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Iron ores for blast furnace feedstocks — Determination of the reducibility by the rate of reduction index

Minerais de fer pour charges de hauts fourneaux — Détermination de la réductibilité à partir de la vitesse de réduction

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Contents

Page

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 fifth edition cancels and replaces the fourth edition (ISO 4695:2015), of which it constitutes a minor revision to correct Formula B.3 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 have to be considered in conjunction with other tests used to evaluate the quality of iron ores as feedstocks for blast furnace processes.

This document can 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 ores for blast furnace feedstocks — Determination of the reducibility by the rate of reduction index

CAUTION — This document can involve hazardous 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 extent to and ease with which oxygen can be removed from iron ores, when reduced under conditions resembling those prevailing in the reduction zone of a blast furnace.

This document is applicable to lump ores, sinters and 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 2597-1, *Iron ores — Determination of total iron content — Part 1: Titrimetric method after tin(II) chloride reduction*

ISO 2597-2, *Iron ores — Determination of total iron content — Part 2: Titrimetric methods after titanium(III) chloride reduction*

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

ISO 9035, *Iron ores — Determination of acid-soluble iron(II) content — Titrimetric method*

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 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 test portion is isothermally reduced in a fixed bed, at 950 °C, using a reducing gas consisting of CO and N_2, and the mass loss of the test portion is recorded continuously or at specified time intervals until its degree of reduction reaches 65 %. The rate of reduction is calculated at the oxygen/iron ratio of 0,9.

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, sinters and lump ores shall be –12,5 mm + 10,0 mm.

A test sample of at least 2,5 kg, on a dry basis, of the sized material 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

Collect each test portion by taking ore particles at random.

NOTE Manual methods of division recommended in ISO 3082, such as riffling, can be applied to obtain the test portions.

At least five test portions, each of approximately 500 g (± the mass of one particle) shall be prepared from the test sample: four test portions for testing and one for chemical analysis.

Weigh the test portions to the nearest 1 g and register the mass of each test portion on its recipient label.

6 Apparatus

6.1 The test apparatus shall comprise the following:

a) ordinary laboratory equipment, such as an oven, hand tools, a time-control device and safety equipment;

b) reduction tube assembly;

c) furnace, equipped with a balance for permitting the mass loss of the test portion to be read at any time during the test;

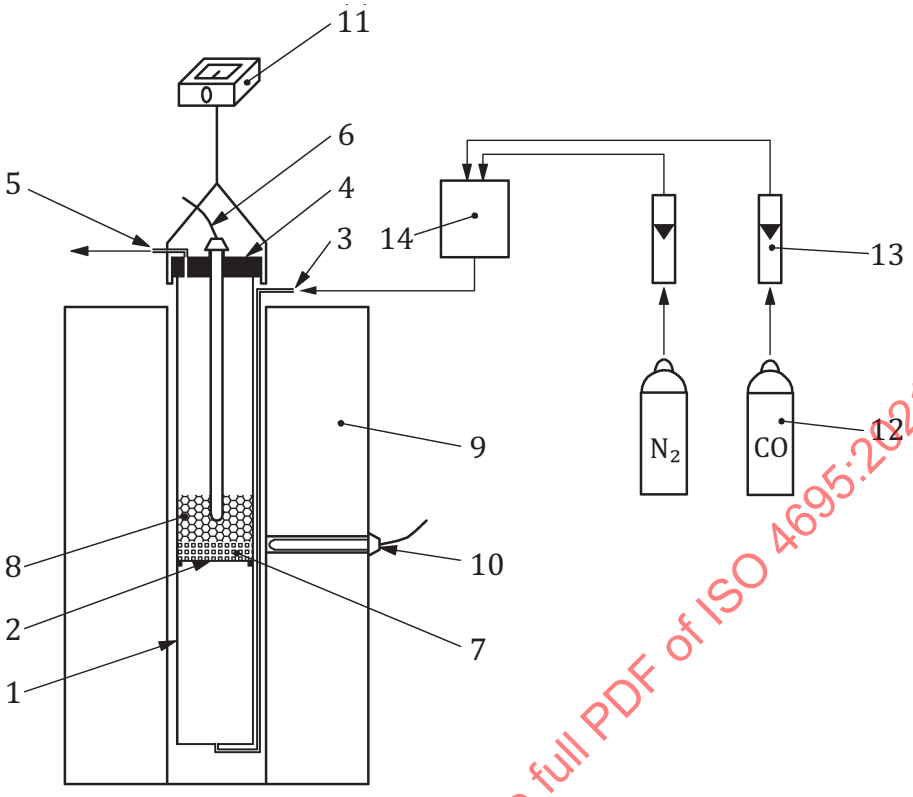
d) system to supply the gases and regulate the flow rates;

e) weighing device.

