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Nickel oxide — Determination of nickel content — Electrolytic deposition method

Oxyde de nickel — Dosage du nickel — Méthode par dépôt électrolytique

Reference number
ISO 12169:1996(E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 12169 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*, Subcommittee SC 3, *Analysis of nickel and ferronickel.*

Annexes A to C form an integral part of this International Standard.

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Nickel oxide — Determination of nickel content — Electrolytic deposition method

1 Scope

This International Standard specifies a gravimetric method for the determination of nickel content of nickel oxide and partially reduced nickel oxide in the range of 70 % to 95 % (m/m).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

3 Principle

Dissolution of the test portion in a solution of nitric acid and potassium chlorate. Removal of nitric acid by fuming with sulfuric acid. Decomposition of any insoluble residue by fusion with potassium pyrosulfate.

Electrolytic deposition of nickel from an ammoniacal solution and weighing of the deposit.

Determination of co-deposited cobalt and copper by redissolution of the nickel plate and measurement of cobalt and copper by atomic absorption spectrometry. Subtraction of the found mass of cobalt and copper from the mass of the impure nickel deposit.

Determination of residual nickel in the ammoniacal electrolyte solution by atomic absorption spectrometry and addition of the found mass of nickel to the mass of the nickel deposit.

Correction of the net nickel deposit for the moisture content of the sample.

4 Reagents

During analysis, unless otherwise stated, use only reagents of recognised analytical grade and only grade 2 water as specified in ISO 3696.

4.1 Hydrochloric acid, ρ_{20} = 1,18 g/ml, diluted (1+1).

4.2 Sulfuric acid, ρ_{20} = 1,84 g/ml, diluted (1+1).

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

4.4 Hydrofluoric acid, ρ_{20} = 1,13 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.

4.5 Ammonia solution, ρ_{20} = 0,89 g/ml.

4.6 Potassium chlorate, reagent grade, $KClO_3$.

WARNING — Potassium chlorate is a strong oxidant and may explode, if contaminated.

4.7 Potassium pyrosulfate, reagent grade, $K_2S_2O_7$.

4.8 Dimethylglyoxime, solution.

Dissolve 1 g of dimethylglyoxime $[C(:NOH)CH_3]_2$ in 100 ml of methanol.

5 Apparatus

Ordinary laboratory glassware, and

5.1 Analytical balance, semimicro, readability of 0,01 mg.

5.2 Electrolytic deposition assembly (see figure 1).

5.3 Platinum cathode and anode. Preferred perforated plate cathode, height 41 mm, diameter 41 mm. Coil type anode.

6 Sampling and sample preparation

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

6.2 The laboratory sample is normally in the form of a homogeneous powder and no further preparation of the sample is necessary.

