

# Nickel, atomic absorption spectrometric, direct

## Parameters and Codes:

Nickel, dissolved, I-1499-85 ( $\mu\text{g/L}$  as Ni): 01065

Nickel, total recoverable, I-3499-85 ( $\mu\text{g/L}$  as Ni): 01067

Nickel, suspended recoverable, I-7499-85 ( $\mu\text{g/L}$  as Ni): 01066

Nickel, recoverable-from-bottom-material, dry wt, I-5499-85 ( $\mu\text{g/g}$  as Ni): 01068

## 1. Application

1.1 This method may be used to analyze water and water-suspended sediment containing at least 100  $\mu\text{g/L}$  of nickel. Sample solutions containing more than 1,000  $\mu\text{g/L}$  need either to be diluted or to be read on a less expanded scale. Sample solutions containing less than 100  $\mu\text{g/L}$  and brines need to be analyzed by the atomic absorption spectrometric chelation-extraction method, providing that the interference limits discussed in that method are not exceeded.

1.2 Suspended recoverable nickel is calculated by subtracting dissolved nickel from total recoverable nickel.

1.3 This method may be used to analyze bottom material containing at least 5.0  $\mu\text{g/g}$  of nickel. Sample solutions containing more than 1,000  $\mu\text{g/L}$  need either to be diluted or to be read on a less expanded scale.

1.4 Total recoverable nickel in water-suspended sediment needs to undergo preliminary digestion-solubilization by method 1-3485, and recoverable nickel in bottom material needs to undergo preliminary digestion-solubilization by method 1-5485 before being determined.

## 2. Summary of method

Nickel is determined by atomic absorption spectrometry by direct aspiration of the sample into an air-acetylene flame without preconcentration or pretreatment.

## 3. Interferences

Individual concentrations of sodium (9,000  $\text{mg/L}$ ), potassium (9,000  $\text{mg/L}$ ), calcium (4,000  $\text{mg/L}$ ), magnesium (4,000  $\text{mg/L}$ ), sulfate (9,000

$\text{mg/L}$ ), and cadmium, lead, copper, zinc, cobalt, and chromium (10,000  $\mu\text{g/L}$  each) do not interfere. Higher concentrations of each constituent were not investigated.

## 4. Apparatus

4.1 *Atomic absorption spectrometer* equipped with electronic digital readout and automatic zero and concentration controls.

4.2 Refer to the manufacturer's manual to optimize instrument for the following:

Grating ----- Ultraviolet

Wavelength ----- 232.0 nm

(NOTES 1 and 2)

Source (hollow-cathode

lamp) ----- Nickel

Oxidant ----- Air.

Fuel ----- Acetylene

Type of flame ----- Oxidizing

NOTE 1. Setting the proper wavelength for nickel is critical because of the presence of an ion line at 231.6 nm. By aspirating a standard that gives approx. 50-percent absorption, the 232.0 nm-absorption line can easily be set.

NOTE 2. The 352.4-nm wavelength, less susceptible to nonatomic absorbance, may be used. The resulting analytical curve is more nearly linear; however, the procedure reduces sensitivity.

## 5. Reagents

5.1 *Nickel standard solution I*, 1.00 mL = 200  $\mu\text{g}$  Ni: Dissolve 0.2000 g Ni powder in a minimum amount of dilute  $\text{HNO}_3$ . Heat to increase rate of dissolution. Add 10.0 mL

concentrated HNO<sub>3</sub> (sp gr 1.41) and dilute to 1,000 mL with demineralized water.

5.2 *Nickel standard solution II*, 1.00 mL = 20 µg Ni: Dilute 100 mL nickel standard solution I and 1 mL concentrated HNO<sub>3</sub> (sp gr 1.41) to 1,000 mL with demineralized water.

5.3 *Nickel standard working solutions*: Prepare a series of at least six standard working solutions containing from 100 to 1,000 µg/L of nickel by appropriate dilution of nickel standard solution II with acidified water. Prepare fresh daily.

5.4 *Water, acidified*: Add 1.5 mL concentrated HNO<sub>3</sub> (sp gr 1.41) to 1 L of demineralized water.

## 6. Procedure

Aspirate the blank (acidified water) to set the automatic zero control. Use the automatic concentration control to set the concentrations of standards. Use at least six standards. Calibrate the instrument each time a set of samples is analyzed and check calibration at reasonable intervals.

## 7. Calculations

7.1 Determine the micrograms per liter of dissolved or total recoverable nickel in each sample from the digital display or printer while aspirating each sample. Dilute those samples containing concentrations of nickel that exceed the working range of the method and multiply by the proper dilution factors.

7.2 To determine micrograms per liter of suspended recoverable nickel, subtract dissolved-nickel concentration from total-recoverable-nickel concentration.

7.3 To determine micrograms per gram of nickel in bottom-material samples, first determine the micrograms per liter of nickel as in paragraph 7.1; then

$$\text{Ni } (\mu\text{g/g}) = \frac{\mu\text{g/L Ni} \times \frac{\text{mL of original digest}}{1,000}}{\text{Wt of sample}}$$

## 8. Report

8.1 Report nickel, dissolved (01065), total-recoverable (01067), and suspended-recoverable

(01066), concentrations to the nearest 100 µg/L.

8.2 Report nickel, recoverable-from-bottom-material (01068), concentrations as follows: less than 100 µg/g, nearest 10 µg/g and above, two significant features.

## 9. Precision

9.1 Precision for dissolved nickel for 16 samples within the range of 6.2 to 36.2 µg/L (NOTE 3) may be expressed as follows:

$$S_T = 0.431X + 2.88$$

where

$S_T$  = overall precision, micrograms per liter, and

$X$  = concentration of nickel, micrograms per liter.

The correlation coefficient is 0.7593.

NOTE 3. Precision data for nickel are below the reporting level of 100 µg/L. Samples were not available that contained greater nickel concentrations; however, precision should improve at greater concentrations.

9.2 Precision for dissolved nickel for five of the 16 samples expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean (µg/L)	Relative standard deviation (percent)
5	6.2	18
11	10.5	80
6	10.8	15
6	20.0	46
19	36.2	47

9.3 It is estimated that the percent relative standard deviation for total recoverable and suspended recoverable nickel and for nickel in bottom material will be greater than that reported for dissolved nickel.

9.4 Precision for total recoverable nickel expressed in terms of the percent relative standard for two water-suspended sediments is as follows:

Number of laboratories	Mean (µg/L)	Relative standard deviation (percent)
10	26.2	58
10	35.9	53