SLOVENSKI STANDARD
SIST EN ISO 11652:2022

01-maj-2022

Jeklo in železo - Določevanje kobalta - Metoda s plamensko atomsko absorpcijsko spektrometrijo (ISO 11652:1997)

Steel and iron - Determination of cobalt content - Flame atomic absorption spectrometric method (ISO 11652:1997)

Stahl und Eisen - Bestimmung des Cobaltgehaltes - Flammenatomabsorptionsspektrometrisches Verfahren (ISO 11652:1997)

Aciers et fontes - Dosage du cobalt - Méthode par spectrométrie d'absorption atomique dans la flamme (ISO 11652:1997)

Ta slovenski standard je istoveten z: **EN ISO 11652:2022**

ICS:

77.080.10	Železo	Irons
77.080.20	Jekla	Steels

SIST EN ISO 11652:2022 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 11652

March 2022

ICS 77.080.01

English Version

Steel and iron - Determination of cobalt content - Flame atomic absorption spectrometric method (ISO 11652:1997)

Aciers et fontes - Dosage du cobalt - Méthode par spectrométrie d'absorption atomique dans la flamme (ISO 11652:1997)

Stahl und Eisen - Bestimmung des Cobaltgehaltes - Flammenatomabsorptionsspektrometrisches Verfahren (ISO 11652:1997)

This European Standard was approved by CEN on 20 March 2022.

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, 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: Rue de la Science 23, B-1040 Brussels

Contents

Page

European foreword

The text of ISO 11652:1997 has been prepared by Technical Committee ISO/TC 17 "Steel" of the International Organization for Standardization (ISO) and has been taken over as EN ISO 11652:2022 by Technical Committee CEN/TC 459/SC 2 "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 2022, and conflicting national standards shall be withdrawn at the latest by September 2022.

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

Any feedback and questions on this document should be directed to the users' national standards body. A complete listing of these bodies can be found on the CEN website.

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Endorsement notice

The text of ISO 11652:1997 has been approved by CEN as EN ISO 11652:2022 without any modification.

INTERNATIONAL STANDARD

ISO 11652

First edition
1997-08-15

Steel and iron — Determination of cobalt content — Flame atomic absorption spectrometric method

Aciers et fontes — Dosage du cobalt — Méthode par spectrométrie d'absorption atomique dans la flamme

Reference number
ISO 11652:1997(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 11652 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition.*

Annex A forms an integral part of this International Standard. Annexes B and C are for information only.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

Printed in Switzerland

Steel and iron – Determination of cobalt content – Flame atomic absorption spectrometric method

1 Scope

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the cobalt content in steel and iron.

The method is applicable to cobalt contents between 0,003 % (*m*/*m*) and 5,0 % (*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:—[1], *Laboratory glassware — One-mark volumetric flasks.*

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

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

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

ISO 5725-3:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method.*

ISO 14284 :1996, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition.*

3 Principle

Dissolution of a test portion in hydrochloric, nitric and perchloric acids.

Spraying of the solution into an air-acetylene flame.

Spectrometric measurement of the atomic absorption of the 240,7 nm spectral line emitted by a cobalt hollow cathode lamp.

---

1) To be published. (Revision of ISO 1042:1983)

4 Reagents

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

4.1 Pure iron, containing less than 0,000 3 % (*m*/*m*) cobalt.

4.2 Pure nickel, containing less than 0,000 3 % (*m*/*m*) cobalt.

4.3 Hydrochloric acid, ρ about 1,19 g/ml.

4.4 Nitric acid, ρ about 1,40 g/ml.

4.5 Perchloric acid, ρ about 1,67 g/ml.

4.6 Cobalt, standard solutions.

4.6.1 Standard solution A, corresponding to 1,0 g of Co per litre.

Weigh, to the nearest 0,001 g, 1,000 g of metallic cobalt [purity > 99,9 % (*m*/*m*) Co]. Transfer to a 250 ml beaker. Add 15 ml of water and 15 ml of nitric acid (4.4). Cover the beaker with a watch-glass, heat gently until complete dissolution has taken place and boil to remove oxides of nitrogen.

Cool to room temperature, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution A contains 1,0 mg of Co.

4.6.2 Standard solution B, corresponding to 0,2 g of Co per litre.

Transfer 20,0 ml of the standard solution A (4.6.1) to a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution B contains 0,2 mg of Co.

4.6.3 Standard solution C, corresponding to 0,08 g of Co per litre.

Transfer 8,0 ml of the standard solution A (4.6.1) to a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.

Prepare this standard solution C immediately before use.

1 ml of this standard solution C contains 0,08 mg of Co.

5 Apparatus

All volumetric glassware shall be class A, in accordance with ISO 385-1, ISO 648 or ISO 1042 as appropriate.

Ordinary laboratory apparatus, and

5.1 Atomic absorption spectrometer, equipped with a cobalt hollow cathode lamp and supplied with air and acetylene sufficiently pure to give a steady clear fuel-lean flame, free from water and oil, and free from cobalt.

The atomic absorption spectrometer used will be satisfactory if, after optimization according to 7.3.4, the limit of detection and characteristic concentration are in reasonable agreement with the values given by the manufacturer and if it meets the precision criteria given in 5.1.1 to 5.1.3.

It is also desirable that the instrument should conform to the additional performance requirements given in 5.1.4.

5.1.1 Minimum precision (see A.1)

Calculate the standard deviation of 10 measurements of the absorbance of the most concentrated calibration solution. The standard deviation shall not exceed 1,5 % of the mean absorbance.

Calculate the standard deviation of 10 measurements of the absorbance of the least concentrated calibration solution (excluding the zero member). The standard deviation shall not exceed 0,5 % of the mean absorbance of the most concentrated calibration solution.

5.1.2 Limit of detection (see A.2)

This is defined as twice the standard deviation of 10 measurements of the absorbance of a solution containing the appropriate element of a concentration level selected to give an absorbance just above that of the zero member.

The limit of detection of cobalt in a matrix similar to the final test solution shall be better than 0,05 µg of Co per millilitre, for wavelength 240,7 nm.

5.1.3 Graph linearity (see A.3)

The slope of the calibration graph covering the top 20 % of the concentration range (expressed as a change in absorbance) shall not be less than 0,7 times the value of the slope for the bottom 20 % of the concentration range (expressed as a change in absorbance) determined in the same way.

For instruments with automatic calibration using two or more standards, it shall be established prior to the analysis, by obtaining absorbance readings, that the above requirements for graph linearity are fulfilled.

5.1.4 Characteristic concentration (see A.4)

The characteristic concentration of cobalt in a matrix similar to the final test portion solution shall be better than 0,3 µg of Co per millilitre, for wavelength 240,7 nm.

5.2 Ancillary equipment

A strip chart recorder and/or digital readout device is recommended to evaluate the criteria of 5.1.1 to 5.1.3 and for all subsequent measurements.

Scale expansion may be used until the noise observed is greater than the readout error and is always recommended for absorbances below 0,1. If scale expansion has to be used and the instrument does not have the means to read the value of the scale expansion factor, the value can be calculated by measuring the absorbances of a suitable solution with and without scale expansion and simply dividing the signal obtained.

A background corrector equipped with a deuterium hollow cathode lamp is advisable for the analysis of highly alloyed steels, in order to eliminate interference from an FeO molecular absorption band at the cobalt wavelength.

6 Sampling

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

7 Procedure

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