SLOVENSKI STANDARD
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Železove rude – Določevanje žvepla – 3. del: Metoda s sežiganjem/infrardeča metoda

Iron ores -- Determination of sulfur content -- Part 3: Combustion/infrared method

Minerais de fer -- Dosage du soufre -- Partie 3: Méthode par combustion et infrarouge

Ta slovenski standard je istoveten z: **ISO 4689-3:2004**

ICS:

73.060.10	Železove rude	Iron ores

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INTERNATIONAL STANDARD

ISO 4689-3

First edition
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Iron ores — Determination of sulfur content —

Part 3: Combustion/infrared method

Minerais de fer — Dosage du soufre —

Partie 3: Méthode par combustion et infrarouge

Reference number
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 4689-3 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

This first edition, in part cancels and replaces ISO 4690:1986, which has been technically revised.

ISO 4689 consists of the following parts, under the general title *Iron ores — Determination of sulfur content*:

— *Part 2: Combustion/titration method*

— *Part 3: Combustion/infrared method*

The following part is under preparation:

— *Part 1: Barium sulfate gravimetric method*

Introduction

This part of ISO 4689 is a new procedure. ISO 4689-2 was originally published as ISO 4690:1986, *Iron ores — Determination of sulfur content — Combustion method*. Under a policy of rationalization of the numbering system used in ISO/TC 102, it has been decided to re-designate ISO 4690:1986 as ISO 4689-2. It was further decided to introduce a combustion/infrared method, numbered ISO 4689-3, i.e. this part of ISO 4689.

When next revised, ISO 4689:1986, *Iron ores — Determination of sulfur content — Barium sulfate gravimetric method*, will be re-designated 4689-1.

Iron ores — Determination of sulfur content —

Part 3:
Combustion/infrared method

WARNING — This part of ISO 4689 may involve hazardous materials, operations and equipment. This part of ISO 4689 does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this part of ISO 4689 to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 4689 specifies a combustion/infrared method, using a high-frequency induction furnace, for the determination of the sulfur content of iron ores.

This method is applicable to sulfur contents between 0,002 % (mass fraction) and 0,25 % (mass fraction) in natural iron ores, iron ore concentrates and agglomerates, including sinter products. The method is not applicable to iron ores containing more than 1,0 % (mass fraction) of combined water. The apparatus, of which the metal filter is equipped with a heating device, can be applied to iron ores containing less than 3,0 % (mass fraction) of combined water.

2 Normative references

The following referenced documents are indispensable for the application 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 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements*

ISO 648:1977, *Laboratory glassware — One-mark pipettes*

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*

ISO 3082:2000, *Iron ores — Sampling and sample preparation procedures*

ISO 7764:1985, *Iron ores — Preparation of predried test samples for chemical analysis*

3 Principle

The sample is mixed with a flux containing iron, tin and tungsten and heated in a high-frequency induction furnace, using oxygen as a supporting fuel and carrier gas.

The evolved sulfur dioxide is carried by oxygen into the cell of an infrared detector, where the absorption scale is read.

4 Reagents

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

4.1 Metallic iron, particle size 0,2 mm to 1,2 mm and containing less than 0,001 % sulfur.

4.2 Magnesium perchlorate, $Mg(ClO_4)_2$ anhydrous, free flowing, particle size 0,5 mm to 2 mm.

4.3 Iron(III) oxide, containing less than 0,003 % sulfur.

4.4 Metallic tin, particle size 0,2 mm to 1,2 mm, containing less than 0,001 % sulfur.

4.5 Metallic tungsten, particle size 0,2 mm to 1,2 mm, containing less than 0,001 % sulfur.

4.6 Soda-asbestos, particle size 0,5 mm to 2 mm.

4.7 Sodium hydroxide, particle size 0,5 mm to 2 mm.

4.8 Standard sulfur solution, prepared as follows.

Dry 8 g of potassium sulfate (purity > 99,9 % (mass fraction) to constant mass at 105 °C to 110 °C and cool in a desiccator. Weigh the dried masses of potassium sulfate specified in Table 1 and dissolve each portion in water. Cool, transfer each portion to separate 100 ml volumetric flasks and dilute to volume with water.

Table 1 — Sulfur standard solution (calibration series)

Solution No.	Mass of potassium sulfate	Concentration of sulfur
	g	mg/ml
1	0,543 5	1,00
2	1,087 0	2,00
3	1,902 2	3,50
4	2,717 4	5,00

5 Apparatus

Ordinary laboratory apparatus, including one-mark pipettes and one-mark volumetric flasks complying with the specifications of ISO 648:1977 and ISO 1042 respectively as well as the following.

5.1 Oxygen supply.

5.2 Absorption tower, containing soda-asbestos (4.6) or sodium hydroxide (4.7).

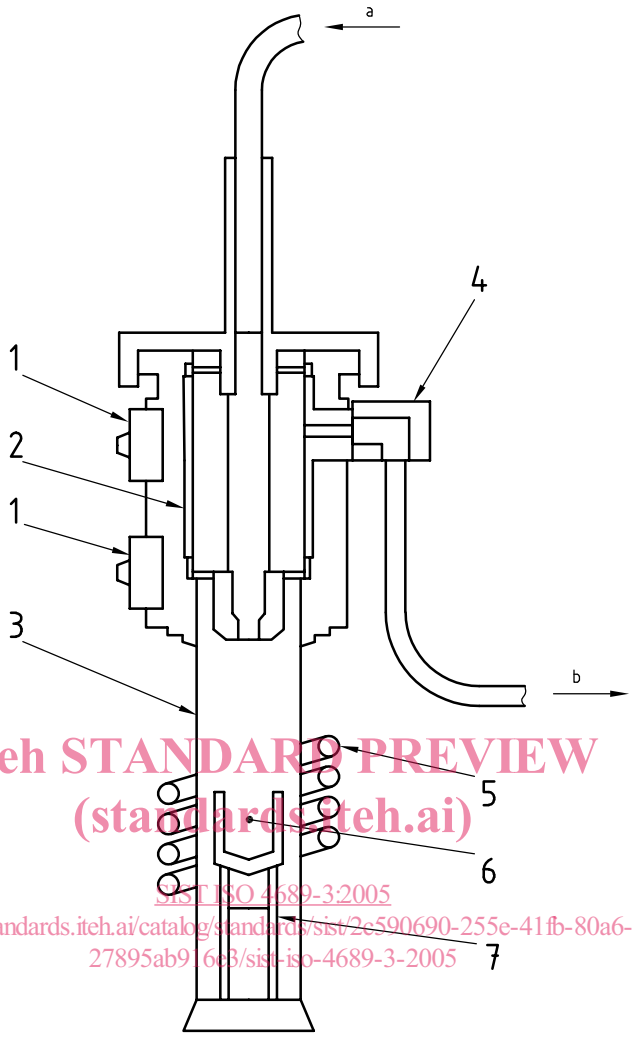
5.3 Drying tower, containing magnesium perchlorate (4.2).

5.4 High-frequency induction furnace, having a heating induction coil (height 30 mm to 55 mm, 4 to 5 rolls), capable of heating to a temperature of 1 200 °C.

An example of a combustion furnace, with combustion gas-refining section, is given in Figure 1.

5.5 Combustion tube, quartz.

5.6 Refractory combustion crucible.



Key

1 metal filter
2 heating device
3 quartz combustion tube
4 dust trap
5 high-frequency induction coil
6 crucible
7 holder

[a] Oxygen.

[b] Combustion gas.

Figure 1 — Example of combustion furnace with combustion gas-refining section