INTERNATIONAL STANDARD

**ISO 4938**

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# Steel and iron — Determination of nickel content — Gravimetric or titrimetric method

*Aciers et fontes — Détermination du nickel — Méthode gravimétrique ou titrimétrique*

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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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information.

The committee responsible for this document is ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

This second edition cancels and replaces the first edition (ISO 4938:1988), which has been technically revised.

# Steel and iron — Determination of nickel content — Gravimetric or titrimetric method

## 1 Scope

This International Standard specifies a method for the determination of nickel in steel and iron by gravimetry or titrimetry.

The method is applicable to nickel contents from 1 % to 30 % (mass fraction).

## 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — Single-volume pipettes*

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*

ISO 14284, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*

## 3 Principle

Dissolution of a test portion with appropriate acids.

Precipitation of the nickel as nickel-dimethylglyoxime.

— Cobalt, if present, is oxidized by potassium hexacyanoferrate(lll).

— Copper, if present with cobalt, preferably is removed by controlled potential electrolysis.

Acid dissolution of the precipitate and filtration of the solution, followed by a second precipitation of the nickel as nickel dimethylglyoxime.

In the case of the gravimetric determination, weighing the dried dimethylglyoxime precipitate.

In the case of the titrimetric determination, acid dissolution of the precipitate, addition of excess EDTA.$Na_2$ solution and back titration of the excess EDTA.$Na_2$ by zinc solution using xylenol orange as an indicator.

In both cases, determination of residual nickel in the filtrate(s) by atomic absorption spectrometry (see Annex C).

## 4 Reagents

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade and only distilled grade 2 water as specified in ISO 3696 or water of equivalent purity.

### 4.1 **Sodium hydrogen sulphate** ($NaHSO_4$).

**4.2 Ethanol**, 95 % (volume fraction).

**4.3 Acetic acid**, glacial, $\rho$ approximately 1,05 g/ml.

**4.4 Hydrofluoric acid**, $\rho$ approximately 1,15 g/ml.

**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 contact with skin, wash well with water, apply a topical gel containing 2,5 % (mass fraction) calcium gluconate and seek immediate medical treatment.**

**4.5 Nitric acid,** $\rho$ approximately 1,40 g/ml.

**4.6 Perchloric acid,** $\rho$ approximately 1,54 g/ml.

**WARNING — Perchloric acid vapour can cause explosions in the presence of ammonia, nitrous fumes or organic material in general.**

**4.7 Sulphuric acid**, $\rho$ approximately 1,84 g/ml.

**4.8 Ammonia solution**, $\rho$ approximately 0,90 g/ml.

**4.9 Hydrochloric acid**, $\rho$ approximately 1,19 g/ml, diluted 1 + 1.

Add 500 ml of hydrochloric acid ($\rho$ approximately 1,19 g/ml) to 500 ml of water.

**4.10 Hydrochloric acid**, $\rho$ approximately 1,19 g/ml, diluted 1 + 99.

Add 10 ml of hydrochloric acid ($\rho$ approximately 1,19 g/ml) to 990 ml of water.

**4.11 Nitric acid**, $\rho$ approximately 1,40 g/ml, diluted 2 + 3.

Add 200 ml of nitric acid (4.5) to 300 ml of water.

**4.12 Perchloric acid**, $\rho$ approximately 1,54 g/ml, diluted 1 + 49.

Add 10 ml of perchloric acid (4.6) to 490 ml of water.

**4.13 Ammonia solution**, $\rho$ approximately 0,90 g/ml, diluted 1 + 1.

Add 500 ml of ammonia solution (4.8) to 500 ml of water.

**4.14 Ammonia solution**, $\rho$ approximately 0,90 g/ml, diluted 1 + 3.

Add 100 ml of ammonia solution (4.8) to 300 ml of water.

**4.15 Hydrochloric/nitric acids mixture.**

Mix three volumes of hydrochloric acid ($\rho$ approximately 1,19 g/ml) with one volume of nitric acid (4.5).

Prepare this mixture immediately prior to use.

**4.16 Ammonium acetate**, 200 g/l solution.

**4.17 Ammonium citrate buffer solution**.

Dissolve 500 g of citric acid monohydrate ($C_6H_8O_7 \cdot H_2O$) in 675 ml of ammonia solution (4.8) and dilute to 1 l with water.

Filter before use.

**4.18 Citric acid**, 500 g/l solution.

Dissolve 500 g of citric acid monohydrate ($C_6H_8O_7 \cdot H_2O$) in water and dilute to 1 l with water.

