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Iron ores for blast furnace feedstocks — Determination of reduction under load

Minerais de fer pour charges de hauts fourneaux — Détermination de la réduction sous charge

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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).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 3, *Physical testing*.

This fourth edition cancels and replaces the third edition (ISO 7992:2015), which has been technically revised to adjust the apparatus described in the previous edition according to the original text.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document 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 document 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 reduction under load

CAUTION — This document can involve hazardous operations and equipment. This document does not purport to address all of the safety issues associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 Scope

This document specifies a method to provide a relative measure for evaluating the structural stability of iron ores when reduced under conditions resembling those prevailing in the reduction zone of a blast furnace.

This document is applicable to lump ores and hot-bonded pellets.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2597-1, *Iron ores — Determination of total iron content — Part 1: Titrimetric method after tin(II) chloride reduction*

ISO 2597-2, *Iron ores — Determination of total iron content — Part 2: Titrimetric methods after titanium(III) chloride reduction*

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

ISO 9035, *Iron ores — Determination of acid-soluble iron(II) content — Titrimetric method*

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.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

The test portion is isothermally reduced in a fixed bed, at 1 050 °C, under static load, using a reducing gas consisting of CO, H_2 and N_2, until a degree of reduction of 80 % is obtained. The differential gas pressure across the bed and the change in the test bed height are measured at 80 % reduction.

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 and lump ores shall be –12,5 mm +10,0 mm.

A test sample of at least 6,0 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 five test portions, each of approximately 1 200 g (±the mass of one particle) shall be prepared from the test sample: four test portions for testing and one for chemical analysis.

Weigh the test portions to the nearest 1 g and register the mass of each test portion on its recipient label.

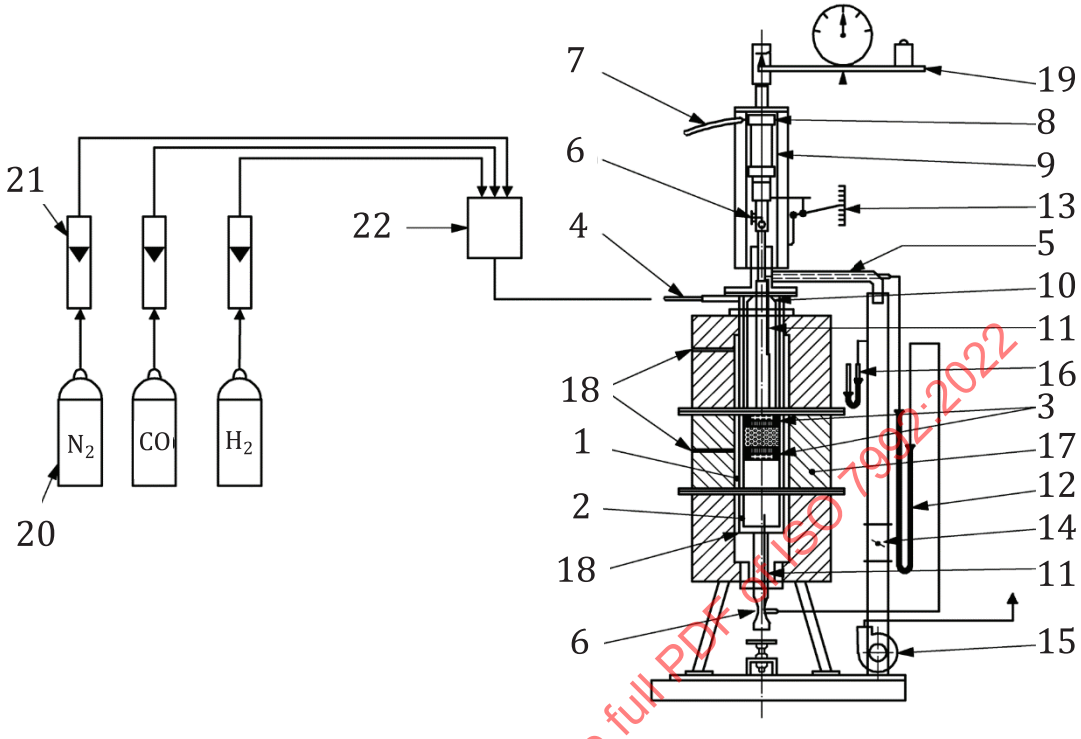
6 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

