

INTERNATIONAL STANDARD

ISO
4696-1

First edition
1996-10-01

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

Part 1:

Reaction with CO, CO₂ and H₂

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

Partie 1: Réaction avec CO, CO₂ et H₂



Reference number
ISO 4696-1:1996(E)

Foreword

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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 4696-1 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Subcommittee SC 3, *Physical testing*.

Together with part 2, 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. Annex B is for information only.

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International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Introduction

This part of ISO 4696 describes a test method for evaluating the disintegration behaviour of iron ores under specific conditions relevant to the low-temperature zone in the blast furnace for ironmaking.

It describes a test method for evaluating the disintegration behaviour of iron ore under specific conditions. The specific conditions are:

- a test sample having a specified size range;
- a specified test sample mass;
- isothermal reduction;
- reduction in a fixed bed;
- reduction with a specified gas composition and flow;
- tumbling in a drum having specified dimensions.

The results of this test should be considered in conjunction with the results of other reduction tests, e.g. ISO 4695, ISO 4698 and ISO 7215 (see annex B), particularly those conducted at high temperatures.

ISO 4696-2 describes an alternative method adopting different test conditions.

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

Part 1:

Reaction with CO, CO₂ and H₂

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 500 °C.

The method is applicable to sized 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 3081:1986, *Iron ores — Increment sampling — Manual method*.

ISO 3083:1986, *Iron ores — Preparation of samples — Manual method*.

3 Principle

A test portion with a specified size range is subjected to static reduction at a temperature of 500 °C using reducing gas consisting of CO, CO₂, H₂ and N₂.

After 1 h reduction time, the test portion is cooled to a temperature below 100 °C and tumbled by using a small tumbler drum for 300 revolutions in total. It is then sieved with test sieves having square mesh apertures of 6,30 mm, 3,15 mm and 500 µm.

The reduction-disintegration index (RDI-1) is calculated as a quantitative measure of the degree of disintegration of an iron ore that has been reduced and then tumbled: the percentage masses of material greater than 6,30 mm, less than 3,15 mm and less than 500 µm, respectively, are related to the total mass of the test portion after reduction and before tumbling.

4 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)¹⁾.

4.1 Composition of reducing gas

The reducing gas shall consist of

CO	20 % (V/V) ± 0,5 % (V/V)
CO ₂	20 % (V/V) ± 0,5 % (V/V)
H ₂	2,0 % (V/V) ± 0,5 % (V/V)
N ₂	58 % (V/V) ± 0,5 % (V/V)

4.2 Purity of reducing gas

Impurities in the reducing gas shall not exceed

O ₂	0,1 % (V/V)
H ₂ O	0,2 % (V/V)

4.3 Flow rate of reducing gas

The reducing-gas flow rate shall, during the test period, be maintained at 20 l/min ± 1 l/min.

4.4 Temperature of test

The reducing gas shall be preheated before entering the test portion to maintain the test portion at 500 °C ± 5 °C during the entire test period.

5 Apparatus

Figure 1 shows a schematic example of the test apparatus which shall consist of the following:

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

5.2 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 2 shows an example of such a reduction tube.

5.3 Electrically heated furnace, having a heating capacity sufficient to maintain the entire test portion and the gas entering the bed at 500 °C, and being equipped with a heating element suitable for the specified temperature.

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

5.4 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 3 shows an example of such a tumbler drum.

5.5 Test sieves, having square mesh apertures of the following nominal sizes:

16,0 mm; 12,5 mm; 10,0 mm; 6,30 mm; 3,15 mm and 500 μm .

