INTERNATIONAL STANDARD

ISO 4696-2

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Iron ores for blast furnace feedstocks — Determination of low-temperature reduction-disintegration indices by static method —

Part 2: Reduction with CO and N2

Minerais de fer pour charges de hauts fourneaux — Détermination des indices de désagrégation par réduction à basse température par méthode statique —

Partie 2: Réduction avec CO et N2

Reference number
ISO 4696-2:2015(E)

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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).

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 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 third edition cancels and replaces the second edition (ISO 4696-2:2007), which has been technically revised to address the care needed during hand sieving, to introduce the mechanical sieving and to exclude the reference to ISO 4701.

ISO 4696 consists of the following parts, under the general title *Iron ores for blast furnace feedstocks — Determination of low-temperature reduction-disintegration indices by static method*:

— *Part 1: Reduction with CO, CO_2, H_2 and N_2*

— *Part 2: Reduction with CO and N_2*

Introduction

This part of ISO 4696 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 and direct reduction processes.

This part of ISO 4696 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 low-temperature reduction-disintegration indices by static method —

Part 2:
Reduction with CO and N2

CAUTION — This International Standard can 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 part of ISO 4696 specifies a method to provide a relative measure for evaluating the degree of size degradation of iron ores when reduced with carbon monoxide and nitrogen, under conditions resembling those prevailing in the low-temperature reduction zone of a blast furnace.

This part of ISO 4696 is applicable to lump ores, 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 3082, *Iron ores — Sampling and sample preparation procedures*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

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 550 °C, using a reducing gas consisting of CO and N_2, for 30 min. The reduced test portion is tumbled in a specific tumble drum for 900 revolutions and then sieved with a sieve having square openings of 2,8 mm. The reduction-disintegration index (RDI) is calculated as the mass percentage of material –2,8 mm.

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 + 16,0 mm.

A test sample of at least 2 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 four test portions, each of approximately 500 g (± the mass of one particle) shall be prepared from the test sample.

Weigh the test portions to the nearest 0,1 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;

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

e) tumble drum;

f) test sieves;

g) 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 600 °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 600 °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 550 °C ± 5 °C.

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

6.5 Tumble drum, made of steel, at least 5 mm thick, having an internal diameter of 130 mm and an inside length of 200 mm. Two equally spaced steel lifters (200 mm long, 20 mm high, and 2 mm thick) shall be mounted longitudinally inside the drum. These can be mounted on a frame that can be inserted inside the vessel from one end. One end of the drum shall be closed and the other open. A close-fitting lid shall be held in place on the opening to ensure a dust-tight seal. The drum shall be replaced in any case when the thickness of the vessel wall is reduced to 3 mm in any area, and the lifters when their height is reduced to less than 18 mm.

Figure 3 shows an example of a tumble drum.

6.6 Rotation equipment, capable of ensuring that the drum attains full speed in one revolution, rotates at a constant speed of 30 r/min ± 1 r/min and stops within one revolution. The equipment shall be fitted with a revolution counter and with an automatic device for stopping the drum after a predetermined number of revolutions.

6.7 Test sieves, conforming to ISO 3310-1 or ISO 3310-2 and having square apertures of 2,8 mm nominal size.

6.8 Weighing device, capable of weighing the test sample and test portions to an accuracy of 0,1 g.

7 Test conditions

7.1 General

Volumes and flow rates of gases 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 % ± 0,5 % (volume fraction)
N_2	70,0 % ± 0,5 % (volume fraction)

7.2.2 Purity

Impurities in the reducing gas shall not exceed the following:

H_2	0,01 % (volume fraction)
Total impurities	0,1 % (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 550 °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 550 °C ± 5 °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

Take, at random, one of the test portions prepared in 5.2. 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 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.

Insert the reduction tube into the furnace (6.3).

Connect the gas-supply system (6.4).

