



Standard Test Methods for Nickel in Water ¹

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

1. Scope*

1.1 These test methods ^{2, 3, 4} cover the atomic absorption determination of nickel in water and wastewaters. Three test methods are given as follows:

	Concentration Range	Sections
Test Method A—Atomic Absorption, Direct	0.1 to 10 mg/L	7-16
Test Method B—Atomic Absorption, Chelation-Extraction	10 to 1000 µg/L	17-26
Test Method C—Atomic Absorption, Graphite Furnace	5 to 100 µg/L	27-36

1.2 Test Methods A, B, and C have been used successfully with reagent grade water and natural waters. Evaluation of Test Method C was also made in condensate from a medium Btu coal gasification process. It is the user’s responsibility to ensure the validity of these test methods for other 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 hazards statements, see **Note 4, 11.7.1, 21.9, 23.7, and 23.10.**

1.4 Two former colorimetric test methods were discontinued. Refer to **Appendix X1** for historical information.

2. Referenced Documents

2.1 ASTM Standards:

- D 858** Test Methods for Manganese in Water ⁵
- D 1066** Practice for Sampling Steam ⁵

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 Chilton, J. M., “Simultaneous Colorimetric Determination of Copper, Cobalt, and Nickel as Diethyldithiocarbamates,” *Analytical Chemistry*, Vol 25, 1953, pp. 1274–1275.

³ Platte, J. A., and Marcy, V. M., “A New Tool for the Water Chemist,” *Industrial Water Engineering*, May 1965.

⁴ Brown, E., Skougstad, M. W., and Fishman, M. J., “Methods for Collection and Analysis of Water Samples for Dissolved Minerals and Gases,” *Techniques of Water-Resources Investigations of the U.S. Geological Survey*, Book 5, Chapter , 1970, p. 115.

⁵ *Annual Book of ASTM Standards*, Vol 11.01.

- D 1068** Test Methods for Iron in Water ⁵
- D 1129** Terminology Relating to Water ⁵
- D 1192** Specification for Equipment for Sampling Water and Steam in Closed Conduits ⁵
- D 1193** Specification for Reagent Water ⁵
- D 1687** Test Methods for Chromium in Water ⁵
- D 1688** Test Methods for Copper in Water ⁵
- D 1691** Test Methods for Zinc in Water ⁵
- D 2777** Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water ⁵
- D 3370** Practices for Sampling Water from Closed Conduits ⁵
- D 3557** Test Methods for Cadmium in Water ⁵
- D 3558** Test Methods for Cobalt in Water
- D 3559** Test Methods for Lead in Water ⁵
- D 3919** Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry ⁵
- D 4841** Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents ⁵
- D 5810** Guide for Spiking into Aqueous Samples ⁵
- D 5847** Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis ⁶

3. Terminology

3.1 Definitions:

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

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *total recoverable nickel*—an arbitrary analytical term relating to the recoverable forms of nickel that are determinable by the digestion method that is included in these test methods.

4. Significance and Use

4.1 Elemental constituents in potable water, receiving water, and wastewater need to be identified for support of effective pollution control programs. Test Methods A, B, and C provide the techniques necessary to make such measurements.

4.2 Nickel is considered to be relatively nontoxic to man and a limit for nickel is not included in the EPA National

⁶ *Annual Book of ASTM Standards*, Vol 11.02.

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

Interim Primary Drinking Water Regulations.⁷ The toxicity of nickel to aquatic life indicates tolerances that vary widely and that are influenced by species, pH, synergistic effects, and other factors.

4.3 Nickel is a silver-white metallic element seldom occurring in nature in the elemental form. Nickel salts are soluble and can occur as a leachate from nickel-bearing ores. Nickel salts are used in metal-plating and may be discharged to surface or ground waters.

5. Purity of Reagents

5.1 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 where such specifications are available.⁸ 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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification **D 1193**, 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 lessening the bias and precision of the determination. Type II water was specified at the time of the round-robin testing of this test method.

6. Sampling

6.1 Collect the sample in accordance with Practice **D 1066**, Specification **D 1192**, or Practices **D 3370**, as applicable.

