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

UDC 669.243.881 : 543.422 : 546.73 **Ref. No. ISO 7520-1985 (E)**

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

Price based on 3 pages

Foreword

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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).

7.5.1.1 Set the required instrument parameters of the atomic absorption spectrometer according to the manufacturer's instructions using a cobalt hollow cathode lamp at a wavelength of 240,7 nm.

7.5.1.2 Light the burner and adjust the air and acetylene flows to obtain a slightly oxidizing clear non-luminescent flame while aspirating water.

7.5.1.3 Aspirate test solution A (7.3.2) into the flame and note the absorbance reading.

7.5.1.4 Aspirate water and check that the reading returns to zero.

7.5.1.5 Aspirate the set of calibration solutions A (7.4.1) in order of increasing instrument response and note the absorbance readings. Flush the system with water between each reading.

7.5.1.6 Aspirate the blank test solution and note the reading.

7.5.1.7 Repeat the measurement of the test solution, blank and calibration solutions to check the repeatability of the readings.

NOTES

1 Poor repeatability may indicate an instrument malfunction or a variation in the aspiration rate among the solutions.

2 Ensure that the measurement corresponding to the test sample falls between two calibration solutions containing cobalt as close as possible to that being determined.

7.5.2 Expected cobalt content 0,25 to 2,5 % (*m*/*m*)

7.5.2.1 Transfer, using a pipette, 25,0 ml of test solution A (7.3.2) into a 250 ml one-mark volumetric flask. Add 12 ml of lanthanum solution (4.6), 10 ml of perchloric acid (4.4) and 5 ml of hydrochloric acid (4.1). Make up to the mark with water and mix (test solution B).

7.5.2.2 Dilute the blank test (7.2) as directed in 7.5.2.1.

7.5.2.3 Proceed with the determination as directed in 7.5.1 using test solution B (7.5.2.1), blank test (7.5.2.2) and the set of calibration solutions B (7.4.2).

7.6 Preparation of calibration graphs

Plot the instrument reading for each of the calibration solutions less the reading for the zero member against the concentration of cobalt in the calibration solution.

NOTE — Some instruments may be adjusted to give a read-out directly in concentration of the analyte. A graph of instrument response versus concentration should be plotted to check the linearity and validity of the readings.

8 Expression of results

8.1 Calculation

8.1.1 Determine the concentration of cobalt in the test solution and in the blank from the calibration graph.

8.1.2 The cobalt content, expressed as a percentage by mass, is given by the formula

$$\frac{V(\varrho_1 - \varrho_0)}{m} \times 10^{-4} \times f$$

where

ϱ_1 is the concentration, expressed in milligrams per litre, of cobalt in the test solution;

ϱ_0 is the concentration, expressed in milligrams per litre, of cobalt in the blank test;

m is the mass, in grams, of the test portion;

V is the volume, in millilitres, of the test solution;

f is the dilution factor of the test solution ($f = 10$ for test solution B).

8.2 Precision

This International Standard was subjected to a limited interlaboratory test programme involving seven laboratories in four countries. Eight samples of commercial ferronickel covering the range 21 to 41 % (*m*/*m*) nickel and 0,6 to 1,3 % (*m*/*m*) cobalt were analysed.

Repeatability and reproducibility were calculated according to ISO 5725 with the results given in the table. It is expected that r and R will be correspondingly lower for lower cobalt levels.

Table — Results of statistical analysis

Nickel Content [% (*m*/*m*)] **Cobalt content** [% (*m*/*m*)]	20 to 25 0,7	25 to 40 0,7 to 1,3
Standard deviation		
— within laboratory, s_w	0,021	0,044
— between laboratories, s_b	0,013	0,028
Repeatability, $r = 2{,}83 \sqrt{s_w^2}$	0,06	0,12
Reproducibility, $R = 2{,}83 \sqrt{s_w^2 + s_b^2}$	0,07	0,15

9 Special cases

If the test solution from the determination of nickel by the dimethylglyoxime gravimetric method is used, an appropriate aliquot shall be taken and lanthanum added for the determination of cobalt following the principles of 7.5.2.

10 Test report

The test report shall include the following information:

a) the reference of the method used;

b) the results of the analysis;

c) the number of independent replications;

d) any unusual features noted during the analysis;

e) any operation not included in this International Standard or regarded as optional.

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