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Steel and iron — Review of available methods of analysis

Aciers et fontes — Vue d'ensemble des méthodes d'analyse disponibles



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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.

The main task of technical committees is to prepare International Standards, but in exceptional circumstances a technical committee may propose the publication of a Technical Report of one of the following types:

- type 1, when the required support cannot be obtained for the publication of an International Standard, despite repeated efforts;
- type 2, when the subject is still under technical development or where for any other reason there is the future but not immediate possibility of an agreement on an International Standard;
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Technical Reports of types 1 and 2 are subject to review within three years of publication, to decide whether they can be transformed into International Standards. Technical Reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 9769, which is a Technical Report of type 3, was prepared by Technical Committee ISO/TC 17, *Steel*.

This third edition cancels and replaces the second edition (ISO/TR 9769:1990),

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Steel and iron — Review of available methods of analysis

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The main task of ISO technical committees is to prepare International Standards. In exceptional circumstances a technical committee may propose the publication of a technical report of one of the following types:

- type 1, when the necessary support within the technical committee cannot be obtained for the publication of an International Standard, despite repeated efforts;
- type 2, when the subject is still under technical development, requiring wider exposure;
- type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical reports are accepted for publication directly by ISO Council. Technical reports types 1 and 2 are subject to review within three years of publication, to decide if they can be transformed into International Standards. Technical reports type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 9769 was prepared by Technical Committee ISO/TC 17, Steel.

It was decided to publish this document in the form of a technical report type 3.

This technical report was revised in accordance with Resolution No.1/88 taken at the 16th Plenary Meeting of ISO/TC 17 held in 1988-10-10/14.

1 Scope

This Technical Report aims to facilitate reference to the available international standard method(s) for the determination of required element(s) in steel and iron.

In this Technical Report, field of application, method of determination (principle) and precision (see informative annex A) of each standard are stated.

2 List of International Standards

2.1 Al Aluminium

Reference	Ed	Pages	Title
ISO 9658: 1990	1	11	Steel - Determination of aluminium content - Flame atomic absorption spectrometric method

2.2 As Arsenic

to be prepared.

2.3 B Boron

ISO 10153: 1991	1	10	Steel - Determination of boron content - Curcumin spectrophotometric method
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2.4 Bi Bismuth

to be prepared.

2.5 C Carbon

ISO 437:1982	1	4	Steel and cast iron - Determination of total carbon content - Combustion gravimetric method
ISO/TR 4830-4 :1978	1	8	Steel - Determination of low carbon contents - Part 4: Coulometric method after combustion
ISO 9556:1989	1	8	Steel and iron - Determination of total carbon content - Infrared absorption method after combustion in an induction furnace

2.6 Ca Calcium

ISO/DIS 10697-1			Steel - Determination of calcium content by flame atomic absorption spectrometry - Part 1: Determination of acid-soluble calcium content
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2.7 Ce Cerium

to be prepared.

2.8 Co Cobalt

under preparation.

2.9 Cr Chromium

Reference	Ed	Pages	Title
ISO 4936:1984	1	3	Steel and cast iron - Determination of chromium content - Diphenylcarbazide spectrophotometric method
ISO 4937:1986	1	8	Steel and iron - Determination of chromium content - Potentiometric or visual titration method
ISO/DIS 10138			Steel and iron - Determination of chromium content - Flame atomic absorption spectrometric method

2.10 Cu Copper

ISO 4943:1985	1	6	Steel and cast iron - Determination of copper content - Flame atomic absorption spectrometric method
ISO 4946:1984 (Corrected and reprinted:1986)	1	6	Steel and cast iron - Determination of copper content - 2,2'-Diquinolyl spectrophotometric method

2.11 Mg Magnesium

to be prepared.

2.12 Mn Manganese

ISO 629:1982	1	4	Steel and cast iron - Determination of manganese content - Spectrophotometric method
ISO/TR 10281 :1990	1	10	Steel and iron - Determination of manganese content - Flame atomic absorption spectrometric method

2.13 Mo Molybdenum

Reference	Ed	Pages	Title
ISO 4941:1978	1	4	Steels and cast irons - Determination of molybdenum content - Photometric method

2.14 N Nitrogen

ISO 4945:1977	1	6	Steel - Determination of nitrogen content - Spectrophotometric method
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2.15 Nb Niobium

ISO 9441:1988	1	6	Steel - Determination of niobium content - PAR spectrophotometric method
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2.16 Ni Nickel

ISO 4938:1988	1	8	Steel and iron - Determination of nickel content - Gravimetric or titrimetric method
ISO 4939:1984 (Corrected and reprinted:1986)	1	6	Steel and cast iron - Determination of nickel content - Dimethylglyoxime spectrophotometric method
ISO 4940:1985	1	7	Steel and cast iron - Determination of nickel content - Flame atomic absorption spectrometric method

2.17 O Oxygen

to be prepared.

