
International Standard



4692

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Iron ores — Determination of calcium and/or magnesium contents — Flame atomic absorption spectrometric method

Minerais de fer — Dosage du calcium et du magnésium — 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 approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4692 was developed by Technical Committee ISO/TC 102, *Iron ores*, and was circulated to the member bodies in June 1978.

It has been approved by the member bodies of the following countries :

Australia	India	Poland
Austria	Iran	Romania
Bulgaria	Ireland	South Africa, Rep. of
Canada	Italy	Sweden
Chile	Japan	United Kingdom
Czechoslovakia	Korea, Dem. P. Rep. of	USA
Egypt, Arab Rep. of	Mexico	USSR
France	Netherlands	
Germany, F. R.	New Zealand	

No member body expressed disapproval of the document.

Iron ores — Determination of calcium and/or magnesium contents — Flame atomic absorption spectrometric method

1 Scope and field of application

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the calcium and/or magnesium contents of iron ores.

This method is applicable to a concentration range of 0,01 to 10,0 % of calcium and/or to that of 0,01 to 3,0 % of magnesium in natural iron ores, and iron ore concentrates, and agglomerates, including sinter products.

2 References

ISO 2596, *Iron ores — Determination of hygroscopic moisture in analytical samples.*

ISO 3081, *Iron ores — Increment sampling — Manual method.*

ISO 3083, *Iron ores — Preparation of samples.*

3 Principle

Decomposition of the test portion by treatment with hydrochloric acid with the addition of a small amount of nitric acid.

Evaporation to dehydrate silica, followed by dilution and filtration. Ignition of the residue and removal of silica by evaporation with hydrofluoric and sulphuric acids. Fusion with sodium carbonate and dissolution of the cooled melt in the filtrate.

Aspiration of the solution into the flame of an atomic absorption spectrometer using a dinitrogen oxide-acetylene burner for calcium and an air-acetylene burner for magnesium.

Comparison of absorbance values obtained for calcium and/or magnesium with those obtained from the calibration solutions.

NOTE — If a dinitrogen oxide flame is used for the determination of magnesium, the sensitivity is decreased by a factor of about 3.

4 Reagents

During the analysis, use only reagents of recognized analytical reagent grade, and only distilled water or water of equivalent purity.

4.1 Sodium carbonate (Na_2CO_3), anhydrous.

4.2 Hydrochloric acid, ρ 1,19 g/ml.

4.3 Hydrochloric acid, (ρ 1,19 g/ml) diluted 1 + 9.

4.4 Nitric acid, ρ 1,4 g/ml.

4.5 Hydrofluoric acid, 40 % (m/m), ρ 1,13 g/ml, or 48 % (m/m), ρ 1,185 g/ml.

4.6 Sulphuric acid, (ρ 1,84 g/ml) diluted 1 + 1.

4.7 Background solution.

Dissolve 10 g of iron wire (Fe > 99,9 %) free from calcium and magnesium in 50 ml of hydrochloric acid (4.2) and oxidize by dropwise addition of nitric acid (4.4). Evaporate to syrupy consistency. Add 20 ml of hydrochloric acid (4.2) and dilute to 200 ml with water. Dissolve 17 g of sodium carbonate (4.1) in water, add carefully to the iron solution and heat to remove carbon dioxide. Transfer the cooled solution to a 1 000 ml volumetric flask, dilute to volume with water and mix.

4.8 Lanthanum chloride solution.

Dissolve 50 g of lanthanum chloride ($\text{LaCl}_3 \cdot x\text{H}_2\text{O}$) in 50 ml of hydrochloric acid (4.2) and 300 ml of hot water. Cool and dilute to 1 litre.

4.9 Calcium, standard solution, 25 mg of Ca per litre.

Dissolve 1,248 7 g of dried calcium carbonate (CaCO_3) in 100 ml of hydrochloric acid (ρ 1,19 g/ml), diluted 1 + 3. When dissolution is complete, cool, transfer to a 1 000 ml volumetric flask, dilute to volume with water and mix. Transfer 10 ml of this solution to a 200 ml volumetric flask. Dilute to volume with water and mix.

1 ml of this solution contains 25 μg of Ca.

4.10 Magnesium, standard solution, 15 mg of Mg per litre.

Dissolve 0,300 0 g of oxide-free magnesium metal by slowly adding 75 ml of hydrochloric acid (ρ 1,19 g/ml), diluted 1 + 3. When dissolved, cool, transfer to a 1 000 ml volumetric flask, dilute to volume with water and mix. Transfer 10 ml of this solution to a 200 ml volumetric flask, dilute to volume with water and mix.

1 ml of this solution contains 15 μg of Mg.