BS ISO 7215:2015

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

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National foreword

This British Standard is the UK implementation of ISO 7215:2015. It supersedes BS ISO 7215:2007 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee ISE/58, Iron ores.

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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INTERNATIONAL STANDARD

ISO 7215

Fourth edition
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Iron ores for blast furnace feedstocks — Determination of the reducibility by the final degree of reduction index

Minerais de fer pour charges de hauts fourneaux — Détermination de la réductibilité relative par le degré final de l'indice de réduction

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Foreword

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 3, *Physical testing*.

This fourth edition cancels and replaces the third edition (ISO 7215:2007), of which it constitutes a minor revision to contemplate the outcome of the studies on mass definition, as well as minor editorial improvements.

Introduction

This International Standard 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 International Standard 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.

INTERNATIONAL STANDARD ISO 7215:2015(E)

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

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

1 Scope

This International Standard specifies a method to provide a relative measure for evaluating the extent to which oxygen can be removed from iron ores when reduced under conditions resembling those prevailing in the reduction zone of a blast furnace.

This International Standard is applicable to lump ore, sinters, and hot-bonded pellets.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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.

4 Principle

The test portion is isothermally reduced in a fixed bed, at 900 °C, using a reducing gas consisting of CO and N_2, for 180 min. The final degree of reduction is calculated from the oxygen mass loss after 180 min (R_{180}).

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.

The size range for sinters and lump ores shall be - 20,0 mm + 18,0 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 1 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 General

The test apparatus shall comprise the following:

a) ordinary laboratory equipment, such as an oven, hand tools, 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 900 °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 900 °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 mm.

Figure 2 shows an example of a reduction tube.

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 900 °C ± 10 °C.

6.4 Balance, capable of weighing the reduction tube assembly, including the test portion, to an accuracy of 0,5 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 the 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 the following:

CO 30,0 % ± 1,0 % (volume fraction)

N_2 70,0 % ± 1,0 % (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 15 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 5 L/min until the test portion reaches 900 °C and at 15 L/min during 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 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 test 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 at least 5 L/min and commence heating. When the temperature of the test portion approaches 900 °C, increase the flow rate to 15 L/min. Continue heating while maintaining the flow of N_2, until the balance reading is constant and the temperature is constant at 900 °C ± 10 °C for 30 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 15 L/min ± 0,5 L/min to replace the N_2. After 180 min of reduction, record the mass loss of the test portion (Δm), turn off the power and stop the flow of the reducing gas. Introduce N_2 at a flow rate of 5 L/min for 5 min or more to purge the reducing gas from the tube.

NOTE If a reduction versus time curve is required, record the mass loss of the test portion continuously or every 10 min during the first hour and every 15 min during the last 2 h.

In the case of lump ores, the temperature of the test portion should be raised to 900 °C over more than 60 min, to reduce their decrepitation.

9 Expression of results

9.1 Calculation of the degree of reduction (R_{180})

The final degree of reduction, R_{180}, expressed as a percentage by mass, is calculated from the following formula[1)]:

$$R_{180} = \left[\frac{\Delta m}{m_0 \left(0{,}430\ w_2 - 0{,}111\ w_1\right)} \text{ x } 100 \right] \times 100$$

where

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

Δm is the mass loss, in grams, of the test portion after 180 min of reduction;

w_1 is the iron(II) oxide content, as a percentage by mass, of the test portion 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.

Record the result to one decimal place.

9.2 Repeatability for R_{180} and acceptance of test results

Follow the procedure in Annex A by using the repeatability values for R_{180} given in Table 1. The results shall be reported to one decimal place.

Table 1 — Repeatability (r) for R_{180}

Type of iron ore	r %, absolute
Pellets	3,0
Sinter	5,0
Lump ore	5,0

10 Test report

The test report shall include the following information:

a) a reference to this International Standard, i.e. ISO 7215:2015;

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 International Standard or regarded as optional, as well as any incident which could have had an influence on the results;

1) The derivation of the formula is given in Annex B.

h) the final degree of reduction, R_{180};

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

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;

