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Third edition
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Iron ore pellets for blast furnace feedstocks — Determination of the free-swelling index

Boulettes de minerais de fer pour l'alimentation de hauts fourneaux — Détermination de l'indice de gonflement libre

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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).

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For an explanation of 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 3, *Physical testing*.

This third edition cancels and replaces the second edition (ISO 4698:2007), which has been technically revised.

The main changes are as follows:

— the dry medium displacement volumetric method has been included in Annex B.

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

This document concerns one of a number of physical test methods that have been developed to measure various physical parameters and to evaluate the behaviour of iron ores, including reducibility, disintegration, crushing strength, apparent density, etc. This method was developed to provide a uniform procedure, validated by collaborative testing, to facilitate comparisons of tests made in different laboratories.

The results of this test should be considered in conjunction with other tests used to evaluate the quality of iron ores as feedstocks for blast furnace processes.

This document may be used to provide test results as part of a production quality control system, as a basis of a contract, or as part of a research project.

Iron ore pellets for blast furnace feedstocks — Determination of the free-swelling index

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

1 Scope

This document specifies a method to provide a relative measure for evaluating the increase in volume of iron ore pellets, when reduced in an unconstrained bed under conditions resembling those prevailing in the reduction zone of a blast furnace. It specifies the determination of the free-swelling index.

This document is applicable to hot-bonded pellets.

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

ISO 11323, *Iron ore and direct reduced iron — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11323 and the following apply.

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 volume of pellets in the test portion is measured at room temperature, using a volumetric apparatus. The test portion is isothermally reduced under unconstrained conditions in a fixed bed at 900 °C, using a reducing gas consisting of CO and N_2, for 60 min. The volume of the reduced pellets is measured at room temperature. The swelling index is calculated as the difference between the volume of the pellets after and before reduction, expressed as a percentage.

5 Sampling, sample preparation and preparation of test portions

5.1 Sampling and sample preparation

Sampling of a lot and preparation of a test sample shall be in accordance with ISO 3082.

The size range for pellets shall be –12,5 mm + 10,0 mm.

A test sample of at least 1 kg, on a dry basis, of whole-sized pellets shall be obtained.

Oven-dry the test sample to constant mass at 105 °C ± 5 °C and cool it to room temperature before preparation of the test portions.

NOTE Constant mass is achieved when the difference in mass between two subsequent measurements becomes less than 0,05 % of the initial mass of the test sample.

5.2 Preparation of test portions

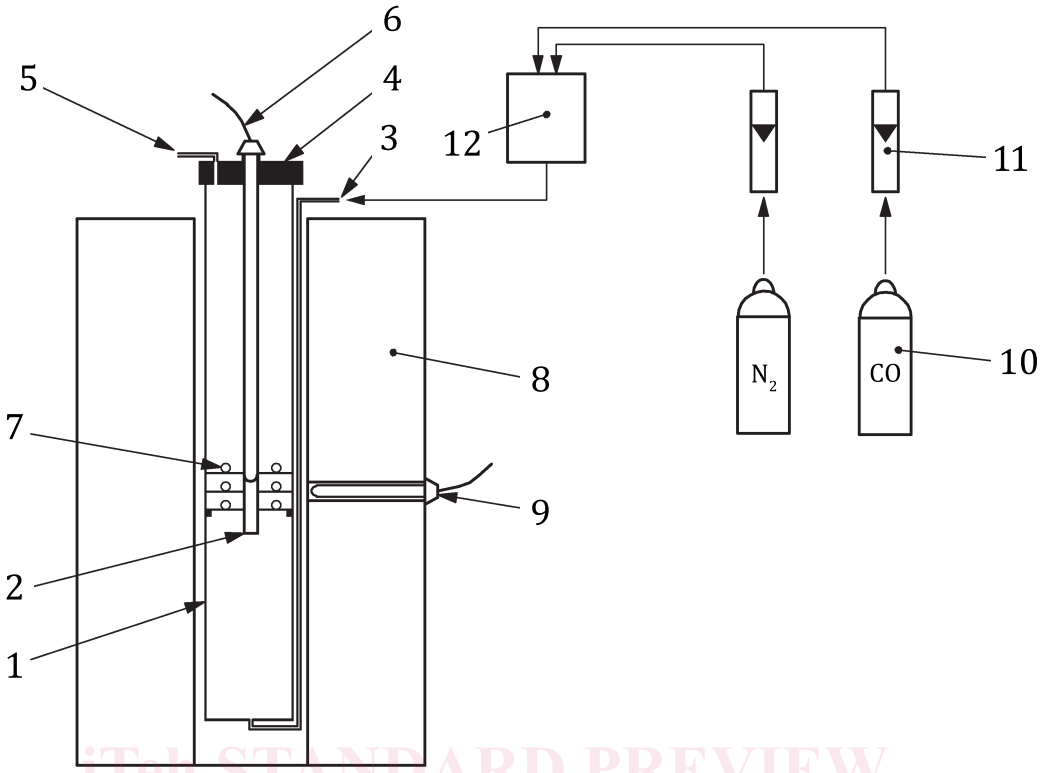
At least four test portions, each one made up of 18 whole pellets, shall be obtained from the test sample by random selection.

