PD ISO/TR 9686:2017

BSI Standards Publication

Direct reduced iron — Determination of carbon and/or sulfur — High-frequency combustion method with infrared measurement

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National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Direct reduced iron — Determination of carbon and/or sulfur — High-frequency combustion method with infrared measurement

Minerais de fer préréduits — Dosage du carbone et/ou du soufre — Méthode par combustion haute fréquence et mesurage par infrarouge

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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).

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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 first edition Technical Report cancels and replaces the second edition International Standard (ISO 9686:2006), which has been technically revised and has been converted to a Technical Report because it is not suitable for determination of carbon or sulfur as a referee method.

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Direct reduced iron — Determination of carbon and/or sulfur — High-frequency combustion method with infrared measurement

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.

1 Scope

This document specifies a method for the determination of the mass fraction of carbon and/or sulfur in direct reduced iron by infrared measurement after high-frequency combustion.

This method is applicable to mass fractions of carbon between 0,05 % and 2,5 %, and/or mass fractions of sulfur between 0,001 % and 0,05 % in direct reduced iron.

2 Normative references

There are no normative references in this document.

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 test portion is combusted in a refractory crucible in a flow of oxygen in the presence of an accelerator, the crucible being inserted in the combustion tube of a high-frequency (HF) furnace.

The carbon present is converted into carbon dioxide and the sulfur into sulfur dioxide. Each gas is measured by infrared absorption, with calibration using barium carbonate and potassium sulfate.

5 Reagents

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

5.1 Oxygen, minimum purity 99,5 % (mass fraction).

The pressure in the furnace should be controlled by a pressure regulator designed especially for the purpose and complying with the manufacturer's specification.

5.2 Magnesium perchlorate, grain size 0,7 mm to 1,2 mm.

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5.3 Accelerator, tungsten (granular form) with known low mass fractions of carbon (<0,002 %) and sulfur (<0,000 5 %).

5.4 Pure iron, or iron of known low mass fractions of carbon and sulfur, as in 5.3.

5.5 Tin capsules, of capacity 0,3 ml, diameter 5 mm, length 17 mm.

5.6 Barium carbonate ($BaCO_3$), fine powder.

Dry at 105 °C for 3 h and cool in a desiccator.

5.7 Sulfur standard solutions.

Dry potassium sulfate (K_2SO_4) at 105 °C for 1 h and cool in a desiccator.

Weigh, to the nearest 0,000 2 g, the amounts of potassium sulfate specified in Table 1.

Transfer to five 100 ml one-mark volumetric flasks, dissolve with 50 ml of water, dilute to volume and mix.

5.8 Ascarite, used only for carbon determination.

Table 1 — Sulfur standard solutions

Sulfur standard solution	Mass of K_2SO_4 g	Sulfur concentration mg/ml
SS 1	0,217 4	0,4
SS 2	0,434 8	0,8
SS 3	0,652 2	1,2
SS 4	0,869 6	1,6
SS 5	2,174 0	4,0

6 Apparatus

Ordinary laboratory apparatus, including micropipettes and one-mark volumetric flasks complying with the specifications of ISO 7550 and ISO 1042, respectively, and the following.

6.1 Commercial carbon/sulfur apparatus

The apparatus required for HF combustion of the samples and the subsequent infrared absorption measurement of evolved carbon dioxide and/or sulfur dioxide may be obtained commercially from a number of manufacturers. Common features of such systems are discussed in Annex C.

The manufacturer's instruction for the operation of their equipment should be followed.

6.2 Ceramic combustion crucibles, required for containing the sample and any additions that may be necessary for the subsequent combustion.

The crucibles should be of precise dimensions for the system, and should adapt to the supporting pedestal post, so that the sample in the crucible will be positioned at the correct height within the induction coil when it is in the raised position.

These crucibles should be pre-ignited in an oxygen flow, in a furnace, for not less than 2 h at 1 350 °C (or at 1 100 °C if only sulfur is to be determined), and then stored in a desiccator or closed container before use.

For pre-ignition, a resistance furnace may be used.

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