INTERNATIONAL STANDARD

ISO 4689-3

Third edition
2017-03

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

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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 (see 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).

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 voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 4689-3:2015), of which it constitutes a minor revision, with the following changes:

— "0,001 %" has been inserted before "sulfur" in 5.1;

— in 6.7:

  — under "Combustion crucible", "Length" has been changed to "Height";

  — under "Combustion crucible", outer diameter has been changed from "10 mm" to "26 mm";

  — under "Combustion crucible", inner diameter has been changed from "26 mm" to "10 mm";

— "0,2 g" has been added after "sulfur" in 8.2;

— Formula (7) and the relevant descriptions in 9.2.4 have been modified to harmonize this clause across all standards for which ISO/TC 102/SC 2 is responsible.

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

Introduction

ISO 4689-2 was originally published as ISO 4690:1986. 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 document.

When next revised, ISO 4689:1986 will be re-designated as ISO 4689-1.

Iron ores — Determination of sulfur content —

Part 3:
Combustion/infrared method

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

1 Scope

This document 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 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 648, *Laboratory glassware — Single-volume pipettes*

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

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*

ISO Guide 35, *Reference materials — General and statistical principles for certification*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

4 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.

5 Reagents

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

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

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

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

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

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

5.6 Sodium hydroxide coated silica, particle size 0,5 mm to 2 mm.

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

5.8 Standard sulfur solution, prepared as follows.

Dry 8 g of potassium sulfate [purity > 99,99 % (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 volumetric flasks and dilute to volume with water.

Table 1 — Sulfur standard solution (calibration series)

Solution no.	Mass of potassium sulfate g	Concentration of sulfur 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

6 Apparatus

Ordinary laboratory apparatus, including one-mark pipettes and one-mark volumetric flasks complying with the specifications of ISO 648 and ISO 1042, respectively, as well as the following. See Figure 1.

6.1 Oxygen supply.

6.2 Absorption tower, containing soda-asbestos (5.6) or sodium hydroxide (5.7).

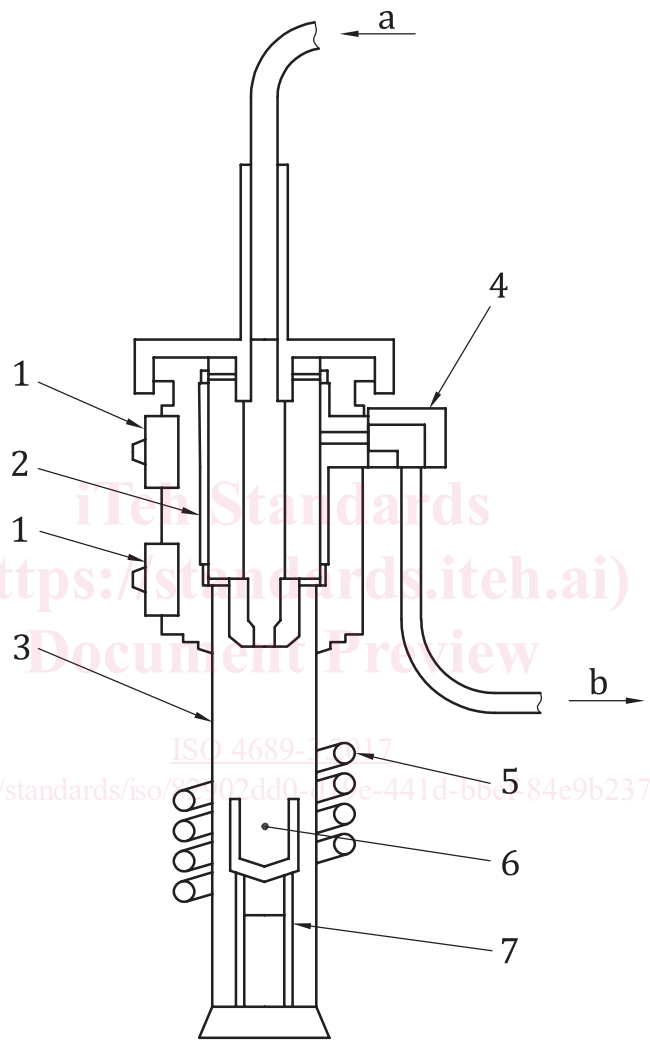
6.3 Drying tower, containing magnesium perchlorate (5.2).

6.4 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.

6.5 Combustion tube, quartz.

6.6 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

6.7 Tin capsule.

The following dimensions may be used as guidelines.

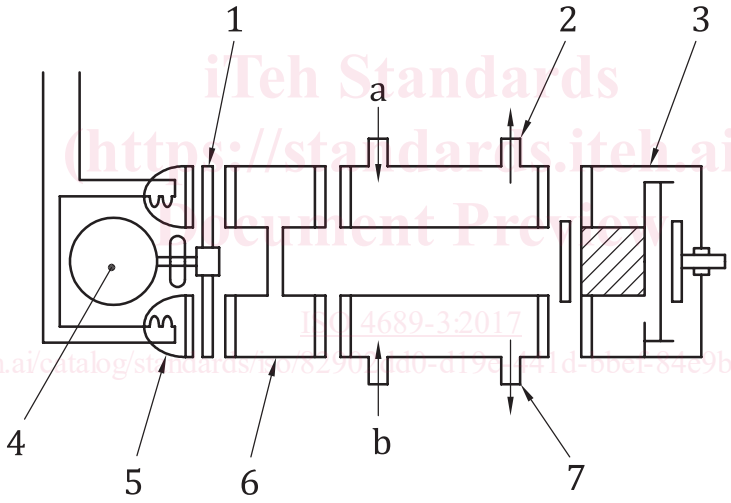
Combustion tube		Combustion crucible		Tin capsule	
Length	140 m to 220 m	Height	26 mm	Diameter	6 mm
Outer diameter	30 mm to 44 mm	Outer diameter	26 mm	Height	18 mm
Inner diameter	26 mm to 37 mm	Inner diameter	10 mm	Mass	0,3 g
				Volume	0,4 ml

6.8 Metal filter.

6.9 Dust trap, containing glass wool.

6.10 Detector for infrared absorption, an example of which is shown in Figure 2.

6.11 Micro-pipette, capacity 100 µl, accurate to 1 µl.



Key

1 chopper
2 measuring cell
3 infrared detector
4 motor
5 infrared source
6 filter cell
7 reference cell
a Combustion gas.
b Oxygen.

Figure 2 — Example of an infrared detection system