INTERNATIONAL STANDARD

ISO 11256

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Iron ore pellets — Determination of clustering of feedstock for direct reduction by gas reforming processes

Boulettes de minerais de fer — Détermination du pouvoir collant des charges utilisées dans les procédés de reforming par réduction directe

Reference number
ISO 11256:1998(E)

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International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

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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 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 11256 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Subcommittee SC 5, *Physical testing for direct reduction*.

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

Introduction

Direct reduction processes are intended to reduce iron ores partially, or almost completely by thermal processes to form high grade feedstocks for iron- and steelmaking. Several kinds of direct reduction processes are in operation worldwide and others are still under development. The behaviour of the iron ores, as feedstock, may vary from process to process. ISO 11256 was prepared addressing specifically the direct reduction by gas reforming processes.

The obtained proportion of generated fines is a relative measure of the disintegration behaviour and the degree of metallization is a 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 for direct reduction processes.

Iron ore pellets — Determination of clustering 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 specifies a test method for evaluating the clustering behaviour of iron ore pellets under conditions that 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 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.*

3 Definitions

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

3.1
cluster
Two or more particles of reduced iron ores stuck together.

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

3.2
clustering
The formation of clusters of iron ore particles when reduced under conditions that resemble the ones prevailing in the direct reduction processes.

4 Principle

Heating of the test portion in an inert atmosphere.

Isothermal reduction of the test portion under load in a fixed bed by reducing gas consisting of H_2, CO, CO_2 and N_2 at a temperature of 850 °C, up to 95 % degree of reduction.

Cooling of the test portion in an inert atmosphere.

Disaggregation of the reduced test portion by tumbling, in a specified drum.

Calculation of the clustering index from the clusters accumulated after specified disaggregation operations.

5 Apparatus

The apparatus shall consist of the following (Figures 1 and 2 show examples of the arrangement).

