**Designation: D1886 − 14**

# Standard Test Methods for Nickel in Water[1]

This standard is issued under the fixed designation D1886; 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 [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 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversion to inch-pound units that are provided for information only and are not considered standard.

1.4 *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 5,11.8.1, 21.11, 23.7, and 23.10.

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

## 2. Referenced Documents

2.1 *ASTM Standards:*[5]

D858 Test Methods for Manganese in Water
D1066 Practice for Sampling Steam
D1068 Test Methods for Iron in Water
D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D1687 Test Methods for Chromium in Water
D1688 Test Methods for Copper in Water
D1691 Test Methods for Zinc in Water
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
D3557 Test Methods for Cadmium in Water
D3558 Test Methods for Cobalt in Water
D3559 Test Methods for Lead in Water
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 these test methods, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *total recoverable nickel, n*—a descriptive term relating to the nickel forms recovered in the acid-digestion procedure specified in these test methods.

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[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, 1970, p. 115.

[5] 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**

## 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 Interim Primary Drinking Water Regulations.[6] 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.[7] 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 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 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 D1066 or Practices D3370, as applicable.

6.2 Samples shall be preserved with $HNO_3$ (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-µm membrane filter before acidification. The holding time for samples may be calculated in accordance with Practice D4841.

NOTE 1—Alternatively, the pH may be adjusted in the laboratory if the sample is returned within 14 days. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. This could reduce hazards of working with acids in the field when appropriate.

## 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.

## 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 D3557), chromium (Test Methods D1687), cobalt (Test Methods D3558), copper (Test Methods D1688), iron (Test Methods D1068), lead (Test Methods D3559), manganese (Test Methods D858), and zinc (Test Methods D1691).

## 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 2—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 3—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 *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.

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[6] 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.

[7] *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.

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

NOTE 4—If a high reagent blank is obtained, distill the HCl or use a spectrograde acid.

NOTE 5—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.

11.3 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid ($HNO_3$).

NOTE 6—If a high reagent blank is obtained, distill the $HNO_3$ or use a spectrograde acid.

11.4 *Nitric Acid* (1 + 499)—Add 1 volume $HNO_3$ (sp gr 1.42) to 499 volumes of water.

11.5 *Nickel Solution, Stock* (1.0 mL = 1.0 mg Ni)—Commercially purchase or dissolve 4.953 g of nickelous nitrate [$Ni(NO_3)_2 \cdot 6H_2O$] in a mixture of 10 mL of $HNO_3$ (sp gr 1.42) and 100 mL of water. Dilute to 1 L with water. A purchased nickel stock solution of appropriate known purity is acceptable.

11.6 *Nickel Solution, Standard* (1 mL = 0.1 mg Ni)—Dilute 100.0 mL of the stock nickel solution and 1 mL of $HNO_3$ to 1 L with water.

11.7 *Oxidant:*

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

11.8 *Fuel:*

11.8.1 *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 345 kPa (50 psig). (**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 $HNO_3$ (1 + 499) as described in 11.6. Prepare the standards (100 mL) each time the test is to be performed or as determined by Practice D4841.

12.2 For total recoverable nickel add 0.5 mL of $HNO_3$ (sp gr 1.42) and proceed as directed in 13.2 through 13.4. For dissolved nickel proceed with 13.5.

12.3 Analyze at least four working standards containing concentrations of nickel that bracket the expected sample concentration, prior to analysis of samples, to calibrate the instrument. Atomize the blank and standards and record the instrument readings. Atomize $HNO_3$ (1 + 499) between each standard.

12.4 Read directly in concentration if this capability is provided with the instrument or prepare an analytical curve by plotting the absorbance versus the concentration for each standard on linear graph paper or use a computer.

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

| | | | |
|---|---|---|---|
| *Reagent Water:* | | | |
| 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 |
| *Natural Water:* | | | |
| 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 |

## 13. Procedure

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

NOTE 7—If only dissolved nickel is to be determined, start with 13.5.

13.2 Add 5 mL of HCl (sp gr 1.19) (11.2) 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 8—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.

NOTE 9—Many laboratories have found block digestion systems a useful way to digest samples for trace metals analysis. Systems typically consist of either a metal or graphite block with wells to hold digestion tubes. The block temperature controller must be able to maintain uniformity of temperature (65°C to 85°C) across all positions of the block. For trace metals analysis, the digestion tubes should be constructed of polypropylene and have a volume accuracy of at least 0.5 %. All lots of tubes should come with a certificate of analysis to demonstrate suitability for their intended purpose.

13.4 Cool and filter the samples through a suitable filter (11.1), 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 $HNO_3$ (1 + 499) between samples.

## 14. Calculation

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

## 15. Precision and Bias[8]

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 D2777 – 77 which was in place at the time of collaborative testing. Under the allowances made in 1.4 of Practice

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[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1038. Contact ASTM Customer Service at service@astm.org.

