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Iron ores for shaft direct-reduction feedstocks — Determination of the reducibility index, final degree of reduction and degree of metallization

Minerais de fer pour charges utilisées dans les procédés par réduction directe — Détermination de l'indice de réductibilité, du degré final de réduction et du degré de métallisation

Reference number
ISO 11258:2007(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

ISO 11258 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 3, *Physical testing*.

This second edition cancels and replaces the first edition (ISO 11258:1999), which has been revised to homogenise with other physical test standards prepared.

Introduction

This International Standard 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 should be considered in conjunction with other tests used to evaluate the quality of iron ores as feedstocks for direct reduction processes.

This International Standard may 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.

INTERNATIONAL STANDARD **ISO 11258:2007(E)**

Iron ores for shaft direct-reduction feedstocks — Determination of the reducibility index, final degree of reduction and degree of metallization

CAUTION — This International Standard may involve hazardous materials, 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 International Standard specifies a method to provide a relative measure for evaluating the extent and ease to which oxygen can be removed from iron ores, when reduced under conditions resembling those prevailing in shaft direct-reduction processes. It specifies the determination of the reducibilty, final degree of reduction and degree of metallization.

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

2 Normative references

The following referenced documents are indispensable for the application of this International Standard. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2597-1:2006, *Iron ores — Determination of total iron content — Part 1: Titrimetric method after tin(II) chloride reduction*

ISO 3082:2000 [1], *Iron ores — Sampling and sample preparation procedures*

ISO 5416:2006, *Direct reduced iron — Determination of metallic iron — 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 11323:2002, *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.

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[1] Under revision to incorporate ISO 10836, *Iron ores — Method of sampling and sample preparation for physical testing*

4 Principle

The test portion is isothermally reduced in a fixed bed, at 800 °C, using a reducing gas consisting of H_2, CO, CO_2 and N_2 and is weighed continuously or at specified time intervals until 90 min of reduction is reached. The rate of reduction is calculated at the oxygen/iron ratio of 0,9 and the final degree of reduction is calculated from the oxygen mass loss after 90 min (R_{90}). The degree of metallization is calculated either by an equation based on R_{90} or by chemical analysis of the reduced product.

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 shall be 50 % − 16,0 mm + 12,5 mm and 50 % − 12,5 mm + 10,0 mm.

The size range for lump ores shall be 50 % − 20,0 mm + 16,0 mm and 50 % − 16,0 mm + 10,0 mm.

A test sample of at least 2,5 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 5 test portions, each of approximately 500 g (± the mass of 1 particle) shall be prepared from the test sample: 4 test portions for testing and 1 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

6.1 General

The test apparatus shall comprise:

a) ordinary laboratory equipment, such as an oven, hand tools, a time-control device and safety equipment;

b) a reduction-tube assembly;

c) a furnace, equipped with a balance for permitting the mass loss of the test portion to be read at any time during the test;

d) a system to supply the gases and regulate the flow rates;

e) a 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 800 °C and resistant to deformation. The internal diameter shall be 75 mm ± 1 mm. A removable perforated plate, made of non-scaling, heat-resistant metal to withstand temperatures higher than 800 °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 its 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 center 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 the gas entering the bed, at 800 °C ± 5 °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 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.

6.6 Gas-supply system, capable of supplying the gases and regulating gas flow rates. It shall be ensured that a frictionless connection between the gas-supply system and the reduction tube does not affect the weight loss determination during reduction.

6.7 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 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:

CO	30 % ± 1 % (volume fraction)
CO_2	15 % ± 1 % (volume fraction)
H_2	45 % ± 1 % (volume fraction)
N_2	10 % ± 1 % (volume fraction)

7.2.2 Purity

Impurities in the reducing gas shall not exceed:

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 50 L/min ± 0,5 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 N_2 shall be maintained at 25 L/min until the test portion reaches 800 °C and at 50 L/min during the temperature-equilibration period. During cooling, it shall be maintained at 25 L/min.

7.4 Temperature of the test portion

The temperature of the entire test portion shall be maintained at 800 °C ± 5 °C during the entire reducing period and, as such, the reducing gas shall be preheated before entering the test portion.

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 before reduction

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 total iron content (w_2) in accordance with ISO 2597-1 or ISO 9507.

8.3 Reduction

Take, at random, another test portion prepared in 5.2 and record its mass (m_0). Place it in the reduction tube (6.2) and level its surface.