Filter before use.

**4.19 Dimethylglyoxime**, 30 g/l solution in alkaline medium.

Dissolve 20 g of potassium hydroxide in 400 ml of water, add 30 g of dimethylglyoxime ($C_4H_8N_2O_2$) and stir until dissolution is complete. Dilute to 1 l with water and mix.

Filter before use.

**4.20 Dimethylglyoxime**, 10 g/l solution in ethanol.

Dissolve 10 g of dimethylglyoxime ($C_4H_8N_2O_2$) in 1 000 ml of ethanol (4.2).

Filter before use.

**4.21 Hydrazine dihydrogen sulphate** ($N_2H_6SO_4$), 100 g/l solution.

**4.22 Potassium hexacyanoferrate(III)**, $K_3[Fe(CN)_6]$, 100 g/l solution.

This solution is stable for approximately 30 days.

1 ml of this solution corresponds approximately to 0,02 g of cobalt and manganese, respectively.

**4.23 Washing water**, adjusted to pH 8 with a few drops of ammonia solution (4.13).

**4.24 Ethylenediaminetetraacetic acid disodium salt** ($EDTA.Na_2$), standard-volumetric solution.

**4.24.1 Preparation of the solution**

Dissolve 6,33 g of ethylenediaminetetraacetic acid disodium salt ($C_{10}H_{14}O_8N_2Na_2 \cdot 2H_2O$) in water, transfer the solution to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution corresponds approximately to 1 mg of nickel.

**4.24.2 Standardization of the solution**

Transfer 25,0 ml of the nickel standard solution (4.24.3) to a 250 ml beaker and add 33 ml of $EDTA.Na_2$ solution (4.24.1). Add 15 ml of ammonium acetate solution (4.16) and dilute to about 150 ml with water. Continue as described in the third paragraph of 7.2.5.

The corresponding concentration, $c$, of the EDTA.$Na_2$ solution (4.24.1), expressed in milligrams of nickel per millilitre, is given by Formula (1):

$$c = \frac{(m_1 \times 25) + (m_2 \times V_1)}{V_2} \quad (1)$$

where

$m_1$ is the mass of nickel contained in 1 ml of the nickel standard solution (4.24.3), in milligrams;

$m_2$ is the mass of nickel corresponding to 1 ml of the zinc standard solution (4.25), in milligrams;

$V_1$ is the volume of the zinc standard solution (4.25) used for the titration, in millilitres;

$V_2$ is the volume of the EDTA.$Na_2$ solution (4.24.1) used for the standardization, in millilitres.

#### 4.24.3 Nickel standard solution

Weigh, to the nearest 0,1 mg, 1,000 g of nickel [Ni > 99,95 % (mass fraction)]. Dissolve in 20 ml of nitric acid (4.11). Boil to remove nitrous fumes, cool, transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 1,0 mg of nickel.

#### 4.25 Zinc standard solution.

Weigh, to the nearest 0,1 mg, 1,114 0 g of zinc [purity > 99,9 % mass fraction] and transfer to a 300 ml beaker. If the zinc is oxidized, it should be cleaned with hydrochloric acid (4.9), water and acetone, and dried for 5 min at 110 °C before use.

Add about 50 ml of water, 20 ml of hydrochloric acid (4.9) and five drops of bromine-saturated water. Cover with a watch glass and heat until the metal is completely dissolved. Continue the heating until the colour of the bromine disappears, cool to room temperature and add 20 ml of glacial acetic acid (4.3). Adjust the pH of the solution to 6,0 ± 0,2 with ammonia solution (4.14). Transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution corresponds to 1,0 mg of Ni and also approximately to 1 ml of the EDTA.$Na_2$ standard volumetric solution (4.24).

**4.26 Xylenol orange**, 1 g/l solution.

Pulverize 0,1 g of xylenol orange ($C_{31}H_{28}N_2O_{13}SNa_4$) with a little portion of water to make a paste. Dilute to 100 ml with water. Filter and store in a brown glass stoppered bottle.

This solution is stable for one week.

## 5 Apparatus

All volumetric glassware shall be Class A in accordance with ISO 648 or ISO 1042, as appropriate.

Ordinary laboratory equipment and the following shall be used.

**5.1 Sintered-glass filter**, complying with porosity grade P16 of ISO 4793.

**5.2 pH-meter**.

**5.3 Apparatus for controlled-potential electrolysis**, with a saturated calomel reference electrode and a platinum electrode.

