
**Iron ores — Static test for low-temperature
reduction-disintegration —**

**Part 2:
Reaction with CO**

*Minerais de fer — Essai statique de désagrégation par réduction à basse
température —*

Partie 2: Réaction avec CO



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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 4696-2 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Subcommittee SC 3, *Physical testing*.

Together with part 1, this part of ISO 4696 cancels and replaces ISO 4696:1984, which has been technically revised.

ISO 4696 consists of the following parts, under the general title *Iron ores — Static test for low-temperature reduction-disintegration*:

- *Part 1: Reaction with CO, CO₂ and H₂*
- *Part 2: Reaction with CO*

Annex A forms an integral part of this part of ISO 4696.

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Introduction

This part of ISO 4696 describes a test method for evaluating the disintegration behaviour of iron ores reduced in a fixed bed under specific conditions. The specific features of this test are:

- a) isothermal reduction in a fixed bed at 550 °C;
- b) tumbling the reduced sample at room temperature;
- c) measurement of the disintegration of a test portion by sieving after tumbling.

The results of this test should be considered in conjunction with the results of other tests, particularly those showing the physical behaviour of materials during reduction.

ISO 4696-1 describes an alternative method adopting different test conditions.

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Iron ores — Static test for low-temperature reduction-disintegration —

Part 2: Reaction with CO

WARNING — This part of ISO 4696 may involve hazardous materials, operations or equipment. It does not purport to address all the safety problems, if any, associated with its use. It is the responsibility of the user of this part of ISO 4696 to establish appropriate safety and health practices and to determine the applicability of any regulatory limitations prior to use.

1 Scope

This part of ISO 4696 specifies a method for testing the disintegration of iron ores by tumbling, at room temperature, a test portion that has been reduced in a fixed bed at a temperature of 550 °C.

The method is applicable to natural iron ores and to iron ore agglomerates such as pellets or sinter.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 4696. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 4696 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3082:1998, *Iron ores — Sampling and sample preparation procedures*.

ISO 3310-1:—¹⁾, *Test sieves — Requirements and tests — Part 1: Metal wire cloth sieves*.

ISO 3310-2:—²⁾, *Test sieves — Requirements and tests — Part 2: Perforated metal plate sieves*.

3 Principle

A test portion with a specified size range is subjected to static reduction at a temperature of 550 °C using reducing gas consisting of carbon monoxide (CO) and nitrogen (N₂).

After 30 min reduction time, the test portion is cooled to a temperature below 100 °C and tumbled by using a small tumbler drum for 900 revolutions in total. It is then sieved with a test sieve having square mesh apertures of 2,8 mm.

1) To be published. (Revision of ISO 3310-1:1990)

2) To be published. (Revision of ISO 3310-2:1990)

The reduction-disintegration index (RDI-2_{-2,8}) is calculated as a quantitative measure of the degree of disintegration of an iron ore that has been reduced and then tumbled: the percentage by mass of material less than 2,8 mm is related to the total mass of the test portion after reduction and before tumbling.

4 Apparatus

The apparatus shall consist of the following:

- a system to supply and regulate the gases;
- a reduction tube;
- an electrically heated furnace to heat the sample to the specified temperature;
- a tumbler drum;
- test sieves;
- a weighing device.

4.1 Reduction tube, made of non-scaling, heat-resisting metal to withstand a temperature of greater than 600 °C. The diameter of the sample bed shall be 75 mm ±1 mm.

Figure 1 shows an example of such a reduction tube.

The test portion may be placed on a perforated plate or, if desired, on balls of alumina on the perforated plate, in order to ensure a uniform flow of gas through the test portion.

4.2 Furnace, having a heating capacity sufficient to maintain the entire test portion and the gas entering the bed at 550 °C, and being equipped with a heating element suitable for the specified temperature.

4.3 Tumbler drum, consisting of a vessel having an internal diameter of 130 mm and an inside length of 200 mm.

Two equally spaced steel lifters 200 mm long, 20 mm wide and 2 mm thick shall be mounted longitudinally inside the drum. These may be mounted on a frame that can be inserted inside the vessel from one end.

One end of the vessel 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.