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

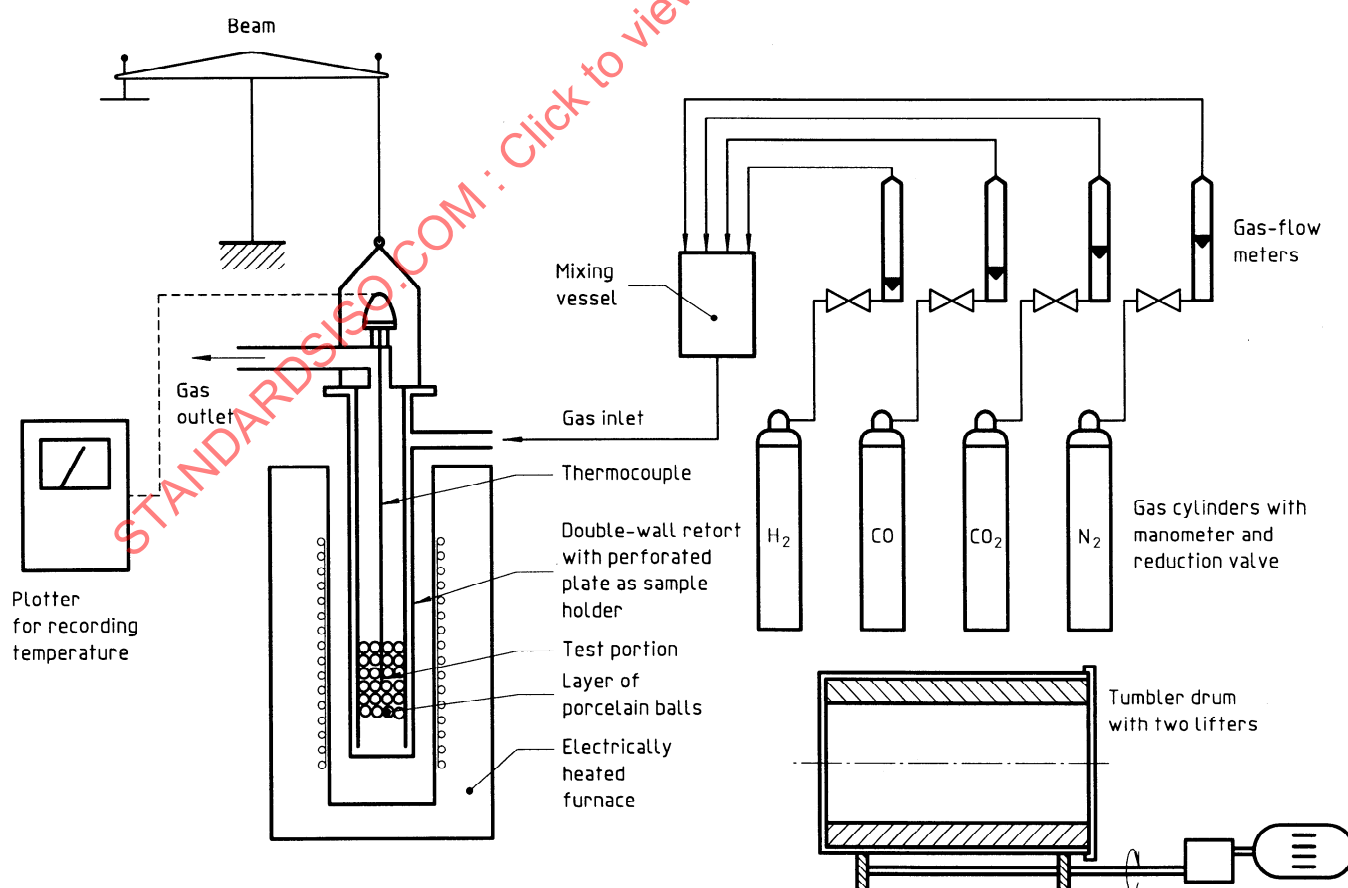
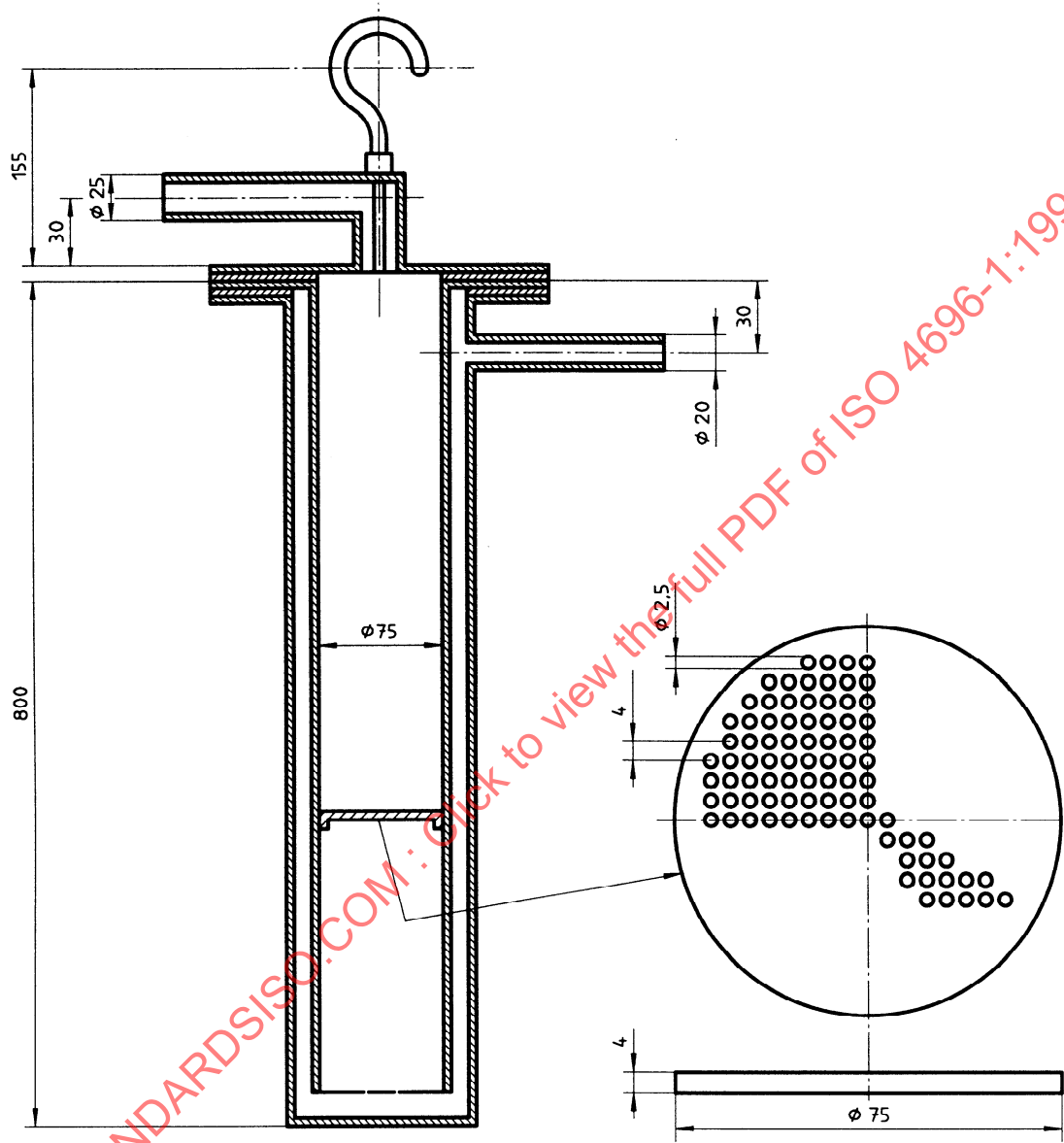