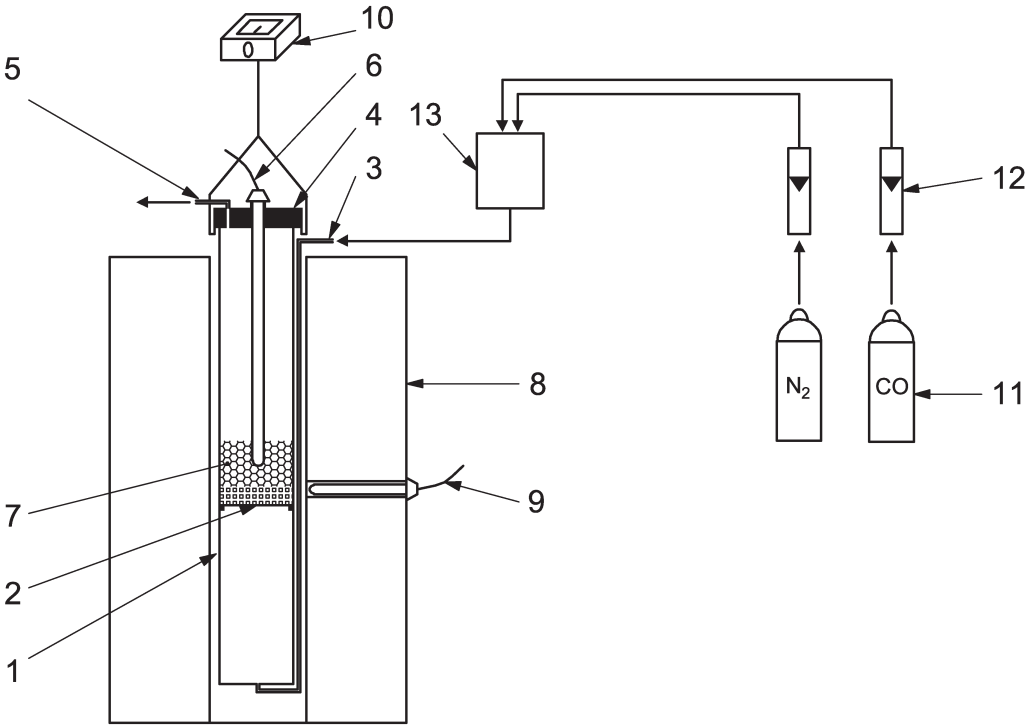
— purity of gases;

— 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.



Key

Reduction tube

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

Furnace

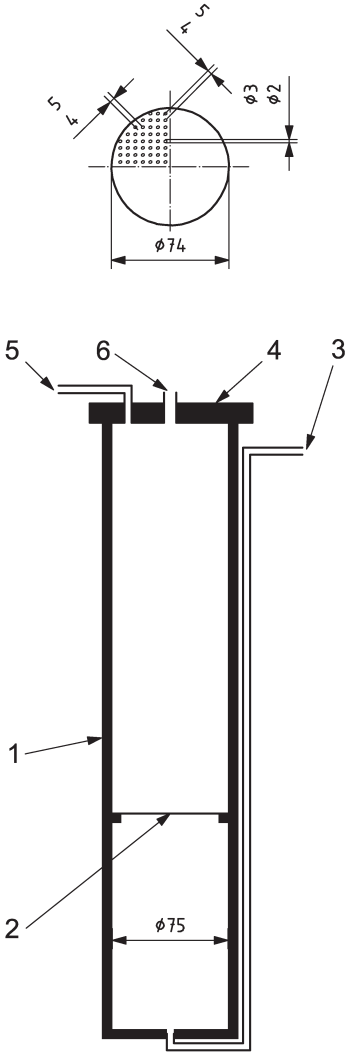
8 electrically heated furnace
9 thermocouple for temperature regulation of furnace
10 balance

Gas supply system

11 gas cylinders
12 gas flow meters
13 mixing vessel

Figure 1 — Example of test apparatus (schematic diagram)

