INTERNATIONAL STANDARD

# ISO 4696-1

First edition
1996-10-01

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

## Part 1: Reaction with CO, $CO_2$ and $H_2$

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

*Partie 1: Réaction avec CO, $CO_2$ et $H_2$*

Reference number
ISO 4696-1:1996(E)

## 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.

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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.

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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, $CO_2$ and $H_2$*

— *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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# 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.

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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.

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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, $CO_2$ and $H_2$

## 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.

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

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## 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, $CO_2$, $H_2$ and $N_2$.

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.

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## 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*) |
| $H_2O$ | 0,2 % (*V*/*V*) |

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### 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.

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### 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.

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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.

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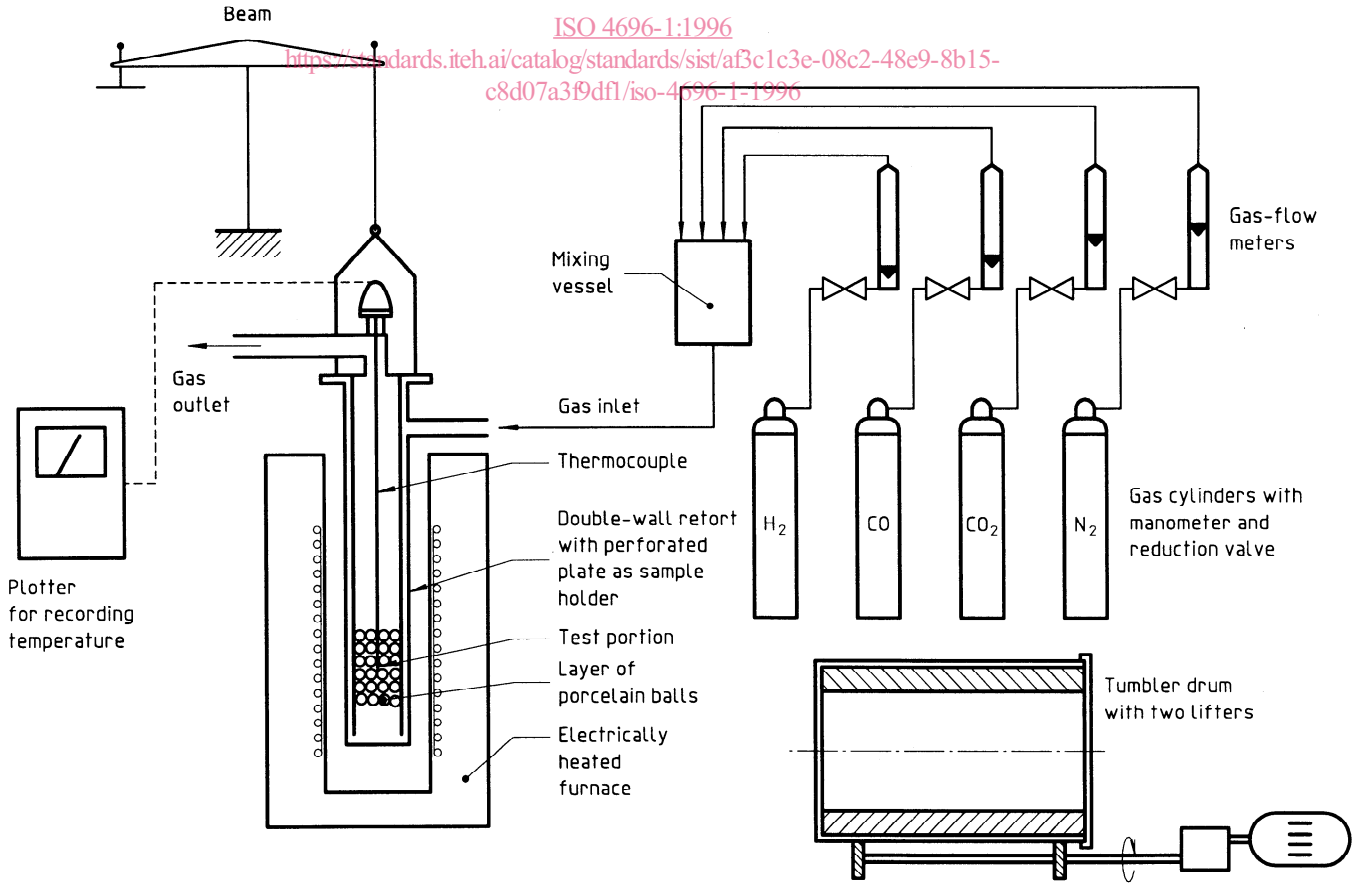
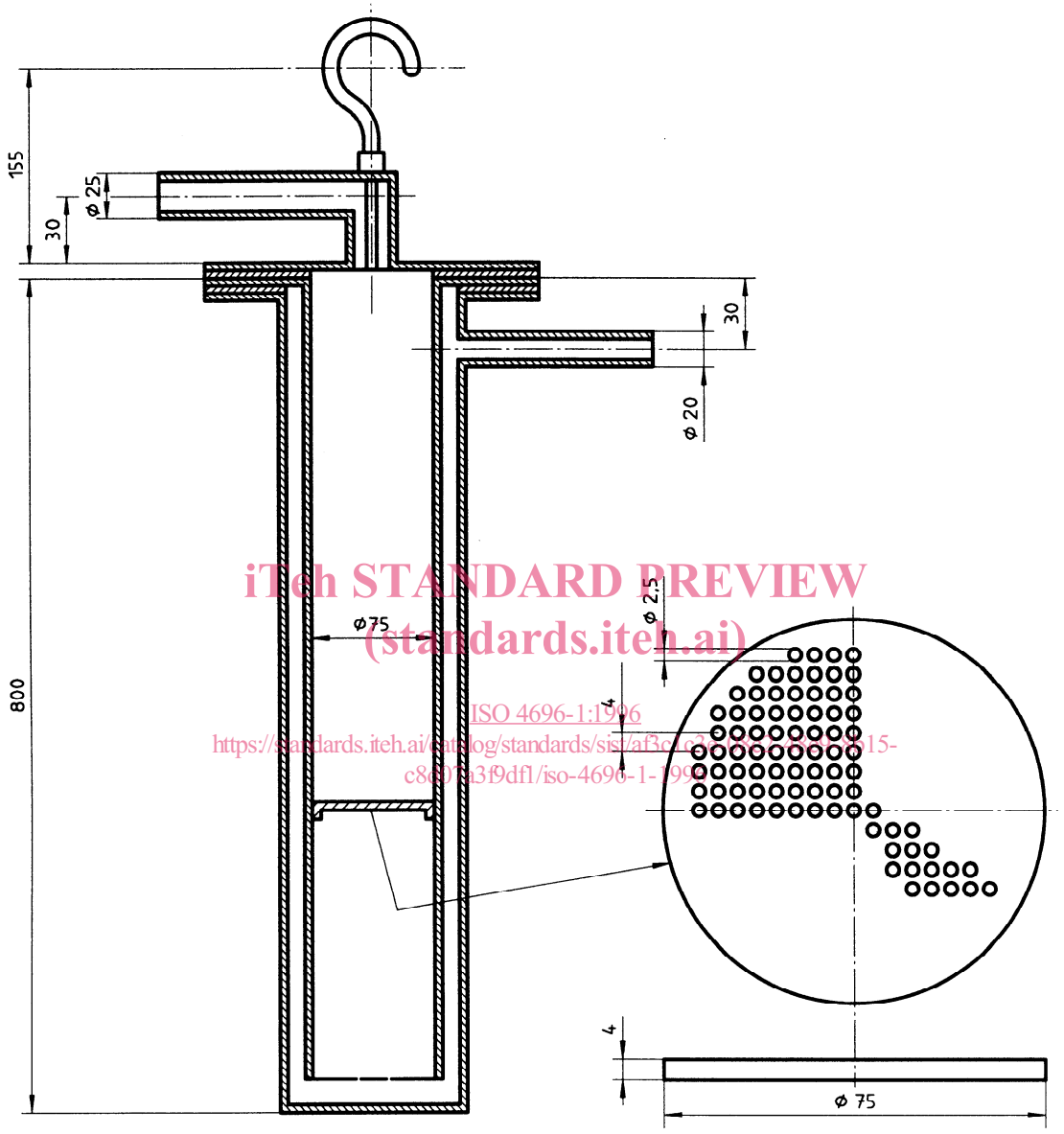