Figure 1 shows an example of the test apparatus.

6.2 Reduction tube, made of non-scaling, heat-resistant metal to withstand temperatures higher than 950 °C and resistant to deformation. The internal diameter shall be 75 mm ± 1 mm. A removable perforated plate, made of non-scaling, heat-resistant metal to withstand temperatures higher than 950 °C, shall be mounted in the reduction tube to support the test portion and to ensure uniform gas flow through it. The perforated plate shall be 4 mm thick, with its diameter 1 mm less than the internal diameter of the tube. The holes in the plate shall be 2 mm to 3 mm in diameter at a pitch centre distance of 4 mm to 5 m.

Figure 2 shows an example of a reduction tube.



Key

Reduction tube

1 reduction tube
2 perforated plate
3 gas inlet
4 lid
5 gas outlet
6 thermocouple for measuring the reduction temperature
7 porcelain ball layer
8 test portion

Furnace

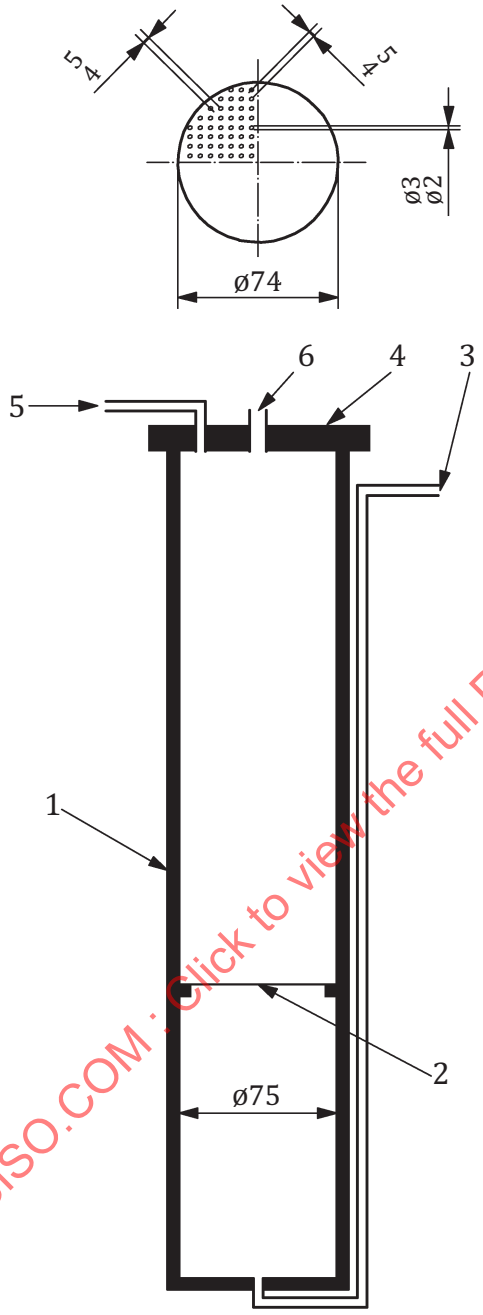
9 electrically heated furnace
10 thermocouple for temperature regulation of furnace
11 balance

Gas supply system

12 gas cylinder
13 gas flow meters
14 mixing vessel

Figure 1 — Example of test apparatus (schematic diagram)

Dimensions in millimetres



Key

1 reduction tube
2 perforated plate
3 opening for gas inlet
4 lid
5 opening for gas outlet
6 opening for thermocouple insertion

NOTE Dimensions not specified in Clause 6 are shown for information only.

Figure 2 — Example of reduction tube (schematic diagram)

6.3 Furnace, having a heating capacity and temperature control able to maintain the entire test portion, as well as the gas entering the bed, at 950 °C ± 10 °C.

6.4 Balance, capable of weighing the reduction tube assembly, including the test portion, to an accuracy of 1 g. The balance shall have an appropriate device to suspend the reduction tube assembly.

6.5 Gas-supply system, capable of supplying the gases and regulating gas flow rates. It shall be ensured that a frictionless connection between the gas-supply system and the reduction tube does not affect the weight loss determination during reduction.

