INTERNATIONAL STANDARD

ISO 4698

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Iron ore pellets — Determination of relative free-swelling index

Boulettes de minerais de fer — Détermination de l'indice relatif de gonflement libre

Reference number
ISO 4698:1994(E)

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.

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 4698 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Sub-Committee SC 3, *Physical testing*.

Annexes A and B form an integral part of this International Standard.

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Introduction

The method for the determination of the free-swelling index is one of several procedures used to evaluate the behaviour of iron ore pellets under specific reducing conditions. The conditions involved in this test are:

reduction under isothermal heating in an unconstrained state using a gaseous reductant;

a sample of specified size range.

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Iron ore pellets — Determination of relative free-swelling index

1 Scope

This International Standard specifies a method for the determination of the free-swelling index during unconstrained reduction of fired iron ore pellets.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3081:1986, *Iron ores — Increment sampling — Manual method.*

ISO 3082:1987, *Iron ores — Increment sampling and sample preparation — Mechanical method.*

ISO 3083:1986, *Iron ores — Preparation of samples — Manual method.*

ISO 3310-1:1990, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth.*

3 Definitions

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

3.1 free-swelling: A volume increase of fired iron ore pellets which occurs during reduction under unconstrained conditions.

3.2 free-swelling test: The determination of the unconstrained volume increase of iron ore pellets which occurs during reduction under specified conditions.

3.3 free-swelling index: A measure of the volume increase of fired iron ore pellets which occurs during reduction under unconstrained conditions, expressed as a percentage.

4 Principle

Determination of swelling of fired iron ore pellets of a specified size range under isothermal reduction, determination of the volume of pellets before and after reduction at a temperature of 900 °C, using a reducing gas consisting of carbon monoxide and nitrogen. Calculation of the free-swelling index, expressed as a percentage, using the difference between the two volumes.

5 Test conditions

5.1 General

The gas volumes and flow rates used in this International Standard are measured at a temperature of 0 °C and at atmospheric pressure [101,325 kPa[1)]].

5.2 Composition of reducing gas

The composition of the reducing gas to be fed to the furnace shall be:

CO: 30 % ± 0,5 % (*V/V*)

N_2: 70 % ± 0,5 % (*V/V*)

1) 1 mmHg = 0,133 3 kPa; 1 atm = 0,101 325 MPa

5.3 Purity of reducing gas

The purity of the reducing gas shall be ensured by confirming that impurities do not exceed the following:

H_2: 0,2 % (*V/V*)

CO_2: 0,2 % (*V/V*)

O_2: 0,1 % (*V/V*)

H_2O: 0,2 % (*V/V*)

5.4 Flow rate of reducing gas

The flow rate of the reducing gas during the test period shall be maintained at 15 l/min ± 1 l/min.