Dimensions in millimetres

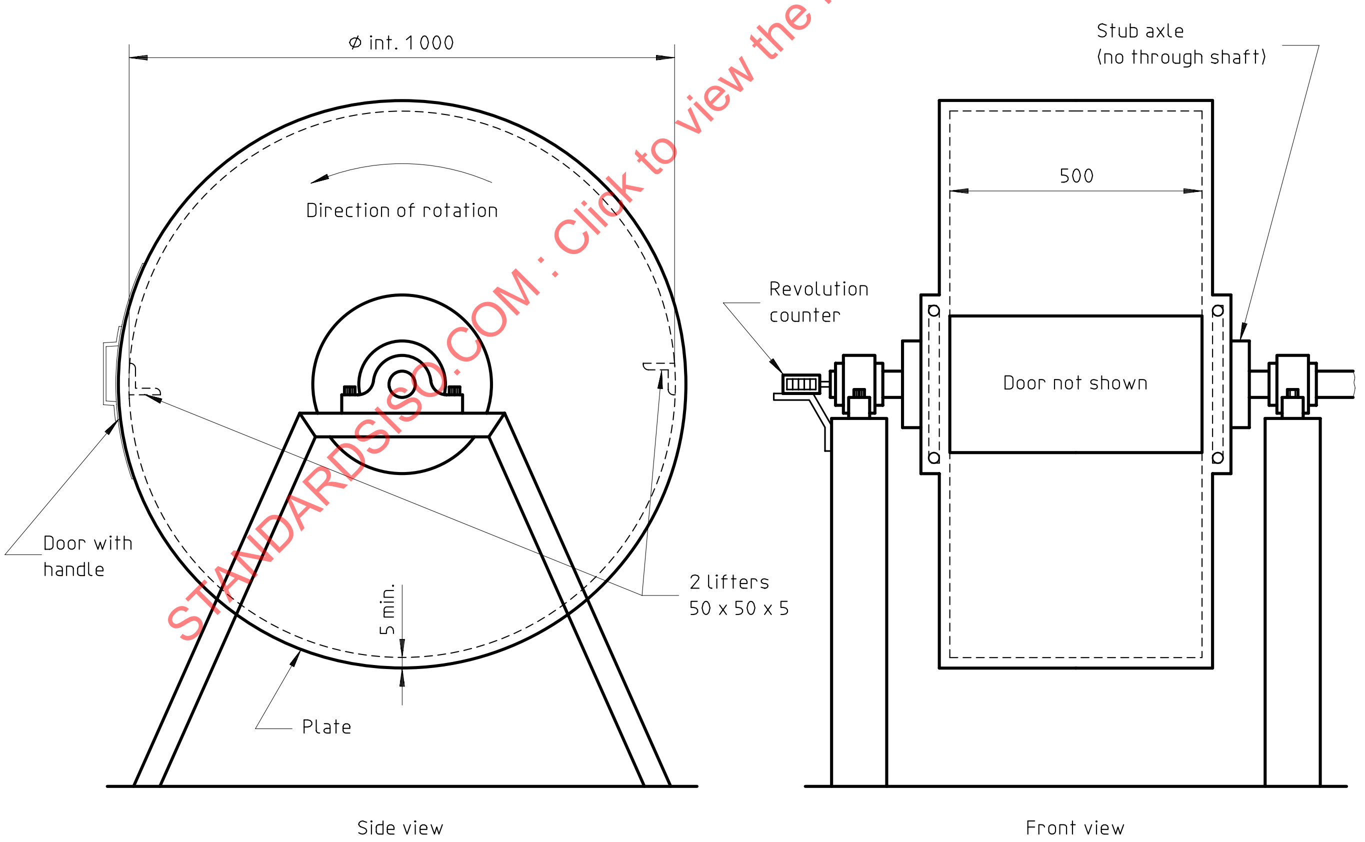


Figure 1 — Example of tumble drum for cluster disaggregation

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 higher than 850 °C, having an internal diameter of 125 mm ± 1 mm. A perforated plate is mounted inside the reduction tube for supporting the test portion. This perforated plate, having a diameter of 120 mm ± 1 mm, shall be 10 mm thick; the holes shall be 3 mm to 4 mm in diameter and separated from each other by 3 mm to 5 mm.

5.3 Loading device, capable of evenly supplying a total static load of 147 kPa to the test portion. The load shall be transferred by means of a ram with rigid perforated foot plate so as to distribute it evenly over the surface of the porcelain pellets placed on top of the test portion. This perforated plate, having a diameter of 120 mm ± 1 mm, shall be 10 mm thick; the holes shall be 3 mm to 4 mm in diameter and separated from each other by 3 mm to 5 mm.

5.4 Weighing device, coupled with the furnace and capable of weighing the reduction tube assembly, including the test portion, to an accuracy of 1 g.

5.5 Weighing device, capable of weighing the test portion before and after the reduction test and the clusters to an accuracy of 1 g.

5.6 Electrically heated furnace, having a heating capacity and controls sufficient to maintain the entire test portion at 850 °C ± 5 °C.

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

5.8 Tumble drum, as shown in Figure 1, consisting of a circular drum of internal diameter 1 000 mm and internal length 500 mm, constructed of steel plate at least 5 mm in thickness. Two equally spaced steel angle lifters, of section 50 mm × 50 mm × 5 mm, of length 500 mm shall be solidly attached longitudinally inside the drum by welding, in such a manner as to prevent accumulation of material between lifter and drum. The drum shall be replaced whenever the thickness of the plate is reduced by wear to 3 mm in any area.

5.9 Rotation equipment, to allow rotation of the tumble drum at a constant rate of 0,416 s^{-1} ± 0,016 s^{-1} (25 rpm ± 1 rpm).

