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Steel — Determination of manganese content — Inductively coupled plasma atomic emission spectrometric method

*Acier — Dosage du manganèse — Méthode par spectrométrie
d'émission atomique de plasma avec couplage inductif*



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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 10278 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 — Determination of manganese content — Inductively coupled plasma atomic emission spectrometric method

1 Scope

This International Standard specifies an inductively coupled plasma atomic emission spectrometric method for the determination of the manganese content in unalloyed steels.

The method is applicable to manganese contents between 0,002 % (*m/m*) and 1,5 % (*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 a mixture of hydrochloric and nitric acids and dilution of the solution to a known volume. If necessary, addition of scandium or yttrium as an internal standard. Nebulization of the solution into an inductively coupled plasma atomic emission spectrometer and measurement of the intensity of the emitted light from manganese at 257,61 nm and, optionally, emitted light from scandium at 361,38 nm or yttrium at 371,03 nm, simultaneously.

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 1 % (*m/m*) of manganese.

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

4.3 Nitric acid, ρ about 1,40 g/ml, diluted 1 + 1.

4.4 Manganese standard solution.

4.4.1 Manganese standard solution A, corresponding to 0,5 g of Mn per litre.

Weigh, to the nearest 0,1 mg, 0,500 g of manganese metal [purity > 99,9 % (*m/m*)] (see note 1) and transfer to a 200 ml beaker. Add 30 ml of hydrochloric acid

(4.2), cover with a watch glass and heat gently to dissolve the manganese. Cool to ambient 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 contains 0,5 mg of Mn.

NOTE 1 High purity manganese metal may contain high levels of oxygen. The oxygen content of the manganese should therefore be checked before it is used.

4.4.2 Manganese standard solution B, corresponding to 0,05 g of Mn per litre.

Transfer 20,0 ml of the manganese standard solution A (4.4.1) to a 200 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,05 mg of Mn.

4.5 Scandium internal standard solution, corresponding to 1 g of Sc per litre.

Weigh, to the nearest 1 mg, 1,534 g of scandium oxide [Sc₂O₃, purity > 99,98 % (m/m)]. Transfer to a 500 ml beaker and dissolve in 40 ml of nitric acid (4.3). Transfer to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this internal standard solution contains 1 mg of Sc.

4.6 Yttrium internal standard solution, corresponding to 1 g of Y per litre.

Weigh, to the nearest 1 mg, 1,270 g of yttrium oxide [Y₂O₃, purity > 99,98 % (m/m)]. Transfer to a 500 ml beaker and dissolve in 50 ml of hydrochloric acid (4.2). Transfer to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this internal standard solution contains 1 mg of Y.

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 emission spectrometer, equipped with an inductively coupled plasma (ICP-AES).

The ICP-AES used will be satisfactory if, after opti-

mization according to 7.4.1, it meets the performance criteria given in 5.1.1 to 5.1.5.

The spectrometer can be either a simultaneous or sequential type. If a sequential spectrometer is equipped with an extra arrangement for simultaneous measurement of the internal standard line (scandium or yttrium), it can be used with the internal standard technique. If the sequential spectrometer is not equipped with this arrangement, an internal standard cannot be used and the alternative technique without an internal standard can be applied.

5.1.1 Practical resolution of the spectrometer (see A.1)

Calculate the bandwidth for Mn 257,61 nm, for Sc 361,38 nm and for Y 371,03 nm. The bandwidth shall be less than 0,040 nm for each line.

5.1.2 Short-term stability

Calculate the standard deviation of ten measurements of the absolute intensity or intensity ratio of the emitted light of the most concentrated calibration solution for manganese. The relative standard deviation shall not exceed 0,9 %.

5.1.3 Long-term stability

Calculate the standard deviation of seven mean values of three measurements of the absolute intensity or intensity ratio of the emitted light of the most concentrated calibration solution for manganese. Each mean value shall be determined every 0,5 h during a total time of 3 h. The relative standard deviation shall not exceed 1,8 % for the absolute intensity technique or 1,2 % for the internal standard technique.

5.1.4 Background equivalent concentration and detection limit (see A.2)

Calculate the background equivalent concentration (BEC) and detection limit (DL) for the analytical line in a solution containing only the analyte element. The value shall be below the values indicated in table 1.

Table 1 — BEC and DL values

Analytical line nm	Background equivalent concentration mg/l	Detection limit mg/l
Mn 257,61	0,6	0,02
Sc 361,38	—	—
Y 371,03	—	—