5.5 Reducing temperature

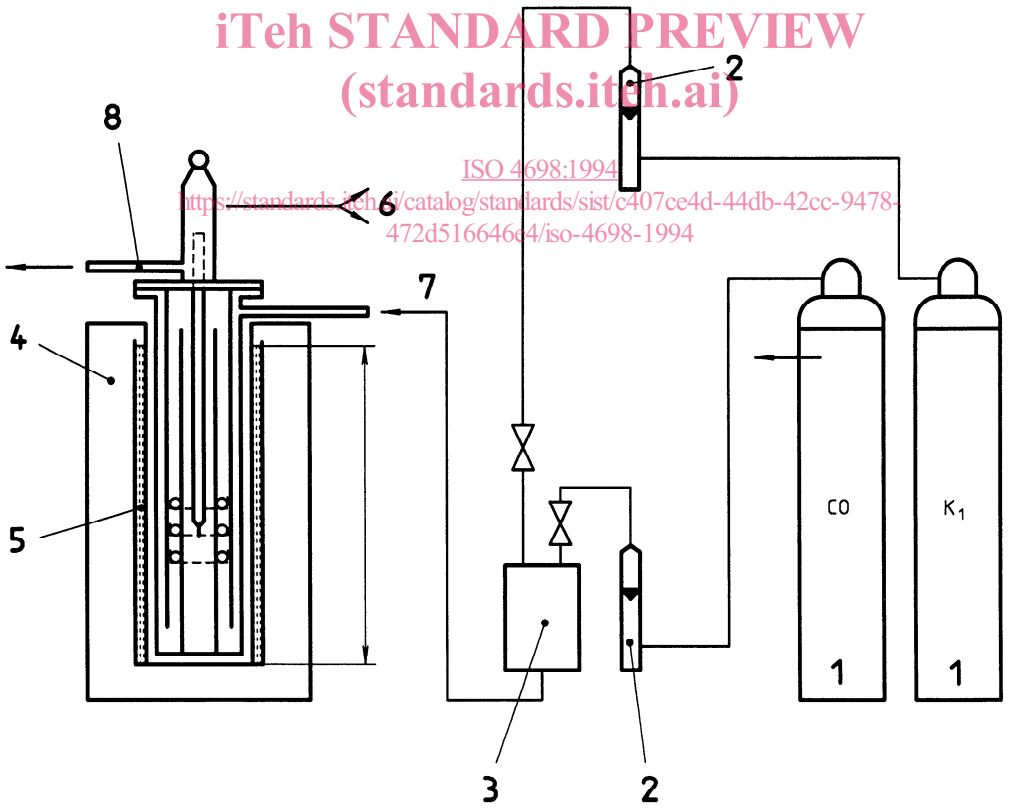
The temperature of the test portion shall be 900 °C ± 10 °C during the test period. The reducing gas shall be preheated before feeding to the furnace in order to maintain this temperature.

6 Apparatus (see figure 1)

6.1 Furnace, electrically heated, with a heating capacity that is sufficient to maintain the entire test portion and the gas contacting it at 900 °C ± 10 °C.

6.2 Reduction tube, made of non-scaling, heat-resistant metal to withstand a temperature higher than 900 °C. The internal diameter of the reduction tube shall be 75 mm ± 1 mm. The details of the reduction tube with the test portion holder are shown in figure 2.

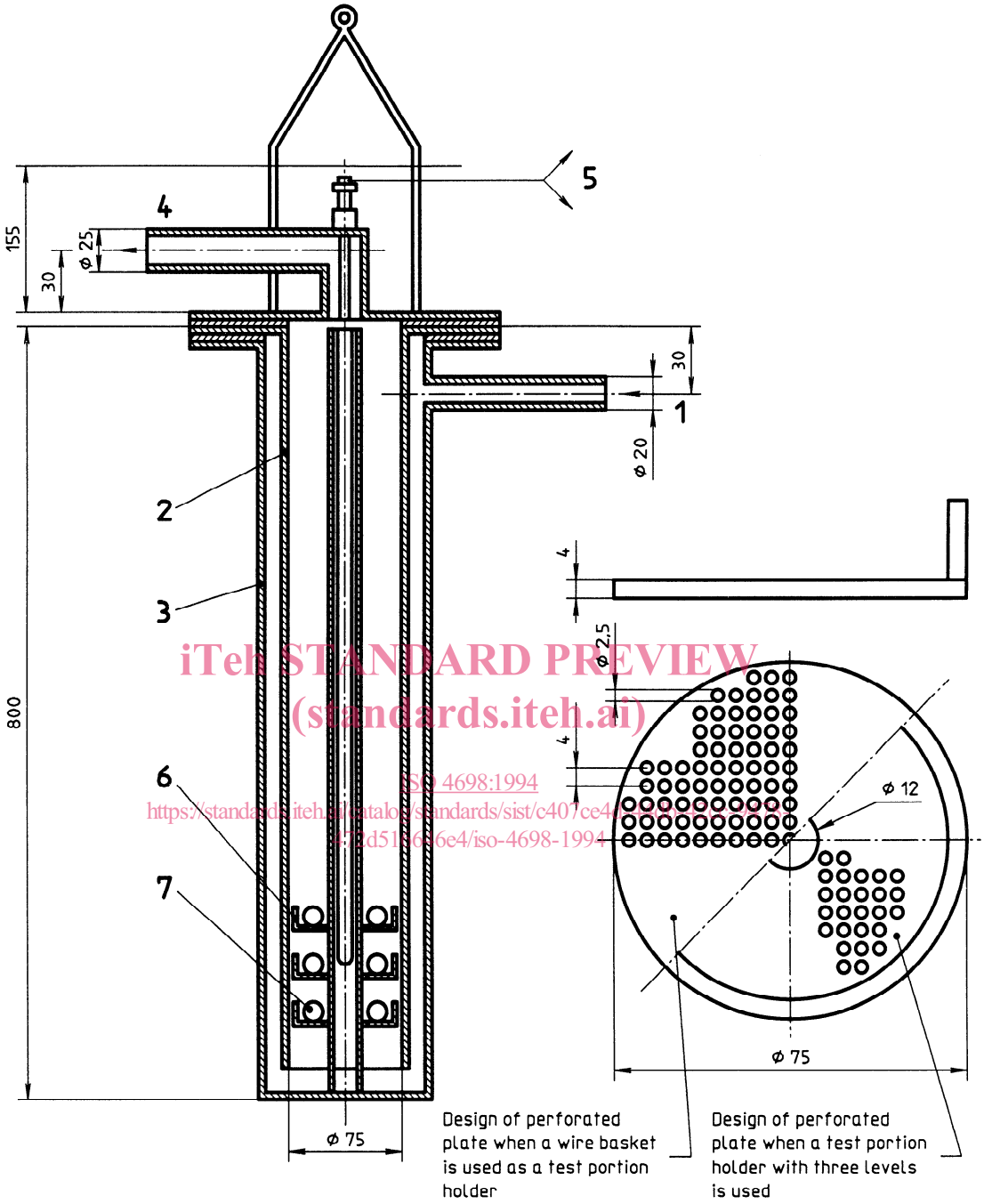
6.3 Test portion holder, a wire basket made of non-scaling, heat-resistant metal to withstand a temperature higher than 900 °C. It shall have room for 18 pellets at three levels, each level holding six pellets in the size range of 10,0 mm to 12,5 mm.