NOTE 1 In order to achieve a more uniform gas flow, a double-layer bed of porcelain balls sized between 10,0 mm and 12,5 mm may be placed between the perforated plate and the test portion.

Close the top of the reduction tube. Connect the thermocouple, ensuring that its tip is in the centre of the test portion.

Insert the reduction tube into the furnace (6.3) and suspend it centrally from the balance (6.5), ensuring that there is no contact with the furnace wall or heating elements.

Connect the gas-supply system (6.6).

Pass a flow of N_2 through the test portion at a rate of at least 25 L/min ± 0,5 L/min and commence heating. When the temperature of the test portion approaches 800 °C, increase the flow rate to 50 L/min ± 0,5 L/min. Continue heating while maintaining the flow of the N_2 until the mass of the test portion is constant and the temperature is constant at 800 °C ± 5 °C for 10 min.

NOTE 2 In the case of lump ores, the temperature of the test portion should be raised to 800° C over more than 60 min to reduce decrepitation.

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 in accordance with the safety codes of each country.

Record the mass of the test portion (m_1) and immediately introduce the reducing gas at a flow rate of 50 L/min ± 0,5 L/min to replace the N_2. Record the mass of the test portion (m_t) 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), turn off the power and replace the reducing gas by N_2 at a flow rate of 25 L/min. The flow of N_2 should be continued until the test portion is below 50 °C.

8.4 Chemical analysis after reduction

Pulverise the entire reduced material and determine its total iron content (w_t) in accordance with ISO 2597-1 or ISO 9507, and its metallic iron content (w_0) in accordance with ISO 5416.

NOTE If desired, the mass percentage of combined carbon in the reduced test portion can be determined by applying ISO 9686:2006, *Direct reduced iron — Determination of carbon and/or sulfur — High-frequency combustion method with infrared measurement.*

9 Expression of results

9.1 Calculation of the final degree of reduction (R_{90})

The final degree of reduction, R_{90}, expressed as a percentage by mass, is calculated from the following equation [2]:

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

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, expressed as a percentage by mass, of the test portion 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, expressed as a percentage by mass, of the test portion prior to the test, determined in accordance with ISO 2597-1 and ISO 9507.

Record the result to one decimal place.

9.2 Calculation of the reducibility indices $\frac{\mathrm{d}R}{\mathrm{d}t}_{(R=40)}$ and $\frac{\mathrm{d}R}{\mathrm{d}t}_{(R=90)}$

9.2.1 Reduction curve

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

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

where

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

Using the reduction curve, obtain and express the reducibility indices to 2 decimal places.

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[2] The derivation of the formula is given in Annex B.

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 %.

The reducibility index $\frac{\mathrm{d}R}{\mathrm{d}t}_{(R=40)}$ for 40 % reduction (O/Fe = 0,9), expressed as the rate of reduction, in %/min, is calculated from the following equation [3]:

$$\frac{\mathrm{d}R}{\mathrm{d}t}_{(R=40)} = \frac{33{,}6}{t_{60} - t_{30}}$$

where

t_{30} is the time, in minutes, to attain a degree of reduction of 30 %;

t_{60} is the time, in minutes, to attain a degree of reduction of 60 %;

33,6 is a constant.

9.2.3 Reducibility index for 90 % reduction

Read off, from the reduction curve, the time, in minutes, to attain degrees of reduction of 80 % and 95 %.

The reducibility index $\frac{\mathrm{d}R}{\mathrm{d}t}_{(R=90)}$ for 90 % reduction (O/Fe = 0,15), expressed as the rate of reduction, in %/min, is calculated from the following equation:

$$\frac{\mathrm{d}R}{\mathrm{d}t}_{(R=90)} = \frac{13{,}9}{t_{95} - t_{80}}$$

where

t_{80} is the time, in minutes, to attain a degree of reduction of 80 %;

t_{95} is the time, in minutes, to attain a degree of reduction of 95 %;

13,9 is a constant.

9.3 Calculation of the degree of metallization (M)

9.3.1 Determination of the degree of metallization from chemical analysis (M)

The degree of metallization, M, expressed as a percentage by mass, is calculated from the following equation:

$$M = \frac{w_0}{w_t} \times 100$$

where

w_0 is the metallic iron content, expressed as a percentage by mass, of the reduced test portion;

w_t is the total iron content, expressed as a percentage by mass, of the reduced test portion.

