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Stål och järn – Bestämning av kalcium och magnesium – Optisk emissionsspektrometri med induktivt kopplat plasma (ISO 13933:2014, IDT)

Steel and iron – Determination of calcium and magnesium – Inductively coupled plasma atomic emission spectrometric method (ISO 13933:2014, IDT)

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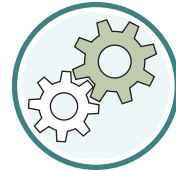
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Foreword

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 $850\text{ °C} \pm 10\text{ °C}$ 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

The wavelength of the internal standard element chosen shall not interfere with the analytical wavelengths, nor should the internal element wavelength be interfered by elements present in the test solution. It is, however, recommended to use Y 371,03 nm or Y 360,07 nm. This wavelength is free of interferences from the elements.

Table 1 — Analytical wavelengths together with interfering elements

Element	Wavelength nm	Possible interferences
Ca	393,66	none
Mg	279,55	none
Y	371,03	none
	360,07	none

Other elements can be used as internal standards, but they shall not be present in the sample, and shall not interfere with the elements to be determined at the appropriate wavelengths. Likewise, the elements present in the test solution shall not interfere with the internal standard at the wavelength chosen. The excitation requirements of the internal standard should be similar to those of the elements to be determined.

5.1.2 Minimum practical resolution of the spectrometer

Calculate the bandwidth, according to [A.1](#), for the wavelength used. The bandwidth shall be less than 0,030 nm.

5.1.3 Minimum short-term precision

Calculate the short-term precision according to [A.2](#). The relative standard deviation (RSD) shall not exceed 1 % of the mean absolute or ratioed intensities for concentrations 100 to 1 000 times the limit of detection (LOD) ([5.1.4](#)). For concentrations that are 10 to 100 times the LOD ([5.1.4](#)), the RSD shall not exceed 5 %.

5.1.4 Limit of Detection (LOD) and Limit of Quantification (LOQ)

Calculate the LOD and LOQ, according to [A.3](#), for the analytical wavelengths used. The calculated values shall be below the values given in [Table 2](#).

Table 2 — Limit of Detection (LOD) and Limit of Quantification (LOQ)

Element	Wavelength nm	LOD mg/l	LOQ mg/l
Ca	393,66	0,003 4	0,010
Mg	279,55	0,004 6	0,014

5.1.5 Linearity of the calibration curve

The linearity of the calibration curve is checked by calculating the correlation coefficient. This coefficient shall be higher than 0,999.

5.2 Polytetrafluoroethylene (PTFE) beakers with PTFE cover

NOTE For the determination of magnesium, glass beakers and watch-glass covers can be used.

5.3 Volumetric flask, of capacity 100 ml, made of polypropylene or polyethylene terephthalate (PET).

5.4 Filter, 0,22 µm pore size, 47 mm diameter PTFE, or polycarbonate filter membrane.

5.5 Suction filtration system, including a flask, a filter funnel, and a vacuum filtration device.

6 Sampling

Sampling shall be carried out in accordance with ISO 14284 or with an appropriate national standard for steels and cast irons.

7 Procedure

WARNING — Perchloric acid vapour can cause explosions in the presence of ammonia, nitrous fumes, or organic matter in general.

All glassware and plasticware shall first be washed with hydrochloric acid (4.3), then with water. The quantities of calcium and magnesium in the glassware and plasticware can be checked by measuring the intensity of the emitted light of water poured into glassware and plasticware after washing with acid and rinsing with water. If contamination from calcium and magnesium is present, the glassware and plasticware are unsuitable and should be replaced or cleaned again.

For each set of determinations, all reagents, including water, calibration and test solutions, shall be from the same batch.

7.1 Test portion

Place the sample or the pure iron in a PTFE beaker. Add 10 ml of hydrochloric acid (4.5) and swirl the beaker gently. Discard the hydrochloric acid solution and rinse the sample thoroughly with water. Repeat the rinsing, firstly with anhydrous ethanol and then with acetone. Once dried, weigh 0,50 g of the test sample to the nearest 0,001 g.

7.2 Blank test

In parallel with the determination and following the same procedure, carry out a blank test using the same quantities of all the reagents as for the test portion including pure iron (4.1).

7.3 Determination

7.3.1 Preparation of the test solution

- a) Place the test portion into a PTFE beaker.
- b) Add 6 ml of hydrochloric acid (4.2) and 3 ml of nitric acid (4.6). Heat gently until the acid action ceases. Add 3 ml of hydrofluoric acid (4.7) and heat at approximately 90 °C for 15 min.
- c) Cool the solution and add 5 ml of perchloric acid (4.8). Heat and evaporate until white fumes appear. Cover the beaker with a PTFE cover, and continue heating at a temperature at which a steady reflux of white perchloric acid fumes is maintained along the walls of the beaker. Continue heating until the residual volume is about 1 ml.

NOTE It is recommended to cover the beaker with a PTFE cover only for samples with high carbon content in order to dissolve the free carbon completely during the perchloric acid fuming step.

- d) Allow to cool, add 10 ml of the mixture of hydrochloric and nitric acid (4.9), and heat gently to dissolve the salts.
- e) Cool the solution to room temperature. Transfer the solution quantitatively into a 100 ml one-mark polypropylene or polyethylene terephthalate volumetric flask (5.3). If the internal standard