# 6 Sampling

Carry out sampling in accordance with ISO 14284 or appropriate national standards for steel and iron.

# 7 Procedure

## 7.1 Test portion

The test portion shall be selected so that the amount of nickel to be precipitated is within the range of 25 mg to 70 mg for the gravimetric determination and 25 mg to 40 mg for the titrimetric determination.

For example, if 3,5 % (mass fraction) nickel content is expected, weigh approximately a 1 g test portion. All weighings shall be to the nearest 0,1 mg.

## 7.2 Determination

### 7.2.1 Preparation of the test solution

Place the test portion (7.1) in a beaker of suitable capacity (e.g. 400 ml for test portions up to 2,5 g and 600 ml for test portions above 2,5 g). Add 30 ml of hydrochloric/nitric acids mixture (4.15) for test portions up to 2,5 g and 50 ml for other test portions. Cover the beaker with a watch-glass and heat at 50 °C to 60 °C until reaction is complete and then add 0,5 ml to 1 ml of hydrofluoric acid (4.4). Add 30 ml of perchloric acid (4.6) for test portions up to 2,5 g and 50 ml for other test portions.

Increase the temperature to approximately 180 °C and evaporate the solution until copious white fumes of perchloric acid are evolved.

Cover the beaker with a dry watch-glass and continue fuming until the chromium, if present, is completely oxidized (at this stage, the solution gets the orange dichromate colour).

Remove the beaker from the heat source and allow to cool. Add 100 ml of water and heat to dissolve the salts. Boil for about 5 min to eliminate chlorine compounds.

Filter through a rapid filter paper to remove graphite and oxides of silicon, tungsten, niobium and tantalum. Collect the filtrate in an 800 ml beaker and wash eight to ten times with hot perchloric acid (4.12) and then, wash twice with water.

NOTE This solution is the main solution.

Transfer the filter and residue to a platinum crucible. Dry, ash and ignite at a temperature of 900 °C. Treat the residue with hydrofluoric acid (4.4), evaporate and then fuse the residue by carefully heating the crucible with a small amount of sodium hydrogen sulphate (4.1).

Allow the crucible to cool, dissolve the melted residue in hot water and add the solution obtained to the main solution.

### 7.2.2 First nickel precipitation

**7.2.2.1** The following applies to test portions containing less than 5 mg of copper and less than 5 mg of cobalt.

Dilute the test solution prepared in 7.2.1 to approximately 400 ml with water and add 50 ml of citric acid solution (4.18). Neutralize with ammonia solution (4.13) and re-acidify slightly with hydrochloric acid (4.9). Heat to 90 °C and directly pour into the solution, 10 ml of dimethylglyoxime solution (4.20) for each 10 mg of nickel present.

Neutralize the solution with ammonia solution (4.13), add 2 ml excess and stir well. Let the solution stand at approximately 65 °C for about 2 h. Cool rapidly to room temperature.

Filter through a rapid 12,5 cm hardened paper and wash six to eight times with the cold washing water (4.23).

**7.2.2.2** The following applies to test portions containing more than 5 mg of copper.

Follow the procedure as described in 7.2.2.1 but increase the amount of dimethylglyoxime solution (4.20) for the precipitation. Add 10 ml for each 10 mg of nickel present and add 30 ml in excess.

**7.2.2.3** The following applies to test portions containing more than 5 mg of cobalt.

Evaporate the test solution prepared in 7.2.1 or 7.2.2.4 to 100 ml approximately.

Transfer the solution into a 600 ml beaker containing 100 ml of the ammonium citrate buffer solution (4.17) and 65 ml of ammonia solution (4.8). After rinsing the original beaker with water, wash once with 15 ml of ammonia solution (4.13) and add the washing to the test solution.

Add an amount of potassium hexacyanoferrate(III) solution (4.22) sufficient to oxidize the cobalt and manganese present (6 ml for each 0,1 g of cobalt and manganese) plus 10 % excess. Stir well (the solution should now be red) and, using a pH-meter (5.2), adjust the solution to pH 8,0 ± 0,2 with ammonia solution (4.8) or acetic acid (4.3). Add 50 ml of ethanol (4.2) and 100 ml of dimethylglyoxime solution (4.19) and stir well. Let the solution stand at room temperature for 4 h, checking that the solution remains at pH 8.

Filter through a rapid 12,5 cm hardened paper and wash six to eight times with the cold washing water (4.23).

