
**Steel and iron — Determination of
calcium and magnesium — Inductively
coupled plasma atomic emission
spectrometric method**

*Aciers et fontes - Détermination du calcium et du magnésium -
Méthode par spectrométrie d'émission atomique avec plasma induit*





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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 procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

Steel and iron — Determination of calcium and magnesium — Inductively coupled plasma atomic emission spectrometric method

1 Scope

This International Standard specifies a method for determination of calcium and magnesium contents in iron, cast iron, steel, and alloyed steel by inductively coupled plasma (ICP) atomic emission spectrometry.

The method is applicable to the determination of calcium and magnesium contents (mass fraction) in the range of 0,000 5 % to 0,006 % and 0,000 5 % to 0,20 %, respectively.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single-volume pipettes*

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

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

ISO 14284, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*

3 Principle

A test portion is dissolved in a hydrochloric, nitric and hydrofluoric acid mixture and fumed with perchloric acid. Hydrochloric acid, nitric acid, and an internal standard element (if used) are added and the solution is diluted to a known volume. The solution is filtered if necessary, nebulized into an ICP spectrometer and the intensity of the emitted light from each element measured simultaneously with the intensity of the light emitted by the internal standard element.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade with very low calcium and magnesium contents and only grade 2 water as specified in ISO 3696.

4.1 Pure iron, containing less than 0,5 µg/g of calcium and magnesium.

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

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

4.4 Hydrochloric acid, ρ about 1,19 g/ml, diluted 1 + 4.

4.5 Hydrochloric acid, ρ about 1,19 g/ml, diluted 1 + 100.

4.6 Nitric acid, ρ about 1,42 g/ml.

4.7 Hydrofluoric acid, ρ about 1,14 g/ml.

4.8 Perchloric acid, ρ about 1,67 g/ml.

4.9 Acid mixture.

Mix two volumes of hydrochloric acid (4.2), one volume of nitric acid (4.6), and three volumes of water.

4.10 Calcium standard solutions.

4.10.1 Calcium stock solution, corresponding to 1 000 mg of calcium per litre.

Dry several grams of calcium carbonate [purity $\geq 99,9$ % (mass fraction)] in an oven at $100\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for at least 1 h and cool to room temperature in a desiccator. Weigh, to the nearest 0,000 1 g, 2,497 g of the dried product into a 400 ml beaker, add 20 ml of hydrochloric acid (4.3), cover with a watch-glass and heat gently until the product is completely dissolved. Cool to room temperature and transfer the solution into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this stock solution contains 1,000 mg of calcium.

4.10.2 Calcium standard solution A, corresponding to 100 mg of calcium per litre.

Transfer 10,00 ml of calcium stock solution (4.10.1) into a 100 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid (4.4). Dilute to the mark with water and mix.

1 ml of this standard solution contains 0,100 mg of calcium.

4.10.3 Calcium standard solution B, corresponding to 10 mg of calcium per litre.

Transfer 10,00 ml of calcium standard solution A (4.10.2) into a 100 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid (4.4). Dilute to the mark with water and mix.

1 ml of this standard solution contains 0,010 mg of calcium.

4.10.4 Calcium standard solution C, corresponding to 1,0 mg of calcium per litre.

Transfer 10,00 ml of calcium standard solution B (4.10.3) into a 100 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid (4.4). Dilute to the mark with water and mix.

1 ml of this standard solution contains 0,001 mg of calcium.

4.11 Magnesium standard solutions

4.11.1 Magnesium stock solution, corresponding to 1 000 mg of magnesium per litre.

Weigh, to the nearest 0,000 1 g, 1,000 g of pure magnesium [purity $\geq 99,9$ % (mass fraction)] and transfer into a 250 ml beaker. Add 20 ml of water, then add hydrochloric acid (4.3) drop by drop while swirling until the acid action ceases and continue adding hydrochloric acid (4.3) to a total volume of 20 ml. Cover with a watch-glass and heat to boil for 10 min. After cooling, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this stock solution contains 1,000 mg of magnesium.

4.11.2 Magnesium standard solution A, corresponding to 100 mg of magnesium per litre.

Transfer 10,00 ml of magnesium stock solution ([4.11.1](#)) into a 100 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid ([4.4](#)). Dilute to the mark with water and mix.

1 ml of this standard solution contains 0,100 mg of magnesium.

4.11.3 Magnesium standard solution B, corresponding to 10 mg of magnesium per litre.

Transfer 10,00 ml of magnesium standard solution A ([4.11.2](#)) into a 100 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid ([4.4](#)). Dilute to the mark with water and mix.

1 ml of this standard solution contains 0,010 mg of magnesium.

4.11.4 Magnesium standard solution C, corresponding to 1,0 mg of magnesium per litre.

Transfer 10,00 ml of magnesium standard solution B ([4.11.3](#)) into a 100 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid ([4.4](#)). Dilute to the mark with water and mix.

1 ml of this standard solution contains 0,001 mg of magnesium.

4.12 Yttrium internal standard solution, corresponding to 100 mg of yttrium per litre.

Calcine several grams of yttrium oxide [purity $\geq 99,9$ % (mass fraction)] in a muffle furnace at $850\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$ for at least 40 min and then cool to room temperature in a desiccator. Weigh 0,127 0 g of the calcined product into a 400 ml beaker, add 10 ml of hydrochloric acid ([4.3](#)), cover with a watch-glass, and heat gently until the product is completely dissolved. Cool to room temperature and transfer the solution to a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this standard solution contains 0,100 mg of yttrium.

5 Apparatus

All volumetric glassware shall be class A and calibrated in accordance with ISO 385, ISO 648, or ISO 1042, as appropriate.

Ordinary laboratory apparatus and the following shall be used.

5.1 Inductively coupled plasma atomic emission spectrometer

The inductively coupled plasma atomic emission spectrometer used will be satisfactory if, after optimizing according to [7.4](#), it meets the performance criteria given in [5.1.2](#) to [5.1.4](#).

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

5.1.1 Analytical wavelengths

This method does not specify any particular emission line. It is mandatory that each laboratory carefully investigates the wavelengths available on its own equipment to find the most suitable one regarding sensitivity and absence of interferences.

In [Table 1](#), however, several suggestions are given together with possible interferences. These wavelengths have been carefully investigated