

INTERNATIONAL STANDARD

**ISO
12169**

First edition
1996-04-01

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)

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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.

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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Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

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, $\rho_{20} = 1,18$ g/ml, diluted (1+1).

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

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

4.4 Hydrofluoric acid, $\rho_{20} = 1,13$ 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, $\rho_{20} = 0,89$ 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, $\text{K}_2\text{S}_2\text{O}_7$.

4.8 Dimethylglyoxime, solution.

Dissolve 1 g of dimethylglyoxime $[\text{C}(\text{:NOH})\text{CH}_3]_2$ 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 riffing.

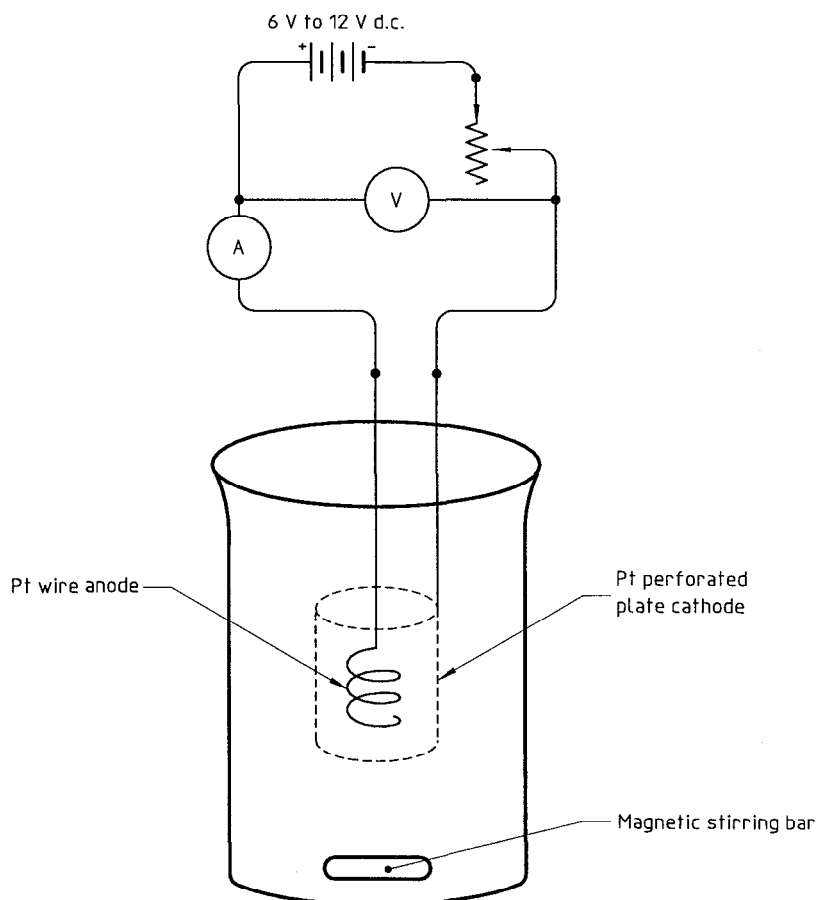


Figure 1 — Electrolytic deposition assembly

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 ($\rho_{20} = 1,41$ 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 ($\rho_{20} = 1,41$ 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 ZETEX¹⁾ 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.

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.