INTERNATIONAL STANDARD

ISO 11258

First edition
1999-08-01

Iron ores — Determination of reducibility and metallization of feedstock for direct reduction by gas reforming processes

Minerais de fer — Détermination de la réductibilité et de la métallisation des charges utilisées dans les procédés de reforming par réduction directe

Reference number
ISO 11258:1999(E)

Contents

Page

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 11258 was prepared by Technical Committee ISO/TC 102, *Iron ores and direct reduced iron*, Subcommittee SC 5, *Physical testing of direct reduction feedstock and DRI*.

Annex C forms an integral part of this International Standard. Annexes A and B are for information only.

Introduction

Direct reduction processes are intended to partially or almost completely reduce iron ores by thermal processes in order to form high grade feedstocks for iron- and steelmaking. Several kinds of direct reduction process are in operation worldwide and others are still under development. The behaviour of the iron ores, as feedstock, may vary from process to process. This International Standard was prepared in order to specifically address direct reduction by gas reforming processes.

The obtained reducibility index is a relative measure of the reducibility behaviour, and the degree of metallization is a relative measure of the metallization behaviour of the iron ore.

The results of this test should be considered in conjunction with the results of other tests used to evaluate the quality of iron ores as feedstock for direct reduction processes.

Iron ores — Determination of reducibility and metallization of feedstock for direct reduction by gas reforming processes

WARNING — This International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems 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 International Standard describes a test method for evaluating the reducibility and metallization behaviour of iron ore pellets and lumps under conditions which resemble the ones prevailing in direct reduction by gas reforming processes.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 2597-1:1994, *Iron ores — Determination of total iron content — Part 1: Titrimetric methods after tin (II) chloride reduction.*

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

ISO 5416:1997, *Direct reduced iron — Determination of metallic iron content — Bromine-methanol titrimetric method.*

ISO 9035:1989, *Iron ores — Determination of acid-soluble iron (II) content — Titrimetric method.*

ISO 9507:1990, *Iron ores — Determination of total iron content — Titanium (III) chloride reduction methods.*

ISO 9508:1990, *Iron ores — Determination of total iron content — Silver reduction titrimetric method.*

ISO 10836:1994, *Iron ores — Method of sampling and sample preparation for physical testing.*

ISO 11323:1996, *Iron ores — Vocabulary.*

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

3 Terms and definitions

For the purposes of this International Standard the terms and definitions given in ISO 11323 and the following apply.

3.1
degree of reduction
extent to which oxygen has been removed from iron oxides, expressed as the ratio of oxygen removed to oxygen originally combined with iron, relative to the iron (III)-state

4 Principle

Heating of the test portion in an inert atmosphere.

Isothermal reduction of the test portion at a specified size range in a fixed bed, at a temperature of 800 °C, using a reducing gas consisting of H_2, CO, CO_2 and N_2.

Continuous weighing or intermittent weighing of the test portion at specified time intervals for 90 min reduction in total.

Cooling of the test portion in an inert atmosphere.

Calculation of the degree of reduction, after reduction for 90 min, relative to the iron (III)-state and the reducibility index at the oxygen:iron ratio of 0,9.

Calculation of the degree of metallization from the chemical analysis of the reduced test portion or from the final degree of reduction (R_{90}).

5 Apparatus

The apparatus shall consist of the following (Figure 1 and Figure 2 show an example of the arrangement).