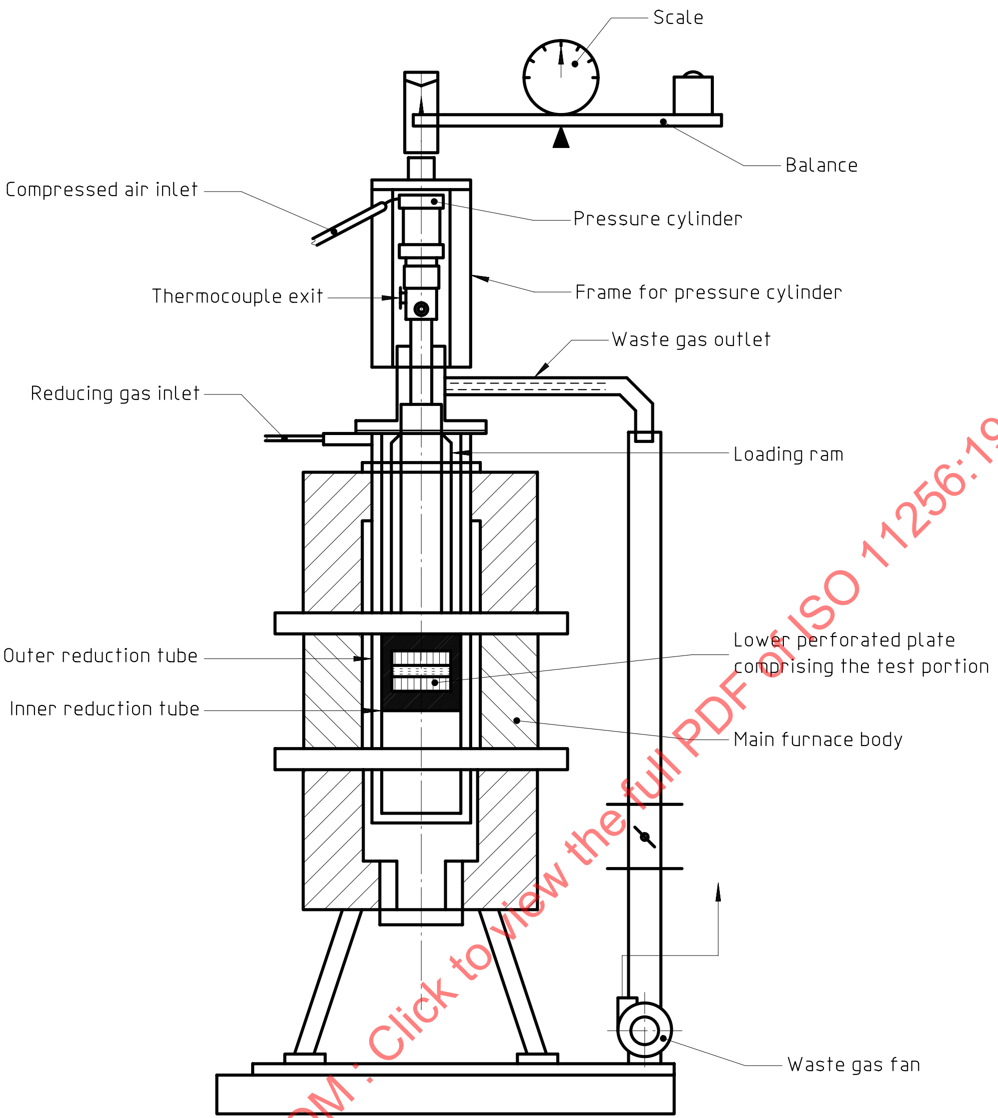


Figure 2 — Example of reduction apparatus for the determination of clustering

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.

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

The reducing gas flow rate shall, during the reduction test, be maintained at 40 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 a temperature of 850 °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 850 °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 six test portions, each of approximately 2 000 g mass shall be prepared as follows.

Collect each test portion by taking ore particles at random. The target mass of the test portion is 2 000 g ± the mass of one particle.

NOTE If the mass of the test portion deviates from 2 000 g, either add or remove particles one by one at random to reach a mass as close as possible to 2 000 g.

For example, if the mass of the test portion is 1 990 g and one more particle has a mass of 25 g, then the choice lies between 1 990 g and 2 015 g. The last particle should not be included in the test portion because the lower mass (1 990 g) is closer to the target mass (2 000 g) than the greater mass (2 015 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.