**TABLE 2 Determination of Bias, Direct Aspiration (Test Method A)**

| Amount Added, mg/L | Amount Found, mg/L | Bias, mg/L | Bias, % | Statistically Significant (95 % Confidence Level) |
|---|---|---|---|---|
| *Reagent Water:* | | | | |
| 8.0 | 7.74 | −0.26 | −3 | yes |
| 0.8 | 0.84 | +0.04 | +5 | yes |
| 4.0 | 3.93 | −0.07 | −2 | no |
| *Natural Water:* | | | | |
| 8.0 | 7.74 | −0.26 | −3 | yes |
| 0.8 | 0.84 | +0.04 | +5 | yes |
| 4.0 | 3.87 | −0.13 | −3 | yes |

D2777 – 13, 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 four working standards containing concentrations of nickel that bracket the expected sample concentration, prior to analysis of samples, to calibrate the instrument (see 12.3). The calibration correlation coefficient shall be equal to or greater than 0.990.

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 ±15 % of the known concentration. Analyze a calibration blank to verify system cleanliness.

16.2.3 If calibration cannot be verified, recalibrate the instrument.

16.2.4 It is recommended to analyze a continuing calibration blank (CCB) and continuing calibration verification (CCV) at a 10 % frequency. The results should fall within the expected precision of the method or ±15 % of the known concentration.

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 complete analytical test method including any sample preservation and pretreatment steps.

16.3.3 Calculate the mean and standard deviation of the seven values and compare to the acceptable ranges of bias in Table 2. This study should be repeated until the recoveries are within the limits given in Table 1. If a concentration other than the recommended concentration is used, refer to Practice D5847 for information on applying the *F* test and *t* test in evaluating the acceptability of the mean and standard deviation.

16.4 *Laboratory Control Sample (LCS):*

16.4.1 To ensure that the test method is in control, prepare and analyze a LCS containing a known concentration of nickel with each batch (laboratory-defined or twenty samples). The laboratory control samples for a large batch should cover the analytical range when possible. The LCS must be taken through all of the steps of the analytical method including sample preservation and pretreatment. The result obtained for a mid-range LCS shall fall within ±15 % of the known concentration.

16.4.2 If the result is not within these limits, analysis of samples is halted until the problem is corrected, and either all the samples in the batch must be reanalyzed, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

16.5 *Method Blank:*

16.5.1 Analyze a reagent water test blank with each laboratory-defined batch. The concentration of nickel found in the blank should be less than 0.5 times the lowest calibration standard. If the concentration of nickel is found above this level, analysis of samples is halted until the contamination is eliminated, and a blank shows no contamination at or above this level, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

16.6 *Matrix Spike (MS):*

16.6.1 To check for interferences in the specific matrix being tested, perform a MS on at least one sample from each laboratory-defined batch by spiking an aliquot of the sample with a known concentration of nickel and taking it through the analytical method.

16.6.2 The spike concentration plus the background concentration of nickel must not exceed the high calibration standard. The spike must produce a concentration in the spiked sample that is 2 to 5 times the analyte concentration in the unspiked sample, or 10 to 50 times the detection limit of the test method, whichever is greater.

16.6.3 Calculate the percent recovery of the spike (P) using the following formula:

$$P = 100[A(V_s + V) - BV_s]/CV$$

where:

$A$ = analyte known concentration (mg/L) in spiked sample,

$B$ = analyte known concentration (mg/L) in unspiked sample,

$C$ = known concentration (mg/L) of analyte in spiking solution,

$V_s$ = volume (mL) of sample used, and

$V$ = volume (mL) of spiking solution added.

16.6.4 The percent recovery of the spike shall fall within the limits, based on the analyte concentration, listed in Test Method D5810, Table 1. If the percent recovery is not within these limits, a matrix interference may be present in the sample selected for spiking. Under these circumstances, one of the following remedies must be employed: the matrix interference must be removed, all samples in the batch must be analyzed by a test method not affected by the matrix interference, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

NOTE 10—Acceptable spike recoveries are dependent on the concentration of the component of interest. See Test Method D5810 for additional information.

16.7 *Duplicate:*

16.7.1 To check the precision of sample analyses, analyze a sample in duplicate with each laboratory-defined batch. If the concentration of the analyte is less than five times the detection limit for the analyte, a matrix spike duplicate (MSD) should be used.

16.7.2 Calculate the standard deviation of the duplicate values and compare to the precision in the collaborative study using an $F$ test. Refer to 6.4.4 of Practice D5847 for information on applying the $F$ test.

