SLOVENSKI STANDARD
SIST EN 27520:2009

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Feronikelj - Določ evanje kobalta - Plamenska atomska absorpcijska spektrometriț na metoda (ISO 7520:1985)

Ferronickel - Determination of cobalt content - Flame atomic absorption spectrometric method (ISO 7520:1985)

Ferronickel - Bestimmung des Cobaltgehaltes - Atomabsorptions-Spektralfotometrisches Verfahren (ISO 7520:1985)

Ferro-nickel - Dosage du cobalt - Méthode par spectrométrie d'absorption atomique dans la flamme (ISO 7520:1985)

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English version

Ferronickel - Determination of cobalt content - Flame atomic absorption spectrometric method (ISO 7520:1985)

Ferro-nickel - Dosage du cobalt - Méthode par spectrométrie d'absorption atomique dans la flamme (ISO 7520:1985)

Ferronickel - Bestimmung des Cobaltgehaltes - Atomabsorptions-Spektralfotometrisches Verfahren (ISO 7520:1985)

This European Standard was approved by CEN on 1991-11-06 and is identical to the ISO standard as referred to.
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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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CEN

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

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Foreword

On the proposal of the CEN Central Secretariat, the Technical Board has decided by resolution BT C17/1990 to submit the International Standard

ISO 7520:1985 : Ferronickel - Determination of cobalt content - Flame atomic absorption spectrometric method

to the formal vote.

This European Standard EN 27520 was approved by CEN on 1991-09-24.

According to the CEN/CENELEC Internal Regulations, the follwing countries are bount to implement this European Standard :

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

Endoresement notice

The text of the International Standard ISO 7520:1985 was approved by CEN as a European Standard without any modifications.

International Standard 7520

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Ferronickel — Determination of cobalt content — Flame atomic absorption spectrometric method

Ferro-nickel — Dosage du cobalt — Méthode par spectrométrie d'absorption atomique dans la flamme

First edition — 1985-11-15

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Descriptors : nickel, ferronickel, chemical analysis, determination of content, cobalt, atomic absorption method.

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 7520 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Printed in Switzerland

Ferronickel — Determination of cobalt content — Flame atomic absorption spectrometric method

1 Scope and field of application

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the cobalt content of ferronickel in the range 0,025 to 2,5 % (m/m).

2 References

ISO 385/1, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 648, *Laboratory glassware — One-mark pipettes.*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks.*

ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*

ISO 6352, *Ferronickel — Determination of nickel content — Dimethylglyoxime gravimetric method.*

3 Principle

Dissolution of a test portion in a nitric acid-hydrochloric acid mixture. Precipitation of silica by dehydration in perchloric acid. Removal of silica by filtration. Addition of lanthanum for elimination of potential interferences. Determination of cobalt by atomic absorption spectrometry in an air-acetylene flame at a wavelength of 240,7 nm.

NOTE — This analysis can be carried out on solutions which have been used for the determination of nickel by ISO 6352.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Hydrochloric acid, ϱ_{20} = 1,19 g/ml.

4.2 Hydrochloric acid, ϱ_{20} = 1,19 g/ml, diluted 1+9.

4.3 Nitric acid, ϱ_{20} = 1,41 g/ml, diluted 1+1.

4.4 Perchloric acid, ϱ_{20} = 1,61 g/ml [72 % (m/m)].

4.5 Hydrofluoric acid, ϱ_{20} = 1,14 g/ml, diluted 1+1.

WARNING — Hydrofluoric acid is extremely irritating and corrosive to skin and mucous membranes, producing severe skin burns which are slow to heal. In case of skin contact wash well with water and seek medical advice.

4.6 Lanthanum, solution, containing 200 g of La per litre.

Weigh 250 g of lanthanum chloride hexahydrate ($LaCl_3 \cdot 6H_2O$) and transfer to a 600 ml beaker. Add 25 ml of hydrochloric acid (4.1) and 300 ml of water. Stir to complete dissolution. Filter, if necessary, into a 500 ml one-mark volumetric flask, make up to the mark with water and mix.

4.7 Nickel plus iron, matrix solution, containing 12 g of Ni and 28 g of Fe per litre.

4.7.1 Weigh 12,0 g of high purity nickel powder [containing less than 0,001 % (m/m) cobalt] into an 800 ml beaker. Add 50 ml of water and 50 ml of nitric acid (ϱ_{20} = 1,41 g/ml). When the initial reaction subsides, stir and heat to complete dissolution. Dilute to about 250 ml with water.

4.7.2 Weigh 28,0 g of high purity iron powder [containing less than 0,001 % (m/m) cobalt] into an 800 ml beaker. Add 100 ml of hydrochloric acid diluted 1+1. Carefully add 50 ml of nitric acid (ϱ_{20} = 1,41 g/ml) and heat to complete dissolution and oxidation of iron. Dilute to about 250 ml.

4.7.3 Carefully combine the nickel solution (4.7.1) with the iron solution (4.7.2). Filter into a 1 000 ml one-mark volumetric flask, make up to the mark with water and mix.