6 Apparatus

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

6.1 Ordinary laboratory equipment, such as an oven, hand tools, a time-control device and safety equipment.

Figure 1 shows an example of the test apparatus.



Key

	Reduction tube		**Furnace**
1	reduction-tube wall	8	electrically heated furnace
2	test portion holder	9	thermocouple for temperature regulation of furnace
3	gas inlet		**Gas-supply system**
4	lid	10	gas cylinder
5	gas outlet	11	gas flow meter
6	thermocouple for measuring the reduction temperature	12	mixing vessel
7	test portion		

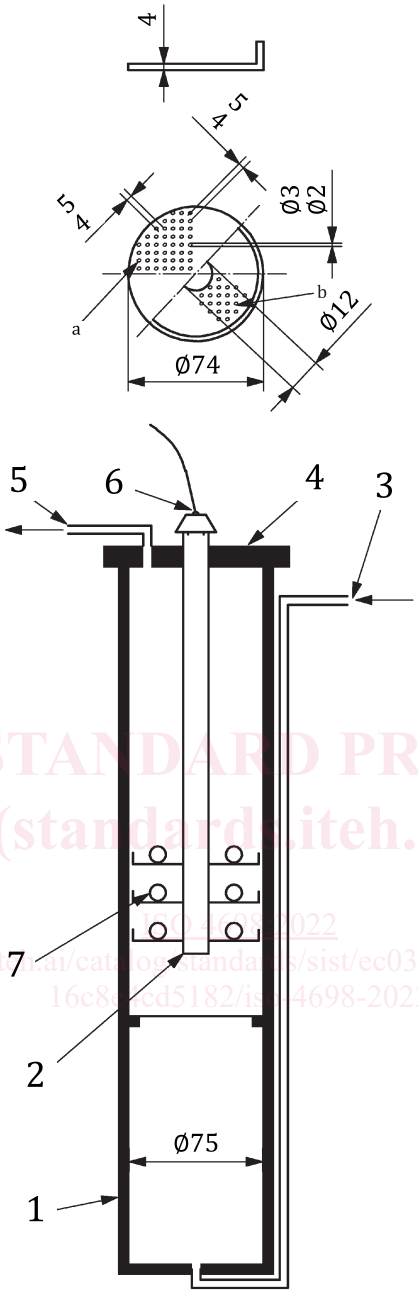
Figure 1 — Example of test apparatus (schematic diagram)

6.2 Reduction tube, made of non-scaling, heat-resistant metal to withstand temperatures higher than 900 °C and resistant to deformation. The internal diameter shall be 75 mm ± 1 mm.

6.3 Test portion holder, a wire basket made of non-scaling, heat-resistant metal to withstand temperatures higher than 900 °C. It shall comprise three levels, each made to receive six pellets, for a total of 18 pellets. Alternatively, the test portion holder may be made by welding a tube to the centre of three perforated plates, mutually parallel and equally spaced. The tube shall be top-opened and bottom-closed to allow the thermocouple insertion up to the middle of the test portion. The set shall be made of non-scaling, heat-resistant metal to withstand temperatures higher than 900 °C. The perforated plate shall be 4 mm thick, with its diameter 1 mm less than the tube internal diameter, the holes in the plate shall be 2 mm to 3 mm in diameter, at a pitch centre distance of 4 mm to 5 mm.

Figure 2 shows an example of a reduction tube with the test portion holder.