6.1 Ordinary laboratory equipment, such as an oven, hand tools, a time-control device and safety equipment.

6.2 Reduction tube, with a double wall made of non-scaling, heat-resistant metal to withstand temperatures higher than 1 050 °C and resistant to deformation. The internal diameter of the inner reduction tube shall be 125 mm ± 1 mm. A removable perforated plate, made of non-scaling, heat-resistant metal to withstand temperatures higher than 1 050 °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 10 mm thick, with a diameter 1 mm less than the internal diameter of the tube. The holes in the plate shall be 3 mm to 4 mm in diameter, at a pitch centre distance of 5 mm to 6 mm. The internal diameter of the outer reduction tube shall be enough to allow gas flow preheating before entering the inner reduction tube.

Figure 1 shows an example of the test apparatus.



Key

Reduction tube

1 outer reduction tube
2 inner reduction tube
3 upper and lower perforated plates comprising test portion
4 gas inlet
5 gas outlet
6 thermocouple for measuring the reduction temperature

Loading device

7 compressed air inlet
8 pressure cylinder
9 frame for pressure cylinder
10 loading ram

Device for measuring differential gas pressure

11 differential gas pressure upper and lower probes
12 differential gas pressure manometer

Height-measuring device

13 linear scale

Waste gas

14 throttle valve
15 waste-gas fan
16 suction gauge

Furnace

18 electrically heated furnace
19 furnace wall thermocouples
20 balance

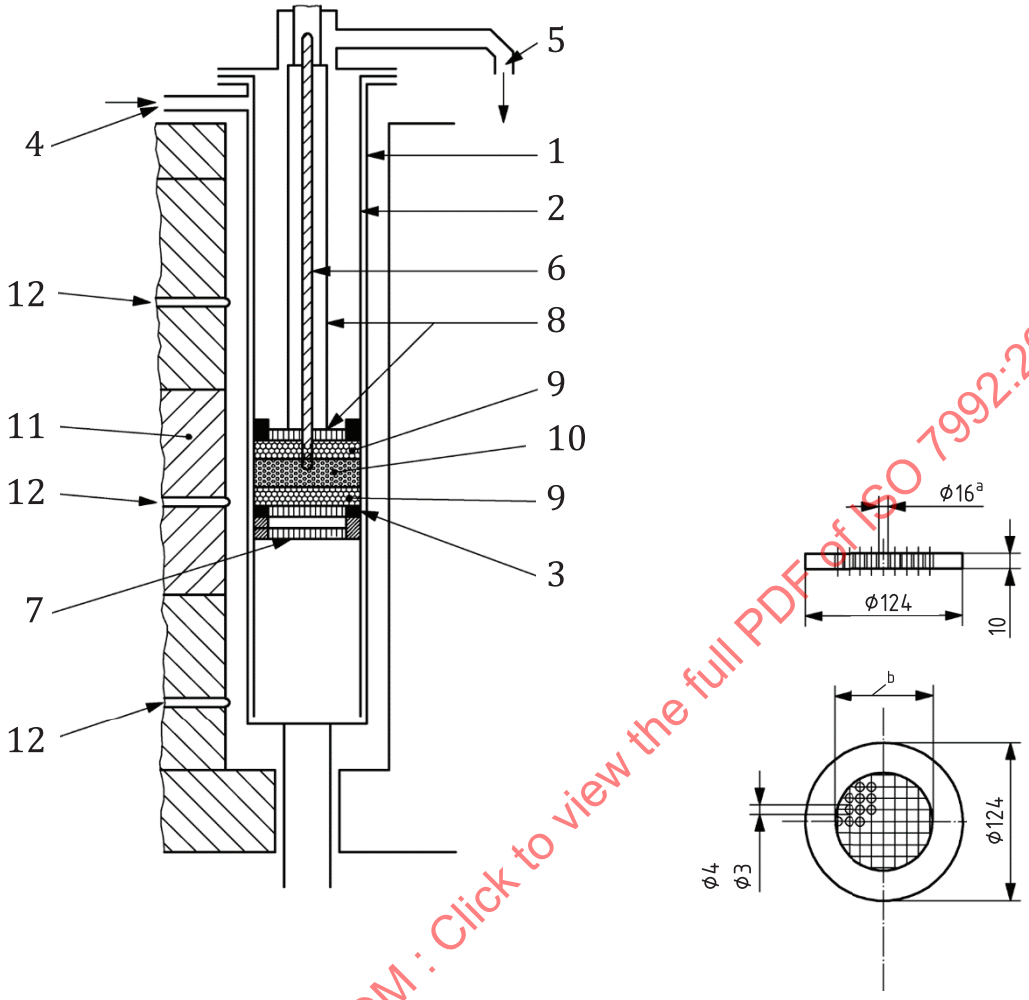
Gas supply system

20 gas cylinders
21 gas flow meters
22 mixing vessel

Figure 1 — Example of the test apparatus (schematic diagram)

Figure 2 shows an example of a reduction tube.

Dimensions in millimetres



Key

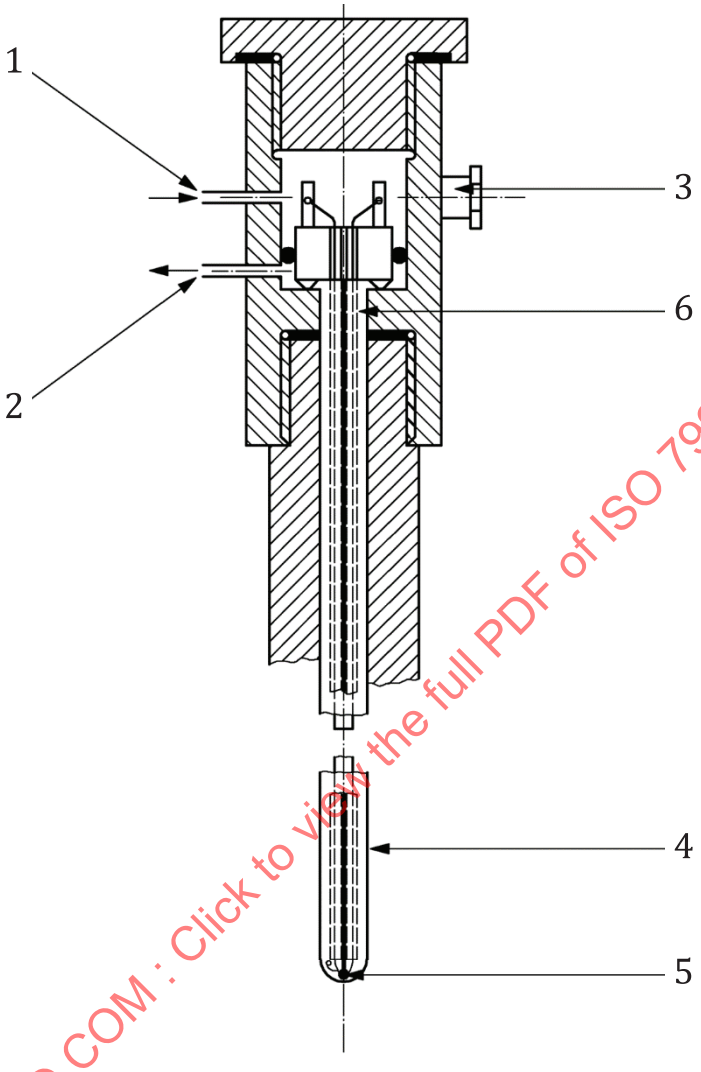
Reduction tube

1	outer reduction tube	8	loading ram with rigid perforated footplate
2	inner reduction tube (Ø 125 mm)	9	porcelain balls (two layers)
3	removable perforated plate	10	test portion (1 200 g)
4	opening for gas inlet	11	furnace wall
5	opening for gas outlet	12	furnace wall thermocouples (upper, medial and lower)
6	thermocouple for measuring the reduction temperature	a	Ø 16 for thermocouple entrance.
7	perforated support	b	14 holes × 5 or 6 pitch.