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

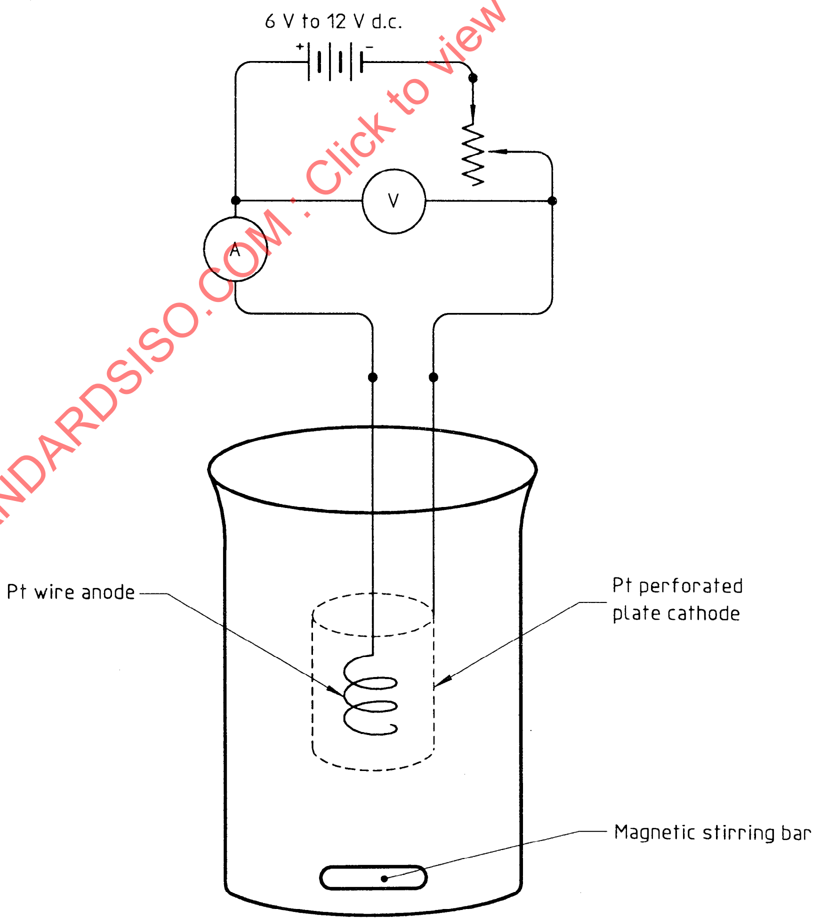

Figure 1 — Electrolytic deposition assembly

7 Procedure

7.1 Test portion and preparation of test solution

7.1.1 Weigh, to the nearest 0,001 g, approximately 2 g of the test sample and transfer it to a tall form 400 ml pyrex beaker.

7.1.2 Add 1 g of potassium chlorate (4.6) and moisten with 20 ml of water, rinsing the inside wall of the beaker. Add 60 ml of nitric acid (ρ_{20} = 1,41 g/ml) and cover the beaker. Heat on a hotplate to apparent dissolution (1 h to 2 h). Remove from the hotplate.

7.1.3 Add another 0,5 g of potassium chlorate (4.6) and 30 ml of nitric acid (ρ_{20} = 1,41 g/ml). Swirl and heat for 1 h. Cool and rinse the walls of the beaker.

7.1.4 Add 25 ml of sulfuric acid (4.2) and evaporate to dryness, but do not bake. To the cooled residue add 40 ml of water and 15 ml of sulfuric acid (4.2). Cover and heat to dissolve the salts.

Do not stir the solution when the anhydrous nickel sulfate solids begin to separate.

NOTE 1 To avoid bumping and mechanical losses in the early fuming stage, it is recommended to cover the hot plate with a glass fibre or asbestos pad. Asbestos-free ZETEX[1] fabric has been found suitable.

7.1.5 Filter any insoluble residue through a Whatman #40 filter paper into a tall form 400 ml beaker and save the filtrate.

NOTE 2 Some special forms of nickel oxide are readily soluble in dilute acids and do not require addition of potassium chlorate. Such oxides are dissolved in 60 ml of the nitric acid (4.3). A clear solution obtained in 7.1.4 does not have to be filtered and is directly processed as directed in clause 7.1.7.

7.1.6 Transfer the filter with the residue to a platinum crucible. Dry, char and ignite to oxidize the carbon. Cool, add 0,5 ml of sulfuric acid (4.2) and 2 ml of hydrofluoric acid (4.4). Carefully evaporate to dryness and fuse the residue with 2 g of potassium pyrosulfate (4.7). Allow the melt to cool and dissolve it in 20 ml to 30 ml of water. Combine with the filtrate from 7.1.5.

7.1.7 Dilute the clear solution to 150 ml and proceed with the electrolytic deposition as directed in 7.2.

7.2 Electrolytic deposition of nickel, cobalt and copper

7.2.1 Place a magnetic stirring bar in the solution (7.1.7) and, while stirring, bring the solution up to 250 ml with the ammonia solution (4.5).

7.2.2 Weigh the clean blank cathode and attach it to the negative buss of the electrolytic assembly with the coil anode already attached. Make sure that the power is off during this operation.