2.18 P Phosphorus

ISO 2732:1984	2	3	Steel and cast iron - Determination of phosphorus content - Phosphovanadomolybdate spectrophotometric method
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2.19 Pb Lead

to be prepared.

2.20 S Sulfur

Reference	Ed	Pages	Title
ISO 671:1982	1	5	Steel and cast iron - Determination of sulfur content - Combustion titrimetric method
ISO 4934:1980	1	5	Steel and cast iron - Determination of sulfur content - Gravimetric method
ISO 4935:1989	1	7	Steel and iron - Determination of sulfur content - Infrared absorption method after combustion in an induction furnace

2.21 Sb Antimony

under preparation.

2.22 Si Silicon

ISO 439:1982	1	3	Steel and cast iron - Determination of total silicon - Gravimetric method
ISO 4829-1:1986	1	7	Steel and cast iron - Determination of total silicon content - Reduced molybdosilicate spectrophotometric method - Part 1: Silicon contents between 0,05 and 1,0 %
ISO 4829-2:1988	1	5	Steel and iron - Determination of total silicon content - Reduced molybdosilicate spectrophotometric method - Part 2: Silicon contents between 0,01 and 0,05 %

2.23 Sn Tin

to be prepared.

2.24 Ta Tantalum

to be prepared.

2.25 Ti Titanium

ISO/DIS 10280			Steel and iron - Determination of titanium content - Di antipyrilmethane spectrophotometric method
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2.26 V Vanadium

Reference	Ed	Pages	Title
ISO 4942:1988	1	6	Steel and iron - Determination of vanadium content - N-BPHA spectrophotometric method
ISO 4947:1986	1	6	Steel and cast iron - Determination of vanadium content - Potentiometric titration method
ISO 9647:1989	1	8	Steel and iron - Determination of vanadium content - Flame atomic absorption spectrometric method

2.27 W Tungsten

to be prepared.

2.28 Zr Zirconium

to be prepared.

3 Synopsis of method

3.1 Al Aluminium

3.1.1 ISO 9658: 1990 Flame atomic absorption spectrometric method

(1) Scope

Determination of acid-soluble and/or total aluminium contents between 0,005 % ($\frac{m}{m}$) and 0,20 % ($\frac{m}{m}$) in non-alloyed steel.

(2) Definition

Acid-soluble aluminium for the method is defined as the aluminium dissolved in the hydrochloric and nitric acids mixture as specified in the procedure.

(3) Principle

Dissolution of a test portion in dilute hydrochloric and nitric acids.

Fusion of the acid-insoluble material with orthoboric acid-potassium carbonate mixture.

Spraying of the solution into a dinitrogen monoxide acetylene flame.

Spectrometric measurement of the atomic absorption of the 309,3 nm spectral line emitted by an aluminium hollow cathode lamp.

(4) Precision: see informative annex A.

Figure A.1.1; Precision for the determination of acid-soluble aluminium content

Figure A.1.2; Precision for the determination of total aluminium content

3.2 As Arsenic

to be prepared.

3.3 B Boron

3.3.1 ISO 10153: 1991 Curcumin spectrophotometric method

(1) Scope

Determination of boron contents between 0,000 5 % (m/m) and 0,012 % (m/m) in steel.

(2) Principle

Dissolution of a test portion in hydrochloric and nitric acids.

Decomposition of boron compounds (nitrides etc.) with orthophosphoric and sulfuric acids at a temperature of 290°C.

Formation of a coloured complex between orthoboric acid and curcumin in buffered acetic acid medium.

Spectrophotometric measurement at a wavelength of about 543 nm.

(3) Precision: see information annex A.

Figure A.3.1; Precision for the determination of boron content by ISO 10153

3.4 Bi Bismuth

to be prepared.

3.5 C Carbon

3.5.1 ISO 437: 1982, Combustion gravimetric method

(1) Scope

Determination of total carbon contents not less than 0,1 % (m/m) in steel and cast iron.

(2) Principle

Combustion of a test portion at a high temperature (1 200°C to 1 350°C) in a current of pure oxygen, if necessary in the presence of a flux and transformation of carbon into carbon dioxide.