16.7.3 If the result exceeds the precision limit, the batch must be reanalyzed or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

16.8 *Independent Reference Material (IRM):*

16.8.1 In order to verify the quantitative value produced by the test method, analyze an Independent Reference Material (IRM) submitted as a regular sample (if practical) to the laboratory at least once per quarter. The concentration of the IRM should be in the concentration mid-range for the method chosen. The value obtained must fall within the control limits established by the laboratory.

## TEST METHOD B—ATOMIC ABSORPTION, CHELATION-EXTRACTION

### 17. Scope

17.1 This test method covers the determination of dissolved and total recoverable nickel and has been used successfully with reagent water, tap water, river water, artificial seawater and a synthetic (NaCl) brine.

17.2 This test method is applicable in the range from 10 to 1000µ g/L of nickel. The range may be extended upward by dilution of the sample.

### 18. Summary of Test Method

18.1 Nickel is determined by atomic absorption spectrophotometry. The element, either dissolved or total recoverable, is chelated with pyrrolidine dithiocarbamic acid and extracted with chloroform. The extract is evaporated to dryness, treated with hot nitric acid to destroy organic matter, dissolved in hydrochloric acid, and diluted to a specified volume with water. The resulting solution is then aspirated into the air-acetylene flame of the spectrophotometer. The digestion procedure summarized in 8.1 is used for total recoverable nickel. The same chelation-extraction procedure is used for cadmium (Test Methods D3557), cobalt (Test Methods D3558), copper (Test Methods D1688), iron (Test Methods D1068), lead (Test Methods D3559), and zinc (Test Methods D1691).

### 19. Interferences

19.1 See 9.1.

### 20. Apparatus

20.1 All apparatus described in Section 10 are required.

### 21. Reagents and Materials

21.1 *Bromphenol Blue Indicator Solution* (1 g/L)—Dissolve 0.1 g of bromphenol blue in 100 mL of 50 % ethanol or isopropanol.

21.2 *Carbon Disulfide* ($CS_2$).

21.3 *Chloroform* ($CHCl_3$).

21.4 *Filter Paper*—See 11.1.

21.5 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl) (see Note 4 and Note 5).

21.6 *Hydrochloric Acid* (1 + 2)—Add 1 volume of hydrochloric acid (HCl), sp gr 1.19, to 2 volumes of water.

21.7 *Hydrochloric Acid* (1 + 49)—Add 1 volume of hydrochloric acid (HCl), sp gr 1.19, to 49 volumes of water.

21.8 *Nickel Solution, Stock* (1.0 mL = 200 µg Ni)—Commercially purchase or dissolve 0.9906 g of nickelous nitrate ($Ni(NO_3)_2 \cdot 6H_2O$) in water containing 1 mL of $HNO_3$ (sp gr 1.42) and dilute to 1 L with water. A purchased nickel stock solution of appropriate known purity is also acceptable.

21.9 *Nickel Solution, Standard* (1.0 mL = 2.0 µg Ni)—Dilute 10.0 mL of nickel solution, stock and 1 mL of $HNO_3$ (sp gr 1.42) to 1 L with water. This standard is used to prepare working standards at the time of analysis.

21.10 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid ($HNO_3$) (see Note 6).

21.11 *Pyrrolidine Dithiocarbamic Acid-Chloroform Reagent*—Add 36 mL of pyrrolidine to 1 L of $CHCl_3$. Cool the solution and add 30 mL of $CS_2$ (21.2) in small portions, swirling between additions. Dilute to 2 L with $CHCl_3$. The reagent can be used for several months if stored in a cool, dark place. (**Warning—**All components of this reagent are highly toxic. Carbon disulfide is also highly flammable. Prepare and use in a well-ventilated hood. Avoid inhalation and direct contact.)

21.12 *Sodium Hydroxide Solution* (100 g/L)—Dissolve 100 g of sodium hydroxide (NaOH) in water and dilute to 1 L. (**Warning—**This is a very exothermic reaction.)

21.13 *Oxidant*—See 11.7.

21.14 *Fuel*—See 11.8.

### 22. Standardization

22.1 Prepare a blank and sufficient standards containing from 0.0 to 100 µg of nickel by diluting 0.0 to 50.0-mL portions of nickel standard solution to 100 mL with water (21.9). 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.

22.2 For total recoverable nickel use 125-mL beakers or flasks, add 0.5 mL of $HNO_3$ (sp gr 1.42) (21.10) and proceed as directed in 23.2 through 23.15. For dissolved nickel use 250-mL separatory funnels and proceed as directed in 23.5 through 23.15. All samples and standards should be treated the same.

22.3 Read directly in concentration if this capability is provided with the instrument or construct an analytical curve by plotting the absorbances of standards versus micrograms of nickel or prepare a linear plot.

## 23. Procedure

23.1 Measure a volume of a well-mixed acidified sample containing less than 100 µg of nickel (100-mL maximum) into a 125-mL beaker or flask and adjust the volume to 100 mL with water.