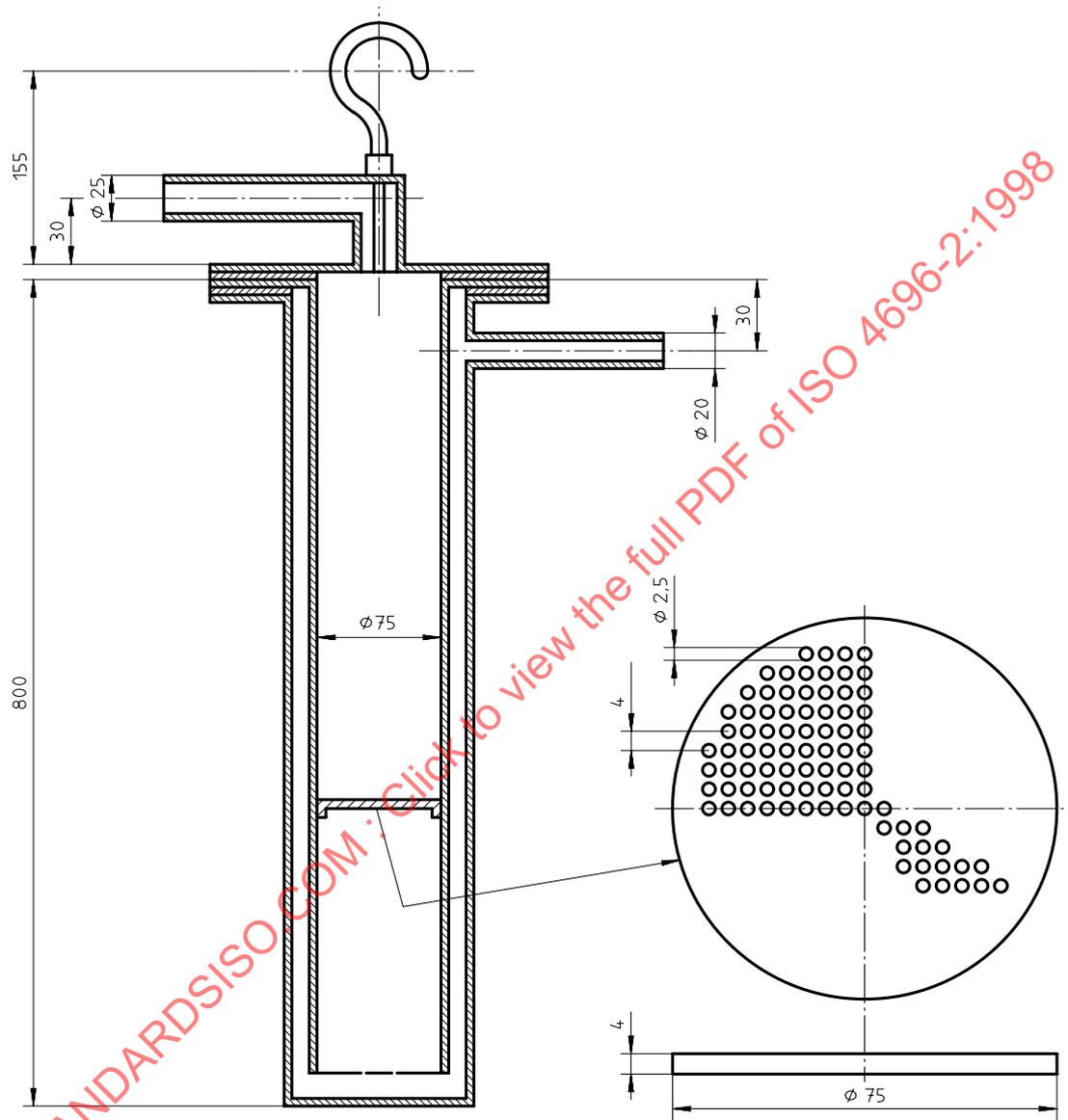
Figure 2 shows an example of such a tumbler drum.

4.4 Test sieves, having square mesh apertures of the following nominal sizes in accordance with ISO 3310-1 or ISO 3310-2:

22,4 mm; 20,0 mm; 16,0 mm; 12,5 mm; 10,0 mm; 2,8 mm.

