UDC 669.14:543.422:546.74

Chemische analyse van ijzer en staal
Bepaling van het gehalte aan nikkel in staal en ijzer met behulp van atomaire-absorptiespectrometrie (vlamtechniek)

Chemical analysis of ferrous materials – Determination of nickel in steels and irons – Flame atomic absorption spectrometric method

NEDERLANDSE NORM

NEN-EN 10 136

1e druk, april 1990

De tekst is gelijk aan die van Euronorm 136.

Dit document bevat de officiële Engelse versie van de Europese norm EN 10 136, januari 1989.

De Europese norm EN 10 136 heeft de status van Nederlandse norm.

Normcommissie 342 38 "Staal- en ijzeranalyse"

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Key words: Iron and steel products, steels, cast iron, chemical analysis, determination of content, nickel, atomic absorption spectrophotometry, flame photometry

English version

Chemical analysis of ferrous materials Determination of nickel in steels and irons Flame atomic absorption spectrometic method

Analyse chimique des matériaux sidérurgiques. Dosage du nickel dans les aciers et les fontes. Méthode par spectrométrie d'absorption atomique dans la flamme.

Chemische Analyse von Eisenwerkstoffen. Bestimmung von Nickel in Stahl. Flammenatomabsorptionsspektrometrisches Verfahren.

This European Standard was accepted by CEN on 1989-01-15.
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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: Rue Bréderode 2, B-1000 Brussels

Ref. No. EN 10 136:1989 E

Brief History

This European Standard takes over the content of EURONORM 136-83 "Chemical analysis of ferrous materials – Determination of nickel in steels and irons – Flame atomic absorption spectrometric method" prepared by ECISS/TC 20 "Methods of chemical analysis"; the Secretariat of which is allocated to the Dansk Standardiseringsrad (DS).

It has been submitted to the CEN Formal Vote following the decision of the Coordinating Commission (COCOR) of the European Committee for Iron and Steel Standardization on 1987-11-24/25.

It has been adopted and ratified by CEN BT on 1988-11-05.

According to the Common CEN/CENELEC Rules, following countries are bound to implement this European Standard:

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxemburg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

Contents

1 SCOPE AND FIELD OF APPLICATION

This European Standard specifies a method for the determination of nickel in steels and irons by means of flame atomic absorption spectrometry.

The method is applicable to steels and irons with nickel contents of 0.003 to 2.0% (m/m).

2 REFERENCE

EURONORM 18 - Selection and preparation of samples and test pieces for steel and iron and steel products.

General guidelines for the application of flame atomic absorption spectrometric methods are in course of preparation.

3 PRINCIPLE

Dissolution of a test portion in a mixture of appropriate acids and fuming with perchloric acid.

Spraying of the solution into an air-acetylene flame. Determination of the nickel by means of the spectrometric measurement of the atomic absorption of the 232.0 nm or 352.5 nm line emitted by a nickel hollow cathode lamp.

The instrument is calibrated by addition of a nickel standard solution to a similar matrix to that of the test solution.

NOTE – At the wavelength of 352.5 nm the signal-to-noise ratio is higher than at a wavelength of 232.0 nm. Generally, use of the 352.5 nm line will lead to a better reproducibility.

However, as the sensitivity at 352.5 nm is less than the sensitivity at 232.0 nm, with some instruments the use of the longer wavelength will be impossible when analysing low nickel contents.

4 REAGENTS

During the analysis use only reagents of recognized analytical reagent quality and having a very low nickel content, and only distilled water or water of equivalent purity.

Carefully check the nickel content of all reagents.

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

4.1 Iron of high purity, with a nickel content < 0.0005% (m/m)

4.2 Hydrochloric acid–nitric acid mixture

Mix three volumes of hydrochloric acid, ρ 1.19 g/ml approximately, one volume of nitric acid, ρ 1.40 g/ml approximately and two volumes of water. This mixture is to be prepared immediately before use.

4.3 Nitric acid–perchloric acid mixture

Mix 100 ml of nitric acid, ρ1.40 g/ml approximately with 800 ml of perchloric acid, ρ 1.54 g/ml approximately. Dilute to 1 l with water and mix.

NOTE – Perchloric acid (ρ 1.67 g/ml approximately) may also be used. 100 ml of perchloric acid (ρ 1.54 g/ml approximately) is equivalent to 79 ml of perchloric acid ρ 1.67 g/ml approximately).

4.4 Nickel stock solution, corresponding to 1 mg of nickel per ml approximately

Weigh, to the nearest 0.001 g, about 0.5 g of high purity nickel (≥ 99.9% pure). Transfer the weighed mass to a 400 ml beaker and dissolve in 25 ml of nitric acid (ρ 1.40 g/ml approximately diluted 1 + 1 (v/v)). Boil to remove oxides of nitrogen. Cool and transfer the solution to a 500 ml volumetric flask, dilute to the mark with water and mix. Calculate the concentration of nickel in this stock solution, in mg/ml.

4.5 Nickel reference solution, corresponding to 40 µg of nickel per ml approximately

Transfer 10.0 ml of nickel stock solution (4.4) to a 250 ml volumetric flask, dilute to the mark with water and mix. Calculate the concentration of nickel in the reference solution, in µg/ml.

5 APPARATUS

Ordinary laboratory equipment 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 characteristic concentration are in reasonable agreement with the values given by the manufacturer and it meets the following performance requirements.

5.1.1 Minimum precision

The standard deviation of 10 measurements of the absorbency of the most concentrated solution shall not exceed 1.0% of the mean absorbance.

The standard deviation of 10 measurements of the absorbance of the least concentrated calibration solution (excluding the zero calibration solution) shall not exceed 0.5% of the mean absorbency of the most concentrated calibration solution.

For example, if the top and bottom calibration solutions represent 0.1% and 0.01% nickel in the sample, the precision called for (as two standard deviations) would be 0.002% and 0.001% nickel respectively, assuming curve linearity.

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