SLOVENSKI STANDARD
SIST EN 26352:2009

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Feronikelj - Doloļ evanje niklja - Gravimetriļ na metoda z dimetilglioksimom (ISO 6352:1985)

Ferronickel - Determination of nickel content - Dimenthylglyoxime gravimetric method (ISO 6352:1985)

Ferronickel - Bestimmung des Nickelgehaltes - Gravimetrisches Verfahren mit Dimethylglyoxim (ISO 6352:1985)

Ferro-nickel - Dosage du nickel - Méthode gravimétrique à la diméthylglyoxime (ISO 6352:1985)

Ta slovenski standard je istoveten z: **EN 26352:1991**

ICS:

77.100	Železove zlitine	Ferroalloys

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Descriptors : Ferroalloys, ferronickel, chemical analysis, determination of content, nickel, gravimetric analysis, dimethylglyoxime

English version

Ferronickel - Determination of nickel content - Dimenthylglyoxime gravimetric method (ISO 6352:1985)

Ferro-nickel - Dosage du nickel - Méthode gravimétrique à la diméthylglyoxime (ISO 6352:1985)

Ferronickel - Bestimmung des Nickelgehaltes - Gravimetrisches Verfahren mit Dimethylglyoxim (ISO 6352:1985)

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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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 6352:1985 : Ferronickel - Determination of nickel content - Dimenthylglyoxime gravimetric method

to the formal vote.

This European Standard EN 26352 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.

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Endoresement notice

The text of the International Standard ISO 6352:1985 was approved by CEN as a European Standard without any modifications.

International Standard 6352

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

Ferronickel — Determination of nickel content — Dimethylglyoxime gravimetric method

Ferro-nickel — Dosage du nickel — Méthode gravimétrique à la diméthylglyoxime

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Descriptors : ferroalloys, ferronickel, chemical analysis, determination of content, nickel, gravimetric analysis, dimethylglyoxime.

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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 6352 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 nickel content — Dimethylglyoxime gravimetric method

1 Scope and field of application

This International Standard specifies a gravimetric method for the determination of the nickel content of ferronickel in the range 15 to 60 % (*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.*

3 Principle

Dissolution of a test portion in nitric acid. Precipitation of silica by dehydration in perchloric acid. Removal of silica by filtration. Precipitation of nickel from a tartro-ammoniacal medium by an ethanolic solution of dimethylglyoxime. A second precipitation of nickel and weighing after drying at 150 °C. Determination of residual nickel in the filtrates by atomic absorption spectrometry.

4 Reagents

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

4.1 Acetic acid, ϱ_{20} = 1,05 g/ml, diluted 1 + 1.

4.2 Ammonium hydroxide, ϱ_{20} = 0,925 g/ml.

4.3 Dimethylglyoxime, 10 g/l solution in ethanol.

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

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

4.6 Nitric acid, ϱ_{20} = 1,41 g/ml.

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

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

4.9 Tartaric acid, 500 g/l solution.

4.10 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.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Filtration crucible, fritted glass, of approximately 10 to 20 µm pore diameter.

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

5.3 Pipettes, of capacities 50 and 100 ml, in accordance with ISO 648, class A.

5.4 Volumetric flasks, of capacities 200 and 1 000 ml, in accordance with ISO 1042, class A.

5.5 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 granules, millings or drillings 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 600 ml glass beaker (5.2).

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 crucible

7.3.1 Filter through the crucible (5.1) a hot mixture of 20 ml of hydrochloric acid (4.4), 10 ml of nitric acid (4.6) and 30 ml of water. Wash the crucible with warm water until all the acid has been removed.

7.3.2 Dry the crucible in an oven at 150 °C for 2 h. Cool in a desiccator for 60 min and weigh quickly.

NOTES

1 This procedure is used to condition a new crucible or to clean a used crucible after the analysis is completed.

2 For highest accuracy, the crucible and precipitate (7.5.9) should be weighed, as closely as possible, under the same temperature and humidity conditions as the empty crucible.

7.4 Preparation of test solution

7.4.1 Dissolve the test portion (7.1) by adding 25 ml of water followed by 50 ml of nitric acid diluted 1 + 1. 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 (5.5). Attack the test portion by adding successively 25 ml of water, 40 ml of nitric acid (4.7) and 10 ml of hydrochloric acid (4.4). To obtain complete dissolution of the sample, add, at the end of effervescence, 10 ml of hydrofluoric acid (4.10) and 40 ml of perchloric acid (4.8). Heat until evolution of fumes of perchloric acid. Allow to cool and transfer the solution quantitatively to a glass beaker (5.2). Heat to 260 °C until abundant white fumes of perchloric acid are obtained. Reflux at this temperature for 20 min and proceed as directed in 7.4.2, "Remove the beaker…".

7.4.2 When the metal is dissolved, add 40 ml of perchloric acid (4.8) 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 (4.4) 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, make up the filtrate to the mark with water and mix thoroughly.

7.5 Determination

7.5.1 Pipette from the test solution into a 600 ml beaker, an aliquot containing 60 to 120 mg of nickel, and dilute to 300 ml with water. Use a 100 ml aliquot for samples containing less than 30 % (*m*/*m*) nickel and 50 ml for samples containing more than 30 % (*m*/*m*) nickel.

