
International Standard



4940

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Steel and cast iron — Determination of nickel content — Flame atomic absorption spectrometric method

Aciers et fontes — Dosage du nickel — Méthode par spectrométrie d'absorption atomique dans la flamme

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Foreword

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International Standard ISO 4940 was prepared by Technical Committee ISO/TC 17, *Steel*.

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Steel and cast iron — Determination of nickel content — Flame atomic absorption spectrometric method

1 Scope and field of application

This International Standard specifies a method for the determination of nickel in steel and cast iron by means of flame atomic absorption spectrometry.

The method is applicable to nickel contents in the range 0,002 to 0,5 % (m/m).

2 Reference

ISO/R 377, *Selection and preparation of samples and test pieces for wrought steel*.

3 Principle

Dissolution of a test portion in a mixture of appropriate acids followed by evaporation to fuming with perchloric acid.

Spraying of the solution into an air-acetylene flame. Spectrometric measurement of the atomic absorption of the 352,5 nm spectral line emitted by a nickel hollow cathode lamp.

NOTE — With some instruments it is not possible to obtain sufficient sensitivity at the wavelength of 352,5 nm for low concentrations of nickel near the bottom end of the application range and in such cases the alternative wavelength of 232,0 nm must be used.

At the wavelength of 352,5 nm, the signal-to-noise ratio is higher than at a wavelength of 232,0 nm. In general, use of the 352,5 nm line will give better reproducibility.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and having a very low nickel content and only distilled water or water of equivalent purity.

If possible, use only freshly prepared distilled or deionized water.

4.1 Iron of high purity, nickel content < 0,000 5 % (m/m).

4.2 Hydrochloric acid-nitric acid mixture.

Mix three parts by volume of hydrochloric acid (ρ about 1,19 g/ml), one part by volume of nitric acid (ρ about 1,40 g/ml), and two parts by volume of water.

Prepare this mixture immediately before use.

4.3 Nitric acid-perchloric acid mixture.

Mix 100 ml of nitric acid (ρ about 1,40 g/ml) with 800 ml of perchloric acid (ρ about 1,54 g/ml). Dilute to 1 litre and mix.

NOTE — Perchloric acid (ρ about 1,67 g/ml) may also be used. 100 ml of perchloric acid (ρ about 1,54 g/ml) is equivalent to 79 ml of perchloric acid (ρ about 1,67 g/ml).

4.4 Nickel, standard solution.

4.4.1 Stock solution, corresponding to 1 g of Ni per litre.

Weigh, to the nearest 0,000 1 g, 0,500 0 g of high purity nickel [$\geq 99,9$ % (m/m) pure], and dissolve in 25 ml of nitric acid (ρ about 1,40 g/ml, diluted 1 + 1). Boil to remove nitrous fumes. Cool and transfer the solution to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

4.4.2 Standard solution, corresponding to 40 mg of Ni per litre.

Transfer 10,0 ml of the stock solution (4.4.1) into a 250 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 40 μ g of Ni.

Prepare this standard solution immediately before use.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Atomic absorption spectrometer

A nickel hollow cathode lamp; supplies of air and acetylene sufficiently pure to give a steady clear fuel-lean flame, free from water and oil, and free from nickel.

The atomic absorption spectrometer used will be satisfactory if after optimization according to 7.3.4 the limit of detection and