Dimensions in millimetres



Key

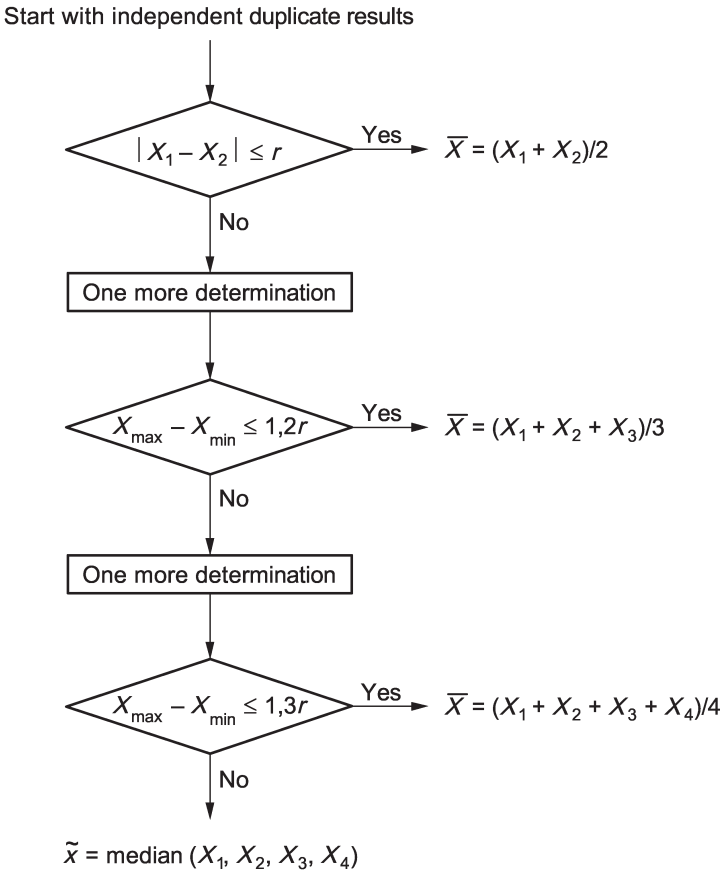
1 reduction tube wall
2 perforated plate
3 opening for gas inlet
4 lid
5 opening for gas outlet
6 opening for thermocouple insertion

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

Figure 2 — Example of reduction tube (schematic diagram)

Annex A
(normative)

Flowsheet of the procedure for the acceptance of test results



r: see Table 1

Annex B
(informative)

Derivation of the formula for final degree of reduction

B.1 Basic formula

The formula for R_f given in 9.1 is derived from the basic formula:

$$R_f = \frac{m_f}{m_3} \times 100 \tag{B.1}$$

where

m_f is the mass loss, in grams, of oxygen during 180 min of reduction;

m_3 is the mass, in grams, of oxygen combined with iron before reduction.

B.2 Derivation of working formula

The iron oxides contained in the sample are considered to be hematite (Fe_2O_3), magnetite (Fe_3O_4), and iron(II) oxide (FeO). The total mass of oxygen, m_3, in Formula (B.1) can be obtained from the masses of Fe_2O_3 and FeO in the test sample before reduction. Therefore, m_3 is given by Formula (B.2), after the total iron content, w_2, and the iron(II) oxide content, w_1, of the test sample have been determined according to the relevant International Standards.

$$m_3 = m_4 + m_5$$

$$= m_0 \left(w_3 \frac{3A_O}{2A_{Fe}} + w_1 \frac{A_O}{M} \right) \times \frac{1}{100} \tag{B.2}$$

where

m_4 is the mass, in grams, of oxygen in Fe_2O_3;

m_5 is the mass, in grams, of oxygen in FeO;

m_0 and w_1 have the same meanings as in 9.1;

w_3 is the iron content, as a percentage by mass, in Fe_2O_3;

A_O is the relative atomic mass of oxygen, 16,00;

A_{Fe} is the relative atomic mass of iron, 55,85;

M is the relative molecular mass of iron(II) oxide, 71,85.

Noting that

$$\Delta_f = m_1 - m_2$$

$$w_3 = w_2 - \frac{A_{Fe}}{M} w_1$$

where

m_1 is the mass, in grams, of the test portion immediately before starting the reduction;

m_2 is the mass, in grams, of the test portion after 180 min of reduction;

w_1 and w_2 have the same meanings as in 9.1.

Substituting m_3 from Formula (B.2) into Formula (B.1), the final degree of reduction, R_f, expressed as a percentage, is given by Formula (B.3):

$$R_f = \frac{(m_1 - m_2) \times 100}{m_0 \left[\left(w_2 - \frac{A_{Fe}}{M} w_1 \right) \frac{3A_O}{2A_{Fe}} + \frac{A_O}{M} w_1 \right] \times \frac{1}{100}} \tag{B.3}$$

$$R_f = \left\{ \frac{m_1 - m_2}{m_0 \left[\left(w_2 - \frac{55,85}{71,85} w_1 \right) \frac{48,00}{111,70} + \frac{16,00}{71,85} w_1 \right]} \times 100 \right\} \times 100$$

$$R_{180} = \left[\frac{\Delta m}{m_0 (0,430\ w_2 - 0,111\ w_1)} \text{ x } 100 \right] \times 100$$

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