7.2.3 Fully immerse the electrodes in the solution, cover the beaker with a split watchglass, and turn on the assembly exhaust fan (if available). Turn the power on and electrolyse the solution as follows:

0,2 ampere/sample, overnight — no stirring.

Next day rinse the split watchglass and the sides of the beaker with water. Bring up to 250 ml with the ammonia solution (4.5) and, while stirring, continue electrolysis at 0,5 ampere/sample for 30 min.

Increase to 1,0 ampere/sample and electrolyse for 60 min with stirring.

7.2.4 Test for completion of the deposition by withdrawing 1 ml to 2 ml of the electrolyte and placing the solution on a spot plate. Mix with a drop or two of the dimethylglyoxime solution (4.8). If no detectable pink develops, the electrolysis is complete.

7.2.5 Turn off the magnetic stirrer and, without interrupting the current, wash the cathode with water as it is withdrawn from the electrolyte. Rinse the cathode in two successive beakers with methanol and dry it for a few moments at 100 °C. Turn off the power to the electrolytic assembly. Retain the electrolyte and determine the residual nickel content in it as directed in 7.3.

7.2.6 Cool and weigh the cathode, and record the mass. Subtract the mass of the blank cathode (7.2.2) to obtain the net mass of the impure nickel deposit.

NOTE 3 There is evidence that under certain electrolytic conditions, for instance in fast plating, platinum may dissolve from the coil anode and may partly deposit on the cathode. This may be checked by weighing the coil anode before and after the electrolytic deposition procedure. No loss in mass has been found when using the described procedure.

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[1] ZETEX is the trade-name of a product supplied by Newtex Industries Inc., USA. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

7.3 Determination of nickel in spent electrolyte

Evaporate the spent electrolyte (7.2.5) to incipient dryness on a hotplate. Do not bake. Add 20 ml of hydrochloric acid (4.1) and water to dissolve the salts. Transfer the solution to a 250 ml one-mark volumetric flask, make up to the mark with water and mix. Determine the residual nickel content in this solution by atomic absorption spectrometry as directed in annex A.

7.4 Determination of copper and cobalt in electrodeposited nickel

Place the weighed cathode (7.2.6) in a beaker and add 100 ml of nitric acid (4.3). Cover the beaker and heat to dissolve the deposit. Maintain hot for about 30 min. Wash and remove the cathode. Evaporate just to dryness, cool and add 25 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml). Digest in a covered beaker until the frothing ceases. Evaporate just to dryness, cool, add a further 25 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml) and repeat the evaporation. Dissolve the salts in 20 ml of hydrochloric acid (4.1), heating if necessary. Cool and transfer the solution to a 250 ml one-mark volumetric flask. Bring to volume with water and mix. Determine the cobalt and copper contents in this solution by atomic absorption spectrometry as directed in annex B.

7.5 Determination of moisture

Determine the moisture content of the test sample by weighing a separate sample portion in a pre-weighed drying bottle. Weigh, to the nearest 0,000 1 g, approximately 2 g of the test sample. Dry the test portion in the open bottle at 105 °C for 4 h. Cover the bottle, cool it in a desiccator and weigh again. Record the masses before and after the drying.

8 Expression of results

8.1 Calculation

8.1.1 Calculate the moisture content of the test portion, ω_{H_2O}, as a percentage by mass by using the formula:

$$\omega_{H_2O} = \frac{100\,(m_{w1} - m_{w2})}{m_0}$$

where

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

m_{w1} is the mass, in grams, of the weighing bottle and test portion before drying;

m_{w2} is the mass, in grams, of the weighing bottle and test portion after drying.

