrate). The nitrite test method (Test Method D 1254) used a reagent which is considered to be a potential carcinogen. The nitrate test method (Test Method D 992) has been shown to have relatively large errors when used in

[1] These test methods are under the jurisdiction of ASTM Committee D-19 on Water and are the responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

Current edition approved June 10, 1999. Published November 1999. Originally published as D 3867 – 79. Last previous edition D 3867 – 90.

[2] Methods similar to these appear in *Methods of Chemical Analysis of Water and Wastes*, 2nd edition, U.S. Environmental Protection Agency.

[3] Discontinued; see 1983 *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 11.01.

[5] *Annual Book of ASTM Standards*, Vol 11.02.

[6] Discontinued; see 1980 *Annual Book of ASTM Standards*, Part 31.

[7] *Annual Book of ASTM Standards*, Vol 03.05.

[8] *Annual Book of ASTM Standards*, Vol 03.06.

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

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