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Iron ores — Determination of sulfur content — Part 1: Barium sulfate gravimetric method

Minerais de fer — Dosage du soufre — Partie 1: Méthode gravimétrique au sulfate de baryum

~~Draft Technical Specification~~

Contents

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 (www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Field Code Changed

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 102, *Iron Ores*, Subcommittee SC 02, *Chemical analysis*.

This second edition cancels and replaces the first edition (ISO 4689:1986), which has been technically revised.

The main ~~change is~~changes are as follows:

— The decomposition method of samples ~~was~~has been changed from the original acid dissolution and alkali melting method to soda-zinc oxide semi melting method.

A list of all parts in the ISO 4689 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

In this ~~revision~~document, a new way of melting the samples for determination of sulfur content by barium sulfate gravimetric method is specified as follows:

— Samples are semi-molten with sodium carbonate and zinc oxide~~;~~.

— The sulfate ions are leached by electromagnetic stirring and separated from the iron and other interference elements such as lead, antimony, bismuth, tin, silicon, titanium, etc.

This method is also suitable for complex natural and artificial sample analysis.

This ~~revision will form~~document consists of Part 1 of the ISO 4689 series, *Iron ores — Determination of sulfur content*.

Iron ores — Determination of sulfur content — Part 1: Barium sulfate gravimetric method

1 Scope

This document 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 a mass fraction of sulfur from 0,01 % to 3,9 % in natural iron ores, iron ore concentrates and agglomerates including sinter products.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2596, *Iron ores — Determination of hygroscopic moisture in analytical samples — Gravimetric, Karl Fischer and mass-loss methods*

ISO 3082, *Iron ores — Sampling and sample preparation procedures*

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

The analysis principle is as follows.

a) Decomposition of a test portion by treatment with a soda-zinc oxide flux mixture to a semi-molten state.

b) Water leaching of sulfate ion with electro-magnetic stirring and filtration of the insoluble residue.

c) Transformation of sulfate ion to barium sulfate through adjustment of the acidity and addition of barium chloride solution. ~~Filtration of barium sulfate and gravimetric determination.~~

d) Filtration of barium sulfate and gravimetric determination.

e) Removal of the interference of Cr, Sn, P through hydrogen peroxide, citric acid and calcium carbonate.

5 Reagents

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

5.1 Sodium carbonate-zinc oxide mixed flux, grind Na_2CO_3 to −100 mesh, blend Na_2CO_3 and ZnO by 3:2 (mass ratio~~)~~.

5.2 Calcium carbonate.

5.3 Hydrofluoric acid, ρ 1,15 g/ml.

5.4 Hydrochloric acid solution, mix ρ=1,15 g/ml hydrochloric acid and distilled water by 1:1 (volume ratio~~)~~.

5.5 Sulfuric acid solution, mix ρ=1,84 g/ml sulfuric acid and distilled water by 1:1 (volume ratio~~)~~.

5.6 Hydrogen peroxide, 30 % (mass fraction~~)~~.

5.7 Ethanol (ethyl~~)~~.

5.8 Citric acid solution, 50 % (mass fraction~~)~~.

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

5.10 Silver nitrate, 10 g/l solution.

5.11 Methyl orange, 1 g/l solution.

5.12 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 l of water, cover and heat to boiling point. Keep warm on a water bath for a minimum of 2 h and allow cooling to room temperature overnight. Store the solution in a plastic bottle and, before each use, filter the required volume through a close-texture filter paper.

5.13 Hydrochloric acid wash solution, containing barium chloride. Filter 10 ml of barium chloride solution (5.12) through a close-texture filter paper, add 40 ml hydrochloric acid (5.4), dilute to about 1,000 ml.

6 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

6.1 Glass plate suction funnel, 150 ml.

An example ~~of a figure~~ is given in Annex D, Figure D.1.

6.2 Water filtration membrane, 0,2 µm.

6.3 Sand core filter, 300 ml.

An example ~~of a figure~~ is given in Annex D, Figure D.2.

7 Sampling and samples

7.1 Laboratory sample

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

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

7.2 Preparation of test samples

7.2.1 General

Depending on the ore type, proceed in accordance with either 7.2.2 or 7.2.3.

7.2.2 Ores having significant contents of combined water or oxidizable compounds

Prepare an air-equilibrated test sample in accordance with ISO 2596 with the following types of ore:

a) Processed ores containing metallic iron;

b) Natural or processed ores in which the sulfur content is higher than 0,2 % mass fraction;

c) Natural or processed ores in which the content of combined water is higher than 2,5 % mass fraction.

7.2.3 Ores outside the scope of 7.2.2

Prepare a predried test sample as follows.

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 ~~±~~ ± 2 ~~°C as specified~~ °C in accordance with ISO 7764.