TC 102

International Standard ISO 7215

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Iron ores — Determination of relative reducibility

Minerais de fer — Détermination de la réductibilité relative

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 7215 was prepared by Technical Committee ISO/TC 102, *Iron ores.*

Iron ores — Determination of relative reducibility

0 Introduction

The relative reducibility test is one of several procedures used to evaluate the behaviour of natural and processed iron ores under specific conditions. The specific conditions involved in this test are: isothermal reduction; reduction in a fixed bed; reduction by means of carbon monoxide; and a sample having a specified size range.

The results of this test should only be considered in conjunction with the results of other tests, particularly those showing the physical behaviour of materials during reduction.

Both annex A, giving mathematical derivations of formulae for relative reducibility, and annex B, giving a list of International Standards relating to reduction tests of iron ores, are included for information only.

1 Scope and field of application

This International Standard specifies a method for determining the reducibility in relative terms of natural and processed iron ores.

2 References

ISO 2597, *Iron ores — Determination of total iron content — Titrimetric methods.* 1)

ISO 3081, *Iron ores — Increment sampling — Manual method.* 2)

ISO 3082, *Iron ores — Increment sampling and sample preparation — Mechanical method.* 3)

ISO 3083, *Iron ores — Preparation of samples — Manual method.* 4)

ISO 3310/1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth.*

3 Definitions

For the purpose of this International Standard, the following definitions apply.

3.1 reducibility (isothermal reduction): A measure of the ease with which oxygen combined with iron can be removed from natural or processed iron ores by a reducing gas.

3.2 relative reducibility: The final degree of reduction attained after a prescribed period of time and under other specified conditions.

4 Principle

Isothermal reduction using carbon monoxide of the test portion placed on a balance in a fixed bed at 900 °C for 3 h. Heating and cooling in an inert atmosphere.

Calculation of the degree of reduction from the loss in mass and the total iron and iron(II) contents of the unreduced material.

5 Reducing gas

Volumes and flow rates of gases used in this International Standard are measured at a temperature of 0 °C and at atmospheric pressure (101,325 kPa).

5.1 Composition

The reducing gas shall consist of

CO	30 ± 1,0 % (*V*/*V*)
N_2	70 ± 1,0 % (*V*/*V*)

5.2 Purity

Impurities in the reducing gas shall not exceed

H_2	0,2 % (*V*/*V*)
CO_2	0,2 % (*V*/*V*)
O_2	0,1 % (*V*/*V*)
H_2O	0,2 % (*V*/*V*)

1) At present at the stage of draft. (Revision of ISO 2597-1973.)

2) At present at the stage of draft. (Revision of ISO 3081-1973.)

3) At present at the stage of draft.

4) At present at the stage of draft. (Revision of ISO 3083-1973.)

6 Apparatus

Ordinary laboratory equipment, and

6.1 Test sieves, having square mesh openings of 10,0; 12,5; 16,0; 19,0; and 22,4 mm nominal size and conforming to ISO 3310/1.

6.2 Electrically heated reduction furnace, with tube assembly, gas supply and flow rate regulating system (see figure 1), and equipped with a balance permitting the oxygen loss of the sample to be read at any time during the test.

6.2.1 The tube assembly (see figure 2) shall consist of

a) a reduction tube made of non-scaling, heat-resistant metal capable of withstanding a temperature greater than 910 °C and having an internal diameter of 75 ± 1 mm;

b) a device to connect the reduction tube to the weighing device;

c) a perforated plate mounted within the reduction tube to support the test portion; the plate shall be 4 mm thick; the holes shall be 2 to 3 mm in diameter and the pitch between holes shall be 4 to 5 mm;

d) heat exchange medium, for example alumina balls, placed in the bottom under the perforated plate of the reduction tube, to a depth of 100 mm, to preheat the gas.

6.2.2 The furnace shall have a heating capacity sufficient to maintain the entire test portion and the gas entering the bed at 900 ± 10 °C.

6.2.3 The weighing device shall be capable of weighing the reduction tube assembly, including the test portion, to an accuracy of 0,5 g. The weighing device shall be checked for accuracy and sensitivity at regular intervals.

7 Sampling and samples

7.1 Sample for relative reducibility test

The test sample shall be prepared according to ISO 3082[1)] or ISO 3083[1)] from the sample for physical testing which has been taken in accordance with ISO 3081[1)] or ISO 3082. A quantity of sample sufficient to provide at least five 500 g test portions shall be prepared.

The test sample shall be oven dried at 105 ± 5 °C for at least 2 h, cooled in a desiccator to room temperature and sieved to obtain material of the desired size range for testing.

The size range for pellets shall be 10,0 to 12,5 mm. However, the test portion may be made more representative of the consignment by selecting a more appropriate size range by agreement between the parties concerned, but in such cases the permissible tolerance given in 10.2.2 is not applicable.