Express the result to one decimal place.

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[3] The derivation of the formula is given in Annex C.

9.3.2 Determination of the degree of metallization from R_{90} (M_R)

During the preparation of the reduced test portion for chemical analysis, some reoxidation can occur. If the iron(II) oxide content before reduction (w_1) is less than 2 %, the chemical analysis can be avoided and the degree of metallization M_R, expressed as a percentage by mass, can be calculated directly from R_{90} using the following equation [4]:

$$M_R = 1{,}43 \times R_{90} - 43$$

Express the result to one decimal place.

9.4 Repeatability for R_{90} and acceptance of test results

Follow the procedure in Annex A by using the repeatability values for R_{90} given in Table 1. The results shall be reported to one decimal place.

Table 1 — Repeatability (r) for R_{90}

Type of iron ore	r %, absolute
Pellets	2,0
Lump ores	3,5

10 Test report

The test report shall include the following information:

a) a reference to this International Standard, i.e. ISO 11258:2007;

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) details of any operation and any test conditions not specified in this International Standard or regarded as optional, as well as any incident which may have had an influence on the results;

h) the reducibility indices $\frac{dR}{dt}_{(R=40)}$ and $\frac{dR}{dt}_{(R=90)}$;

i) the degree of metallization, M or M_R;

j) the final degree of reduction, R_{90};

k) the total iron and iron(II) contents of the test portion before reduction.

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[4] The derivation of this formula is given in Annex C.

11 Verification

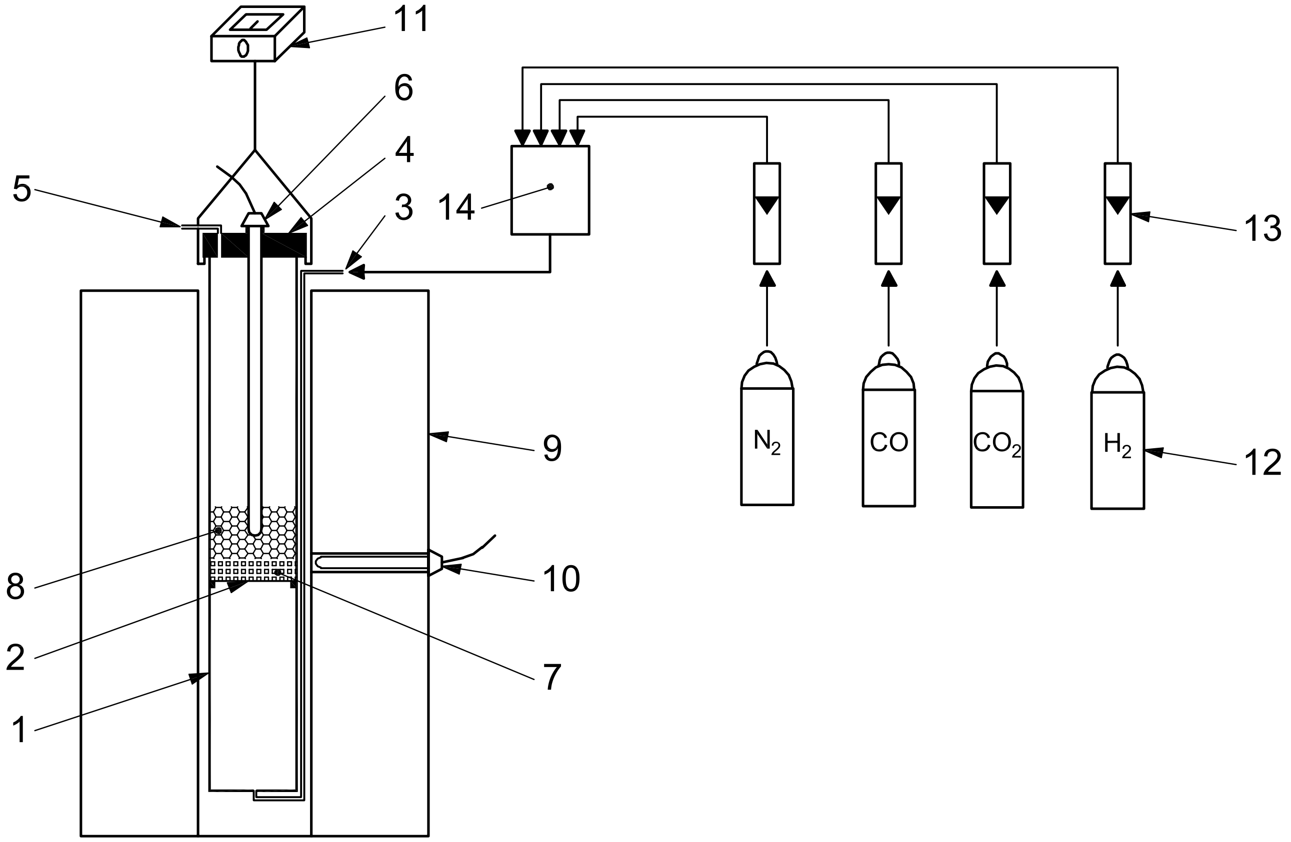
Regular checking of the apparatus is essential to assure test-result reliability. The frequency of checking is a matter for each laboratory to determine.

