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

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



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Foreword

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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 10700 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.

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

1 Scope

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

The method is applicable to manganese 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 and nitric acids followed by evaporation with perchloric acid until white fumes appear.

Spraying of the solution into an air-acetylene flame.

Spectrometric measurement of the atomic absorption of the 279,5 nm spectral line emitted by a manganese 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, free from manganese or with a known low manganese content.

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

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

4.4 Nitric acid, ρ about 1,40 g/ml.

4.5 Perchloric acid, ρ about 1,54 g/ml (see note 1).

NOTE 1 Perchloric acid (ρ about 1,67 g/ml) may also be used. 100 ml of perchloric acid (ρ about 1,54 g/ml) is equivalent to 79 ml of perchloric acid (ρ about 1,67 g/ml).

4.6 Background solution.

WARNING — See clause 7.

Weigh, to the nearest 0,01 g, 10,00 g of the pure iron (4.1). Place in a 1 litre beaker. Add 200 ml of hydrochloric acid (4.2), cover the beaker with a watch glass and heat gently until the iron is dissolved. Then oxidize by adding 50 ml of nitric acid (4.4) in small portions. Add 150 ml of perchloric acid (4.5). Heat strongly and evaporate until dense white fumes of perchloric acid appear. Continue fuming for 15 min at a temperature that maintains a steady reflux of white perchloric acid fumes on the walls of the beaker. Allow to cool, add 300 ml of water and heat gently to dissolve the salts.

Transfer quantitatively to a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this background solution contains 0,010 g of Fe.

4.7 Manganese, standard solution.

4.7.1 Manganese stock solution, corresponding to 1,00 g of Mn per litre.

Weigh, to the nearest 0,1 mg, 1,00 g of pure manganese metal [purity $\geq 99,9\%$ (*m/m*)]. Place in a 250 ml beaker and add 40 ml of hydrochloric acid (4.2). Cover with a watch glass and heat gently until the manganese has dissolved. Cool, 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 contains 1,00 mg of Mn.

4.7.2 Manganese standard solution, corresponding to 0,02 g of Mn per litre.

Transfer 20,0 ml of the stock solution (4.7.1) to a 1 000 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 0,02 mg of Mn.

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 manganese 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 manganese.

The atomic absorption spectrometer used will be satisfactory if, after optimization according to 7.3.5, the limit of detection and characteristic concentration are in reasonable agreement with the values given by the manufacturer and 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 of this solution.

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 at a concentration level selected to give an absorbance just above that of the zero member.

The limit of detection of manganese in a matrix similar to the final test portion solution shall be less than 0,02 µg of Mn per millilitre.

5.1.3 Graph linearity (see A.3)

The value of 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.