INTERNATIONAL STANDARD

ISO 4689-2

Third edition
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Iron ores — Determination of sulfur content —

Part 2: Combustion/titration method

Minerais de fer — Dosage du soufre —

Partie 2: Méthode par combustion et titration

Reference number
ISO 4689-2:2017(E)

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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.

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 4689-2:2015), which constitutes a minor revision with the following changes:

— 6.7: first line, after "approximately", insert "10 g";

— 9.2.4: modify Formula (7) and the relevant descriptions, to harmonize this subclause 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

This document 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 this document as ISO 4689-2. It was further decided to introduce a combustion/infrared method, numbered ISO 4689-3.

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

Iron ores — Determination of sulfur content —

Part 2:
Combustion/titration 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/titration method 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 results are not affected by the presence of fluoride.

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 385, *Laboratory glassware — Burettes*

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 tungsten(VI) oxide and heated at 1 200 °C in a resistance furnace, using nitrogen as a carrier gas.

The evolved sulfur dioxide is absorbed in dilute hydrochloric acid solution containing starch and potassium iodide, and the solution is titrated continuously during evolution with a standard volumetric solution of potassium iodate.

5 Reagents

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

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

5.2 Tungsten(VI) oxide, (WO_3).

5.3 Tin(II) chloride, $SnCl_2·2H_2O$, particle size 0,5 mm to 2 mm.

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

5.5 Hydrochloric acid, ρ, 1,16 g/ml to 1,19 g/ml, diluted 1 + 66.

5.6 Potassium iodide, KI, 3 % (mass fraction) solution.

5.7 Starch, 2 % (mass fraction) solution.

Make a suspension of 2,0 g of starch in 10 ml of water, add to 50 ml of boiling water and stir. Cool, dilute to 100 ml and mix.

Prepare this solution immediately prior to use.

5.8 Potassium iodate, KIO_3, standard volumetric solution, 0,001 042 mol/l.

The potassium iodate should be previously dried for 2 h at 130 °C.

Weigh, to the nearest 0,000 2 g, 0,223 g of dried potassium iodate and dissolve in water. Cool, transfer to a 1 l volumetric flask, dilute to volume and mix.

1 ml of this standard volumetric solution is equivalent to 0,10 mg of sulfur.

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 Nitrogen supply.

6.2 Drying tower, containing sodium hydroxide coated silica (5.4) and magnesium perchlorate (5.1).

6.3 Flowmeter, graduated from 0 to 1 l/min.

6.4 Resistance furnace, capable of maintaining a temperature of 1 200 °C ± 25 °C.

6.5 Refractory combustion tube, capable of withstanding a temperature of 1 200 °C ± 25 °C.

6.6 Refractory combustion boat, with loose-fitting lid, or a suitable refractory combustion boat capable of accommodating a porous cartridge, as shown in Figure 2 a) or b) respectively.

The following dimensions can be used as guidelines.

Combustion boat		Cartridge	
Length	80 mm	Length	50 mm
Width	13 mm	Internal diameter	15 mm
Depth	9 mm		

6.7 Tin(II) chloride absorption tube, of narrow glass tubing containing approximately 10 g of solid tin(II) chloride (5.3), suitably plugged with inert wool to prevent the reagent being blown into the absorption vessel.

A U-tube is suitable. Where necessary, this is inserted in the train at the exit of the combustion tube at point X in Figure 1.

If the sample is known or thought to contain chloride, e.g. in the form of sodium chloride, chlorapatite, or scapolite, the chlorine formed during combustion of the sample should be removed by passing the gas stream through solid tin(II) chloride in a suitable tube or vessel (6.7) prior to absorption and titration. If numerous samples having chloride contents >1 (mass fraction) are to be analysed, it is advisable to determine the absorption capacity of the tin(II) chloride in order to be able to judge the most suitable interval for reagent replacement. In such cases, a larger absorption vessel may be preferred.