The conditions of the following items shall be checked:

- weighing device;
- reduction tube;
- temperature control and measurement devices;
- balance;
- gas flowmeters;
- purity of gases;
- recording system;
- 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.



Key

Reduction tube

1	reduction-tube wall
2	perforated plate
3	gas inlet
4	lid
5	gas outlet
6	thermocouple for measuring the reduction temperature
7	porcelain ball layer
8	test portion

Furnace

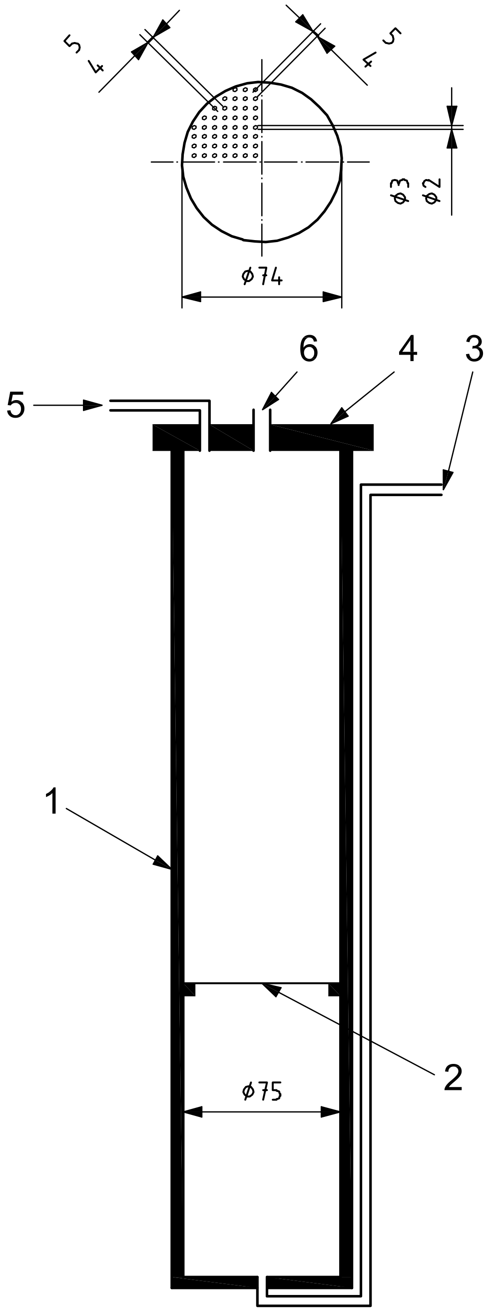
9	electrically heated furnace
10	thermocouple for temperature regulation of furnace
11	balance

Gas-supply system

12	gas cylinders
13	gas flowmeters
14	mixing vessel

Figure 1 — Example of test apparatus (schematic diagram)

