INTERNATIONAL STANDARD

ISO 4938

Second edition
2016-02-01

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

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

Reference number
ISO 4938:2016(E)

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Foreword

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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, ρ approximately 1,05 g/ml.

4.4 Hydrofluoric acid, ρ 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, ρ approximately 1,40 g/ml.

4.6 Perchloric acid, ρ 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, ρ approximately 1,84 g/ml.

4.8 Ammonia solution, ρ approximately 0,90 g/ml.

4.9 Hydrochloric acid, ρ approximately 1,19 g/ml, diluted 1 + 1.

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

4.10 Hydrochloric acid, ρ approximately 1,19 g/ml, diluted 1 + 99.

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

4.11 Nitric acid, ρ 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, ρ 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, ρ 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, ρ 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 (ρ 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.