4.5 Weighing device, of adequate load capacity and accurate to 0,1 g.

Dimensions in millimetres

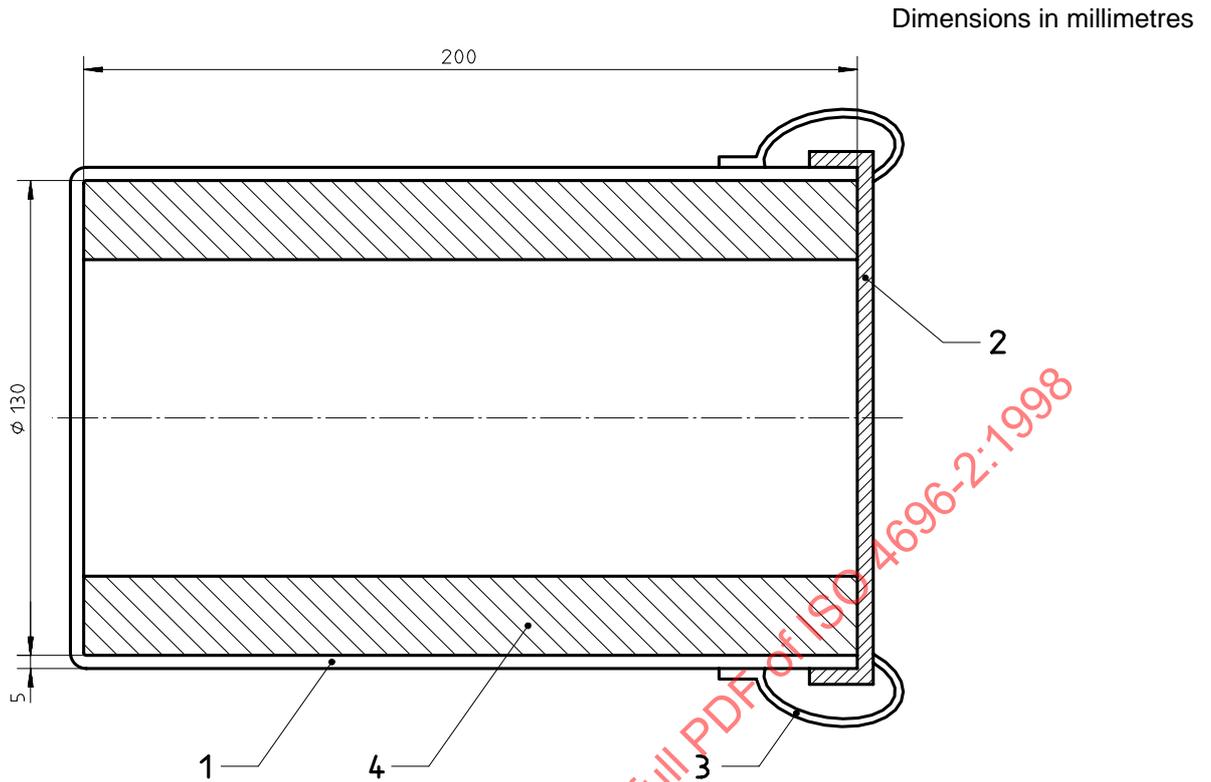


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Perforated plate
 Hole diameter: 2,5 mm
 Pitch between holes: 4 mm
 Number of holes: 241
 Total hole area: 1 180 mm²
 Thickness of plate: 4 mm

NOTE — The dimensions not specified in clause 4 are shown for information only.

Figure 1 — Example of reduction tube



Key

- 1 Vessel
- 2 Lid
- 3 Clamps
- 4 Frame with lifters
Lifters: 20 mm wide by 2 mm thick
Material: plain carbon steel

Figure 2 — Example of tumbler drum

5 Test conditions

Gas volumes and flow rates used in this part of ISO 4696 are as measured at a temperature of 0 °C and at atmospheric pressure (101,325 kPa) ³⁾.

5.1 Composition of reducing gas

The reducing gas shall consist of:

CO 30 % (V/V) ± 0,5 % (V/V);

N₂ 70 % (V/V) ± 0,5 % (V/V).

Impurities in the reducing gas shall not exceed:

Total impurities 0,1 % (V/V);

H₂ 0,01 % (V/V).

5.2 Flow rate of reducing gas

The reducing-gas flow rate shall, during the test period, be maintained at 15 l/min ± 0,5 l/min.

³⁾ 1 mmHg = 0,133 3 kPa; 1 atm = 0,101 325 MPa.

5.3 Temperature of test

The reducing gas shall be preheated before entering the test portion to maintain the test portion at $550\text{ °C} \pm 10\text{ °C}$ during the entire test period.