6.2 Samples shall be preserved with HNO₃(sp gr 1.42) to a pH of 2 or less immediately at the time of collection, normally about 2 mL/L. If only dissolved nickel is to be determined, the sample shall be filtered through a 0.45- μ m membrane filter before acidification. The holding time for samples may be calculated in accordance with Practice **D 4841**.

TEST METHOD A—ATOMIC ABSORPTION, DIRECT

7. Scope

7.1 This test method covers the determination of dissolved and total recoverable nickel and has been used successfully with reagent water, tap water, river water, lake water, ground water, a refinery effluent, and a wastewater.

7.2 This test method is applicable in the range from 0.1 to 10 mg/L of nickel. The range may be extended upward by dilution of the sample.

⁷ EPA Publication No. EPA-570/9-76-003 was originally published in 1976, and amended in 1980. Contact the Environmental Protection Agency, 401 "M" ST., S.W., Washington, DC 20406 for availability.

⁸ *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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

8. Summary of Test Method

8.1 Nickel is determined by atomic absorption spectrophotometry. Dissolved nickel is determined by aspirating the filtered sample directly with no pretreatment. Total recoverable nickel is determined by aspirating the sample following hydrochloric-nitric acid digestion and filtration. The same digestion procedure is used for cadmium (Test Methods **D 3557**), chromium (Test Methods **D 1687**), cobalt (Test Methods **D 3558**), copper (Test Methods **D 1688**), iron (Test Methods **D 1068**), lead (Test Methods **D 3559**), manganese (Test Methods **D 858**), and zinc (Test Methods **D 1691**).

9. Interferences

9.1 Sodium, potassium, sulfate, and chloride (9000 mg/L each), calcium, magnesium and iron (4000 mg/L each), nitrate (2000 mg/L), and cadmium, lead, copper, zinc, cobalt, and chromium (10 mg/L each) do not interfere.

NOTE 1—Background correction by techniques such as a continuum source, nonabsorbing lines, or chelation-extraction, may be necessary for low levels of nickel for some types of water. Instrument manufacturer's instructions for use of the specific correction technique should be followed.

10. Apparatus

10.1 *Atomic Absorption Spectrophotometer*, for use at 232.0 nm.

NOTE 2—The manufacturer's instructions should be followed for all instrumental parameters. Wavelengths other than 232.0 nm may be used if they have been determined to be equally suitable.

10.2 *Nickel Hollow-Cathode Lamp*—Multielement hollow-cathode lamps are available and also have been found satisfactory.

10.3 *Pressure-Reducing Valves*—The supplies of fuel and oxidant shall be maintained at pressures somewhat higher than the controlled operating pressure of the instrument by suitable valves.

11. Reagents and Materials

11.1 *Hydrochloric Acid (sp gr 1.19)*—Concentrated hydrochloric acid (HCl).

NOTE 3—If a high reagent blank is obtained, distill the HCl or use a spectrograde acid.

NOTE 4—When HCl is distilled, an azeotropic mixture is formed (~6 N HCl). Therefore, whenever concentrated HCl is used in the preparation of reagents or in the procedure, use twice the volume of the distilled HCl.

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

NOTE 5—If a high reagent blank is obtained, distill the HNO₃ or use a spectrograde acid.

11.3 *Nitric Acid (1 + 499)*—Add 1 volume HNO₃(sp gr 1.42) to 499 volumes of water.

11.4 *Nickel Solution, Stock (1.0 mL = 1.0 mg Ni)*—Dissolve 4.953 g of nickelous nitrate [Ni(NO₃)₂·6H₂O] in a mixture of 10 mL of HNO₃(sp gr 1.42) and 100 mL of water. Dilute to 1 L with water.

11.5 *Nickel Solution, Standard (1 mL = 0.1 mg Ni)*—Dilute 100.0 mL of the stock nickel solution and 1 mL of HNO₃ to 1 L with water.

11.6 Oxidant:

11.6.1 *Air*, which has been passed through a suitable filter to remove oil, water, and other foreign substances is the usual oxidant.

