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

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



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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.

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 10138 was prepared by Technical Committee ISO/TC 17, *Steel*.

The publication of this International Standard gives rise to the cancellation of ISO 4936:1984.

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

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

1 Scope

This International Standard specifies a flame atomic absorption spectrometric method for the determination of chromium in non-alloy and low-alloy steels and iron.

The method is applicable to chromium contents between 0,002 % (*m/m*) and 2,0 % (*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 377-2:1989, *Selection and preparation of samples and test pieces of wrought steels — Part 2: Samples for the determination of the chemical composition*.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements*.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks*.

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

ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests*.

3 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.

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 2 % (*m/m*) chromium.

4.2 Potassium hydrogen sulfate (KHSO_4).

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

4.4 Nitric acid, ρ about 1,40 g/ml.

4.5 Hydrofluoric acid, ρ about 1,15 g/ml.

4.6 Sulfuric acid, ρ about 1,84 g/ml.

4.7 Sulfuric acid, ρ about 1,84 g/ml, diluted 1 + 3.

4.8 Background solution.

Weigh, to the nearest 0,01 g, 10,00 g of the pure iron (4.1) into a 1 litre beaker. Add 10 g of potassium hydrogen sulfate (4.2) and 150 ml of hydrochloric acid (4.3). Heat gently until dissolved, then oxidize with 30 ml of nitric acid (4.4) added in small portions. Allow to cool, transfer to a 250 ml one-mark volumetric flask, dilute to the mark with water and mix.

25 ml of this background solution contains 1,000 g of Fe.

4.9 Chromium, standard solutions.

4.9.1 Stock solution, corresponding to 1,0 g of Cr per litre.

Weigh, to the nearest 0,001 g, 1,000 g of metallic chromium [purity > 99,9 % (*m/m*) Cr]. Place in a 250 ml beaker, add 40 ml of hydrochloric acid (4.3), cover the beaker with a watch-glass and dissolve by heating. Cool to room temperature, transfer the solution quantitatively to a 1000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this stock solution contains 1,0 mg of Cr.

4.9.2 Standard solution A, corresponding to 400 mg of Cr per litre.

Transfer 100,0 ml of the stock solution (4.9.1) to a 250 ml one-mark volumetric flask, dilute to the mark with water and mix.

Prepare this standard solution immediately before use.

1 ml of this standard solution contains 400 µg of Cr.

4.9.3 Standard solution B, corresponding to 100 mg of Cr per litre.

Transfer 10,0 ml of the stock solution (4.9.1) into a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.

Prepare this standard solution immediately before use.

1 ml of this standard solution contains 100 µg of Cr.

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 Platinum crucible, of capacity 30 ml.

5.2 Atomic absorption spectrometer.

A chromium hollow cathode lamp; supplies of dinitrogen monoxide and acetylene sufficiently pure to give a steady clear fuel-lean flame, free from water and oil, and free from chromium.

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 5.2.1 to 5.2.3.

It is also desirable that the instrument should conform to the additional performance requirements given in 5.2.4.

5.2.1 Minimum precision (see clause 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.

5.2.2 Limit of detection (see clause 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 chromium in a matrix similar to the final test portion solution shall be better than 0,05 µg of Cr per millilitre for wavelength 357,9 nm and better than 0,15 µg of Cr per millilitre for wavelength 425,4 nm.

5.2.3 Graph linearity (see clause 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.