

International Standard



4689

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Iron ores — Determination of sulfur content — Barium sulfate gravimetric method

Minerais de fer — Dosage du soufre — Méthode gravimétrique au sulfate de baryum

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4689 was prepared by Technical Committee ISO/TC 102, *Iron ores*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Iron ores — Determination of sulfur content — Barium sulfate gravimetric method

1 Scope and field of application

This International Standard specifies a barium sulfate gravimetric method for the determination of the sulfur content of iron ores.

This method is applicable to a concentration range of 0,01 to 1,0 % (*m/m*) of sulfur in natural iron ores, and iron ore concentrates and agglomerates including sinter products.

2 References

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

ISO 3082, *Iron ores — Increment sampling and sample preparation — Mechanical method*.¹⁾

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

ISO 7764, *Iron ores — Preparation of predried test samples for chemical analysis*.

3 Principle

Decomposition of a test portion by treatment with potassium chlorate and hydrochloric and nitric acids followed by evaporation to dryness. Dissolution of the salts in hydrochloric acid and filtration of the insoluble residue. Removal of the major portion of the iron in the filtrate by extraction with methyl isobutyl ketone.

Ignition of the insoluble residue and removal of silicon dioxide by evaporation with hydrofluoric and nitric acids. Fusion of the residue with sodium carbonate followed by leaching and filtration. Acidification of the filtrate and combination with the main solution.

Reduction of any remaining iron to the bivalent state, adjustment of the acidity and addition of barium chloride solution. Filtration of barium sulfate and gravimetric determination.

4 Reagents

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

4.1 Potassium chlorate (KClO_3), powder.

4.2 Sodium carbonate (Na_2CO_3), anhydrous.

4.3 Zinc.

Use zinc with the lowest available sulfur content and with a particle size of 1 to 3 mm.

4.4 Hydrochloric acid, ρ 1,16 to 1,19 g/ml.

4.5 Hydrochloric acid, ρ 1,16 to 1,19 g/ml, diluted 2 + 1.

4.6 Hydrochloric acid, ρ 1,16 to 1,19 g/ml, diluted 1 + 1.

4.7 Hydrochloric acid, ρ 1,16 to 1,19 g/ml, diluted 2 + 100.

4.8 Nitric acid, ρ 1,4 g/ml.

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

4.10 Acid mixture: hydrochloric acid (4.4) + nitric acid (4.8), 4 + 1.

NOTE — Do not store this mixture; prepare immediately before use.

4.11 Barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$), 100 g/l solution.

Dissolve 100 g of crystalline barium chloride dihydrate ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in 1 litre of water, cover and heat to boiling point. Keep warm on a water bath for a minimum of 2 h and allow to cool to room temperature overnight. Store the solution in a plastics bottle and before each use, filter the required volume through a close-texture filter paper.

4.12 Hydrochloric acid wash solution, containing barium chloride.

Filter 10 ml of barium chloride solution (4.11) through a close-texture filter paper, and dilute to 1 000 ml with hydrochloric acid solution (4.7).

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4.13 Sodium nitrate (NaNO_3), saturated solution.

4.14 Sodium carbonate (Na_2CO_3), 20 g/l solution.

Store in a plastics bottle.

4.15 Ammonium thiocyanate (NH_4SCN), 100 g/l solution.

4.16 Silver nitrate, 20 g/l solution.

4.17 Methyl orange, 0,1 g/100 ml solution.

Dissolve 0,10 g of methyl orange in 100 ml of water.

4.18 Methyl isobutyl ketone [$\text{CH}_3\text{COCH}_2\text{CH}(\text{CH}_3)_2$ (4-Methylpentan-2-one)].

Pre-treat as follows before use : transfer 200 ml of methyl isobutyl ketone and 100 ml of hydrochloric acid solution (4.6) to a 500 ml separating funnel, and shake thoroughly for about 1 min. Allow the layers to separate and discard the lower aqueous layer.

5 Apparatus

Ordinary laboratory apparatus.

6 Sampling and samples

6.1 Laboratory sample

For analysis, use a laboratory sample of minus 100 μm particle size which has been taken in accordance with ISO 3081 or ISO 3082 and prepared in accordance with ISO 3082 or ISO 3083. In the case of ores with significant contents of combined water or oxidizable compounds, use a particle size of minus 160 μm .

NOTE — A guideline on significant contents of combined water and oxidizable compounds is incorporated in ISO 7764.

6.2 Preparation of predried test samples

Thoroughly mix the laboratory sample and, taking multiple increments, extract a test sample in such a manner that it is representative of the whole contents of the container. Dry the test sample at $105 \pm 2^\circ\text{C}$ as specified in ISO 7764. (This is the predried test sample.)

7 Procedure

7.1 Number of determinations

Carry out the analysis at least in duplicate in accordance with annex A, independently, on one predried test sample.

NOTE — The expression “independently” means that the second and any subsequent result is not affected by the previous result(s). For this particular analytical method, this condition implies that the repetition of the procedure shall be carried out either by the same operator at a different time or by a different operator, including, in either case, appropriate recalibration.

7.2 Blank test and check test

In each run, one blank test and one analysis of a certified reference material of the same type of ore shall be carried out in parallel with the analysis of the ore sample(s) under the same conditions. A predried test sample of the certified reference material shall be prepared as specified in 6.2.

NOTE — The certified reference material should be of the same type as the sample to be analysed and the properties of the two materials should be sufficiently similar to ensure that in either case no significant changes in the analytical procedure become necessary.

When the analysis is carried out on several samples at the same time, the blank value may be represented by one test, provided that the procedure is the same and the reagents used are from the same reagent bottles.

When the analysis is carried out on several samples of the same type of ore at the same time, the analytical value of one certified reference material may be used.

7.3 Test portion

Taking several increments, weigh, to the nearest 0,000 2 g, the amount of the predried test sample (6.2) specified in the table.

NOTE — The test portion should be taken and weighed quickly in order to avoid reabsorption of moisture.

Table — Mass of test portion and volume of acid mixture (4.10) required for decomposition

Sulfur content, w_S % (m/m)	Mass of test portion g	Volume of acid mixture ml
$0,01 < w_S < 0,1$	5,0	70
$0,1 < w_S < 0,5$	2,0	40
$0,5 < w_S < 1,0$	1,0	25

7.4 Determination

7.4.1 Decomposition of the test portion

Place the test portion (7.3) in a 500 ml beaker, add 1 g of potassium chlorate (4.1) and 3 to 4 ml of water, and mix thoroughly.

Add, in accordance with the table, the required volume of acid mixture (4.10) and heat gently to decompose the test portion. Evaporate the solution to dryness on a hot-plate heated to about 150°C , cool and add 10 ml of hydrochloric acid (4.4).