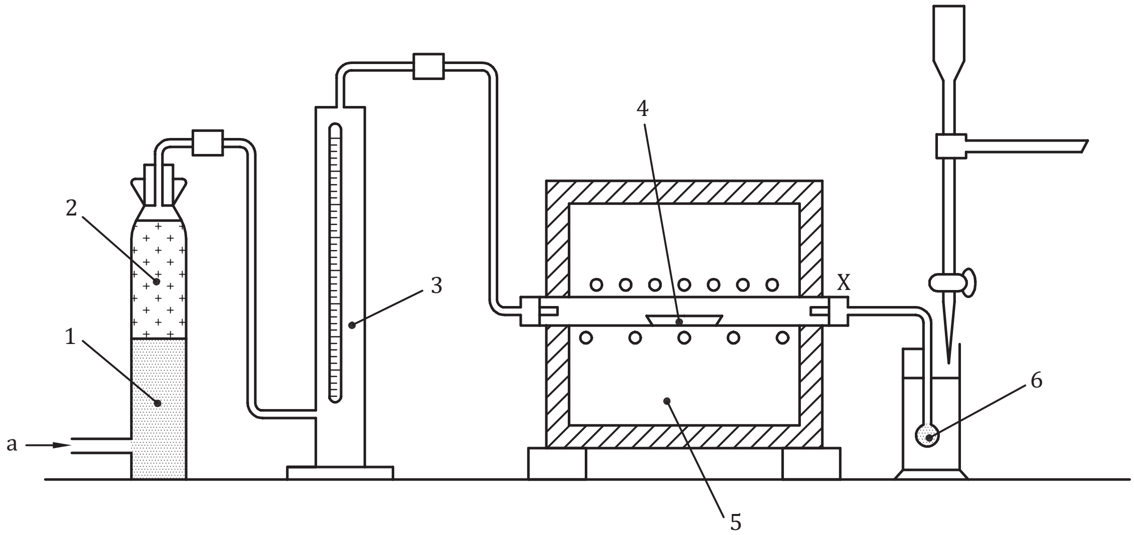
The tin(II) chloride absorption tube should also be fitted if the chloride content of the test sample is unknown.

6.8 Cylinder, 100 ml, tall form, to hold the absorbing solution.

6.9 Bubbler, multi-hole, to reach to the bottom of the cylinder (6.8).

6.10 Burette, conforming to ISO 385, graduated in divisions of 0,05 ml.

For determination of low sulfur contents [<0,005 % (mass fraction)], alternative titration devices such as a piston burette or a suitable micro-syringe capable of delivering small, precise aliquots of titrant may be used.



Key

1 sodium hydroxide coated silica

2 anhydrous magnesium perchlorate

3 flowmeter

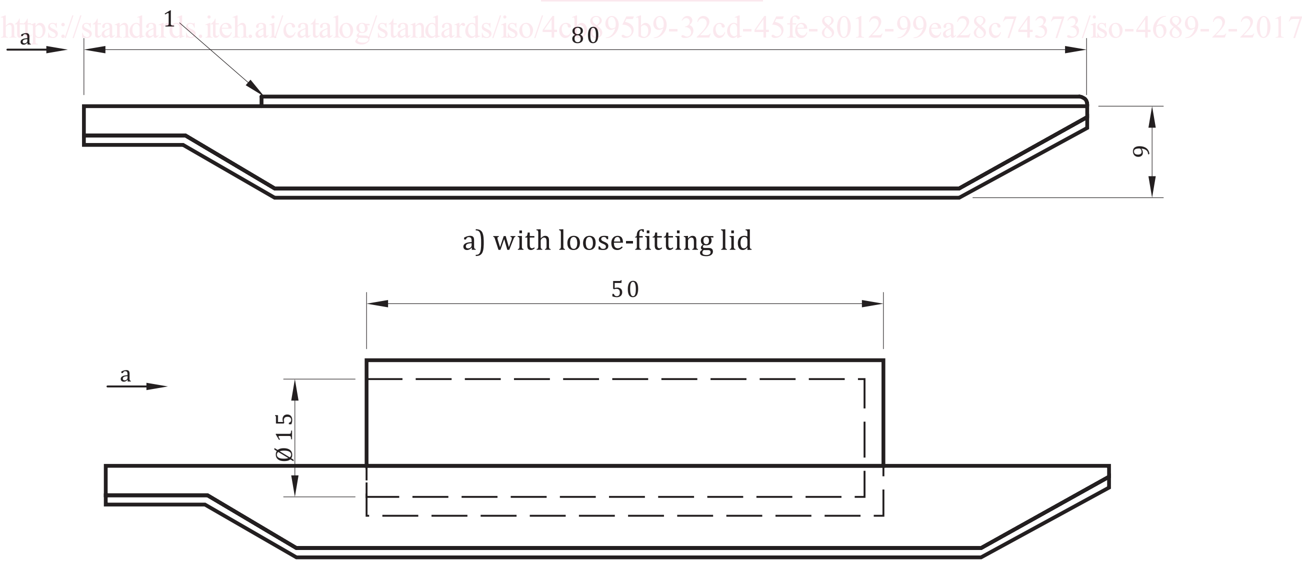
4 combustion boat

5 resistance furnace

6 multihole bubbler

a Nitrogen.

Figure 1 — Combustion apparatus



Key

1 loose-fitting lid

a Gas flow.

Figure 2 — Combustion boat

