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

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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 ($BaCl_2 \cdot 2H_2O$), 100 g/l solution.

Dissolve 100 g of crystalline barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) 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).

1) At present at the stage of draft.