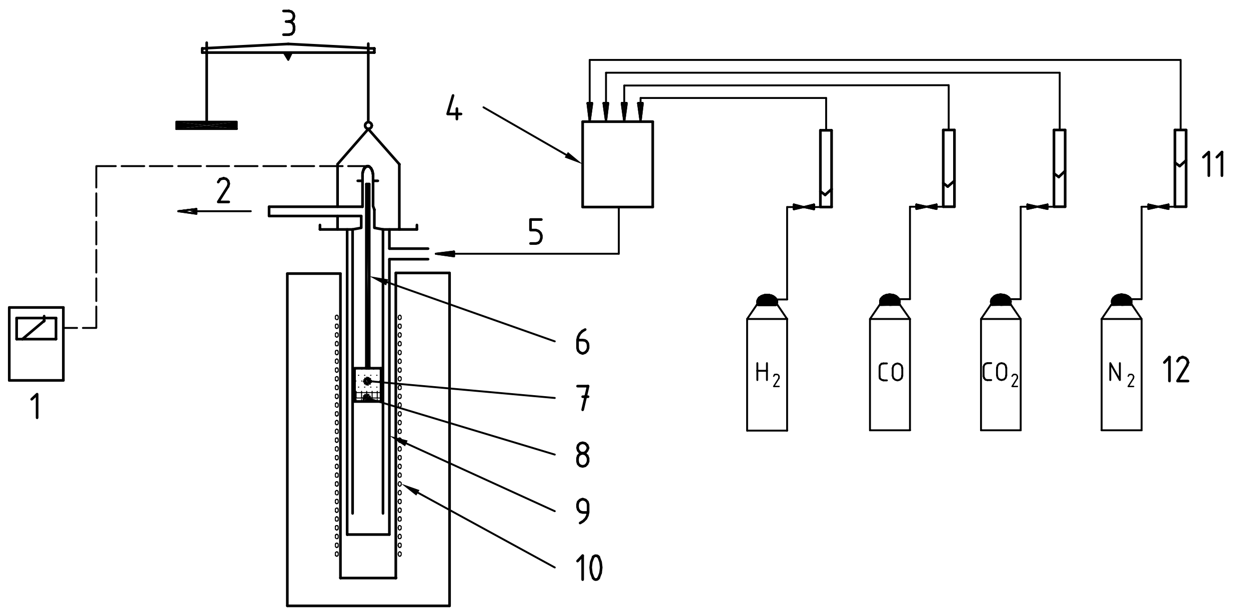
5.1 Gas supply system, capable of supplying the gases and regulating the gas flow rates, freely suspended and connected to the tube in such a way that weighing is not affected.

5.2 Reduction tube, made of non-scaling, heat-resisting metal to withstand temperatures greater than 800 °C and having an internal diameter of 75 mm ± 1 mm. A perforated plate is mounted inside the reduction tube to support the test portion.

5.3 Weighing device, capable of weighing the reduction tube, including the test portion, to an accuracy of 1 g.

5.4 Electrically heated furnace, having a heating capacity and controls sufficient to maintain the entire test portion and the gas entering the bed at 800 °C ± 5 °C.

5.5 Test sieves conforming to ISO 3310-1 and having square openings of the following nominal aperture size: 16 mm; 12,5 mm and 10 mm.