Dimensions in millimetres



Key

1	reduction-tube wall	6	opening for thermocouple insertion
2	test portion holder	7	test portion
3	opening for gas inlet	a	Design of perforated plate when a wire basket is used as the test portion holder.
4	lid	b	Design of perforated plate when a test portion holder with three levels is used.
5	opening for gas outlet		

NOTE Dimensions not specified in the apparatus clause are shown for information only.

Figure 2 — Example of a reduction tube with test portion holder (schematic diagram)

6.4 Furnace, having a heating capacity and temperature control able to maintain the entire test portion, as well as the gas entering the bed, at 900 °C ± 10 °C.

6.5 Gas-supply system, capable of supplying the gases and regulating gas flow rates.

6.6 Volumetric apparatus, capable of measuring the volume of the test portion to an accuracy of 0,2 ml.

Annex B shows examples of volumetric apparatus.

7 Test conditions

7.1 General

Volumes and flow rates of gases are measured at a reference temperature of 0 °C and at a reference atmospheric pressure of 101,325 kPa.

7.2 Reducing gas

7.2.1 Composition

The reducing gas shall consist of:

CO 30,0 % ± 0,5 % (volume fraction)

N_2 70,0 % ± 0,5 % (volume fraction)

7.2.2 Purity

Impurities in the reducing gas shall not exceed:

H_2 0,2 % (volume fraction)

CO_2 0,2 % (volume fraction)

O_2 0,1 % (volume fraction)

H_2O 0,2 % (volume fraction)

7.2.3 Flow rate

The flow rate of the reducing gas, during the entire reducing period, shall be maintained at 15 l/min ± 1 l/min.

7.3 Heating and cooling gas

Nitrogen (N_2) shall be used as the heating and cooling gas. Impurities shall not exceed 0,1 % (volume fraction).

The flow rate of N_2 shall be maintained at 10 l/min until the test portion reaches 900 °C, and at 15 l/min during the temperature-equilibration period. During cooling, it shall be maintained at 5 l/min.

7.4 Temperature of the test portion

The temperature of the entire test portion shall be maintained at 900 °C ± 10 °C during the entire reducing period and, as such, the reducing gas shall be preheated before entering the test portion.

8 Procedure

8.1 Number of determinations for the test

Carry out the test as many times as required by the procedure in Annex A.

8.2 Reduction

Determine the volume of the test portion (V_0) to an accuracy of 0,2 ml, in accordance with one of the methods specified in Annex B.

Place six pellets on each of the three levels of the test portion holder (6.3) and place it in the reduction tube (6.2). Close the top of the reduction tube. Connect the thermocouple, ensuring that its tip is in the centre of the test portion. Close the top of the reduction tube and insert it in the furnace.

Connect the gas-supply system (6.5).

Pass a flow of N_2 through the test portion at a rate of at least 5 l/min and commence heating. When the temperature of the test portion approaches 900 °C, increase the flow to 15 l/min ± 1 l/min. After reaching 900 °C ± 10 °C, maintain the test portion under these conditions for 15 min.

DANGER — Carbon monoxide and the reducing gas, which contains carbon monoxide, are toxic and therefore hazardous. Testing shall be carried out in a well-ventilated area or under a ventilation hood. Precautions should be taken for the safety of the operator.

Introduce the reducing gas at a flow rate of 15 l/min ± 1 l/min to replace the N_2. After 60 min of reduction, turn off the power.

Some pellets show a higher degree of swelling within a shorter reduction time than 60 min. Therefore, a shorter reduction time may be used as an alternative when appropriate, e.g. 40 min.

Replace the reducing gas with N_2 at a flow rate of 5 l/min. Remove the reduction tube from the furnace (6.4) while maintaining the flow of N_2 until the test portion reaches room temperature (below 50 °C).

8.3 Volume determination

Remove the test portion from the reduction tube and immediately determine and register the total volume of the test portion (V_1), applying the same method used for the determination of V_0.

9 Expression of results

9.1 Calculation of the free-swelling index (V_{FS})

The free-swelling index, V_{FS}, expressed as a percentage, is calculated from Formula (1):

$$V_{FS} = \frac{V_1 - V_0}{V_0} \times 100 \quad (1)$$

where

V_0 is the volume, in millilitres, of the test portion before reduction;

V_1 is the volume, in millilitres, of the test portion after reduction.

Record the result to one decimal place.