[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.

8 Procedure

8.1 Number of determinations

Carry out the test in duplicate on one ore sample.

8.2 Other determinations

Use one of the test portions prepared in clause 7 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 clause 7, weigh it using the weighing device (5.5) to the nearest 1 g and register its mass (m_0).

8.4 Reduction

Place a double layer of porcelain pellets sized between 10 mm and 12,5 mm on the perforated plate in the reduction tube (5.2), in order to achieve uniform gas flow through the test portion, then level its surface.

Place some ceramic fibre wool along the inner wall of the reduction tube to avoid the test portion sticking to the wall. Spread the test portion (see 8.3) on the top of the porcelain pellets and level its surface.

Place a further double layer of porcelain pellets sized between 10 mm and 12,5 mm upon the test portion.

Close the top of the reduction tube with the loading device (5.3). Insert the reduction tube assembly into the furnace (5.6) and suspend it centrally from the weighing device (5.4) ensuring that there is no contact with the furnace wall or heating elements.

Connect the thermocouple, ensuring that its tip is in the central position (middle height) of the test portion.

Connect the gas supply system (5.1) and the compressed air to the loading device.

Pass a flow of nitrogen through the reduction tube at a flow rate of approximately 20 l/min and commence heating. When the temperature of the test portion approaches 850 °C, increase the flow of nitrogen to 40 l/min and continue heating at 850 °C for 10 min.

> **WARNING: Hydrogen, carbon monoxide, and reducing and waste gases which contain 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 for the safety of the operator, according to local or national safety codes, shall be taken.**

Record the mass of the test portion (m_1), record the time and immediately introduce the reducing gas at a flow rate of 40 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 one hour of reduction evenly apply a load of 147 kPa ± 2 kPa, to the test portion.

Terminate the reduction by introducing nitrogen at a flow rate of 20 l/min when the degree of reduction reaches 95 % and record the time.

The degree of reduction is calculated as follows[3] :

$$R_t = \left(\frac{0{,}111\, w_1}{0{,}430\, w_2} + \frac{m_1 - m_t}{m_0 \times 0{,}430\, w_2} \times 100 \right) \times 100 \qquad \text{...(1)}$$

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

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_t is the mass, 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, and 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, ISO 9507 or ISO 9508.

Remove the load, continue passing nitrogen at a flow rate of 20 l/min and cool the test portion to below 50 °C.

8.5 Disaggregation operations

Carefully remove the reduced test portion from the reduction tube and immediately register its total mass (m_r) by means of the weighing device (5.5). During this operation some individual pellets usually separate from the clusters. Remove these pellets and register the mass of the clusters (cm_1). This step is considered to be the first disaggregation operation.

NOTE The removal of the test portion from the reduction tube is a critical step and care should be taken to avoid its untimely disaggregation.

Carefully place the clusters inside the tumble drum (5.8) and rotate it for a total of 35 revolutions divided in seven disaggregation operations of five revolutions each. After each disaggregation operation the mass of the remaining clusters is measured by means of the weighing device (5.5) and recorded as a series (cm_2, cm_3, ..., cm_8). Any individual pellets that are separated from the clusters shall be removed prior to the next disaggregation operation.

9 Expression of results

9.1 Clustering index (*CI*)

Calculate the clustering index *CI*, as a percentage, by the following formula:

$$CI = \frac{100}{8 \times m_r} \times \sum_{i=1}^{8} cm_i \quad \text{...(2)}$$

where

m_r is the total mass, in grams, of the test portion after reduction;

cm_i is the mass, in grams, of the clusters after the i^{th} disaggregation operations.

9.2 Number of tests and permissible tolerances

The permissible tolerance r shall be 27 % of the mean value.

If the absolute difference between the paired results (X_1 and X_2) meets the permissible tolerance (r) the test is terminated and the mean value of these results shall be reported. If not, the flowsheet given in annex A shall be followed.

The result shall be rounded off to the nearest whole number.