Key

1 Plotter for recording temperature

2 Gas outlet

3 Beam

4 Mixing vessel

5 Gas inlet

6 Thermocouple

7 Test portion

8 Layer of porcelain balls

9 Double wall retort with perforated plate as sample holder

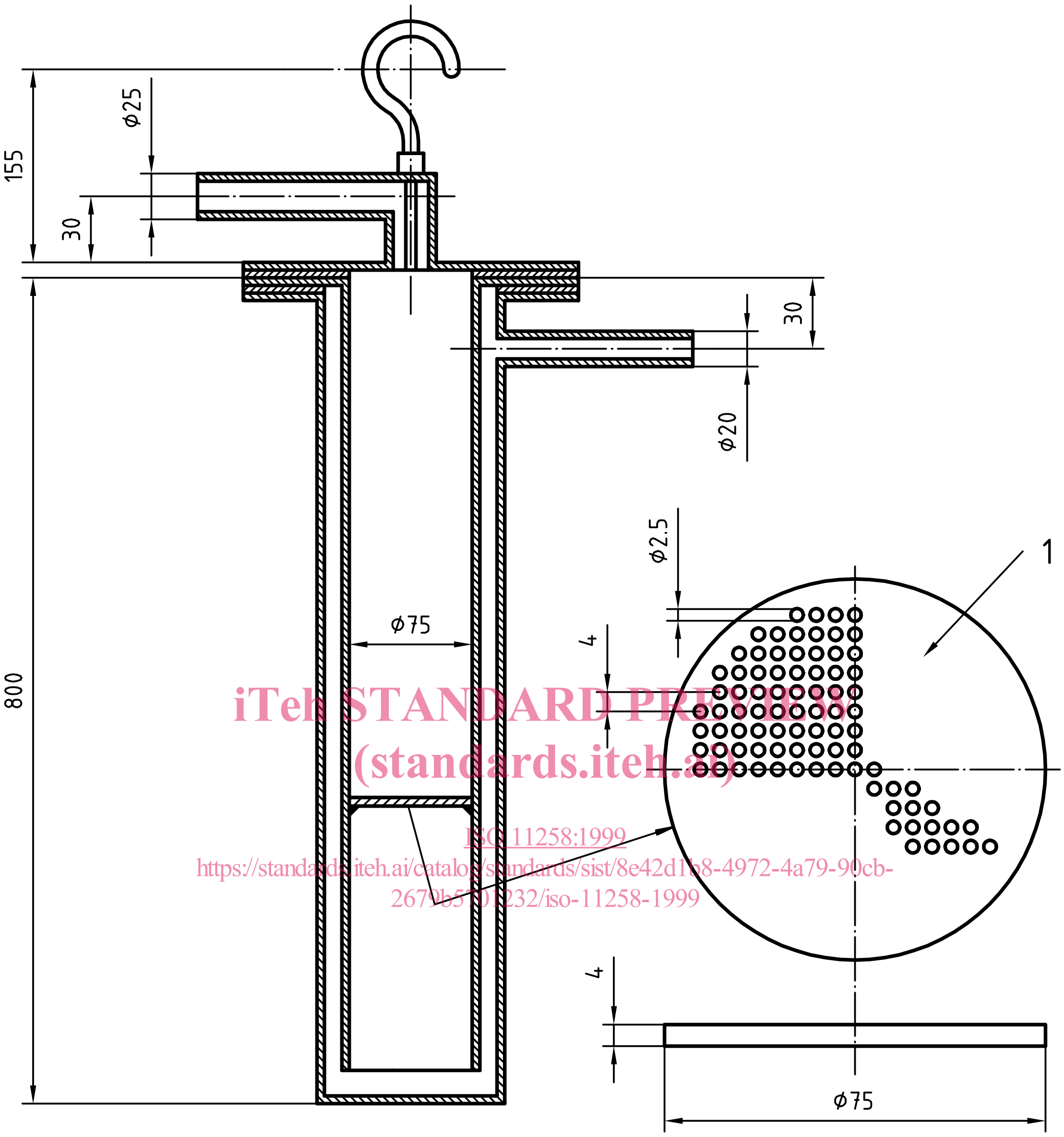
10 Electrically heated furnace

11 Gas flow meters

12 Gas cylinders with manometers and reduction valve

Figure 1 — Arrangement of a test unit

Dimensions in millimetres



NOTE — The dimensions specified in clause 5 are shown for information only.

Key

1:

Hole diameter:	2,5 mm
Pitch between holes:	4,0 mm
Number of holes:	241
Total hole area:	11,8 cm^2
Thickness of plate:	4 mm

Figure 2 — Example of reduction tube

6 Test conditions

6.1 General

Volumes and flow rate of gases used in this International Standard are as measured at a temperature of 0 °C and at an atmospheric pressure of 101,325 kPa (1,013 25 bar).

6.2 Composition of reducing gas

The reducing gas shall consist of:

— H_2 45 % ± 1,0 % (*V/V*)

— CO 30 % ± 1,0 % (*V/V*)

— CO_2 15 % ± 1,0 % (*V/V*)

— N_2 10 % ± 1,0 % (*V/V*)

6.3 Purity of reducing gas

Impurities in the reducing gas shall not exceed

— O_2 0,1 % (*V/V*)

— H_2O 0,2 % (*V/V*)

6.4 Flow rate of reducing gas

Flow rate of the reducing gas shall, during the test period, be maintained at 50 l/min ± 0,5 l/min.

6.5 Purity of heating and cooling gas

Impurities in nitrogen shall not exceed 0,1 % (*V/V*).

6.6 Temperature of test

The test portion shall be reduced at 800 °C ± 5 °C.

The reducing gas shall be preheated while entering the reduction tube to maintain the temperature within the reduction tube and hence the entire test portion at 800 °C ± 5 °C, during the entire reduction period.

7 Sampling and sample preparation

The sampling and the preparation of test samples and test portions shall be in accordance with ISO 10836 [2)].

The test sample shall be oven dried at 105 °C ± 5 °C and cooled to room temperature before the preparation of the test portions.

At least five test portions each of approximately 500 g mass shall be prepared.

Collect each test portion by taking ore particles at random. The target mass of the test portion is 500 g ± the mass of one particle.

[2)] ISO 10836:1994 does not yet include test sample preparation for this test method. Subclause 7.2.3 of ISO 10836:1994 can be applied with the sieves adjusted accordingly.

NOTE If the mass of the test portion deviates from 500 g, either add or remove particles one by one at random to reach a mass as close as possible to 500 g.

For example, if the mass of the test portion is 490 g and one more particle has a mass of 25 g, then the choice lies between 490 g and 515 g. The last particle should not be included in the test portion because the lower mass (490 g) is closer to the target mass (500 g) than the greater mass (515 g).

The size range for pellets shall be 10 mm to 16 mm, being 50 % between 10 mm and 12,5 mm and 50 % between 12,5 mm and 16 mm.

