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TECHNICAL REPORT

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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

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- 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).

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

This second edition cancels and replaces the first edition (ISO/TR 9769 : 1988), of which it constitutes a minor revision.

Annexes A and B of this Technical Report are for information only.

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Steel and iron — Review of available methods of analysis

1 Scope and field of application

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		11	Steel - Determination of aluminium content - Flame atomic absorption spectrometric method

2.2 As Arsenic

to be prepared

2.3 B Boron

under preparation

2.4 Bi Bismuth

to be prepared

2.5 C Carbon

ISO 437:1982		4	Steel and cast iron - Determination of total carbon content - Combustion gravimetric method
ISO/TR 4830-1 :1978		6	Steel - Determination of low carbon contents - Part I: Manometric (low-pressure) method after combustion
ISO/TR 4830-2 :1978		10	Steel - Determination of low carbon contents - Part II: Titrimetric method after combustion
ISO/TR 4830-3 :1978		8	Steel - Determination of low carbon contents - Part III: Conductimetric measurement after combustion
ISO/TR 4830-4 :1978		8	Steel - Determination of low carbon contents - Part IV: Coulometric method after combustion
ISO 9556:1989		8	Steel and iron - Determination of total carbon content - Infrared absorption method after combustion in an induction furnace

2.6 Ca Calcium

under preparation

2.7 Ce Cerium

to be prepared

2.8 Co Cobalt

to be prepared

2.9 Cr Chromium

Reference	Ed	Pages	Title
ISO 4936:1984		3	Steel and cast iron - Determination of chromium content - Diphenylcarbazide spectrophotometric method
ISO 4937:1986		8	Steel and iron - Determination of chromium content - Potentiometric or visual titration method

2.10 Cu Copper

ISO 4943:1985		6	Steel and cast iron - Determination of copper content - Flame atomic absorption spectrometric method
ISO 4946:1984 (Corrected and reprinted:1986)		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		4	Steel and cast iron - Determination of manganese content - Spectrophotometric method
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2.13 Mo Molybdenum

ISO 4941:1978		4	Steels and cast irons - Determination of molybdenum content - Photometric method
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2.14 N Nitrogen

Reference	Ed	Pages	Title
ISO 4945:1977		6	Steel - Determination of nitrogen content - Spectrophotometric method

2.15 Nb Niobium

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

ISO 4938:1988		8	Steel and iron - Determination of nickel content - Gravimetric or titrimetric method
ISO 4939:1984 (Corrected and reprinted:1986)		6	Steel and cast iron - Determination of nickel content - Dimethylglyoxime spectrophotometric method
ISO 4940:1985		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		5	Steel and cast iron - Determination of sulphur content - Combustion titrimetric method
ISO 4934:1980		5	Steel and cast iron - Determination of sulphur content - Gravimetric method
ISO 4935:1989		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		3	Steel and cast iron - Determination of total silicon - Gravimetric method
ISO 4829-1:1986		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		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

under preparation

2.26 V Vanadium

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

(1) Scope

Determination of acid-soluble and/or total aluminium content between 0,005 % (m/m) and 0,20 % (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

under preparation

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 content 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 4830-1: 1978, Part I: Manometric (low-pressure) method after combustion

(1) Scope

Determination of carbon content between 0,002 % (m/m) and 0,1 % (m/m) in steel

(2) Principle

Combustion of a test portion at a high temperature (1 200°C to 1 350°C, depending on the type of material) in a current of pure oxygen, converting the carbon to carbon dioxide

Entrainment of the carbon dioxide by the current of oxygen, elimination of oxides of sulphur and of water vapour, and separation of the carbon dioxide by solidification in a freezing trap immersed in liquid oxygen

After elimination of oxygen from the measuring system, vaporization of the carbon dioxide, by raising the temperature, into a calibrated volume which has been evacuated

Determination of the carbon content of the test portion by measuring the pressure in the volume which has previously been calibrated using known volumes of carbon dioxide

3.5.3 ISO/TR 4830-2: 1978, Part II: Titrimetric method after combustion

(1) Scope

Determination of carbon content between 0,005 % (m/m) and 0,1 % (m/m) in pure iron and steel

(2) Principle

Combustion of a test portion in an electric resistance furnace in a current of oxygen, in the presence of a fluxing agent which encourages the combustion, converting the carbon to carbon dioxide, which is absorbed in a mixture of pyridine and 2-aminoethanol or of formdimethylamide and 2-aminoethanol, and which is at the same time titrated using thymol blue as indicator

Calculation of the carbon content from the consumption of the standard volumetric solution; this solution being standardized against calcium carbonate (primary standard), which is heated to a temperature between 1 250°C and 1 400°C in the same furnace

3.5.4 ISO/TR 4830-3: 1978, Part III: Conductimetric method after combustion

(1) Scope

Determination of carbon content between 0,005 % (m/m) and 0,1 % (m/m) in steels

(2) Principle

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

Entrainment of the carbon dioxide by the current of oxygen, separation from the other volatile oxides and absorption by a dilute solution of sodium hydroxide

Deduction of the carbon content of the test portion from measurement of the variation in conductivity of the sodium hydroxide solution with respect to a reference

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

(1) Scope

Determination of carbon content between 0,005 % (m/m) and 0,1 % (m/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.

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

(1) Scope

Determination of total carbon content between 0,003 % (m/m) and 4,5 % (m/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

under preparation

3.7 Ce Cerium

to be prepared

3.8 Co Cobalt

to be prepared

3.9 Cr Chromium

3.9.1 ISO 4936: 1984, Diphenylcarbazide spectrophotometric method

(1) Scope

Determination of chromium content 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 content 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

3.10 Cu Copper

3.10.1 ISO 4943: 1985, Flame atomic absorption spectrometric method

(1) Scope

Determination of copper content between 0,004 % (m/m) and 0,5 % (m/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 content between 0,02 % (m/m) and 5 % (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 content between 0,01 % (m/m) and 4 % (m/m) in steel and cast iron

(2) Principle

Dissolution of a test portion in sulphuric-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

3.13 Mo Molybdenum

3.13.1 ISO 4941: 1978, Photometric method

(1) Scope

Determination of molybdenum content 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

Quantitative formation of a coloured compound of molybdenum, in the presence of thiocyanate, iron(II) and/or copper(II) ions and extraction of this compound using n-butyl acetate

Photometric measurement of the coloured compound at a wavelength of about 470 nm

NOTE - When the conditions of the procedure are respected, the coefficient of molecular absorption is $18\,930 \pm 60$.

3.14 N Nitrogen

3.14.1 ISO 4945: 1977, Spectrophotometric method

(1) Scope

Determination of nitrogen content between 0,002 % (m/m) and 0,050 % (m/m) in non-alloy and low-alloy steels