NOTE 11—If only dissolved nickel is to be determined measure a volume of filtered and acidified sample containing less than 10.0 µg of nickel (100-mL maximum) into a 250-mL separatory funnel, and start with 23.5.

23.2 Add 5 mL of HCl (sp gr 1.19) (21.5) to each sample.

23.3 Heat the samples on a steam bath or hotplate until the volume has been reduced to 15 to 20 mL, making certain that the samples do not boil. See Note 9.

NOTE 12—For brines and samples with high levels of suspended matter, the amount of reduction in volume is left to the discretion of the analyst.

23.4 Cool and filter the samples through a suitable filter (21.4), such as fine-textured, acid-washed, ashless paper into 250-mL separatory funnels. Wash the filter paper two or three times with water and bring to about 100 mL of volume.

23.5 Add 2 drops of bromphenol blue indicator solution (21.1) and mix.

23.6 Adjust the pH by addition of NaOH (100 g/L) solution (21.12) until a blue color persists. Add HCl (1 + 49) (21.7) by drops until the blue color just disappears; then add 2.5 mL of HCl (1 + 49) in excess. The pH at this point should be 2.3.

NOTE 13—The pH adjustment in 23.6 may be made with a pH meter instead of using an indicator.

23.7 Add 10 mL of pyrrolidine dithiocarbamic acid-chloroform reagent (21.11) and shake vigorously for 2 min. (**Warning—**All components of this reagent are highly toxic. Carbon disulfide is also highly flammable. Prepare and use in a well-ventilated hood. Avoid inhalation and direct contact.)

23.8 Plug the tip of the separatory funnel with cotton, allow the phases to separate, and drain the $CHCl_3$ phase into a 100-mL beaker.

23.9 Repeat the extraction with 10 mL of $CHCl_3$ (21.3) and drain the $CHCl_3$ layer into the same beaker.

NOTE 14—If color still remains in the $CHCl_3$ extract, reextract the aqueous phase until the $CHCl_3$ layer is colorless.

**TABLE 3 Precision and Concentration, Chelation–Extraction (Test Method B)**

| | | | |
|---|---|---|---|
| *Reagent Water:* | | | |
| Concentration (*X*), µg/L | 784.2 | 56.8 | 398.5 |
| $S_T$ | 146.4 | 15.2 | 35.1 |
| $S_O$ | 41.02 | 9.18 | 22.97 |
| *Natural Water:* | | | |
| Concentration (*X*), µg/L | 794.4 | 53.6 | 399.4 |
| $S_T$ | 133.9 | 11.2 | 37.7 |
| $S_O$ | 70.9 | 12.6 | 11.1 |

23.10 Place the beaker on a hotplate set at low heat or on a steam bath, and evaporate to near dryness. Remove beaker from heat and allow residual solvent to evaporate without further heating. (**Warning—**Perform in a well-ventilated hood.) See Note 9.

23.11 Hold the beaker at a 45° angle and slowly add dropwise 2 mL of $HNO_3$ (sp gr 1.42) (21.10), rotating the beaker to effect thorough contact of the acid with the residue. (**Warning—**If acid is added to the beaker in a vertical position, a violent reaction will occur accompanied by high heat and spattering.)

23.12 Place the beaker on a hotplate set at low heat in a well-ventilated hood, or on a steam bath, and evaporate to near dryness. Remove beaker from heat and allow residual solvent to evaporate without further heating. See Note 9.

23.13 Add 2 mL of HCl (1 + 2) (21.6) to the beaker, and heat, while swirling for 1 min.

NOTE 15—If a precipitate appears when the hydrochloric acid (1 + 2) is added to the dried residue, obtain a fresh supply of pyrrolidine which has a different lot number or redistill the pyrrolidine just before preparing the pyrrolidine dithiocarbamic acid-chloroform reagent (21.11) and repeat the procedure with a different aliquot of the sample.

23.14 Cool and quantitatively transfer the solution to a 10-mL volumetric flask and bring to volume with water.

23.15 Aspirate each sample and record the scale reading or concentration.

## 24. Calculation

24.1 Determine the weight of nickel in each sample by referring to 22.3. Calculate the concentration of nickel in micrograms per litre as follows:

$$\text{Nickel, } \mu\text{g/L} = (1000/A) \times B$$

where:

*1000* = 1000 mL / litre

*A* = volume of original sample, mL, and

*B* = weight of nickel sample, µg.

## 25. Precision and Bias[5]

25.1 The precision of this test method was tested by six laboratories in reagent water, natural waters and in synthetic brines. One laboratory reported data from two operators. The precision of this test method is shown in Table 3; the bias is shown in Table 4.