6.6 Weighing device, capable of weighing the test sample and test portions to an accuracy of 1 g.

7 Test conditions

7.1 General

Volumes and flow rates of gases used are as measured at a reference temperature of 0 °C and at a reference atmospheric pressure of 101,325 kPa (1,013 25 bar).

7.2 Reducing gas

7.2.1 Composition

The reducing gas shall consist of

CO 40,0 % ± 0,5 % (volume fraction)

N_2 60,0 % ± 0,5 % (volume fraction)

7.2.2 Purity

Impurities in the reducing gas shall not exceed the following:

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 50 l/min ± 0,5 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 25 l/min until the test portion reaches 950 °C, and at 50 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 950 °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 Chemical analysis

Take, at random, one of the test portions prepared in 5.2 and use it for the determination of the iron(II) oxide content (w_1) in accordance with ISO 9035 and the total iron content (w_2) in accordance with ISO 2597-1 or ISO 2597-2.

8.3 Reduction

Take, at random, another portion prepared in 5.2 and record its mass (m_0). Place it in the reduction tube (6.2) and level its surface.

NOTE In order to achieve a more uniform gas flow, a double-layer bed of porcelain balls sized between 10,0 mm and 12,5 mm can be placed between the perforated plate and the test portion.

Close the top of the reduction tube. Connect the thermocouple, ensuring that its tip is in the centre of the test portion, as shown in Figure 1.

Insert the reduction tube into the furnace (6.3) and suspend it centrally from the balance (6.4) ensuring that there is no contact with the furnace wall or heating elements.

Connect the gas-supply system (6.5).

Pass a flow of N_2 through the test portion at a rate of 25 l/min and commence heating. When the temperature of the test portion approaches 950 °C, increase the flow rate to 50 l/min. Continue heating while maintaining the flow of N_2, until the balance reading is constant and the temperature is constant at 950 °C ± 10 °C 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 hood. Precautions should be taken for the safety of the operator, in accordance with the safety codes of each country.

Tare the balance, start the time control device and immediately introduce the reducing gas at a flow rate of 50 l/min ± 0,5 l/min to replace the N_2. Record the mass loss of the test portion (Δm_t) at least every 3 min for the first 15 min and thereafter at 10 min intervals.

Calculate the degree of reduction, R_t, relative to the iron(III) state, after t min, as shown in Formula (1):

$$R_t = \left(\frac{0{,}111\, w_1}{0{,}430\, w_2} + \frac{\Delta m_t}{m_0 \times 0{,}430\, w_2} \times 100 \right) \times 100 \qquad (1)$$

where

m_0 is the mass, in grams, of the test portion;

Δm_t is the mass loss, in grams, of the test portion after reduction time t;

w_1 is the iron(II) oxide content, as a percentage by mass, of the test sample prior to the test, determined in accordance with ISO 9035; it is calculated from the iron(II) content by multiplying it by the oxide conversion factor FeO/Fe(II) = 1,286;

w_2 is the total iron content, as a percentage by mass, of the test portion prior to the test, determined in accordance with ISO 2597-1 or ISO 2597-2.

When the degree of reduction reaches 65 %, turn off the power and stop the flow of the reducing gas. If after 4 h, 65 % of oxygen loss has not been achieved, the reduction can be stopped. Introduce N_2 at a flow rate of 5 l/min for 5 min or more to purge the reducing gas from the tube.

9 Expression of results

9.1 Calculation of the reducibility index

Reducibility index: $\left(\frac{dR}{dt}\right)$ $(O/Fe = 0,9)$

Prepare the reduction curve by plotting the degree of reduction R_t against time t.

Read off, from the reduction curve the time, in minutes, to attain degrees of reduction of 30 % and 60 %.

The reducibility index, expressed as the rate of reduction at the atomic ratio of O/Fe of 0,9[1)] in %/min, is calculated from Formula (2):

$$\frac{dR}{dt}(O/Fe = 0,9) = \frac{33,6}{t_{60} - t_{30}} \quad (2)$$

where

t_{30} is the time, in minutes, to attain a degree of reduction of 30 %;

t_{60} is the time, in minutes, to attain a degree of reduction of 60 %;

33,6 is a constant.

Record the result to two decimal places.

NOTE 1 The derivation of the equation is given in Annex B.