Dimensions in millimetres



Key

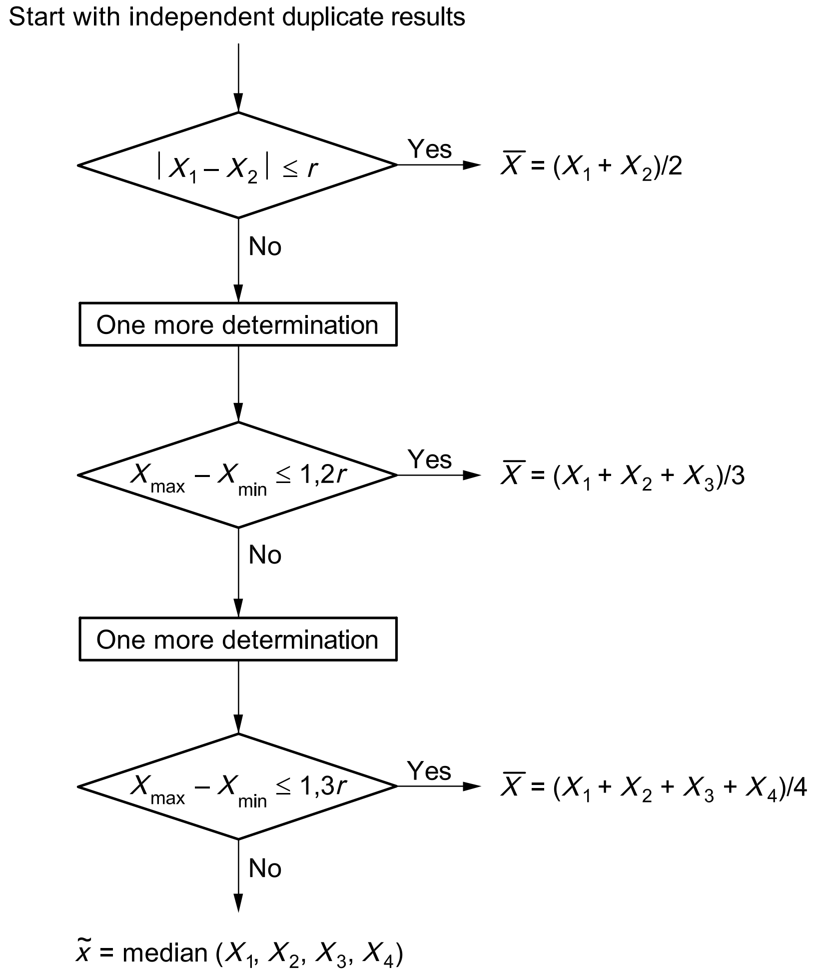
1 reduction-tube wall
2 perforated plate
3 opening for gas inlet
4 lid
5 opening for gas outlet
6 opening for thermocouple insertion

NOTE Dimensions not specified in the apparatus clause are shown for information only.

Figure 2 — Example of reduction tube (schematic diagram)

Annex A
(normative)

Flowsheet of the procedure for the acceptance of test results



r: see Table 1

Annex B
(informative)

Derivation of equation for reducibility

The “Degree of reduction” describes the extent to which oxygen has been removed from iron oxides, and is defined generally as shown below:

$$\text{Degree of reduction} = \frac{\text{Oxygen removed from iron oxide}}{\text{Original oxygen combined with iron}} \tag{B.1}$$

The equation in 9.1 is derived on the assumption that all oxygen combined with iron is present in the form of hematite (Fe_2O_3), whereas, for most iron ore materials, some magnetite (Fe_3O_4), wustite (FeO), and metallic iron is also present. Therefore, the degree of reduction is estimated from the loss in mass of the test portion during reduction plus the difference between the theoretical oxygen content of the original sample, based on all iron being associated as Fe_2O_3, and the true oxygen content based on actual amounts of Fe_2O_3, Fe_3O_4 and FeO in the sample.

$$R_t = \frac{m_0 w_1 \times \dfrac{8}{71{,}85}}{m_0 w_2 \times \dfrac{48}{111{,}7}} \times 100 + \frac{m_1 - m_t}{m_0 \times \dfrac{w_2}{100} \times \dfrac{48}{111{,}7}} \times 100 \tag{B.2}$$

The equation in 9.2.2 is derived on the assumption that the rate of oxygen removed from iron ore is a first-order reaction with respect to the prevailing oxygen concentration.

$$-\frac{\mathrm{dO}}{\mathrm{d}t} = k \times \mathrm{O}_v \tag{B.3}$$

$$\mathrm{dO} = -\mathrm{d}R \times \frac{\mathrm{O}_{\text{total}}}{100} \tag{B.4}$$

$$\frac{\mathrm{O}_v}{\mathrm{O}_{\text{total}}} = 1 - \frac{R}{100} \tag{B.5}$$

where

O_v is the prevailing oxygen content;

$\mathrm{O}_{\text{total}}$ is the total oxygen combined with iron (as Fe_2O_3);

R is the degree of reduction.

