Nickel, atomic absorption spectrophotometry, graphite furnace

Parameters and Codes:
Nickel, dissolved, I-2503-89 (µg/L as Ni): 01065
Nickel, whole water recoverable, 1-4503-89 (µg/L as Ni): 01067

1. Application

1.1 This method is used to determine nickel in samples of water and water-suspended sediment with a specific conductance not greater than 10,000 µS/cm. With Zeeman background correction and a 20-µL sample, the method is applicable in the range from 1 to 25 µg/L. Sample solutions that contain nickel concentrations greater than 25 µg/L must be diluted or be analyzed by an alternate method. This method was implemented in the National Water Quality Laboratory in May 1989.

1.2 The analytical range and detection limit can be increased or possibly decreased by varying the volume of sample injected or the instrumental settings.

1.3 Whole water recoverable nickel in samples of water-suspended sediment must undergo preliminary digestion by method I-3485 before being determined.

2. Summary of method

Nickel is determined by atomic absorption spectrophotometry in conjunction with a graphite furnace containing a graphite platform (Hinderberger and others, 1981). A sample is placed on the graphite platform, and the sample then is evaporated to dryness, charred, and atomized using high-temperature ramping. The absorption signal produced during atomization is recorded and compared with standards.

3. Interferences

3.1 Interferences for samples with specific conductances less than 10,000 µS/cm normally are small. In addition, the use of the graphite platform reduces the effects of many interferences.

3.2 Special precautionary measures to prevent contamination need to be used during sample collection and laboratory determination.

4. Apparatus

4.1 *Atomic absorption spectrophotometer,* for use at 232.0 nm and equipped with Zeeman background correction, digital integrator to quantitate peak areas, graphite furnace with temperature programmer, and automatic sample injector. The programmer needs to have high-temperature ramping and controlled argon-flow capabilities.

4.1.1 Refer to the manufacturer's manual to optimize operations and instrumental performance. A 20-µL sample with a 25-µg/L concentration of nickel should yield a signal of approximately 0.17 absorbance-second. This absorbance signal is based on nickel's characteristic mass of 13.0 pg for a signal of 0.0044 absorbance-second. A 20-µL sample generally requires 30 seconds at 130°C to dry. Samples that have a complex matrix might require a longer drying or charring time. Peak shapes may be used to detect insufficient drying, charring, or atomization times or temperatures.

4.1.2 *Graphite furnace,* capable of reaching a temperature of 2,600°C sufficient to atomize the nickel. **Warning**: dial settings frequently are inaccurate, and newly conditioned furnaces need to be temperature-calibrated.

4.1.3 *Graphite tubes and platforms,* pyrolytically coated graphite tubes and platforms are suggested.

4.2 *Labware.* Many trace metals at small concentrations adsorb rapidly to glassware. To preclude this problem, fluorinated ethylene propylene (FEP) or Teflon labware may be used. Alternatively, glassware, particularly flasks and pipets, can be treated with silicone anti-wetting agent such as Surfacil (Pierce Chemical Co.) according to the manufacturer's instructions. Check autosampler cups for contamination. Lancer polystyrene disposable cups are satisfactory after acid rinsing. Alternatively, reusable Teflon or FEP cups can be purchased.

4.3 *Argon,* standard, welder's grade, commercially available. Nitrogen also can be used if recommended by the instrument manufacturer.

5. Reagents

5.1 *Nickel standard solution I*, 1.00 mL = 1,000 µg Ni: A commercially prepared and certified nickel standard can be used. An alternate method is to dissolve 1.0000 g nickel wire in a minimum of dilute HNO_3. Heat to increase rate of dissolution. Add 4 mL ultrapure concentrated HNO_3 (sp gr 1.41), Ultrex or equivalent, and dilute to 1,000 mL with water.

5.2 *Nickel standard solution II,* 1.00 mL = 10.0 µg Ni: Dilute 10.0 mL nickel standard solution I to 1,000 mL (NOTE 1).

NOTE 1. Use acidified water (paragraph 5.7) to make dilutions. Store all standards in sealed Teflon or FEP containers. Rinse each container twice with a small volume of standard solution before filling the storage container. Standards stored for 6 months in FEP containers yielded values equal to freshly prepared standards.

5.3 *Nickel standard solution III,* 1.00 mL = 1.00 µg Ni: Dilute 100 mL nickel standard solution II to 1,000 mL with acidified water. Prepare fresh monthly.