The size range for sinter and ore shall be 19,0 to 22,4 mm. Because of the inherent heterogeneity of some ores and sinter, the size range 10,0 to 12,5 mm may be used by agreement between the parties concerned, but in such cases the permissible tolerance given in 10.2.2 is not applicable. The 10,0 to 12,5 mm test sample shall be prepared as follows.

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

After sieving, the test material shall be kept in a desiccator until testing and only particles taken at random shall be used to constitute a test portion.

7.2 Sample for chemical analysis

A 500 g test portion shall be reserved for the determination of total iron content and iron(II) content.

8 Test conditions

8.1 Flow rate of reducing gas

The reducing gas flow rate shall, during the test period, be maintained at 15 ± 0,5 l/min.

8.2 Temperature of test

The reducing gas shall be preheated before entering the test portion to maintain the test portion at 900 ± 10 °C during the entire test period.

9 Procedure

Carry out the test in duplicate on one ore sample.

Simultaneously with the test, determine the total iron content in accordance with ISO 2597, and the iron(II) content.

9.1 Test portion

Weigh, to the nearest 1 g, approximately 500 g (± 1 particle) of the test sample (mass m_0).

9.2 Reduction

Place the test portion in the reduction tube so that the surface is even.

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

1) At present, these International Standards do not specify any requirements applicable to this International Standard.

Pass a flow of inert gas through the reduction tube at a flow rate of approximately 5 l/min and start heating. When the temperature of the test portion approaches 900 °C increase the flow rate to 15 l/min and continue heating at 900 °C for 30 min. Record the mass of the test portion (mass m_1). Introduce the reducing gas to replace the inert gas at a flow rate of 15 l/min.

CAUTION — Carbon monoxide and the reducing gas, which contains carbon monoxide, are toxic and therefore hazardous. During the following procedure, 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.

At the end of 3 h of reduction, determine the mass of the test portion (mass m_2) and turn off the power. Then, for safety reasons, introduce again inert gas at a flow rate of 5 l/min to replace the reducing gas in the tube. Maintain the inert gas flow until the test portion is cooled to below 100 °C.

NOTES

1 If reduction versus time curves are required, record the mass of the test portion every 10 min during the first hour and every 15 min during the last two hours.

2 In the case of lump ores, the temperature of the test portion should be raised to 900 °C over more than 60 min to reduce decrepitation of the lump ore.

3 If physical tests, such as crushing strength, are to be performed on the reduced test portion, the flow of inert gas after reduction should be continued until the test portion reaches room temperature.

10 Expression of results

10.1 Calculation of the degree of reduction

The degree of reduction attained after 3 h (referred to as the final degree of reduction), R_f, expressed as a percentage, is given by the equation [1]

$$R_f = \left[\frac{m_1 - m_2}{m_0 \, (0{,}430 \, w_2 - 0{,}111 \, w_1)} \right] \times 10^4$$

where

m_0 is the initial 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 3 h of reduction;

w_1 is the iron(II) oxide content, expressed as a percentage by mass, of the test sample prior to the test and is calculated from the iron(II) content by a factor of 1,286;

w_2 is the total iron content, expressed as a percentage by mass, of the test sample prior to the test, determined in accordance with ISO 2597.

Record the final degree of reduction to one place of decimals.

10.2 Number of tests and permissible tolerance

10.2.1 Number of tests

The reduction test shall be carried out in duplicate. If the difference between the paired results of R_f meets the permissible tolerance given in 10.2.2, the test is terminated; if not, another duplicate test shall be carried out.

10.2.2 Permissible tolerance

For a paired result, the difference between the two individual results shall be less than 3 % absolute for pellets and less than 5 % absolute for sinter.

NOTE — A permissible tolerance for lump ore is not specified, because of the inherent heterogeneity, which varies for different ores. For a particular ore, a permissible tolerance may be determined by the parties concerned.

10.3 Calculation of final result

The final degree of reduction, R_f, expressed as a percentage, shall be reported as the arithmetic mean of all test results, rounded to the nearest whole number.

11 Test report

The test report shall contain the following information:

a) reference to this International Standard;

b) description of the test sample;

c) final degree of reduction, R_f;

d) total iron and iron(II) contents of the test sample.

1) The derivation of the equation is given in annex A.