NOTE 2 If a degree of reduction of 60 % is not attained in the test, lower values can be accommodated by Formula (3):

$$\frac{dR}{dt}(O/Fe = 0,9) = \frac{k}{t_y - t_{30}} \quad (3)$$

where

t_y is the time, in minutes, to attain a degree of reduction of y %;

k is a constant depending on y.

1) The atomic ratio O/Fe = 0,9 means a 40 % degree of reduction.

If y = 50 %, then the value of k = 20,2.

If y = 55 %, then the value of k = 26,5.

9.2 Repeatability and acceptance of test results

Follow the procedure in Annex A by using the repeatability values given in Table 1. The results shall be reported to two decimal places.

Table 1 — Repeatability (r)

Type of iron ore	r %/min
Pellets	$0{,}07 \times \overline{dR/dt}$
Sinter	0,17
Lump ore	0,10
NOTE $\overline{dR/dt}$ is the mean value of dR/dt results.	

10 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 4695:2021;

b) all details necessary for the identification of the sample;

c) the name and address of the test laboratory;

d) the date of the test;

e) the date of the test report;

f) the signature of the person responsible for the test;

g) the details of any operation and any test conditions not specified in this document or regarded as optional, as well as any incident which may have had an influence on the results;

h) the reducibility index, dR/dt (O/Fe = 0,9);

i) the total iron and iron(II) contents of the test portion before reduction;

j) the reduction curve, if specified at the time of ordering.

11 Verification

Regular checking of the apparatus is essential to ensure test result reliability. The frequency of checking is a matter for each laboratory to determine.

The conditions of the following items shall be checked:

— weighing device;

— reduction tube;

— temperature control and measurement devices;

— balance;

— gas flow meters;

— purity of gases;

— recording system;

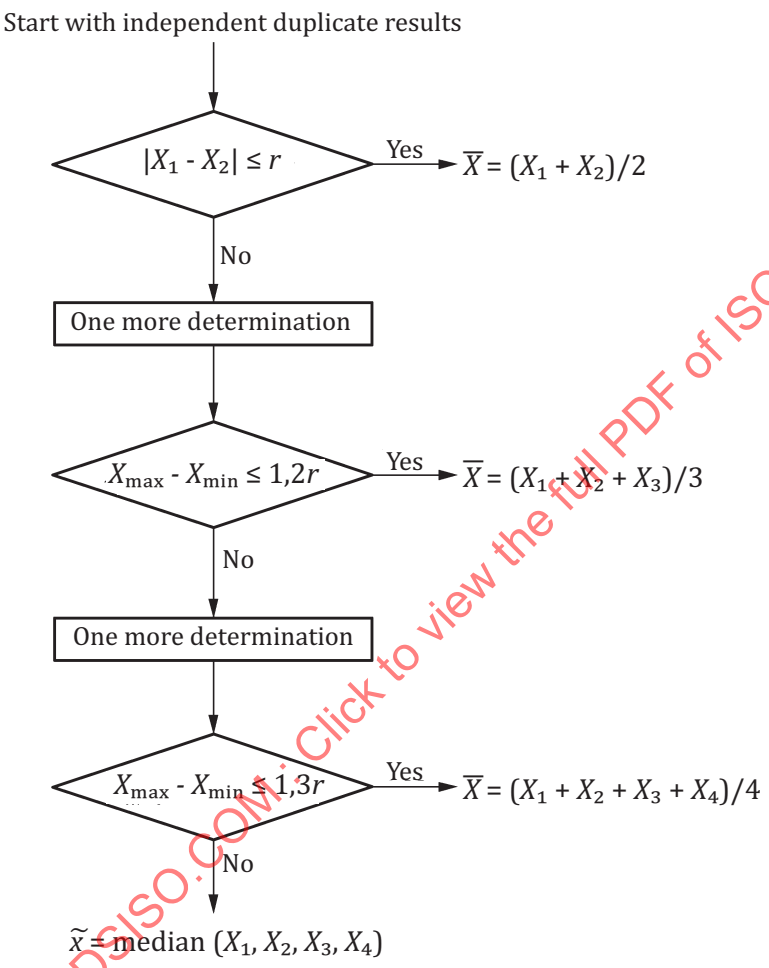
— time-control device.

It is recommended that internal reference material be prepared and used periodically to check test repeatability.

Appropriate records of verification activities shall be maintained.

Annex A
(normative)

Flowsheet of the procedure for the acceptance of test results



NOTE For *r*, see Table 1.

Figure A.1 — Flowsheet of the procedure for the acceptance of test results