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

TABLE 4 Determination of Bias, Chelation-Extraction (Test Method B)

| Amount Added, µg/L | Amount Found, µg/L | Bias, µg/L | Bias, % | Statistically Significant (95 % Confidence Level) |
|---|---|---|---|---|
| *Reagent Water:* | | | | |
| 900 | 784.2 | −115.8 | −12.9 | yes |
| 50 | 56.8 | + 6.8 | + 13.6 | no |
| 400 | 398.5 | −1.5 | −0.4 | no |
| *Natural Water:* | | | | |
| 900 | 794.4 | −105.6 | −11.7 | yes |
| 50 | 53.6 | + 3.6 | + 7.2 | no |
| 400 | 399.4 | −0.6 | −0.2 | no |

25.3 This section on precision and bias conforms to Practice D2777 – 77 which was in place at the time of collaborative testing. Under the allowances made in 1.4 of Practice D2777 – 13, these precision and bias data do meet existing requirements of interlaboratory studies of Committee D19 test methods.

## 26. Quality Control

26.1 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.

26.2 *Calibration and Calibration Verification:*

26.2.1 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 (22.1). The calibration correlation coefficient shall be equal to or greater than 0.990.

26.2.2 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 ±15 % of the known concentration. Analyze a calibration blank to verify cleanliness.

26.2.3 If calibration cannot be verified, recalibrate the instrument.

26.2.4 It is recommended to analyze a continuing calibration blank (CCB) and continuing calibration verification (CCV) at a 10 % frequency. The results should fall within the expected precision of the method or ±15 % of the known concentration.

26.3 *Initial Demonstration of Laboratory Capability:*

26.3.1 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.

26.3.2 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 complete analytical test method including any sample preservation and pretreatment steps.

26.3.3 Calculate the mean and standard deviation of the seven values and compare to the acceptable ranges of bias in Table 4. This study should be repeated until the recoveries are within the limits given in Table 3. If a concentration other than the recommended concentration is used, refer to Practice D5847 for information on applying the F test and t test in evaluating the acceptability of the mean and standard deviation.

26.4 *Laboratory Control Sample (LCS):*

26.4.1 To ensure that the test method is in control, prepare and analyze a LCS containing a known concentration of nickel with each batch (laboratory-defined or twenty samples). The laboratory control samples for a large batch should cover the analytical range when possible. The LCS must be taken through all of the steps of the analytical method including sample preservation and pretreatment. The result obtained for a mid-range LCS shall fall within ±15 % of the known concentration.

26.4.2 If the result is not within these limits, analysis of samples is halted until the problem is corrected, and either all the samples in the batch must be reanalyzed, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

26.5 *Method Blank:*

26.5.1 Analyze a reagent water test blank with each laboratory-defined batch. The concentration of nickel found in the blank should be less than 0.5 times the lowest calibration standard. If the concentration of nickel is found above this level, analysis of samples is halted until the contamination is eliminated, and a blank shows no contamination at or above this level, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

26.6 *Matrix Spike (MS):*

26.6.1 To check for interferences in the specific matrix being tested, perform a MS on at least one sample from each laboratory-defined batch by spiking an aliquot of the sample with a known concentration of nickel and taking it through the analytical method.

26.6.2 The spike concentration plus the background concentration of nickel must not exceed the high calibration standard. The spike must produce a concentration in the spiked sample that is 2 to 5 times the analyte concentration in the unspiked sample, or 10 to 50 times the detection limit of the test method, whichever is greater.

26.6.3 Calculate the percent recovery of the spike (P) using the following formula:

$$P = 100[A(V_s + V) - BV_s]/CV$$

where:

$A$ = analyte known concentration (µg/L) in spiked sample,
$B$ = analyte known concentration (µg/L) in unspiked sample,
$C$ = known concentration (µg/L) of analyte in spiking solution,
$V_s$ = volume (mL) of sample used, and
$V$ = volume (mL) of spiking solution added.

26.6.4 The percent recovery of the spike shall fall within the limits, based on the analyte concentration, listed in Test Method D5810, Table 1. If the percent recovery is not within these limits, a matrix interference may be present in the sample selected for spiking. Under these circumstances, one of the following remedies must be employed: the matrix interference must be removed, all samples in the batch must be analyzed by a test method not affected by the matrix interference, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

NOTE 16—Acceptable spike recoveries are dependent on the concentration of the component of interest. See Test Method D5810 for additional information.

26.7 *Duplicate:*

26.7.1 To check the precision of sample analyses, analyze a sample in duplicate with each laboratory-defined batch. If the concentration of the analyte is less than five times the detection limit for the analyte, a matrix spike duplicate (MSD) should be used.

26.7.2 Calculate the standard deviation of the duplicate values and compare to the precision in the collaborative study using an F test. Refer to 6.4.4 of Practice D5847 for information on applying the F test.