7.5.2 Add 10 ml of tartaric acid solution (4.9) to the aliquot (7.5.1). Pour in, while stirring, ammonium hydroxide (4.2) until the colour of the solution changes from yellow to blue-green (pH is slightly alkaline). The solution must remain clear. Restore the yellow colour by slowly adding sufficient acetic acid (4.1). The pH must be between 4 and 5. Heat the solution to 60 °C.

7.5.3 Pour in, while stirring, 4 ml of dimethylglyoxime solution (4.3) for each 10 mg of nickel estimated to be present. Add 20 ml in excess.

7.5.4 Make the solution slightly ammoniacal (pH of about 10) by addition of sufficient ammonium hydroxide. Stir vigorously for about 30 s and allow the precipitate to settle for 30 min.

7.5.5 Filter the solution through a medium porosity paper. Wash the precipitate five times with warm water (about 40 to 50 °C). Retain the filtrate for processing in 7.5.10.

7.5.6 Dissolve the precipitate through the filter into the beaker used for the first precipitation, using a hot mixture of 20 ml of hydrochloric acid (4.4), 10 ml of nitric acid (4.6) and 30 ml of water. Wash the filter carefully with three 20 ml portions of the acid mixture, following the addition of each portion by washing with warm water. Ensure that all the red precipitate is dissolved and finally wash the filter thoroughly with warm water.

7.5.7 Reprecipitate the nickel by repeating the steps in 7.5.2 to 7.5.4 inclusive but use only 2 ml of tartaric acid solution (4.9) and only 5 ml excess of dimethylglyoxime solution (4.3).

7.5.8 Filter the precipitate on the dried and preweighed fritted glass crucible (7.3.2). Clean the beaker thoroughly and wash the precipitate five times with warm water. Retain the filtrate for processing in 7.5.10.

7.5.9 Dry the crucible and precipitate in an oven at 150 °C for 2 h, cool in a desiccator for 60 min and weigh quickly, under the same conditions as used in 7.3.2.

7.5.10 Combine the filtrates from 7.5.5 and 7.5.8. Evaporate to a viscous consistency. Add 50 ml of hydrochloric acid (4.4) in 10 to 15 ml portions and heat. Add 50 ml of hot water and bring to the boil. Allow to cool and transfer to a 200 ml one-mark volumetric flask. Make up to the mark with water and mix.

7.5.11 Determine the nickel content of the combined filtrates by atomic absorption. See annex A.

NOTES

1 The nickel content of the combined filtrates should not exceed 0,2 % (*m*/*m*) nickel in the original sample.

2 High levels of nickel in the filtrates may indicate a faulty crucible.

3 If the nickel in the filtrate is high, or if the sensitivity of the atomic absorption instrument is very high, it may be necessary to dilute the solution from 7.5.10 for atomic absorption analysis.

8 Expression of results

8.1 Calculation

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

$$20{,}32\ \frac{(m_2 - m_1) \times 1\,000}{m_0 \times V} + w_f\,(\%)$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the empty crucible;

m_2 is the mass, in grams, of the crucible and the nickel dimethylglyoxime precipitate;

V is the volume, in millilitres, of the aliquot part of the test solution taken in 7.5.1;

w_f (%) is the correction for percentage of nickel which is determined in the combined filtrates (7.5.11);

20,32 is the conversion factor, multiplied by 100, of nickel dimethylglyoxime to nickel.

8.2 Precision

8.2.1 Laboratory tests

8.2.1.1 This International Standard was subjected to an interlaboratory test programme involving seven countries, 20 laboratories and 39 analysts. Eight samples of commercial ferronickel covering the range of 21 to 41 % (*m*/*m*) nickel were analysed. Each laboratory was requested to supply two determinations by each of two analysts for each sample.

8.2.1.2 The test was carried out on test solutions of 50 ml for nickel contents lower than 30 % (*m*/*m*) and 25 ml for contents higher than 30 % (*m*/*m*). These solutions became 100 ml and 50 ml, respectively, in the final procedure adopted after completion of the programme (see 7.5.1). It is expected that the repeatability and the reproducibility will be better when the method is used as specified.

8.2.2 Statistical results

Repeatability and two reproducibilities were calculated according to the principles of ISO 5725. The values resulting from this analysis are given in table 1. A report on the interlaboratory tests and statistical analysis is given in annex B.

Table 1 — Results of statistical analysis

Nickel content [% (*m*/*m*)]	15 to 30	31 to 45
Standard deviation		
— within analyst, s_{w1}	0,047	0,066
— between analyst/within laboratory, s_{w2}	0,047	0,095
— between laboratories, s_b	0,054	0,047
Repeatability, $r = 2{,}83\sqrt{s^2_{w1}}$	0,13	0,19
Reproducibility, within laboratory, R_w $R_w = 2{,}83\sqrt{s^2_{w1} + s^2_{w2}}$	0,19	0,33
Reproducibility, between laboratories, R $R = 2{,}83\sqrt{s^2_{w1} + s^2_{w2} + s^2_b}$	0,24	0,35

NOTES TO TABLE 1

1 These values of the repeatability and reproducibility can be retained as normally representative of the performance of the analytical method specified in this International Standard and applied to similar samples.

2 The repeatability and reproducibility data may be used as guidelines for establishing splitting limits.

9 Test report

The test report shall include the following information:

a) the reference to 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.