**7.2.2.4** The following applies to test portions containing cobalt and high concentrations of copper.

To the test portion prepared in 7.2.1, add hydrazine dihydrogen sulphate solution (4.21) drop by drop until the chromium is completely reduced.

Remove the copper by electrolysing the solution at controlled potential, commencing the deposition with a cathode potential of −0,15 V (against standard calomel electrode) and gradually reducing to −0,30 V (against standard calomel electrode). Copper deposition should be complete after about 40 min when the current remains constant at a very low value.

Completion of deposition can be checked by adding about 20 ml of water to the test solution and continuing the electrolysis. Deposition is complete if no copper is deposited on the freshly submerged portion of the cathode after 5 min. Switch off the current to the calomel electrode circuit. Remove the calomel electrode, then remove the platinum electrodes and rinse with water.

Add about 5 ml of nitric acid (4.5) and evaporate the solution until copious white fumes are evolved. Cover the beaker with a dry watch-glass and continue heating until the chromium is completely oxidized. Remove the beaker from the heat source and allow to cool. Dilute the solution with 100 ml of water, heat to dissolve the salts and boil for about 5 min to eliminate chlorine compounds.

Proceed as described in 7.2.2.3.

### 7.2.3 Second nickel precipitation

The second nickel precipitation may be omitted for test portions containing less than 20 mg of copper and less than 25 mg of cobalt if the titrimetric determination is to be used, in which case, proceed as described in 7.2.5.

Place the filter and precipitate obtained in 7.2.2 in the precipitation beaker and cover with a watch glass. Add 15 ml of nitric acid (4.5), 10 ml of sulphuric acid (4.7) and 4 ml of perchloric acid (4.6), and then heat. Increase the temperature to evaporate the solution until there is a strong emission of white fumes (see Note 1). Cool and dilute with water to about 400 ml.

NOTE 1 The elimination of the organic content of the precipitate and filter normally takes 20 min to 30 min.

Add 10 ml of citric acid solution (4.18), neutralize with ammonia solution (4.13) and re-acidify with hydrochloric acid (4.9). Heat the solution to 90 °C and add 10 ml of dimethylglyoxime solution (4.20) for each 10 mg of nickel present.

Neutralize with ammonia solution (4.13) and add 2 ml excess. Stir well and allow to stand at 80 °C for 2 h. Allow the solution to cool slowly to 50 °C, add 50 ml of ethanol (4.2) (see Note 2), mix well and allow to cool to 35 °C.

NOTE 2 Ethanol is added to compensate for the loss due to evaporation and to prevent the possible separation of dimethylglyoxime at lower temperatures.

### 7.2.4 Gravimetric determination

Collect the precipitate obtained in 7.2.3 on a sintered-glass filter (5.1) which has been dried previously at 110 °C, cooled in a desiccator and weighed at 10 min intervals to constant mass. Wash the beaker and crucible 15 times with the washing water (4.23) at 45 °C. Do not allow the precipitate to become dry at any time during the washing procedure.

Dry the crucible and precipitate at 110 °C for 2 h, cool in a desiccator and weigh at 10 min intervals to constant mass.

Combine the filtrates from 7.2.2 and 7.2.4. Evaporate to a viscous consistency. Add 50 ml of hydrochloric acid ($\rho$ approximately 1,19 g/ml), in 10 ml 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.

Determine the nickel content of the combined filtrates by atomic absorption spectrometry (see Annex C).

The nickel content of the combined filtrates should not exceed 0,2 % (mass fraction) nickel in the original sample.

NOTE 1 High levels of nickel in the filtrates might indicate a faulty crucible.

NOTE 2 If the nickel concentration in the filtrates is high or the sensitivity of the atomic absorption spectrometer is very high, it might be necessary to dilute the solution before the atomic absorption measurements.

### 7.2.5 Titrimetric determination

Dissolve the precipitate obtained in 7.2.2 or 7.2.3 (see the following paragraph) from the paper with alternate small additions of nitric acid (4.11) and warm water and collect the solution in the original precipitation beaker.

When continuing from 7.2.3, carry out the following filtration before proceeding in 7.2.5.

Filter through a rapid 12,5 cm hardened filter paper and wash thoroughly with the cold washing water (4.23).

Transfer to a 250 ml beaker and boil for several minutes. After cooling, add 12 ml of the EDTA.$Na_2$ standard volumetric solution (4.24) for each 10 mg of nickel present and then add 3 ml in excess. Record the total volume of the EDTA.$Na_2$ solution added. Add 15 ml of ammonium acetate solution (4.16) and dilute to about 150 ml with water.