NOTE Key numbers do not coincide with the ones in Figures 1 and 3.

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

Figure 3 shows the principle for oxygen flushing of thermocouples to avoid mismeasurement due to carbon deposition.



Key

1 oxygen inlet
2 oxygen outlet
3 thermocouple exit
4 protective tube
5 thermocouple tip
6 inner tube with four borings

NOTE Key numbers do not coincide with the ones in Figures 1 and 2.

Figure 3 — Principle of oxygen flushing of thermocouples to avoid mismeasurement due to carbon deposition

6.3 Loading device, capable of supplying a total static load of 50 kPa ± 2 kPa evenly to the test portion. The load shall be transferred by means of a ram with rigid perforated footplate, so as to distribute it evenly to the surface of the porcelain balls placed on top of the test portion. The footplate shall be 10 mm thick, with a diameter 1 mm less than the internal diameter of the tube. The holes in the plate shall be 3 mm to 4 mm in diameter, at a pitch centre distance of 5 mm to 6 mm.

6.4 Device for measuring differential gas pressure, having a resolution of 0,01 kPa.

6.5 Height-measuring device, having a resolution of 0,1 mm.

6.6 Porcelain balls, having a size range between 10,0 mm and 12,5 mm.

6.7 Furnace, having a heating capacity and temperature control able to maintain the entire test portion, as well as the gas entering the bed, at 1 050 °C ± 10 °C.

6.8 Balance, capable of weighing the reduction tube assembly, including the test portion, to an accuracy of 1 g. The balance shall have an appropriate device to suspend the reduction tube assembly.

6.9 Gas-supply system, capable of supplying the gases and regulating gas flow rates. A frictionless connection between the gas-supply system and the reduction tube shall be ensured to not affect the weight loss determination during reduction.

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

7 Test conditions

7.1 General

Volumes and flow rates of gases used are as measured at a reference temperature of 0 °C and at a reference atmospheric pressure of 101,325 kPa.

7.2 Reducing gas

7.2.1 Composition

The reducing gas shall consist of the following:

CO 40,0 % ± 0,5 % (volume fraction);

H_2 2,0 % ± 0,5 % (volume fraction);

N_2 58,0 % ± 0,5 % (volume fraction).

7.2.2 Purity

Impurities in the reducing gas shall not exceed the following:

CO_2 0,2 % (volume fraction);

O_2 0,1 % (volume fraction);

H_2O 0,2 % (volume fraction).

7.2.3 Flow rate

The flow rate of the reducing gas, during the entire reducing period, shall be maintained at 83 l/min ± 1 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 N2 shall be maintained at 50 l/min until the test portion reaches 1 050 °C and at 83 l/min during temperature-equilibration period. If desired, the test portion may be cooled under nitrogen to below 100 °C. 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 1 050 °C ± 10 °C during the entire reducing period and, as such, the reducing gas shall be preheated before entering the test portion.

7.5 Loading of the test portion

During the entire heating and reducing periods, the test portion shall be subjected to a constant load of 50 kPa ± 2 kPa applied over the surface of the bed.

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 Chemical analysis

Take, at random, one of the test portions prepared in 5.2 and use it for the determination of the iron(II) oxide content (w_1) in accordance with ISO 9035 and the total iron content (w_2) in accordance with ISO 2597-1 or ISO 2597-2.

8.3 Reduction

In order to achieve a more uniform gas flow, place a double-layer bed of porcelain balls (6.6) in the reduction tube (6.2) on the perforated plate and level its surface. Measure the height of the top surface of the porcelain layer.

Take, at random, another test portion prepared in 5.2 and record its mass (m_0). Place it on the bed of porcelain balls and level its surface.

Place a further double layer of the porcelain balls upon the test portion and level its surface. Measure the height of the top surface of this porcelain layer.

Close the top of the reduction tube by connecting the heating assembly containing the loading device (6.3) to the reduction tube. Insert the reduction tube assembly into the furnace (6.7) and suspend it centrally from the balance (6.8), ensuring that there is no contact with the furnace wall or heating elements.