Absorption of the carbon dioxide carried by current of oxygen in soda asbestos contained in a weighed absorption bulb, and determination of the increase in mass.

3.5.2 ISO/TR 4830-4: 1978, Part 4: Coulometric method after combustion

(1) Scope

Determination of carbon contents between 0,005 % ($\underline{\underline{m}}/\underline{\underline{m}}$) and 0,1 % ($\underline{\underline{m}}/\underline{\underline{m}}$) in steels.

(2) Principle

Combustion of a test portion at a high temperature (> 1 250°C; see note below) in a current of pure oxygen, converting the carbon to carbon dioxide.

Entrainment of the carbon dioxide by the current of oxygen, and separation from the other volatile oxides.

Absorption of the carbon dioxide in a barium hydroxide solution containing barium perchlorate: during the absorption, re-establishment of the initial barium hydroxide concentration by electrolysis of the barium perchlorate, the variation in the pH of the solution being followed by a pH meter.

Measurement of the quantity of electricity required for re-establishment of the initial pH, in order to deduce the quantity of barium hydroxide formed and hence the quantity of carbon dioxide neutralized.

The pH of the absorbing solution must always be equal to or greater than 9,0.

NOTE - Certain types of apparatus are incapable of obtaining the temperature of 1 250°C necessary to comply with the requirements of this Technical Report.

3.5.3 ISO 9556: 1989, Infrared absorption method after combustion in an induction furnace

(1) Scope

Determination of total carbon contents between 0,003 % ($\underline{\underline{m}}/\underline{\underline{m}}$) and 4,5 % ($\underline{\underline{m}}/\underline{\underline{m}}$) in steel and iron.

(2) Principle

Combustion of a test portion with accelerator at a high temperature in an induction furnace in a current of pure oxygen and transformation of carbon into carbon dioxide and/or carbon monoxide.

Measurement by infrared absorption of the carbon dioxide and/or carbon monoxide carried by a current of oxygen.

(3) Precision: see informative annex A.

Figure A.5.1; Precision for the determination of total carbon content by ISO 9556

3.6 Ca Calcium

3.6.1 ISO/DIS 10697-1:Flame atomic absorption spectrometric method
- Part 1: Determination of acid-soluble calcium content

(1) Scope

Determination of acid soluble calcium contents between 0,0005 % ($\frac{m}{m}$) and 0,003 % ($\frac{m}{m}$) in steel.

(2) Principle

Dissolution of a test portion in hydrochloric and nitric acids.

Addition of potassium chloride-lanthanum nitrate solution as a spectrochemical buffer.

Spraying of the solution into a dinitrogen monoxide-acetylene flame.

Spectrometric measurement of the atomic absorption of the 422,7 nm spectral line emitted by a calcium hollow cathode lamp.

(3) Precision: see informative annex A.

Figure A.6.1; Precision for the determination of acid-soluble calcium content by ISO/DIS 10697-1.

3.7 Ce Cerium

to be prepared.

3.8 Co Cobalt

under preparation.

3.9 Cr Chromium

3.9.1 ISO 4936: 1984, Diphenylcarbazide spectrophotometric method

(1) Scope

Determination of chromium contents between 0,01 % (m/m) and 0,25 % (m/m) in steel and cast iron.

(2) Principle

Dissolution of a test portion in nitric and perchloric acids.

Oxidation of chromium in a phosphoric perchloric medium by potassium permanganate.

Oxidation of diphenylcarbazide by chromium(VI).

Spectrophotometric measurement of the oxidized form of diphenylcarbazide at a wavelength of about 540 nm.

3.9.2 ISO 4937: 1986, Potentiometric or visual titration method

(1) Scope

Determination of chromium contents between 0,25 % (m/m) and 35 % (m/m) in steel and iron. (If vanadium is present, the visual titration is applicable only to test portions containing less than 3 mg of vanadium.)

(2) Principle

Dissolution of a test solution with appropriate acids.

Oxidation of chromium in an acid medium to chromium(VI) by ammonium peroxydisulfate in the presence of silver sulfate.

Reduction of manganese(VII) by hydrochloric acid.

Reduction of chromium(VI) by ammonium iron(II) sulfate standard solution.

In the case of potentiometric detection, determination of the equivalence point by measurement of the potential variation when the ammonium iron(II) sulfate standard solution is being added.

In the case of visual detection, titration of the excess ammonium iron(II) sulfate by potassium permanganate standard solution which also acts as the indicator.