Adjust the pH of the solution to 6,0 ± 0,2 with ammonia solution (4.14) or hydrochloric acid (4.10) and keep for about 10 min. Add several drops of xylenol orange solution (4.26) as indicator, mix and titrate with the zinc standard solution (4.25) to a red-purple end point.

Take the filtrate from 7.2.2 or combine the filtrates from 7.2.2 and 7.2.4. Proceed as described in 7.2.4 from “Add 50 ml of hydrochloric acid ($\rho$ approximately 1,19 g/ml), …” .

# 8 Expression of results

## 8.1 Methods of calculation

### 8.1.1 Gravimetric determination

The nickel content (mass fraction), $c_{Ni}$, expressed as a percentage (%), is given by Formula (2):

$$c_{Ni} = 20{,}32 \times \frac{m_4 - m_3}{m_0} + w_f \quad (2)$$

where

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

$m_3$ is the mass of the empty crucible, in grams;

$m_4$ is the mass of the crucible and the nickel dimethylglyoxime precipitate, in grams;

20,32 is the conversion factor for nickel dimethylglyoxime to nickel, multiplied by 100;

$w_f$ is the nickel content of the filtrates (see Annex C), expressed as a percentage.

### 8.1.2 Titrimetric determination

The nickel content (mass fraction), $c_{Ni}$, expressed as a percentage (%), is given by Formula (3) and Formula (4):

$$c_{Ni} = \frac{\left(c \times V_4\right) - \left(m_2 - V_3\right)}{m_0 \times 10^3} \times 100 + w_f \quad (3)$$

$$c_{Ni} = \frac{\left(c \times V_4\right) - \left(m_2 - V_3\right)}{m_0 \times 10} + w_f \quad (4)$$

where

$c$ is the nickel equivalent of the EDTA.$Na_2$ standard volumetric solution, in milligrams per millilitre;

$V_3$ is the volume of the zinc standard solution consumed, in millilitres;

$V_4$ is the total volume of the EDTA.$Na_2$ standard volumetric solution added, in millilitres;

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

$m_2$ is the mass of nickel corresponding to 1 ml of the zinc standard solution used, in milligrams;

$w_f$ is the nickel content of the filtrates (see Annex C), expressed as a percentage.

## 8.2 Precision

A planned trial of this method was carried out by 12 laboratories for the gravimetric determination and by 10 laboratories for the titrimetric determination, at seven levels of nickel, each laboratory making four determinations of nickel at each level.

The results obtained were treated statistically in accordance with ISO 5725:1986[1].

Annex A gives information on the samples used and on the experimental precision data.

A graphic presentation of the experimental precision data is given in Annex B.

The smoothed precision data, expressed as a percentage (mass fraction), shown in Table 1 were calculated from the relationships between the nickel content mean values and repeatability and reproducibility experimental data (see Annex A and Annex B).

NOTE The precision data summarized in Table 1 do not take into account the determination of the nickel content in the combined filtrates described in 7.2.4, 7.2.5 and Annex C.

**Table 1 — Precision data (smoothed values)**

| Nickel content % (mass fraction) | Gravimetric determination | | Titrimetric determination | |
|---|---|---|---|---|
| | Repeatability $r$ | Reproducibility $R$ | Repeatability $r$ | Reproducibility $R$ |
| 1,0 | 0,035 | 0,030 | 0,015 | 0,018 |
| 2,0 | 0,042 | 0,040 | 0,024 | 0,031 |
| 5,0 | 0,063 | 0,071 | 0,050 | 0,070 |
| 10,0 | 0,097 | 0,123 | 0,093 | 0,134 |
| 15,0 | 0,132 | 0,174 | 0,136 | 0,198 |
| 20,0 | 0,166 | 0,226 | 0,179 | 0,262 |
| 25,0 | 0,201 | 0,277 | 0,222 | 0,326 |
| 30,0 | 0,235 | 0,329 | 0,265 | 0,390 |

## 9 Test report

The test report shall include the following information:

a) all information necessary for the identification of the sample, the laboratory and the date of analysis or of the test report;

b) method used by reference to this International Standard, i.e. ISO 4938;

c) type of determination carried out (gravimetric or titrimetric);

d) results and unit in which they are expressed;

e) any unusual features noted during the determination;

f) any operation not specified in this International Standard or any optional operation which might have influenced the results.