Connect the thermocouple, ensuring that its tip is at the central position, as shown in Figure 2. Connect the measurement devices for the differential pressure (6.4) and for the change in the height of the test bed (6.5).

Connect the gas-supply system (6.9), the discharge line and the compressed air to the loading device. Apply a load of 50 kPa ± 2 kPa.

Pass a flow of N_2 through the test portion at a rate of 50 l/min and commence heating. When the temperature of the test portion approaches 1 050°C, increase the flow rate to 83 l/min. Continue

heating while maintaining the flow of N_2 until the balance reading is constant and the temperature is constant at 1 050 °C ± 10 °C for 10 min.

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.

Tare the balance, start the time control device and immediately introduce the reducing gas at a flow rate of 83 l/min to replace the N_2. Record the differential pressure across the test bed, the height of the test bed and the mass loss of the test portion (Δm_t) continuously or at least every 5 min for the first 30 min, and thereafter at 10 min intervals.

Calculate the degree of reduction, R_t, relative to the iron(III) state, after t min, as shown in Formula (1):

$$R_t = \left(\frac{0{,}111 w_1}{0{,}430 w_2} + \frac{\Delta m_t}{m_0 \times 0{,}43 w_2} \times 100 \right) \times 100 \quad (1)$$

where

m_0 is the mass, in grams, of the test portion;

Δm_t is the mass loss, in grams, of the test portion after reduction time t;

w_1 is the iron(II) oxide content, as a percentage by mass, of the test sample prior to the test, determined in accordance with ISO 9035, it is calculated from the iron(II) content by multiplying it by the oxide conversion factor FeO/Fe(II) = 1,286;

w_2 is the total iron content, as a percentage by mass, of the test portion prior to the test, determined in accordance with ISO 2597-1 or ISO 2597-2.

NOTE A derivation of Formula (1) is given in Annex B.

When the degree of reduction reaches 80 %, turn off the power and stop the flow of the reducing gas. Remove the load and record the time.

If, after 4 h, 80 % of reduction has not been achieved, the reduction may be stopped.

If any further evaluations are to be performed on the reduced test portion, introduce N_2 at a flow rate of 5 l/min until the test portion reaches room temperature.

9 Expression of results

9.1 Preparation of the reduction curve

Prepare the reduction curve by plotting the degree of reduction R_t against time t.

9.2 Calculation of the differential pressure at 80 % reduction ($\Delta p80$)

The differential pressure at 80 % reduction, $\Delta p80$, expressed in kPa, is calculated as follows.

Plot the differential gas pressure against the degree of reduction and read off, from the curve, the differential pressure ($\Delta p80$) corresponding to 80 % reduction.

Record the result to two decimal places.

9.3 Calculation of the change in the height of the test bed at 80 % reduction ($\Delta h80$)

The change in the height of the test bed at 80 % reduction, $\Delta h80$, expressed as a percentage, is calculated as follows.

Plot the percentage change in the height of the test bed against the degree of reduction and read off, from the curve, the percentage change in the height of the test bed ($\Delta h80$) corresponding to 80 % reduction.

Record the result to one decimal place.

9.4 Repeatability and acceptance of test results

Follow the procedure in Annex A for $\Delta p80$ index by using the repeatability r = 0,30 $\overline{\Delta p}80$ (kPa). The results shall be reported to two decimal places.

10 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 7992;

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 document or regarded as optional, as well as any incident which could have had an influence on the results;

h) the differential gas pressure at 80 % reduction, $\Delta p80$;

i) the change in the height of the test bed at 80 % reduction, $\Delta h80$;

j) the total iron and iron(II) contents of the test portion before reduction;

k) the time taken to reach 80 % reduction;

l) the reducibility dR/dt (O/Fe = 0,9), expressed in percentage per minute.

11 Verification

Regular checking of the 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:

— weighing device;

— reduction tube;

— temperature control and measurement devices;

— loading device;

— gas flow meters;

— purity of gases;

— device for measuring the differential gas pressure;

— height-measuring device;

— cleanliness of porcelain balls;

— balance;

— time-control device.

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

Appropriate records of verification activities shall be maintained.