8.1.2 Calculate the content of nickel in the test portion, ω_{Ni}, as a percentage by mass, corrected for the moisture content, by using the formula:

$$\omega_{Ni} = \frac{10^4\,(m_1 - m_{Cu} - m_{Co} + m_{Ni})}{m_0 \times (100 - \omega_{H_2O})}$$

where

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

m_1 is the mass, in grams, of nickel (plus Cu and Co) plated on the cathode (7.2.6);

m_{Cu} is the mass, in grams, of copper found in the electroplated nickel (7.4);

m_{Co} is the mass, in grams, of cobalt found in the electroplated nickel (7.4);

m_{Ni} is the mass, in grams, of residual nickel found in the electrolyte solution (7.3).

8.2 Precision

8.2.1 Laboratory tests

Nine laboratories in four countries participated in the testing of this procedure using five samples of nickel oxide: NIST-671, NIST-672, NIST-673, S-1003 (acid-soluble black oxide), and S-1900 (refractory sinter).

Each laboratory reported three results for each sample. Two of the three determinations were carried out as duplicates on day 1. The third determination was carried out separately on a different day, day 2.

8.2.2 Statistical analysis

8.2.2.1 Results from the interlaboratory test programme were evaluated according to ISO 5725 [2]. The data were tested for statistical outliers by the Cochran and Grubb tests (see ISO 5725-2).

8.2.2.2 The principle of the Cochran test is that a set of results is an outlier if the within-laboratory variance is too large in relation to others. The Grubb test is to determine if the mean from a laboratory is too far from the other laboratory means. Both tests were applied at the 95 % confidence level.

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[2] ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory test* (now withdrawn), was used to obtain the precision data.

8.2.2.3 From the two values obtained on day 1 the repeatability (r) and reproducibility (R) were calculated at the 95 % confidence level using the procedure specified in ISO 5725 [2]. From the mean value of the duplicates on day 1 and the value obtained on day 2, the within-laboratory reproducibility (R_W) was calculated. The results of this analysis are given in table 1.

8.2.2.4 One laboratory was rejected as a Cochran outlier for sample 5 and as Grubb outliers for samples 3, 4 and 5. Another laboratory was rejected as a Cochran outlier for sample 2 and as a Grubb outlier for sample 1.

9 Interferences

Zinc and cadmium, although not usually present in nickel oxide, would be co-deposited with nickel from ammoniacal solution. If they are known to be present, they shall be determined in the nickel plate solution (7.4) by atomic absorption spectrometry and the mass of impure nickel deposit shall be corrected accordingly.

It has been observed that presence of more than 0,2 mg of Cr(III) in the ammoniacal solution retards the electrodeposition of nickel, especially at high currents and may result in more residual nickel in the spent electrolyte. Contamination of the nickel plate by chromium was, however, insignificant (< 0,01 %).

10 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 replicates;

d) any unusual features noted during the analysis;

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

Table 1 — Results of statistical analysis

Sample	Nickel content, % (m/m) Mean (day 1)	Grand mean (day 1 + day 2)	Precision data Repeatability r	Reproducibility R	R_W
1 (NIST-671)	77,00	76,99	0,316	0,334	0,168
2 (NIST-672)	77,30	77,32	0,068	0,159	0,141
3 (NIST-673)	77,80	77,78	0,099	0,089	0,108
4 (S-1003)	76,85	76,82	0,099	0,203	0,224
5 (S-1900)	76,49	76,49	0,090	0,160	0,126

Annex A
(normative)

Determination of nickel in spent electrolyte by atomic absorption spectrometry

A.1 General

This annex describes the procedure for the determination of residual nickel in the electrolyte after the electrolytic deposition of nickel (see 7.3).

A.2 Reagents

In the analysis use only reagents of recognised analytical grade and only grade 2 water as specified in ISO 3696.

A.2.1 Nickel, standard reference solution (1 000 mg/l).

Weigh, to the nearest 0,001 g, 1 g of nickel metal of 99,9 % (*m*/*m*) minimum purity, transfer to a 600 ml beaker and dissolve in 40 ml of nitric acid (4.3). Evaporate to a viscous syrup, cool and add 20 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml). Digest in a covered beaker until the frothing ceases and evaporate just to dryness. Dissolve the salts in 50 ml of hydrochloric acid (4.1) and transfer the solution to a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix.