# Annex A
(informative)

# Additional information on the international interlaboratory test

Table 1 was derived from the results of the international trial carried out in 1983 on one pig iron and six steel samples in five countries, involving 12 (gravimetric determination) and 10 (titrimetric determination) laboratories.

The results of the trials were reported in document ISO/TC 17/SC 1 N 598, March 1984. Graphical relationship of the precision data is given in Annex B.

The test samples used are shown in Table A.1.

**Table A.1 — Test samples used for the precision test**

| Sample | Nickel content % (mass fraction) | | |
|---|---|---|---|
| | Certified | Found | |
| | | Gravimetric | Titrimetric |
| **1** ECRM 481–1 (pig iron) | 1,19 | 1,183 | 1,190 |
| **2** JSS 151–7 (low-alloy steel) | 2,99 | 2,959 | 2,985 |
| **3** BAM 230–1 (stainless steel) | 5,55 | 5,555 | 5,576 |
| **4** JSS 653–1 (stainless steel) | 13,91 | 13,897 | 13,935 |
| **5** BCS 334 (stainless steel) | 20,60 | 20,478 | 20,560 |
| **6** NBS 348 (high-alloy steel) | 25,8 | 25,719 | 25,751 |
| **7** JK 37 (stainless steel) | 30,82 | 30,743 | 30,802 |

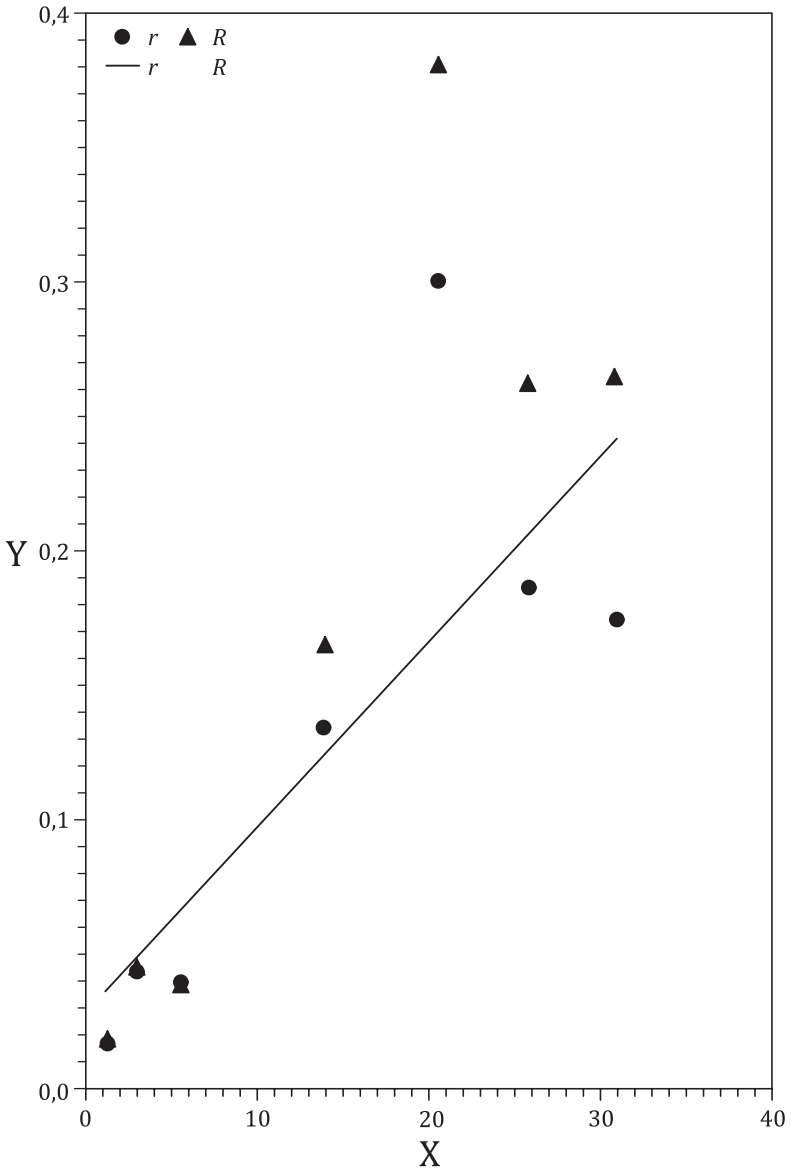
The experimental precision data expressed as a percentage (mass fraction) are shown in Table A.2.