**Figure 1 — Arrangement of a test unit**

Dimensions in millimetres

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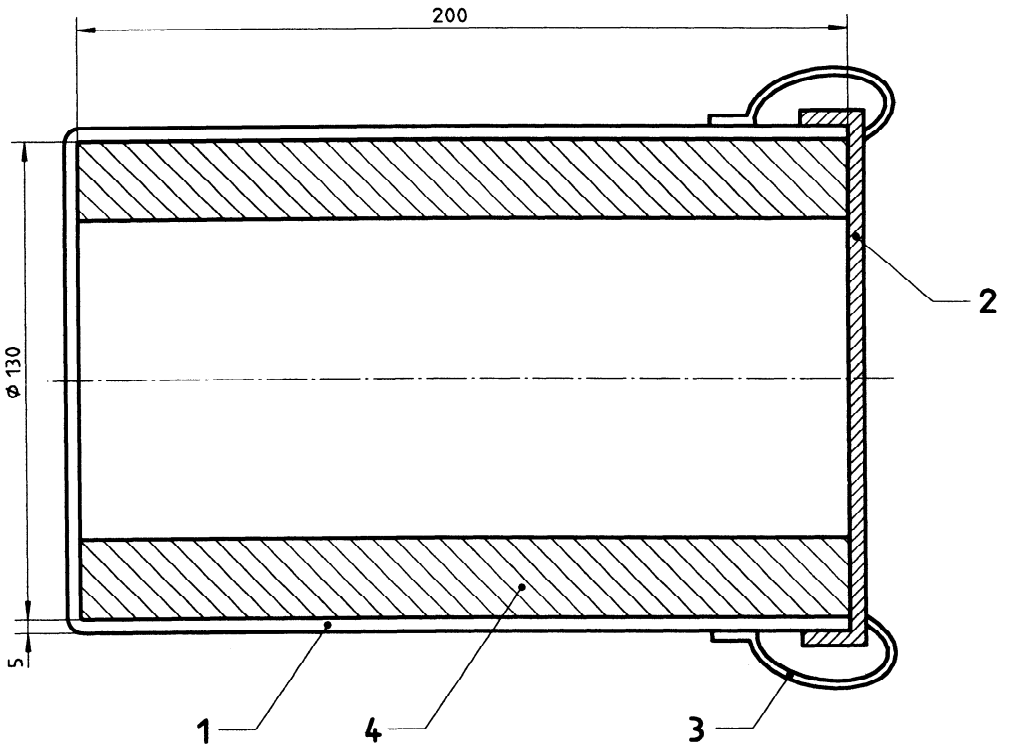
**Perforated plate**
Hole diameter: 2,5 mm
Pitch between holes: 4 mm
Number of holes: 241
Total hole area: 1 180 $mm^2$
Thickness of plate: 4 mm

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

**Figure 2 — Example of reduction tube (5.2)**

Dimensions in millimetres

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

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**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 °C ± 5 °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 riffling, 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.