A.2.1.1 Nickel, standard solution (100 mg/l).

Pipette 25,0 ml of the nickel standard reference solution (A.2.1) into a 250 ml one-mark volumetric flask and add 10 ml of hydrochloric acid (4.1). Make up to the mark with water and mix.

A.3 Apparatus

Ordinary laboratory glassware, and

A.3.1 Burette, of capacity 25 ml, graduated in divisions of 0,1 ml, in accordance with ISO 385-1, class A.

A.3.2 Volumetric flasks, of capacities 250 ml and 1 000 ml, in accordance with ISO 1042, class A.

A.3.3 Pipette, of capacity 25 ml, in accordance with ISO 648, class A.

A.3.4 Atomic absorption spectrometer, equipped with a burner head for an air-acetylene flame and a nickel hollow cathode lamp. The spectrometer used in this method shall meet the instrument performance parameters given in annex C.

A.4 Procedure

A.4.1 Adjustment of atomic absorption spectrometer

Set the required instrument parameters according to the manufacturer's instructions. Adjust the wavelength to 232,0 nm and the nickel lamp current as recommended. Light the burner and adjust the flows of air and acetylene to obtain an oxidizing, clean, nonluminescent flame while aspirating water. Adjust the instrument to zero.

A.4.2 Preparation of the set of calibration solutions

Transfer, using a burette, 0 ml, 5,0 ml, 10,0 ml, 15,0 ml and 20,0 ml of the nickel standard solution (A.2.1.1) to each of five 250 ml one-mark volumetric flasks. Add 20 ml of hydrochloric acid (4.1), make up to the mark with water and mix. These calibration solutions correspond to 0 mg, 0,5 mg, 1,0 mg, 1,5 mg and 2,0 mg of Ni in the 250 ml volume.

A.4.3 Atomic absorption measurements

A.4.3.1 Aspirate each of the calibration solutions (A.4.2), starting with the zero member and note the absorbance reading. Flush the system by aspirating water between each reading.

A.4.3.2 Aspirate the analysed solution (7.3) and note the absorbance reading. Repeat the measurement of calibration solutions and the test solution such that the measurement of the test solution lies between the measurement of two calibration solutions.

A.4.4 Preparation of the calibration graph

Plot the instrument reading against the nickel content of the calibration solutions (see A.4.3.1).

A.5 Expression of results

A.5.1 Using the calibration curve (A.4.4) read the amount of nickel corresponding to the measured absorbance of the test solution (A.4.3.2).

A.5.2 The nickel content of the spent electrolyte, m_{Ni}, expressed as a mass, in grams, to be applied as a correction in 8.1.2, is given by the formula

$$m_{\mathrm{Ni}} = C_{\mathrm{Ni}} \times 10^{-3}$$

where C_{Ni} is the mass, in milligrams, of nickel found in the test solution (7.3).

NOTES

4 For some atomic absorption instruments it may be necessary to use scale expansion.

5 If the nickel content of the analysed solution (7.3) is high, the solution may be diluted and an appropriate correction made.

Annex B
(normative)

Determination of cobalt and copper in electrodeposited nickel by atomic absorption spectrometry

B.1 General

This annex describes the procedure for the determination of cobalt and copper in the electrolytically deposited nickel (see 7.4).

B.2 Reagents

In the analysis use only reagents of recognised analytical grade and only grade 2 water as specified in ISO 3696.

B.2.1 Cobalt, standard reference solution (1 000 mg/l).

Weigh, to the nearest 0,001 g, 1 g of cobalt metal of 99,9 % (*m/m*) minimum purity and transfer to a 600 ml beaker. Add 30 ml of hydrochloric acid (4.1) and heat to complete dissolution. Cool, transfer to a 1 000 ml one-mark volumetric flask and add 25 ml of hydrochloric acid (4.1). Make up to the mark with water, mix and store in a polyethylene bottle.