**Table A.2 — Precision data (experimental data)**

| Sample | Gravimetric | | | Titrimetric | | |
|---|---|---|---|---|---|---|
| | Mean value | $r$ | $R$ | Mean value | $r$ | $R$ |
| 1 | 1,183 | 0,017 1 | 0,018 4 | 1,190 | 0,019 5 | 0,022 4 |
| 2 | 2,959 | 0,043 7 | 0,045 5 | 2,985 | 0,045 2 | 0,057 9 |
| 3 | 5,555 | 0,040 1 | 0,040 2 | 5,576 | 0,046 4 | 0,058 1 |
| 4 | 13,897 | 0,134 | 0,165 | 13,935 | 0,090 | 0,137 |
| 5 | 20,478 | 0,300 | 0,380 | 20,560 | 0,225 | 0,338 |
| 6 | 25,719 | 0,186 | 0,261 | 25,751 | 0,211 | 0,363 |
| 7 | 30,743 | 0,174 | 0,264 | 30,802 | 0,271 | 0,356 |

# Annex B
(informative)

# Graphical representation of precision data

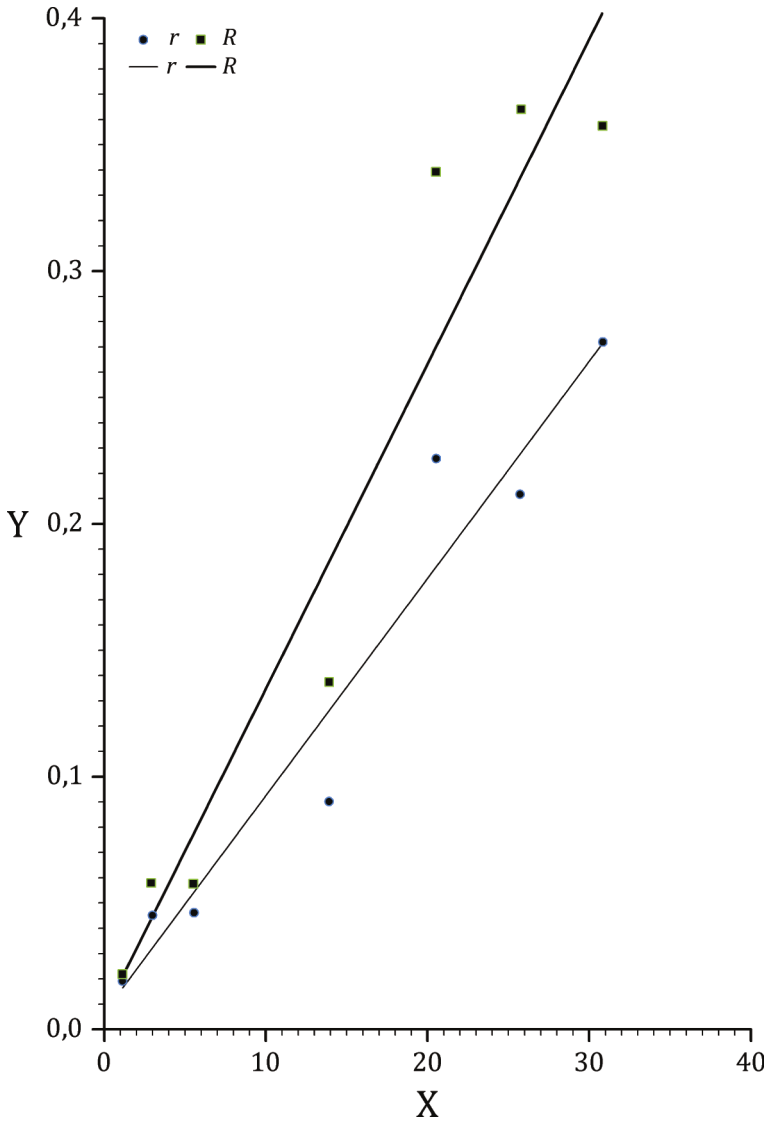



**Key**

X nickel content, % (mass fraction)

Y precision data, %

**Figure B.1 — Relationship between nickel content and repeatability ($r$) and reproducibility ($R$) (gravimetric determination)**




**Key**

X nickel content, % (mass fraction)

Y precision data, %

**Figure B.2 — Relationship between nickel content and repeatability ($r$) and reproducibility ($R$) (titrimetric determination)**

# Annex C
(normative)

# Determination of nickel in combined filtrates by atomic absorption spectrometry

## C.1 General

This Annex describes the procedure for the determination of residual nickel in the combined filtrates obtained during the gravimetric or the titrimetric determination of nickel (see 7.2.2, 7.2.4 or 7.2.5).