From Equations (B.3), (B.4) and (B.5), the rate of reduction is derived:

$$\frac{\mathrm{d}R}{\mathrm{d}t} = k \times \left(1 - \frac{R}{100}\right) \times 100 \tag{B.6}$$

Integration of Equation (B.6) gives

$$\lg\left(1 - \frac{R}{100}\right) = -0{,}434\ kt + C$$

For R between 30 % and 60 %.

$$k=\frac{-\lg(1-60/100)+\lg(1-30/100)}{0{,}434(t_{60}-t_{30})}=\frac{0{,}56}{t_{60}-t_{30}} \tag{B.7}$$

In the case of hematite, the oxygen/iron ratio of 0,9 has the same meaning as R = 40 %. By substituting R = 40 % and Equation (B.7) into Equation (B.6), the value of $\mathrm{d}R/\mathrm{d}t$ (at O/Fe = 0,9) is obtained:

$$\frac{\mathrm{d}R}{\mathrm{d}t}(\mathrm{O/Fe}=0{,}9)=\frac{33{,}6}{t_{60}-t_{30}}$$

Annex C
(informative)

Derivation of equation for degree of metallization from R_{90}

The degree of metallization is the ratio of the content of metallic iron to the total content of iron expressed as a percentage.

The iron content can be expressed as a percentage by mass of the reduced test portion or in absolute terms as follows:

$$M_\mathrm{R} = 100 \times \frac{m_\mathrm{FeM}}{m_\mathrm{FeT}} \qquad \text{(C.1)}$$

where

M_R is the degree of metallization of the test portion after 90 min of reduction, expressed as a percentage by mass;

m_FeM is the mass of metallic iron in the test portion after 90 min of reduction, in grams;

m_FeT is the mass of total iron in the test portion, in grams.

Considering that the amount of iron(III) in the test portion after 90 min of reduction is very small, the following can be assumed:

$$m_\mathrm{FeM} = m_\mathrm{FeT} - M_{\mathrm{Fe}^{2+}} \qquad \text{(C.2)}$$

where

$M_{\mathrm{Fe}^{2+}}$ is the mass of iron(II) in the test portion after 90 min of reduction, in grams

$$M_{\mathrm{Fe}^{2+}} = \left(55{,}8 \big/ 16{,}8 \right) \times m_\mathrm{O} = 3{,}32\ m_\mathrm{O} \qquad \text{(C.3)}$$

where

m_O is the mass of oxygen in $\mathrm{FeO}_{1{,}05}$ in the test portion after 90 min of reduction, in grams

$$m_\mathrm{O} = \left(1 - \frac{R_{90}}{100} \right) \times m_{\mathrm{O},1} \qquad \text{(C.4)}$$

where,

$m_{\mathrm{O},1}$ is the mass of oxygen in the test portion before reduction, in grams

Assuming that the amount of iron(II) oxide content in the test portion is very small

$$m_{\mathrm{O},1} = 0{,}43 \times \mathrm{FeT} \qquad \text{(C.5)}$$

Substituting Equation (C.5) in Equation (C.4)

$$m_{\mathrm{O}} = \left(1 - \frac{R_{90}}{100}\right) \times 0{,}43 \times m_{\mathrm{FeT}} \tag{C.6}$$

Substituting Equation (C.6) in Equation (C.3)

$$M_{\mathrm{Fe}^{2+}} = 3{,}32 \times 0{,}43 \times m_{\mathrm{FeT}} \times \left(1 - \frac{R_{90}}{100}\right) \tag{C.7}$$

Substituting Equation (C.7) in Equation (C.2)

$$m_{\mathrm{FeM}} = m_{\mathrm{FeT}} - 3{,}32 \times 0{,}43 \times m_{\mathrm{FeT}} \times \left(1 - \frac{R_{90}}{100}\right) \tag{C.8}$$

Substituting Equation (C.8) in Equation (C.1)

$$M_{\mathrm{R}} = 100 \times \frac{m_{\mathrm{FeT}} - 3{,}32 \times 0{,}43 \times m_{\mathrm{FeT}} \times \left(1 - \frac{R_{90}}{100}\right)}{m_{\mathrm{FeT}}} \tag{C.9}$$

$$M_{\mathrm{R}} = 1{,}43 \times R_{90} - 43 \tag{C.10}$$

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