10 Test Report

The test report shall contain the following information:

a) reference to this International Standard, i.e. ISO 11256;

b) identification of the sample;

c) name and address of the testing laboratory;

d) date of issue of the test report;

e) the clustering index (*CI*);

f) time to reach 95 % of degree of reduction.

11 Verification

Regular checking of apparatus and procedures is essential to verify the test results. Checks shall be carried out at regular intervals. The frequency of checking is a matter for each laboratory to determine. The following items shall be checked:

a) Test sample preparation:

   1) sieves;

   2) weighing device.

b) Reduction test:

   1) reduction tube condition;

   2) temperature level and distribution in the test portion;

   3) gas composition;

   4) gas flow rate;

   5) weighing device;

   6) load application;

   7) timer;

   8) recording system.

c) Test evaluation:

   1) tumble condition;

   2) condition of lifters;

   3) rotation speed;

   4) revolution counter;

   5) weighing device.

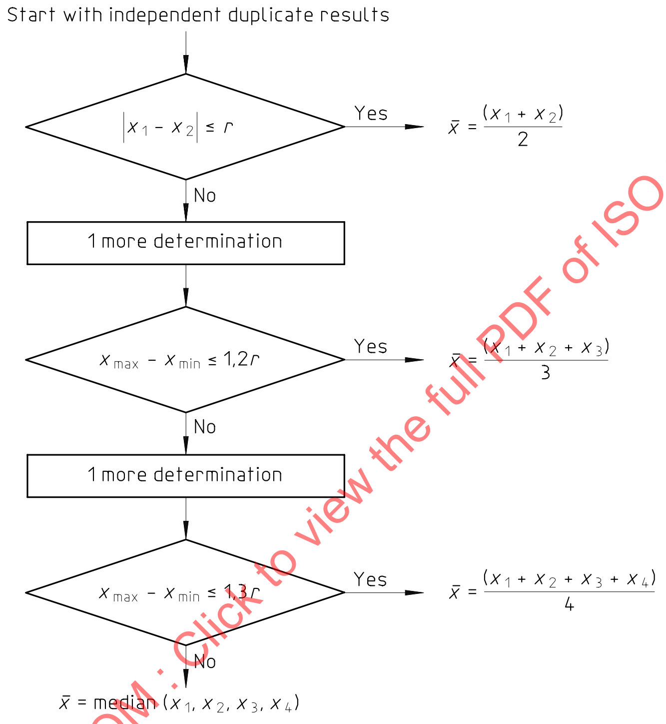
It is recommended that internal reference material be prepared and used periodically to check test repeatability.

Appropriate records of verification activities shall be maintained.

Annex A
(normative)

Flowsheet for the procedure for the acceptance of test results

Attention: *x* hereafter means *CI*



Annex B
(informative)

Example of calculation of the Clustering Index

B.1

Suppose that the mass of a reduced test portion is m_r = 1 442 g, and that after the removal of the individual pellets separated from the clusters, the mass of the remaining cluster is cm_1 = 1 430 g.

Also suppose that after the disaggregation operations, the following set of results was obtained:

	Number of disaggregation operations i	Number of revolutions	Mass of clusters - cm_i (g)
	1	zero	1 430
	2	5	700
	3	5	300
	4	5	150
	5	5	110
	6	5	80
	7	5	60
	8	5	40
TOTAL	8	35	2 870

Being the formula for the calculation of the clustering index (*CI*):

$$CI = \frac{100}{8 \times m_r} \times \sum_{i=1}^{8} cm_i \qquad \text{...(B.1)}$$

The clustering index for this test will be:

$$CI = \frac{100}{8 \times 1442} \times 2\,870 = 24{,}88 \cong 25\ \% \qquad \text{...(B.2)}$$

B.2

Suppose that for the duplicate test a value of *CI* = 32 % was obtained.

$$\overline{CI} = \frac{25 + 32}{2} = 28{,}5\ \% \qquad \text{...(B.3)}$$

According to clause 9, the permissible tolerance r = 27 % relative of the $\overline{CI}$, then r is