## C.2 Reagents

**C.2.1 Hydrochloric acid**, $\rho$ approximately 1,19 g/ml.

**C.2.2 Nickel**, 1g/l standard solution.

Weigh, to the nearest 0,005 g, 1,000 g of nickel metal, [Ni > 99,95 % (mass fraction)], transfer to a 600 ml beaker and dissolve in 40 ml nitric acid (4.5), diluted 1 + 1.

Evaporate to a viscous syrup, cool, re-dissolve the salts in water and transfer to a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix.

1 ml of this standard solution contains 1 mg of nickel.

**C.2.3 Nickel**, 0,1 g/l standard solution.

Transfer 25,0 ml of the nickel standard solution (C.2.2) into a 250 ml one-mark volumetric flask. Make up to the mark with water and mix.

1 ml of this standard solution contains 0,1 mg of nickel.

## C.3 Apparatus

All volumetric glassware shall be grade A in accordance with ISO 648 or ISO 1042, as appropriate.

**Atomic absorption spectrometer**, fitted with an air/acetylene burner and a nickel hollow-cathode lamp.

## C.4 Procedure

### C.4.1 Establishment of the calibration curve

Into each of a series of 200 ml one-mark volumetric flasks, introduce the volumes of nickel standard solution (C.2.3) shown in Table C.1. Add 50 ml of hydrochloric acid (C.2.1), make up to the mark with water and mix.

**Table C.1 — Composition of the calibration solutions**

| Volume of nickel standard solution (C.2.3) ml | Corresponding amount of nickel (7.2.4) mg |
|---|---|
| 0 | 0 |
| 2 | 0,2 |
| 5 | 0,5 |
| 10 | 1,0 |
| 15 | 1,5 |

### C.4.2 Adjustment of the atomic absorption spectrometer

Fit the nickel hollow-cathode lamp (C.3) into the atomic absorption spectrometer (C.3), switch on the current and allow it to stabilize. Adjust the wavelength in the region of 232,0 nm to minimum absorbance. Following the manufacturer's instructions, fit the air/acetylene burner (C.3), light the flame and allow the burner temperature to stabilize. Taking careful note of the manufacturer's instructions regarding the minimum flow rate of acetylene, aspirate the calibration solution of the highest concentration of analyte and adjust the burner configuration and gas flows to obtain maximum absorbance.

### C.4.3 Atomic absorption measurements

Aspirate each of the calibration solutions (C.4.1) in succession, into the flame and measure the absorbance for each. Take care to keep the aspiration rate constant throughout the series of measurements. Aspirate water through the burner after each measurement (see Note below).

NOTE For certain types of apparatus, instead of water, it is preferable to use a solution containing the attack reagents in the same concentrations as in the test solutions.

Aspirate the test solution (7.2.2, 7.2.4 or 7.2.5) and record the absorbance.

Repeat the measurement of calibration solutions and test solution so that the absorbance of each test solution lies between the absorbances of two consecutive calibration solutions.

If the nickel content of the test solution is higher than the highest concentration of the calibration range, the test solution shall be diluted by a known amount and an appropriate correction made.

### C.4.4 Calibration curve

Establish the calibration curve using measured absorbances and corresponding nickel amounts in the calibration solutions. Use appropriate spectrometer software or an offline computer for regression calculations or prepare a graphical representation.

## C.5 Expression of results

Using the calibration curve (C.4.4), read the amount of nickel corresponding to the measured absorbance of the test solution.

The nickel content of the filtrates, $w_f$, to be applied as a correction in 8.1.1 or in 8.1.2, expressed as a percentage (%) of the original sample, is given by Formula (C.1):

$$w_f = \frac{m_5}{m_0 \times 10} \qquad \text{(C.1)}$$

where

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

$m_5$ is the mass of nickel in the filtrate or in the combined filtrates, in milligrams (7.2.2, 7.2.4 or 7.2.5).

NOTE 1 For some atomic absorption spectrometers, it might be necessary to use scale expansion.

NOTE 2 It is not necessary to add dimethylglyoxime to the calibration solutions and evaporate to viscous consistency for mineralization.

# Bibliography

[1] ISO 5725:1986,[1)] *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests*

1) Withdrawn. (Replaced with the ISO 5725 series.)

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