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 550 °C, increase the flow rate to 15 L/min. Continue heating while maintaining the flow of N_2 until the test portion reaches 550 °C ± 10 °C. Allow a period of 15 min for temperature equilibration at 550 °C.

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.

Introduce the reducing gas at a flow rate of 15 L/min ± 0,5 L/min to replace the N_2. After 30 min of reduction, turn off the power. Replace the reducing gas with N_2 at a flow rate of 5 L/min and cool the test portion to a temperature below 100 °C.

8.3 Tumbling

Remove the test portion carefully from the reduction tube. Determine its mass (m_0) and place it in the tumble drum (6.5). Fasten the lid tightly and rotate the drum for a total of 900 revolutions at a rate of 30 r/min ± 1 r/min.

8.4 Sieving

Remove all material from the drum, determine and record the mass and hand sieve with care on 2,8 mm sieve. Determine and record the mass retained on this sieve (m_1). Material lost during tumbling and sieving shall be considered to be part of the –2,8 mm fraction.

NOTE Equivalent mechanical sieving can be used provided that preliminary test programme is carried out according to ISO 3086, being the hand sieving the reference method.

Sieving results are influenced by the sieve shaker characteristics. Therefore, in cases in which two or more laboratories need to compare their results for commercial or research purposes, they should adjust the sieving conditions until they obtain identical results for the same test sample.

9 Expression of results

9.1 Calculation of the reduction-disintegration index (RDI–$2_{-2,8}$)

The reduction-disintegration index, RDI–$2_{-2,8}$, expressed as a percentage by mass, is calculated from Formula (1):

$$\mathrm{RDI}-2_{-2,8} = 100 - \frac{m_1}{m_0} \times 100 \qquad (1)$$

where

m_0 is the mass, in grams, of the test portion after reduction and before tumbling;

m_1 is the mass, in grams, of the fraction retained on the 2,8 mm sieve.

Record each result to one decimal place.

9.2 Repeatability and acceptance of test results

Follow the procedure of Annex A using the repeatability value given in Table 1. The results shall be reported to one decimal place.

Table 1 — Repeatability (*r*)

Mean value of RDI-2 %	*R* (%, absolute)
1	0,45
2	0,88
3	1,29
4	1,68
5	2,05
7	2,73
10	3,60
15	4,66
20	5,21
25	5,30
30	5,30
35	5,30
40	5,30

Table 1 *(continued)*

Mean value of RDI-2 %	*R* (%, absolute)
45	5,30
50	5,30
55	5,30
60	5,30

10 Test report

The test report shall include the following information:

a) a reference to this part of ISO 4696, i.e. ISO 4696-2:2015;

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 part of ISO 4696 or regarded as optional, as well as any incident which may have had an influence on the results;

h) the reduction-disintegration index, $RDI{-}2_{-2,8}$;

i) the type of sieve used;

j) the sieving conditions, e.g. the method of sieving and the sieving time;

k) the total mass of the material inserted into the tumble drum and taken from the tumble drum.

11 Verification

Regular checking of 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:

— sieves;

— weighing device;

— reduction tube;

— temperature control and measurement devices;

— gas flow meters;

— purity of gases;

