BS EN ISO 4938:2016

BSI Standards Publication

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

bsi.

...making excellence a habit.™

Document Sharing Hub

National foreword

This British Standard is the UK implementation of EN ISO 4938:2016. It supersedes BS 6200-3.20.1:1989 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee ISE/102, Methods of Chemical Analysis for Iron and Steel.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2016.
Published by BSI Standards Limited 2016

ISBN 978 0 580 81555 3

ICS 77.080.01

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 March 2016.

Amendments/corrigenda issued since publication

Date	Text affected

Documents
Document Sharing Hub

Full document details: BS EN ISO 4938:2016

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 4938

March 2016

ICS 77.080.01

Supersedes EN 24938:1990

English Version

Steel and iron - Determination of nickel content - Gravimetric or titrimetric method (ISO 4938:2016)

Aciers et fontes - Détermination du nickel - Méthode gravimétrique ou titrimétrique (ISO 4938:2016)

Stahl und Eisen - Bestimmung des Nickelanteils - Gravimetrisches oder titrimetrisches Verfahren (ISO 4938:2016)

This European Standard was approved by CEN on 21 November 2015.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

© 2016 CEN All rights of exploitation in any form and by any means reserved worldwide for CEN national Members.

Ref. No. EN ISO 4938:2016 E

Documents
Document Sharing Hub

Full document details: BS EN ISO 4938:2016

European foreword

This document (EN ISO 4938:2016) has been prepared by Technical Committee ISO/TC 17 "Steel" in collaboration with Technical Committee ECISS/TC 102 "Methods of chemical analysis for iron and steel" the secretariat of which is held by SIS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2016, and conflicting national standards shall be withdrawn at the latest by September 2016.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 24938:1990.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Endorsement notice

The text of ISO 4938:2016 has been approved by CEN as EN ISO 4938:2016 without any modification.

Documents
Document Sharing Hub

Full document details: BS EN ISO 4938:2016

Contents

Page

Foreword ..... **iv**
1 Scope **1**
2 Normative references **1**
3 Principle **1**
4 Reagents **1**
5 Apparatus **4**
6 Sampling **5**
7 Procedure **5**
7.1 Test portion 5
7.2 Determination 5
7.2.1 Preparation of the test solution 5
7.2.2 First nickel precipitation 5
7.2.3 Second nickel precipitation 6
7.2.4 Gravimetric determination 7
7.2.5 Titrimetric determination 7
8 Expression of results **8**
8.1 Methods of calculation 8
8.1.1 Gravimetric determination 8
8.1.2 Titrimetric determination 8
8.2 Precision 8
9 Test report **9**
Annex A (informative) **Additional information on the international interlaboratory test** **10**
Annex B (informative) **Graphical representation of precision data** **11**
Annex C (normative) **Determination of nickel in combined filtrates by atomic absorption spectrometry** **13**
Bibliography **16**

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS EN ISO 4938:2016

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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.

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS EN ISO 4938:2016

INTERNATIONAL STANDARD ISO 4938:2016(E)

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 ($NaHSO_4$).

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS EN ISO 4938:2016

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.

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS EN ISO 4938:2016