26.7.3 If the result exceeds the precision limit, the batch must be reanalyzed or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

26.8 *Independent Reference Material (IRM):*

26.8.1 In order to verify the quantitative value produced by the test method, analyze an Independent Reference Material (IRM) submitted as a regular sample (if practical) to the laboratory at least once per quarter. The concentration of the IRM should be in the concentration mid-range for the method chosen. The value obtained must fall within the control limits established by the laboratory.

## TEST METHOD C—ATOMIC ABSORPTION, GRAPHITE FURNACE

### 27. Scope

27.1 This test method covers the determination of dissolved and total recoverable nickel and has been used successfully with reagent grade water, lake water, river water, well and filtered tap water, and a condensate from a medium Btu coal gasification process. It is the user's responsibility to ensure the validity of this test method to other matrices.

27.2 This test method is applicable in the range from approximately 5 to 100 µg/L nickel using a 20-µL injection. The range can be increased or decreased by varying the volume of sample injected or the instrumental settings. High concentrations may be diluted but preferably should be analyzed by direct aspiration atomic absorption spectrophotometry (see Test Method A). ICP-MS may also be appropriate but at a higher instrument cost. See Test Method D5673.

27.3 The analyst is encouraged to consult Practice D3919 for a general discussion of interferences and sample analysis procedures for graphite furnace atomic absorption spectrophotometry.

### 28. Summary of Test Method

28.1 Nickel is determined by an atomic absorption spectrophotometer used in conjunction with a graphite furnace. A sample is placed in a graphite tube, evaporated to dryness, charred (pyrolyzed or ashed) and atomized. The absorption signal generated during atomization is recorded and compared to standards. A general guide for the application of the graphite furnace is given in Practice D3919.

28.2 Dissolved nickel is determined on a filtered sample with no pretreatment.

28.3 Total recoverable nickel is determined following acid digestion and filtration. Because chlorides interfere with furnace procedures for some metals, the use of hydrochloric acid in any digestion or solubilization step is to be avoided. If suspended material is not present, this digestion and filtration may be omitted.

### 29. Interferences

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

29.2 The following interferences have been reported at the indicated levels:

| Nickel, µg/L | Concomitant Added, mg/L | % Error |
|---|---|---|
| 42 | 10 Fe (Cl) | +23.8 |
| 42 | 100 Fe (Cl) | +66.7 |
| 72 | 10 Cu (0) | +12.5 |
| 72 | 100 Cu (0) | +13.9 |

### 30. Apparatus

30.1 *Atomic Absorption Spectrophotometer,* for use at 232.0 nm with background correction.

NOTE 17—A wavelength other than 232.0 nm may be used if it has been determined to be suitable. Greater linearity may be obtained at high concentrations by using a less sensitive wavelength.

NOTE 18—The manufacturer's instructions should be followed for all instrumental parameters.

30.2 *Nickel Hollow-Cathode Lamp,* a single-element lamp is preferred, but multielement lamps may be used.

30.3 *Graphite Furnace,* capable of reaching temperatures sufficient to atomize the element of interest.

30.4 *Graphite Tubes,* compatible with furnace device. Pyrolytically coated graphite tubes are recommended to eliminate the possible formation of carbides.

30.5 *Pipettes,* microlitre with disposable tips. Sizes may range from 1 to 100 µL, as required.

30.6 *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.

30.7 *Automatic Sampling,* recommended if available.

### 31. Reagents and Materials

31.1 *Filter Paper*—See 11.1.

31.2 *Nickel Solution, Standard* (1.0 mL = 1.0 µg Ni)—Dilute 5.0 mL of nickel solution, stock (see 31.3) and 1 mL of $HNO_3$ (sp gr 1.42) to 1 L with water. This standard is used to prepare working standards at the time of the analysis. A purchased nickel stock solution of appropriate known purity is also acceptable.

31.3 *Nickel Solution, Stock* (1.0 mL = 200 µg Ni)—(See 21.8.)

31.4 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid ($HNO_3$) (see Note 6).

31.5 *Argon,* standard, welders grade, commercially available. Nitrogen may also be used if recommended by the instrument manufacturer.

## 32. Standardization

32.1 Initially, set the instrument according to the manufacturer's specifications. Follow the general instructions as provided in Practice D3919.

## 33. Procedure

33.1 Clean all glassware to be used for preparation of standard solutions or in the solubilization step, or both, by rinsing first with $HNO_3$ (1 + 1) and then with water.

33.2 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. Measure 100.0 mL of each standard and well-mixed sample into 125-mL beakers or flasks. For total recoverable nickel add $HNO_3$ (sp gr 1.42) to each standard and sample at a rate of 5 mL/L and proceed as directed in 33.4 through 33.6.

33.3 If only dissolved nickel is to be determined, filter the sample through a 0.45-µm membrane filter prior to acidification and proceed to 33.6.