4.8 Cobalt, standard solution, corresponding to 0,500 g of Co per litre.

Weigh, to the nearest 0,001 g, 0,500 g of high purity [99,9 % (m/m) Co, minimum] cobalt powder, transfer to a 600 ml beaker and add 40 ml of nitric acid (4.3). Heat to complete dissolution, boil gently to expel oxides of nitrogen, cool and transfer to a 1 000 ml one-mark volumetric flask containing 160 ml of nitric acid (4.3). Make up to the mark with water and mix.

1 ml of this standard solution contains 0,500 mg of Co.

5 Apparatus

Ordinary laboratory glassware, and

5.1 Atomic absorption spectrometer, equipped with a laminar flow burner for an air-acetylene flame and a cobalt hollow cathode lamp.

5.2 Burette, of capacity 50 ml, graduated in divisions of 0,1 ml, in accordance with ISO 385/1, class A.

5.3 Glass beakers, of capacity 600 ml, clean, unetched and flat bottomed.

5.4 Pipettes, of capacities 25 and 50 ml, in accordance with ISO 648, class A.

5.5 Volumetric flasks, of capacities 250; 500; and 1 000 ml, in accordance with ISO 1042, class A.

5.6 Polytetrafluoroethylene (PTFE) beaker, of capacity 600 ml, for samples with a high silicon content.

6 Sampling and samples

6.1 Sampling and preparation of the laboratory sample shall be carried out by normal agreed procedures or, in case of dispute, by the relevant International Standard.

6.2 The laboratory sample normally is in the form of millings, drillings or granules and no further preparation of the sample is necessary.

6.3 If it is suspected that the laboratory sample is contaminated with oil or grease from the milling or drilling process, it shall be cleaned by washing with high purity acetone and drying in air.

6.4 If the laboratory sample contains particles or pieces of widely varying sizes, the test portion should be obtained by riffling.

7 Procedure

WARNING — Fuming perchloric acid is a powerful oxidant and can cause an explosive mixture when in contact with organic materials. All evaporations should be done in fume cupboards suitable for use with perchloric acid.

7.1 Test portion

Weigh, to the nearest 0,001 g, 3,9 to 4,1 g of the laboratory sample and transfer to a glass beaker (5.3).

7.2 Blank test

Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all the reagents.

7.3 Preparation of test solution

7.3.1 Dissolve the test portion (7.1) by adding 25 ml of water followed by 50 ml of nitric acid (4.3). Cover the beaker with a watch-glass and heat gently, if necessary, to complete dissolution.

NOTE — For ferronickel samples containing more than 1 % (m/m) silicon, use a polytetrafluoroethylene beaker (5.6). Attack the test portion by adding successively 25 ml of water, 40 ml of nitric acid (4.3) and 10 ml of hydrochloric acid (4.1). To obtain complete dissolution of the sample, add, at the end of effervescence, 10 ml of hydrofluoric acid (4.5) and 40 ml of perchloric acid (4.4). Heat until evolution of fumes of perchloric acid. Allow to cool and transfer the solution quantitatively to a glass beaker (5.3). Heat at 260 °C until abundant white fumes are obtained. Reflux at this temperature for 20 min and proceed as directed in 7.3.2, "Remove the beaker...".

7.3.2 When the metal is dissolved, add 40 ml of perchloric acid (4.4) and heat at 260 °C until abundant white fumes are obtained. Reflux at this temperature for 20 min. Remove the beaker from the hotplate and allow to cool. Add 20 ml of hydrochloric acid (4.1) and 200 ml of warm water. Filter off the silica using a medium porosity filter paper, collecting the filtrate in a 1 000 ml one-mark volumetric flask. Rinse the beaker and wash the silica precipitate three times with hydrochloric acid diluted 1 + 9 and four times with warm water. Discard the silica precipitate. Add 50 ml of lanthanum solution (4.6) to the filtrate, make up to the mark with water and mix thoroughly (test solution A).

7.4 Preparation of calibration solutions

7.4.1 Set A

7.4.1.1 Transfer six 50,0 ml volumes of the nickel plus iron matrix solution (4.7) to six 150 ml beakers.

7.4.1.2 Add, using a burette (5.2), 0; 1,0; 2,0; 3,0; 5,0; and 10,0 ml respectively of the cobalt standard solution (4.8). Add 20 ml of perchloric acid (4.4) and heat just to the liberation of white fumes. Cool, add 50 ml of water and transfer quantitatively into a 500 ml one-mark volumetric flask containing 10 ml of hydrochloric acid (4.1). Add 25 ml of lanthanum solution (4.6), make up to the mark with water and mix.

7.4.2 Set B

7.4.2.1 Transfer six 5 ml volumes of the nickel plus iron matrix solution (4.7) to six 150 ml beakers.

7.4.2.2 See 7.4.1.2.

7.5 Calibration and determination

7.5.1 Expected cobalt contents 0,025 to 0,25 % (m/m)

Carry out the determination on test solution A (7.3.2) and the set of calibration solutions A (7.4.1).