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

Part 1:

Reduction with CO, CO₂, H₂ and N₂

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

ISO 4696-1:2015

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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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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.

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).

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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 third edition cancels and replaces the second edition (ISO 4696-1:2007), 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, CO₂, H₂ and N₂
- Part 2: Reduction with CO and N₂

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 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.

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

Part 1: Reduction with CO, CO₂, H₂ and N₂

CAUTION — This International Standard may involve hazardous operations and equipment. This 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, carbon dioxide, hydrogen, 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 500 °C, using a reducing gas consisting of CO, CO₂, H₂, and N₂, for 60 min. The reduced test portion is tumbled in a specific tumble drum for 300 revolutions and then sieved with sieves having square openings of 6,30 mm, 3,15 mm, and 500 µm. Three reduction-disintegration indices (RDI) are calculated as the mass percentage of material greater than 6,30 mm, less than 3,15 mm, and less than 500 µm.

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, sinters, and lump ores shall be $- 12,5 \text{ mm} + 10,0 \text{ mm}$.

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 $105 \text{ °C} \pm 5 \text{ °C}$ 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 riffing, can be applied to obtain the test portions.

At least four test portions, each of approximately 500 g (\pm the mass of 1 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.

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6 Apparatus

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6.1 General

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The test apparatus shall comprise the following:

- a) ordinary laboratory equipment, such as an oven, hand tools, a time-control device, 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 $75 \text{ mm} \pm 1 \text{ mm}$. 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 a 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 gas entering the bed, at $500\text{ °C} \pm 5\text{ °C}$.

6.4 Porcelain balls, having a size range between 10,0 mm and 12,5 mm and of sufficient quantity to form a double-layer bed on the perforated plate.

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

6.6 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.

6.7 Rotation equipment, capable of ensuring that the drum attains full speed in one revolution, rotates at a constant speed of $30\text{ r/min} \pm 1\text{ r/min}$ 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.

6.8 Test sieves, conforming to ISO 3310-1 or ISO 3310-2 and having square apertures of the following nominal sizes: 6,30 mm; 3,15 mm, and 500 μm .

6.9 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	$20,0\% \pm 0,5\%$ (volume fraction)
CO ₂	$20,0\% \pm 0,5\%$ (volume fraction)
H ₂	$2,0\% \pm 0,5\%$ (volume fraction)
N ₂	$58,0\% \pm 0,5\%$ (volume fraction)