11.7 Fuel:

11.7.1 *Acetylene*—Standard, commercially available acetylene is the usual fuel. Acetone, always present in acetylene cylinders, can affect analytical results. The cylinder should be replaced at 50 psig (345 kPa). (**Warning**—“Purified” grade acetylene containing a special proprietary solvent rather than acetone should not be used with poly(vinyl chloride) tubing as weakening of the walls can cause a potentially hazardous situation.)

12. Standardization

12.1 Prepare a blank and at least four standard solutions to bracket the expected nickel concentration range of the samples to be analyzed by diluting the standard nickel solution with HNO₃(1 + 499) as described in 11.5. Prepare the standards (100 mL) each time the test is to be performed.

12.2 For total recoverable nickel add 0.5 mL of HNO₃(sp gr 1.42) and proceed as directed in 13.2 through 13.4. For dissolved nickel proceed with 13.5.

12.3 Atomize the blank and standards and record the instrument readings. Atomize HNO₃(1 + 499) between each standard.

12.4 Prepare an analytical curve by plotting the absorbance versus the concentration for each standard on linear graph paper. Alternatively, read directly in concentration if this capability is provided with the instrument.

13. Procedure

13.1 Measure 100.0 mL of a well-mixed acidified sample into a 125-mL beaker or flask.

NOTE 6—If only dissolved nickel is to be determined, start with 13.5.

13.2 Add 5 mL of HCl (sp gr 1.19) to each sample.

13.3 Heat the samples on a steam bath or hotplate in a well-ventilated fume hood until the volume has been reduced to 15 to 20 mL, making certain that the samples do not boil.

NOTE 7—For samples with high levels of suspended matter or dissolved solids, the amount of reduction in volume is left to the discretion of the analyst.

13.4 Cool and filter the samples through a suitable filter, such as fine-textured, acid-washed, ashless paper, into 100-mL volumetric flasks. Wash the filter paper two or three times with water and bring filtrate to volume.

13.5 Atomize each filtered and acidified sample and determine its absorbance or concentration. Atomize HNO₃(1 + 499) between samples.

14. Calculation

14.1 Calculate the concentration of nickel in each sample, in milligrams per litre, using 12.4.

TABLE 1 Precision and Concentration, Direct Aspiration (Test Method A)

<i>Reagent Water:</i>			
Concentration (\bar{X}), mg/L	7.74	0.84	3.93
S_T	0.502	0.102	0.383
S_O	0.261	0.045	0.324
<i>Natural Water:</i>			
Concentration (\bar{X}), mg/L	7.74	0.84	3.87
S_T	0.629	0.108	0.401
S_O	0.420	0.067	0.192

15. Precision and Bias ⁹

15.1 The precision of this test method was tested by eleven laboratories in reagent water, natural waters, a refinery effluent and in a wastewater. Five laboratories reported data for two operators. The precision of this test method is shown in Table 1; the bias is shown in Table 2.

15.2 It is the user’s responsibility to ensure the validity of this test method for waters of untested matrices.

15.3 This section on precision and bias conforms to Practice D 2777 – 77 which was in place at the time of collaborative testing. Under the allowances made in 1.4 of D 2777 – 98, these precision and bias data do meet existing requirements of interlaboratory studies of Committee D19 test methods.

16. Quality Control

16.1 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 nickel.

16.2 Calibration and Calibration Verification

16.2.1 Analyze at least three working standards containing concentrations of nickel that bracket the expected sample concentration, prior to analysis of samples, to calibrate the instrument. The calibration correlation coefficient shall be equal to or greater than 0.990. In addition to the initial calibration blank, a calibration blank shall be analyzed at the end of the batch run to ensure contamination was not a problem during the batch analysis.

16.2.2 Verify instrument calibration after standardization by analyzing a standard at the concentration of one of the calibration standards. The concentration of a mid-range standard should fall within $\pm 15\%$ of the known concentration.

16.2.3 If calibration cannot be verified, recalibrate the instrument.

16.3 Initial Demonstration of Laboratory Capability

16.3.1 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, and so forth, a precision and bias study must be performed to demonstrate laboratory capability.

16.3.2 Analyze seven replicates of a standard solution prepared from an Independent Reference Material containing a midrange concentration of nickel. 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

⁹ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D19-1038.