33.4 Heat the samples at 95°C 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 (see Note 8). (See Note 9.)

33.5 Cool and filter the sample through a suitable filter (31.1) (such as fine-textured, acid-washed, ashless paper) into a 100-mL volumetric flask. Wash the filter paper two or three times with water and bring to volume (see Note 19). The acid concentration at this point should be 0.5 % $HNO_3$.

NOTE 19—If suspended material is not present, this filtration may be omitted, but the sample must still be diluted to 100 mL.

33.6 Inject a measured aliquot of the sample into the furnace device following the directions as provided by the particular instrument manufacturer. Refer to Practice D3919.

## 34. Calculation

34.1 Determine the concentration of nickel in each sample by referring to Practice D3919.

**TABLE 5 Determination of Bias and Overall Precision in Reagent Water (Test Method C)**

| Amount Added, µg/L | Amount Found, µg/L | $S_T$ | Bias, µg/L | Bias, % | Statistically Significant |
|---|---|---|---|---|---|
| 80 | 87.0 | 12.97 | + 7.0 | + 8.8 | no |
| 8.0 | 9.37 | 2.11 | + 1.37 | + 17.1 | yes |
| 30 | 31.6 | 5.49 | + 1.6 | + 5.3 | no |

**TABLE 6 Determination of Bias and Overall Precision in Water of Choice (Test Method C)**

| Amount Added, µg/L | Amount Found, µg/L | $S_T$ | Bias, µg/L | Bias, % | Statistically Significant |
|---|---|---|---|---|---|
| 80 | 81.0 | 9.42 | +1.0 | +1.3 | no |
| 8.0 | 7.58 | 1.54 | −0.42 | −5.3 | no |
| 30 | 30.8 | 5.29 | +0.8 | +2.7 | no |

## 35. Precision and Bias[9]

35.1 The precision of this test method was tested by 12 laboratories in reagent water, lake water, river water, well water, filtered tap water, and condensate from a medium Btu coal gasification process. Two laboratories reported data from two operators. Although multiple injections may have been made, the report sheets provided allowed only for reporting single values. Thus, no single-operator precision data can be calculated. Bias data and overall precision data are given in Table 5 and Table 6.

35.2 These data may not apply to waters of other matrices, therefore, it is the responsibility of the analyst to ensure the validity of this test method for waters of untested matrices.

35.3 This section on precision and bias conforms to Practice D2777 – 77 which was in place at the time of collaborative testing. Under the allowances made in 1.4 of Practice D2777 – 13, these precision and bias data do meet existing requirements of interlaboratory studies of Committee D19 test methods.

## 36. Quality Control

36.1 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.

36.2 *Calibration and Calibration Verification:*

36.2.1 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 (33.2). The calibration correlation coefficient shall be equal to or greater than 0.990. Analyze a calibration blank to verify system cleanliness.

36.2.2 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 ±15 % of the known concentration.

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[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1107. Contact ASTM Customer Service at service@astm.org.

36.2.3 If calibration cannot be verified, recalibrate the instrument.

36.2.4 It is recommended to analyze a continuing calibration blank (CCB) and continuing calibration verification (CCV) at a 10 % frequency. The results should fall within the expected precision of the method or ±15 % of the known concentration.

36.3 *Initial Demonstration of Laboratory Capability:*

36.3.1 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.

36.3.2 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 complete analytical test method including any sample preservation and pretreatment steps.

36.3.3 Calculate the mean and standard deviation of the seven values and compare to the acceptable ranges of bias in Table 5 and Table 6. This study should be repeated until the recoveries are within the limits given in Table 5 and Table 6. If a concentration other than the recommended concentration is used, refer to Practice D5847 for information on applying the *F* test and *t* test in evaluating the acceptability of the mean and standard deviation.

36.4 *Laboratory Control Sample (LCS):*

36.4.1 To ensure that the test method is in control, prepare and analyze a LCS containing a known concentration of nickel with each batch (laboratory-defined or twenty samples). The laboratory control samples for a large batch should cover the analytical range when possible. The LCS must be taken through all of the steps of the analytical method including sample preservation and pretreatment. The result obtained for a mid-range LCS shall fall within ±15 % of the known concentration.

36.4.2 If the result is not within these limits, analysis of samples is halted until the problem is corrected, and either all the samples in the batch must be reanalyzed, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

36.5 *Method Blank:*

36.5.1 Analyze a reagent water test blank with each laboratory-defined batch. The concentration of nickel found in the blank should be less than 0.5 times the lowest calibration standard. If the concentration of nickel is found above this level, analysis of samples is halted until the contamination is eliminated, and a blank shows no contamination at or above this level, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

36.6 *Matrix Spike (MS):*

36.6.1 To check for interferences in the specific matrix being tested, perform a MS on at least one sample from each laboratory-defined batch by spiking an aliquot of the sample with a known concentration of nickel and taking it through the analytical method.