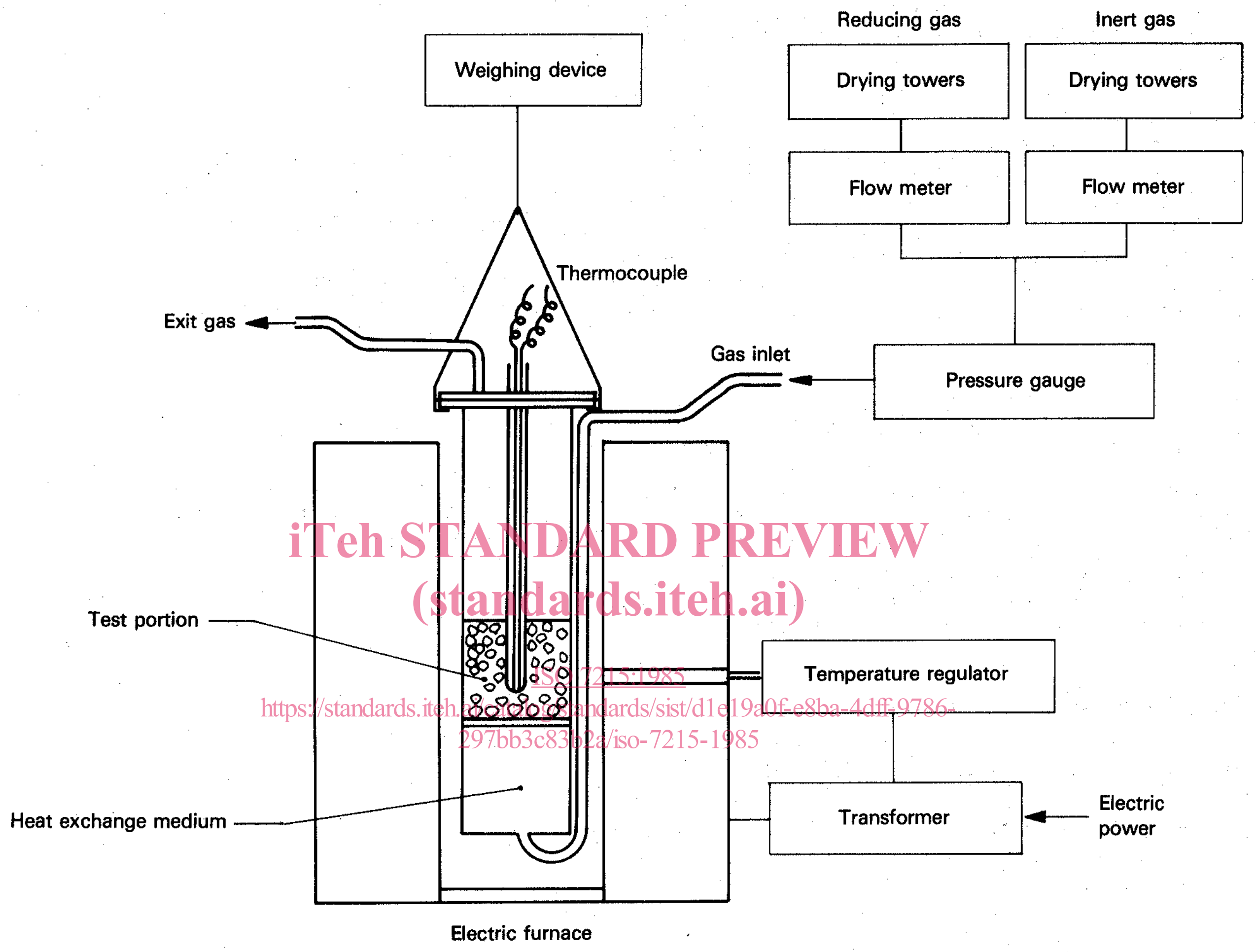
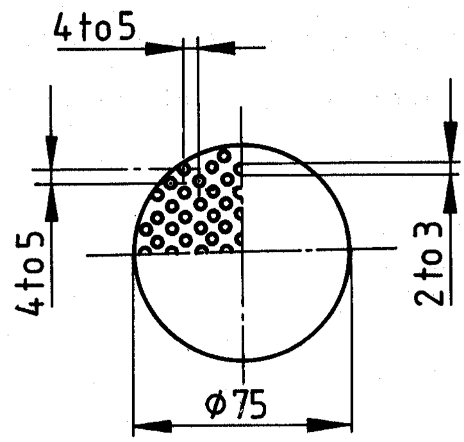