(3) Precision: see informative annex A.

Figures A.9.1 and A.9.2; Precision for the determination of chromium content by ISO 4937

3.9.3 ISO/DIS 10138: Flame atomic absorption spectrometric method

(1) Scope

Determination of chromium contents between 0,002 % ($\underline{m}/\underline{m}$) and 2,0 % ($\underline{m}/\underline{m}$) in non-alloy and low-alloy steels and iron.

(2) Principle

Dissolution of a test portion in hydrochloric acid, followed by oxidation with nitric acid.

Filtration and ignition of the acid insoluble residue
Removal of silica with hydrofluoric acid.

Fusion of the residue with potassium hydrogen sulfate, extraction of the melt in acid and addition of the extract to the reserved filtrate.

Spraying the solution into a dinitrogen monoxide-acetylene flame.

Spectrometric measurement of the atomic absorption of the 357,9 nm or 425,4 nm spectral line emitted by a chromium hollow cathode lamp.

(3) Precision: see informative annex A.

Figure A.9.3; Precision for the determination of chromium content by ISO/DIS 10138

3.10 Cu Copper

3.10.1 ISO 4943: 1985, Flame atomic absorption spectrometric method

(1) Scope

Determination of copper contents between 0,004 % ($\underline{m}/\underline{m}$) and 0,5 % ($\underline{m}/\underline{m}$) in steel and cast iron.

(2) Principle

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

Spraying of the solution into an air-acetylene flame.

Spectrometric measurement of the atomic absorption of the 324,7 nm spectral line emitted by a copper hollow cathode lamp.

(3) Precision: see informative annex A.

Figure A.10.1; Precision for determination of copper content by ISO 4943

3.10.2 ISO 4946: 1984, 2,2'-Diquinolyl spectrophotometric method

(1) Scope

Determination of copper contents between 0,02 % ($\frac{m}{m}$) and 5 % ($\frac{m}{m}$) in steel and cast iron.

(2) Principle

Dissolution of a test portion with appropriate acids.

Fuming with perchloric acid to remove hydrochloric and nitric acids and dehydrate silicic acid.

Reduction of copper(II) to copper(I) in hydrochloric acid solution by means of ascorbic acid.

Formation of a coloured compound of copper(I) with 2,2'-diquinolyl.

Spectrophotometric measurement at a wavelength of about 545 nm.

(3) Precision: see informative annex A.

Figure A.10.2; Precision for determination of copper content by ISO 4946

3.11 Mg Magnesium

to be prepared.

3.12 Mn Manganese

3.12.1 ISO 629: 1982, Spectrophotometric method

(1) Scope

Determination of manganese contents between 0,01 % ($\frac{m}{m}$) and 4 % ($\frac{m}{m}$) in steel and cast iron.

(2) Principle

Dissolution of a test portion in sulfuric-phosphoric acid mixture and nitric acid.

Treatment of the test solution with perchloric acid.

Formation of the permanganate ion by sodium periodate.

Spectrophotometric measurement of the test solution at wavelength of about 545 nm.

3.12.2 ISO/TR 10281: Flame atomic absorption spectrometric method

(1) Scope

Determination of manganese contents between 0,002 % (m/m) and 3,0 % (m/m) in non-alloy and low-alloy steels and iron.

(2) Principle

Dissolution of a test portion with hydrochloric acid followed by oxidation with nitric acid.

Ignition of the residue and removal of silica with hydrofluoric acid.

Fusion of the residue with potassium hydrogen sulfate.

Determination of the manganese by means of the spectrometric measurement of the atomic absorption of the 403,1 nm line emitted by a manganese hollow cathode lamp when the solution is sprayed into an air acetylene flame.

For low levels of manganese the more sensitive line of 279,5 nm may be used.

(3) Precision

Figure A.12.1; Precision for determination of manganese content by ISO/TR 10281

3.13 Mo Molybdenum

3.13.1 ISO 4941: 1978, Photometric method

(1) Scope

Determination of molybdenum contents between 0,003 % (m/m) and 9 % (m/m) in steel and cast iron.

Vanadium and tungsten interfere with the measurement if, because of their contents, the V/Mo ratio is greater than 16 or the W/Mo ratio is greater than 8.

NOTE - Greater V/Mo or W/Mo ratios (up to 300) may, however, be permitted, but in such cases it is necessary to carry out the measurement very quickly after the extraction.

(2) Principle

Dissolution of a test portion in an appropriate mixture of acids and decomposition of the carbides by oxidation.