The size range for lumps shall be 10 mm to 20 mm, being 50 % of the mass percentage between 10 mm and 16 mm and 50 % between 16 mm and 20 mm.

8 Procedure

8.1 Number of determinations

Carry out the test in duplicate on one ore sample.

8.2 Other determinations

Take at random one of the test portions prepared in accordance with clause 7 and use it for the determination of total iron content in accordance with ISO 2597-1, ISO 9507 or ISO 9508 and the iron (II) content in accordance with ISO 9035.

8.3 Test portion

Take at random one of the test portions prepared in accordance with clause 7, weigh it to the nearest 1 g and register its mass (m_0).

8.4 Reduction

Place the test portion in the reduction tube (5.2) and level its surface. In order to achieve a more uniform gas flow, a two-layer bed of porcelain pellets having a size range of 10 mm to 12,5 mm may be placed between the perforated plate and the test portion.

Close the top of the reduction tube. Insert the reduction tube into the furnace (5.4) and suspend it centrally from the weighing device (5.3), ensuring that there is no contact with the furnace or heating elements.

Pass a flow of nitrogen through the reduction tube at a flowrate of approximately 25 l/min and commence heating. When the temperature of the test portion approaches 800 °C, increase the flow of nitrogen to 50 l/min. Continue the heating maintaining the flow of nitrogen until the mass of the test portion is constant and the temperature is constant at 800 °C ± 5 °C.

WARNING — Hydrogen, carbon monoxide and the reducing and waste gas which contains hydrogen and carbon monoxide are toxic and therefore hazardous. During reduction the testing shall be carried out in a well-ventilated area or under a hood. Precautions, according to local or national safety codes, shall be taken for the safety of the operator.

Record the mass of the test portion (m_1) and immediately introduce the reducing gas at a flowrate of 50 l/min to replace the nitrogen. Record the mass of the test portion (m_t) continuously or at least every 3 min for the first 15 min and thereafter at 10 min intervals. After 90 min of reduction, record the mass of the test portion (m_2) and turn off the power.

Replace the reducing gas by nitrogen at a flowrate of 25 l/min. The flow of nitrogen should be continued until the test portion reaches to below 50 °C.

If the degree of metallization is to be obtained from 9.3.1, pulverise the entire reduced test portion and determine the total iron content in accordance with ISO 2597-1, ISO 9507 or ISO 9508, and its metallic iron content in accordance with ISO 5416.

9 Expression of results

9.1 Final degree of reduction (R_{90})

Calculate the degree of reduction after 90 min (referred to as the final degree of reduction), R_{90}, expressed as a percentage, using the following equation[3] :

$$R_{90} = \left(\frac{0{,}111\, w_1}{0{,}430\, w_2} + \frac{m_1 - m_2}{m_0 \times 0{,}430\, w_2} \times 100 \right) \times 100 \qquad (1)$$

where,

- m_0 is the mass, in grams, of the test portion;
- m_1 is the mass, in grams, of the test portion immediately before starting the reduction;
- m_2 is the mass, in grams, of the test portion, after 90 min of reduction;
- w_1 is the iron (II) oxide content, as a percentage by mass, of the test sample prior to the test and is calculated from the iron(II) content, as determined by ISO 9035, 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, ISO 9507 or ISO 9508.

Record the final degree of reduction to one decimal place.

9.2 Reducibility indices $\frac{dR}{dt}_{(R=40)}$ and $\frac{dR}{dt}_{(R=90)}$

9.2.1 Reduction curve

Prepare the reduction curve by plotting the degree of reduction R_t against time t, using the following equation:

$$R_t = \left(\frac{0{,}111\, w_1}{0{,}430\, w_2} + \frac{m_1 - m_t}{m_0 \times 0{,}430\, w_2} \times 100 \right) \times 100 \qquad (2)$$

where m_t is the mass, in grams, of the test portion, after reduction time t.

Using the reduction curve, obtain and record the reducibility indices to two decimal places as follows.

9.2.2 Reducibility index for 40 % reduction

Read off from the reduction curve the time in minutes to attain degrees of reduction of 30 % and 60 %.

Calculate the reducibility index $\frac{dR}{dt}_{(R=40)}$ for 40 % of reduction (O/Fe = 0,9), expressed in %/min, using the following equation:

$$\frac{dR}{dt}_{(R=40)} = \frac{33{,}6}{t_{60} - t_{30}} \qquad (3)$$

3) The derivation of the formula is given in annex A.