B.2.1.1 Cobalt, standard solution (100 mg/l).

Pipette 50,0 ml of the cobalt standard reference solution (B.2.1) into a 500 ml one-mark volumetric flask and add 25 ml of hydrochloric acid (4.1). Make up to the mark with water and mix.

B.2.2 Copper, standard reference solution (1 000 mg/l).

Weigh, to the nearest 0,001 g, 1 g of copper metal of 99,9 % (*m/m*) minimum purity and transfer to a 600 ml beaker. Add 40 ml of nitric acid (4.3) and allow to stand until the reaction ceases. Heat to complete the dissolution and evaporate just to dryness. Cool, add 25 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml), digest in a covered beaker until the frothing ceases and evaporate just to dryness. Dissolve the salts in 50 ml of hydrochloric acid (4.1), heating if necessary. Cool and transfer to a 1 000 ml one-mark volumetric flask. Make up to the mark with water, mix and store in a polyethylene bottle.

B.2.2.1 Copper, standard solution (50 mg/l).

Pipette 25,0 ml of the copper standard reference solution (B.2.2) into a 500 ml one-mark volumetric flask and add 25 ml of hydrochloric acid (4.1). Make up to the mark with water and mix.

B.3 Apparatus

Ordinary laboratory glassware, and

B.3.1 Burette, of capacity 25 ml, graduated in divisions of 0,1 ml, in accordance with ISO 385-1, class A.

B.3.2 Volumetric flasks, of capacities 100 ml, 500 ml and 1 000 ml, in accordance with ISO 1042, class A.

B.3.3 Pipettes, of capacities 10 ml, 20 ml, 25 ml and 50 ml, in accordance with ISO 648, class A.

B.3.4 Atomic absorption spectrometer, equipped with a burner head for an air-acetylene flame and cobalt and copper hollow cathode lamps. The spectrometer used in this method shall meet the instrument performance parameters given in annex C.

B.4 Procedure

B.4.1 Adjustment of atomic absorption spectrometer

Set the required instrument parameters according to the manufacturer's instructions. Adjust the wavelengths to either 240,7 nm for cobalt or 324,8 nm for copper. Set the lamp currents as recommended by the manufacturer. Light the burner and adjust the flows of air and acetylene to obtain an oxidizing, clean, non-luminescent flame while aspirating water. Adjust the instrument to zero.

B.4.2 Preparation of test solutions

B.4.2.1 Determination of cobalt

Transfer an aliquot of 10,0 ml of the solution (7.4) into a 100 ml one-mark volumetric flask. Add 5 ml of hydrochloric acid (4.1), make up to the mark with water and mix.

B.4.2.2 Determination of copper

Transfer an aliquot of 20,0 ml of the solution (7.4) into a 100 ml one-mark volumetric flask. Add 5 ml of hydrochloric acid (4.1), make up to the mark with water and mix.

NOTE 6 If the content of cobalt or copper in the dilute test solution (B.4.2) is high, a smaller aliquot of the original solution (7.4) may be taken.

B.4.3 Preparation of calibration solutions

Transfer, using a burette, 0 ml, 4,0 ml, 8,0 ml, 12,0 ml and 16,0 ml of each of the cobalt and copper standard solutions (B.2.1.1 and B.2.2.1) to each of five 100 ml one-mark volumetric flasks. Add 5 ml of hydrochloric acid (4.1), make up to the mark with water and mix. These calibration solutions correspond to 0 mg, 0,4 mg, 0,8 mg, 1,2 mg and 1,6 mg of cobalt, and 0 mg, 0,2 mg, 0,4 mg, 0,6 mg and 0,8 mg of copper in the 100 ml volume.

B.4.4 Atomic absorption measurement

B.4.4.1 Aspirate each of the calibration solutions (B.4.3) starting with the zero member and note the absorbance reading. Flush the system by aspirating water between each reading.