Figure 1 — Arrangement of a test unit

Dimensions in millimetres

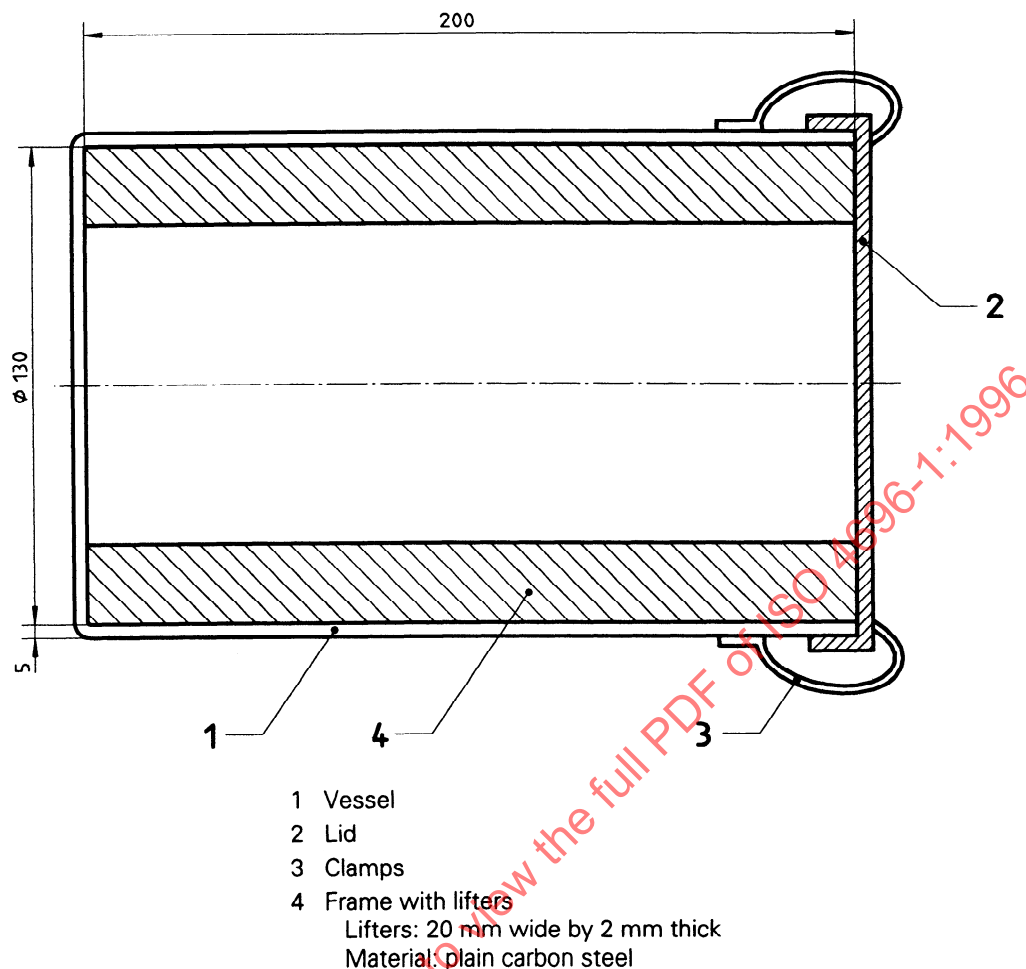


Perforated plate
Hole diameter: 2,5 mm
Pitch between holes: 4 mm
Number of holes: 241
Total hole area: 1 180 mm²
Thickness of plate: 4 mm

NOTE — Dimensions not specified in clause 5 are shown for information only.

Figure 2 — Example of reduction tube (5.2)

Dimensions in millimetres

**Figure 3 — Example of tumbler drum (5.4)**

6 Preparation of test sample

The test sample shall be prepared in accordance with ISO 3083 from the sample for physical testing which has been taken in accordance with ISO 3081.

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) Pellets

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

b) Ore or sinter

The test sample in the size range of 10,0 mm to 12,5 mm shall be prepared as follows:

Screen the sample on a 12,5 mm sieve and carefully crush the +12,5 mm material until it all passes the 16,0 mm sieve. Combine all the fractions and remove, by sieving, the +12,5 mm and -10,0 mm material from the sample.

7 Procedure

7.1 Number of tests

Carry out the test at least in duplicate on each test sample. (See annex A.)