6 Sampling and sample preparation

The sample and test sample shall be taken in accordance with ISO 3082⁴⁾.

The test sample shall be oven-dried at $105\text{ °C} \pm 5\text{ °C}$ for at least 2 h and cooled to room temperature before testing.

The total mass of the test sample shall be approximately 2 kg on a dry basis, being prepared as follows:

a) Sized ore or sinter

The test sample in the size range of 16,0 mm to 20,0 mm shall be prepared as follows:

Screen the sample on a 20,0 mm sieve and carefully crush the +20,0 mm material until it all passes the 22,4 mm sieve. Combine all the fractions and remove, by sieving, the +20,0 mm and -16,0 mm material from the sample.

b) Pellets

The test sample in the size range of 10,0 mm to 12,5 mm shall be obtained by sieving and, after sieving, only pellets taken at random, for example by riffing, shall be used for the test.

7 Procedure

7.1 Number of determinations

Carry out the test at least in duplicate on each test sample.

7.2 Test portion

Weigh, to the nearest 0,1 g, approximately 500 g (\pm the mass of 1 particle) of the test sample. The test portions shall be obtained from the test sample either by means of riffle divider or by the manual increment-division method specified in ISO 3082.

7.3 Reduction

Place the test portion (7.2) in the reduction tube (4.1) so that the surface is even. Close the top of the reduction tube. Then insert the reduction tube into the furnace (4.2).

Replace the air in the tube with inert gas. Heat the test portion and, while heating, pass a flow of inert gas through the test portion at a flow rate of approximately 15 l/min. Continue the heating, while passing inert gas, until the test portion reaches the test temperature of 550 °C.

Unless the characteristics of the furnace and thermocouple are very well known, allow a period of at least 15 min for temperature equilibration at 550 °C.

4) ISO 10836:1994, *Iron ores — Method of sampling and sample preparation for physical testing*, will be revised to specify the procedures of sampling and sample preparation for this part of ISO 4696.

CAUTION — Carbon monoxide and the reducing gas which contains carbon monoxide are toxic and therefore hazardous. The reduction shall be carried out in a well ventilated area or under a hood. Precautions, in accordance with the safety codes of each country, shall be taken for the safety of the operator.

Introduce the reducing gas (see 5.1) at a flow rate of 15 l/min to replace the inert gas and to reduce the test portion. Continue the reduction with the reducing gas for 30 min.

After 30 min reduction time, stop the flow of the reducing gas and cool the test portion to a temperature below 100 °C in the reduction tube under a flow of inert gas.

7.4 Tumbling and sieving

Remove the test portion carefully from the reduction tube, determine the mass (mass m_0) and place it in the tumbler drum (4.3). Fasten the lid tightly and rotate the drum for a total of 900 revolutions at a rate of 30 rev/min \pm 1 rev/min.

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

NOTE — Equivalent mechanical sieving may be used provided that preliminary tests give similar results to hand sieving within the permissible tolerance of 2 % absolute.

8 Expression of results

The reduction-disintegration index, RDI-2_{-2,8}, expressed as a percentage by mass, is calculated from the following equation:

$$\text{RDI-2}_{-2,8} = 100 - \frac{m_1}{m_0} \times 100$$

where

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

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

Repeatability, number of tests and calculation of the final test result are as specified in annex A.

9 Test report

The test report shall include the following information:

- a) a reference to this part of ISO 4696;
- b) all details necessary for identification of the sample;
- c) the total mass of the material inserted into the tumbler drum and taken from the tumbler drum;
- d) the sieving conditions, e.g. the method of sieving and the sieving time;
- e) the reducing-gas composition used;
- f) the result of the test, i.e. the reduction-disintegration index, RDI-2_{-2,8}, expressed to one decimal place;

- g) 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 name and address of the test laboratory;
- i) the signature of the person responsible for the test;
- j) the date of the test.

10 Verification

Regular checking of apparatus and procedures is essential to ensure repeatable results. Checks shall be carried out at regular intervals. The frequency of checking is a matter for each laboratory to determine. The following items shall be checked:

- Test sample preparation
 - sieves
 - weighing device
- Reduction apparatus
 - reduction tube
 - test temperature
 - gas composition
 - gas flow rate
 - timer
- Tumbler
 - tumbler drum
 - speed of rotation
 - revolution counter
 - lifters
 - test sieve
 - weighing device

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

Appropriate records of verification activities shall be maintained.