5.4 *Nickel working solution IV,* 1.00 mL = 0.025 µg Ni: Dilute 25.0 mL nickel standard solution III to 1,000 mL with acidified water. Prepare fresh monthly.

5.5 *Nickel working solution V,* 1.00 mL = 0.010 µg Ni: Dilute 10.0 mL nickel standard solution III to 1,000 mL with acidified water. Prepare fresh monthly.

5.6 *Nitric acid,* concentrated, ultrapure (sp gr 1.41): J.T: Baker Ultrex brand HNO_3 is adequately pure; however, check each lot for contamination. Analyze acidified water (paragraph 5.7) for nickel. Add 1.5 mL concentrated HNO_3 per liter of water, and repeat analysis. Integrated signal should not increase by more than 0.001 absorbance-second.

5.7 *Water, acidified:* Add 4.0 mL ultrapure concentrated HNO_3 (sp gr 1.41*)* to each liter of water.

5.8 *Water:* All references to water shall be understood to mean ASTM Type I reagent water (American Society for Testing and Materials, 1991).

6. Procedure

6.1 The autosampler and the graphite furnace need to be in a clean environment,

6.2 Soak autosampler cups at least overnight in a 1*N* HNO_3 solution.

6.3 Rinse the sample cups twice with sample before filling. Place cups in sample tray and cover. Adjust sampler so that only the injection tip contacts the sample.

6.4 In sequence, inject 20-µL aliquots of blank and a minimum of two standards in duplicate. Construct the analytical curve from the integrated peak areas (absorbance-seconds).

6.5 Similarly, inject and analyze the samples in duplicate. Every tenth sample cup needs to contain either a standard, blank, or a reference material.

6.6 Restandardize as required, although with the use of L'vov platforms, restandardization generally is not necessary. Minor changes of values for known samples usually indicate deterioration of the furnace tube, contact rings, or platform. A major variation usually indicates either auto-sampler malfunction or residue buildup from a complex matrix in a previous sample.

7. Calculation

Determine the micrograms per liter of nickel in each sample from the digital display or printer output. Dilute those samples containing concentrations of nickel that exceed the working range of the method; repeat the analysis, and multiply by the proper dilution factor.

8. Report

Report concentrations of nickel, dissolved (01065), and whole water recoverable (01067), as follows: less than 10 µg/L, the nearest 1 µg/L; 10 µg/L and greater, two significant figures.

9. Precision

9. Analysis of six samples for dissolved nickel by a single operator is as follows:

Number of replicates	Mean (µg/L)	Standard deviation (µg/L)	Relative standard deviation (percent)
5	1.6	0.12	7.5
15	4.2	0.75	17.9
5	5.3	0.52	9.8
18	11.4	0.83	7.3
15	12.1	1.88	15.5
22	48.1	2.82	5.9

9.2 Analysis of three samples for whole water recoverable nickel by a single operator is as follows:

Number of replicates	Mean (µg/L)	Standard deviation (µg/L)	Relative standard deviation (percent)
7	5.9	0.67	11.4
11	13.9	1.51	10.9
10	17.6	0.23	1.3

9.3 The precision and bias for dissolved nickel was tested on several standard reference water samples. A known amount of nickel was added to each sample, and single-operator precision and bias for the samples are as follows:

Amount present (µg/L)	Number of replicates	Amount added (µg/L)	Found (µg/L) (NOTE 2)	Standard deviation (µg/L)	Relative standard deviation (percent)	Percent recovery
4.7	6	22.2	22.2	2.1	9.2	100.0
4.8	6	46.2	46.0	2.4	5.1	99.6
5.1	6	9.2	9.1	0.5	5.0	98.9
10.0	6	23.7	22.9	1.3	5.8	96.6
10.1	6	45.2	43.6	2.9	6.7	96.5

NOTE 2. The amount originally present has been subtracted.

References

American Society for Testing and Materials, 1991, Annual book of ASTM standards, Section 11, Water: Philadelphia, American Society for Testing and Materials, v. 11.01, p. 45-47.

Hinderberger, E.J., Kasser, M.L., and Koirtyohann, S.R., 1981, Furnace atomic absorption analysis of biological samples using the L'vov platform and matrix modification: Atomic Spectroscopy, v. 2, p. 1.