B.4.4.2 Aspirate the test solutions (B.4.2) and note the respective cobalt and copper absorbance readings. For each element, repeat the measurement of calibration solutions and the test solution such that the measurement of the test solution lies between the measurement of two calibration solutions, respectively.

B.4.5 Preparation of calibration graph

Plot the instrument readings against the cobalt and copper contents of the calibration solutions (B.4.4.1).

B.5 Expression of results

B.5.1 Using the calibration curve (B.4.5) read the amounts of cobalt and copper corresponding to the measured absorbances of the test solutions (B.4.4.2).

B.5.2 Calculate the cobalt and copper contents in the electrodeposited nickel, m_{Co} and m_{Cu}, expressed as a mass, in grams, of cobalt and copper, to be applied as corrections in 8.1, by using the formula

$$m_{\mathrm{Co}} = C_{\mathrm{Co}} \times F \times 10^{-3}$$

$$m_{\mathrm{Cu}} = C_{\mathrm{Cu}} \times F \times 10^{-3}$$

where

C_{Co} is the mass, in milligrams, of cobalt found in the analysed test solution (B.4.2.1);

C_{Cu} is the mass, in milligrams, of copper found in the analysed test solution (B.4.2.2);

F is the volume ratio of the test solution (7.4) and the respective aliquot taken in B.4.2.

Annex C
(normative)

Checking of atomic absorption spectrometer performance parameters

C.1 Introduction

The performance of atomic absorption spectrometers of the same or different manufacture may vary from instrument to instrument. It is therefore essential to establish that a particular instrument meets certain performance requirements before it is used in this International Standard.

C.2 Initial instrument adjustments

C.2.1 Set up the atomic absorption spectrometer to operate with an air/acetylene flame using a single slot (normally about 10 cm) laminar flow burner head according to the manufacturer's instructions.

C.2.2 Use a single element hollow cathode lamp as the light source for the element under test. Operate the source as recommended by the manufacturer.

NOTE 7 The use of multi-element lamps is not generally recommended although some binary alloy lamps give a more stable emission than single element lamps.

C.2.3 Light the burner and aspirate water until thermal equilibrium is reached.

C.2.4 Aspirate a mid-range calibration solution of the element under test and adjust the instrument to give optimum absorption. Use the wavelengths specified in the relevant part of annex A or annex B of this International Standard and the slit setting or bandpass recommended by the instrument manufacturer for the element under test.

C.2.5 Flush the burner system by aspirating hydrochloric acid diluted 1 + 19, adjust the instrument zero and proceed with the performance parameter check as directed in C.3.2 and C.3.3.

C.2.6 The spectrometer shall be used in the absorbance mode.

C.3 Instrument performance check

C.3.1 Performance check solutions

The calibration graphs in annex A and annex B of this International Standard are established using five calibration solutions including the zero member. For the instrument performance check, select two pairs of calibration solutions covering the upper and lower end of the respective calibration graph, such that the interval between the two calibration solutions of highest concentration is equal to that between the "zero" solution and the calibration solution of low concentration.

C.3.2 Linearity of instrument response

C.3.2.1 Aspirate the two calibration solutions of highest concentrations of the element under test, record the instrument readings and calculate the difference.

C.3.2.2 Aspirate the zero member solution and the calibration solution of low concentration of the element under test. Record the instrument readings and calculate the difference.

C.3.2.3 Divide the difference calculated in C.3.2.1 by the difference calculated in C.3.2.2. The linearity of the instrument response is acceptable if the ratio is 0,7 or greater.

C.3.2.4 If the ratio is less than 0,7 further adjustments to the instrument may give acceptable results. Otherwise the operating range of the method shall be reduced by lowering the concentration of the calibration solution of highest concentration.

C.3.3 Minimum stability

C.3.3.1 Aspirate hydrochloric acid diluted 1 + 19 and zero the instrument.

C.3.3.2 Aspirate the calibration solution of highest concentration and record the absorbance.