7.2 Test portion

Weigh, to the nearest 0,1 g, approximately 500 g (± 1 particle) of the test sample. This test portion shall be obtained from the test sample either by means of a riffle divider or by a manual increment-division method.

7.3 Reduction

Place a double-layer bed of porcelain pellets, having a size range of 10,0 mm to 12,5 mm, on the perforated plate (see figure 2).

Place the test portion (7.2) on the porcelain pellets in the reduction tube (5.2) and level the surface. Place the thermocouple (see figure 1) in the centre of the test portion. Close the top of the reduction tube. Then insert the reduction tube into the furnace (5.3) and, if desired, attach it to the weighing device of appropriate capacity and accuracy (5.6), ensuring that there is no contact with the furnace or heating elements.

NOTE — The use of a weighing device for continuous monitoring is optional.

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 20 l/min. Continue the heating, while passing inert gas, until the test portion reaches the test temperature of $500\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

Unless the characteristics of the furnace and thermocouple are very well known, allow a period of about 15 min for temperature equilibration at $500\text{ }^{\circ}\text{C}$.

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 4.1) at a flow rate of $20\text{ l/min} \pm 1\text{ l/min}$ to replace the inert gas and to reduce the test portion. Continue the reduction with the reducing gas for 1 h.

After 1 h reduction time, stop the flow of the reducing gas and cool the test portion to a temperature below $100\text{ }^{\circ}\text{C}$ in the reduction tube under a flow of inert gas.

7.4 Tumbling

Remove the test portion carefully from the reduction tube, determine the mass (m_0) and place it in the tumbler drum (5.4). Fasten the lid tightly and rotate the drum for a total of 300 revolutions at a rate of 30 rev/min ± 1 rev/min.

Remove all material from the drum, determine the mass and hand sieve with care on 6,30 mm, 3,15 mm and 500 μm sieves. Determine and record the mass of each fraction retained on the 6,30 mm (m_1), 3,15 mm (m_2) and 500 μm (m_3) sieve. Material lost during tumbling and sieving shall be considered to be less than 500 μm .

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

8 Expression of results

The reduction-disintegration index RDI-1, expressed as a percentage by mass, is calculated from the following equations:

$$\text{RDI-1}_{+6,3} = \frac{m_1}{m_0} \times 100$$

$$\text{RDI-1}_{-3,15} = \frac{m_0 - (m_1 + m_2)}{m_0} \times 100$$

$$\text{RDI-1}_{-0,5} = \frac{m_0 - (m_1 + m_2 + m_3)}{m_0} \times 100$$

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 oversize fraction retained on the 6,30 mm sieve;

m_2 is the mass, in grams, of the oversize fraction retained on the 3,15 mm sieve;

m_3 is the mass, in grams, of the oversize fraction retained on the 500 μm sieve.

Repeatability and number of tests are given in annex A.

9 Test report

The test report shall include the following information:

- a reference to this part of ISO 4696;
- all details necessary for identification of the sample;
- the sieving conditions, e.g. the method of sieving and the sieving time;
- the total mass of the material inserted into the tumbler drum and taken from the tumbler drum;
- the results of the test, i.e. the reduction-disintegration indices expressed to one decimal place;
- 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.

Annex A

(normative)

Repeatability and number of tests

For a paired result, the acceptable values for the range at each stage are indicated in table A.1.

Table A.1 — Mean value of RDI-1 and range

Mean value of RDI-1 % (m/m)	Range ($x_1 - x_2$)		
	A	B	C
100	0	0	0
95	1,0	1,2	1,3
90	2,0	2,4	2,6
85	3,0	3,6	3,9
80	4,0	4,8	5,2
75	4,0	4,8	5,2
50	4,0	4,8	5,2
25	4,0	4,8	5,2
20	4,0	4,8	5,2
15	3,0	3,6	3,9
10	2,0	2,4	2,6
5	1,0	1,2	1,3
0	0	0	0

The computation of the final results is carried out as described in the flow diagram in figure A.1.