36.6.2 The spike concentration plus the background concentration of nickel must not exceed the high calibration standard. The spike must produce a concentration in the spiked sample that is 2 to 5 times the analyte concentration in the unspiked sample, or 10 to 50 times the detection limit of the test method, whichever is greater.

36.6.3 Calculate the percent recovery of the spike (*P*) using the following formula:

$$P = 100[A(V_s + V) - BV_s]/CV$$

where:

$A$ = analyte known concentration (µg/L) in spiked sample,
$B$ = analyte known concentration (µg/L) in unspiked sample,
$C$ = known concentration (µg/L) of analyte in spiking solution,
$V_s$ = volume (mL) of sample used, and
$V$ = volume (mL) of spiking solution added.

36.6.4 The percent recovery of the spike shall fall within the limits, based on the analyte concentration, listed in Test Method D5810, Table 1. If the percent recovery is not within these limits, a matrix interference may be present in the sample selected for spiking. Under these circumstances, one of the following remedies must be employed: the matrix interference must be removed, all samples in the batch must be analyzed by a test method not affected by the matrix interference, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

NOTE 20—Acceptable spike recoveries are dependent on the concentration of the component of interest. See Test Method D5810 for additional information.

36.7 *Duplicate:*

36.7.1 To check the precision of sample analyses, analyze a sample in duplicate with each laboratory-defined batch. If the concentration of the analyte is less than five times the detection limit for the analyte, a matrix spike duplicate (MSD) should be used.

36.7.2 Calculate the standard deviation of the duplicate values and compare to the precision in the collaborative study using an *F* test. Refer to 6.4.4 of Practice D5847 for information on applying the *F* test.

36.7.3 If the result exceeds the precision limit, the batch must be reanalyzed or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

36.8 *Independent Reference Material (IRM):*

36.8.1 In order to verify the quantitative value produced by the test method, analyze an Independent Reference Material (IRM) submitted as a regular sample (if practical) to the laboratory at least once per quarter. The concentration of the IRM should be in the concentration mid-range for the method chosen. The value obtained must fall within the control limits established by the laboratory.

## 37. Keywords

37.1 atomic absorption; chelation; graphite furnace; nickel; water

# APPENDIX

### (Nonmandatory Information)

## X1. RATIONALE FOR DISCONTINUATION OF TEST METHODS

X1.1 Colorimetric Test Methods (Former Test Method A—Carbamate, and Former Test Method B—Glyoxime):

X1.1.1 These test methods were discontinued in 1988. They may be found published in their entirety in the 1988 Annual Book of ASTM Standards, Vol 11.01.

X1.1.2 These test methods cover the determination of nickel in water for samples containing 0.001 to 0.05 mg/L of nickel (carbamate test method) or 0.01 to 5.0 mg/L of nickel (glyoxime test method).

X1.1.3 *Summary of Test Methods:*

X1.1.3.1 *Carbamate*—This test method is based on the formation of a yellow-green complex of nickel with sodium diethyldithiocarbamate, at a pH of 8.5 to 9.0 in the presence of citrate. The color complex is extracted with carbon tetrachloride and the color intensity measured by means of a spectrophotometer. Correction is made for copper up to 0.100 mg/L. Calculations are based on absorbance measurements at wavelengths of 328 and 436 nm.

X1.1.3.2 *Glyoxime*—This test method is based on the formation of a wine-red color complex of nickel with ammoniacal dimethylglyoxime in the presence of iodine. The color is measured directly with a filter photometer or a spectrophotometer.

X1.1.4 These test methods were discontinued because there were insufficient laboratories interested in participating in a collaborative study to obtain the necessary precision and bias data as required by Practice D2777.

## SUMMARY OF CHANGES

Committee D19 has identified the location of selected changes to this standard since the last issue (D1886 – 08) that may impact the use of this standard. (Approved Oct. 1, 2014.)

*(1)* Section 2 was updated.
*(2)* Section 3 was updated.
*(3)* Section 6 was modified to allow for pH of the samples in the laboratory.
*(4)* Sections 11, 21, and 31 were modified to allow for commercial standards, filter paper and missing reagents were added.
*(5)* Sections 12, 22, and 33 were modified with standard and calibration information.
*(6)* Reagent references were added to Sections 13, 21, 22, 23, and 33.
*(7)* Sections 13, 23, and 33 were modified to include note about the use of block digestion systems.
*(8)* 16.2.4, 16.3.2, 16.6.3, 26.2.4, 26.6.3, 36.2.4, and 36.6.3 were modified.
*(9)* Section 27 was modified to inform the user of the possibility of using an ICP-MS.