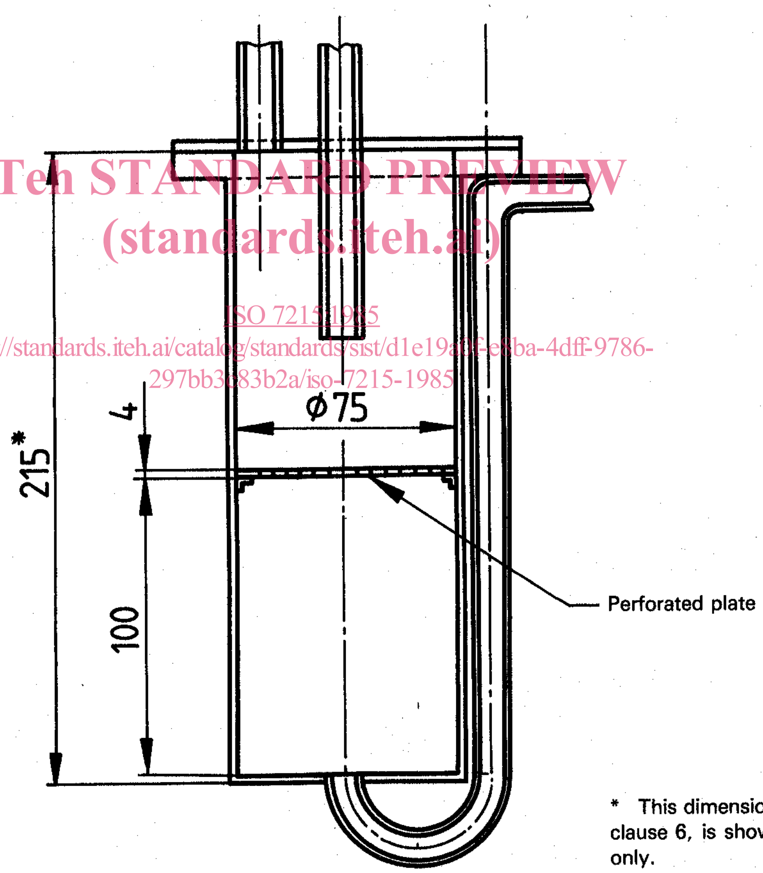
Figure 1 — Schematic diagram of reduction test apparatus

Dimensions in millimetres



Perforated plate

Hole diameter	2 to 3 mm
Pitch between holes	4 to 5 mm
Thickness of plate	4 mm



* This dimension, not specified in clause 6, is shown for information only.

Figure 2 — Schematic diagram of reduction tube assembly

Annex A

Derivation of the equation for final degree of reduction

(This annex is for information only and does not form an integral part of this International Standard.)

A.1 Basic equation

The equation for R_f given in 10.1 is derived from the basic equation

$$R_f = \frac{\Delta m_f}{m_3} \times 100 \qquad \ldots (1)$$

where

Δm_f is the mass loss, in grams, of oxygen during 3 h of reduction;

m_3 is the mass, in grams, of oxygen combined with iron before reduction.

A.2 Derivation of working equation

The iron oxides contained in the sample are considered to be haematite (Fe_2O_3), magnetite ($FeO \cdot Fe_2O_3$) and iron(II) oxide (FeO). The total mass of oxygen, m_3, in equation (1) can be obtained from the masses of Fe_2O_3 and FeO in the test sample before reduction. Therefore, m_3 is given by equation (2), after the total iron content, w_2, and the iron(II) oxide content, w_1, of the test sample have been determined according to the relevant International Standards.

$$m_3 = m_4 + m_5$$

$$= m_0 \left(w_3 \frac{3\,A_O}{2\,A_{Fe}} + w_1 \frac{A_O}{M} \right) \qquad \ldots (2)$$

where

m_4 is the mass, in grams, of oxygen in Fe_2O_3;

m_5 is the mass, in grams, of oxygen in FeO;

m_0 and w_1 have the same meanings as in 10.1;

w_3 is the iron content, expressed as a percentage by mass, in Fe_2O_3;

A_O is the relative atomic mass of oxygen, 16,00;

A_{Fe} is the relative atomic mass of iron, 55,85;

M is the relative molecular mass of iron(II) oxide, 71,85;

Noting that

$$\Delta m_f = m_1 - m_2$$

$$w_3 = w_2 - \frac{A_{Fe}}{M} w_1$$

where m_1, m_2, w_1 and w_2 have the same meanings as in 10.1, and substituting m_3 from equation (2) into equation (1), the final degree of reduction, R_f, expressed as a percentage, is given by the equation

$$R_f = \frac{(m_1 - m_2) \times 100}{m_0 \left[\left(w_2 - \frac{A_{Fe}}{M} w_1 \right) \frac{3\,A_O}{2\,A_{Fe}} + \frac{A_O}{M} w_1 \right] \times \frac{1}{100}}$$

$$= \left\{ \frac{m_1 - m_2}{m_0 \left[\left(w_2 - \frac{55{,}85}{71{,}85} w_1 \right) \frac{48{,}00}{111{,}70} + \frac{16{,}00}{71{,}85} w_1 \right]} \right\} \times 10^4$$

$$= \left[\frac{m_1 - m_2}{m_0\,(0{,}430\,w_2 - 0{,}111\,w_1)} \right] \times 10^4$$