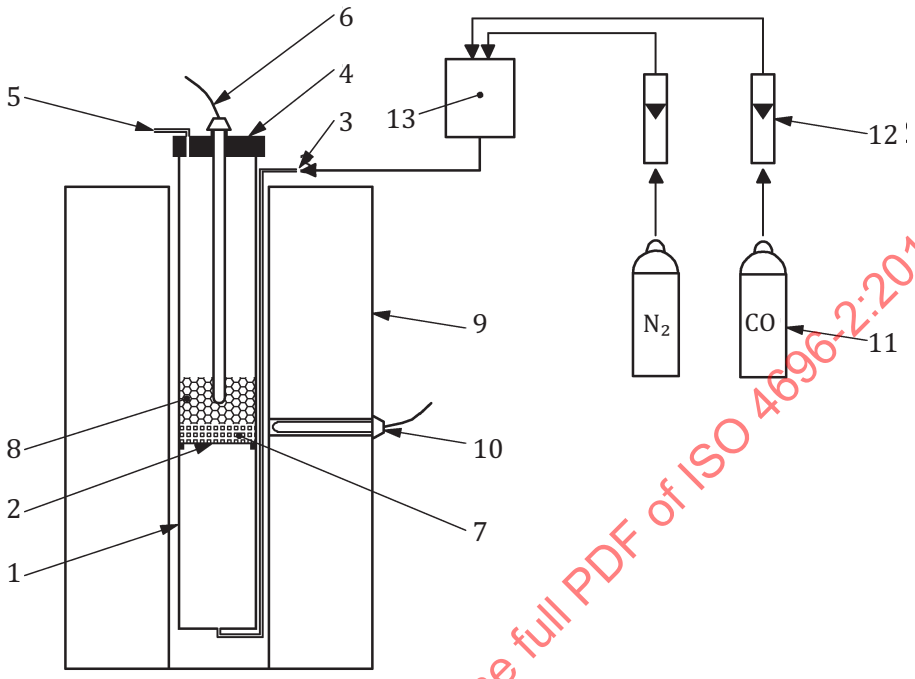
— time-control device;

— tumble drum;

— drum-rotation equipment.

It is recommended that the 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 porcelain ball layer
8 test portion

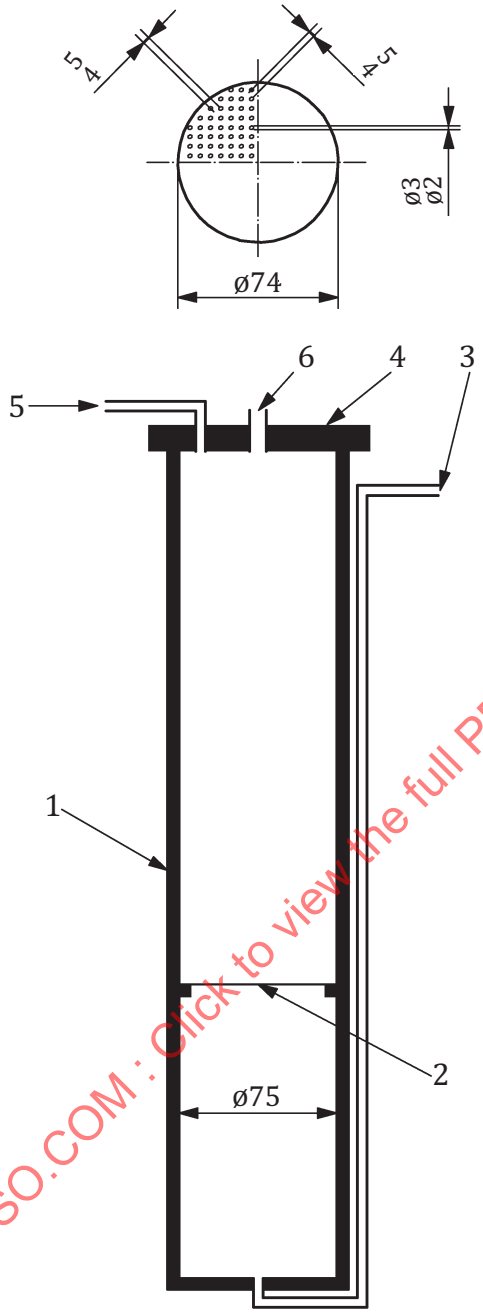
Furnace

9 electrically heated furnace
10 thermocouple for temperature regulation of furnace

Gas supply system

11 gas cylinders
12 gas flow meters
13 mixing vessel

Figure 1 — Example of test apparatus (schematic diagram)



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 Clause 6 are shown for information only.

Figure 2 — Example of a reduction tube (schematic diagram)