Key
1 Gas cylinders
2 Flowmeters
3 Mixing vessel
4 Furnace
5 Test portion
6 Thermocouple
7 Gaz inlet
8 Gas outlet

Figure 1 — Example of apparatus used for the reduction test

Dimensions in millimetres



a) Reduction tube

Key
1 Gas inlet
2 Inner retort
3 Outer retort
4 Gas outlet
5 Thermocouple
6 Test portion holder
7 Test portion

b) Perforated plate/test portion holder

Key	
Hole diameter	2,5 mm
Pitch between holes	4 mm
Number of holes	241
Total hole area	11,8 cm^2
Thickness of plate	4 mm
Border	3 × 7 mm

Figure 2 — Example of reduction tube with test portion holder

6.4 Volumetric apparatus, capable of determining the volume of the test portion to an accuracy of 0,2 ml. Examples of the volumetric apparatus are shown in annex A.

6.5 Test sieves, with square mesh openings of 10,0 mm and 12,5 mm in accordance with ISO 3310-1.

7 Preparation of test sample

7.1 General

Prepare the test sample according to ISO 3083 from the sample for physical testing which has been taken in accordance with ISO 3081 or ISO 3082. Oven dry the test sample at 105 °C ± 5 °C for not less than 2 h and cool to room temperature before testing.

7.2 Sample for free-swelling test

Obtain the test sample, with a mass of approximately 1 kg in the size range of 10,0 mm to 12,5 mm, by sieving. After sieving, use only whole pellets taken at random for the test portion consisting of 18 pellets.

8 Procedure

8.1 Number of determinations

Carry out two separate tests on two test portions taken from one test sample.

8.2 Determination of volume before reduction

Determine the volume of the test portion (V_0) to an accuracy of 0,2 ml, in accordance with one of the methods specified in annex A.

8.3 Reduction

Place six pellets on each of the three levels of the test portion holder and place it in the reduction tube. Seal the top of the reduction tube and insert it into the furnace. Pass a flow of inert gas through the reduction tube at a flow rate of approximately 10 l/min and commence the heating. When the temperature of the test portion approaches 900 °C, increase the flow to 15 l/min ± 1 l/min. Maintain the test portion under these conditions until the temperature is constant at 900 °C ± 10 °C.

CAUTION — Carbon monoxide and the reducing gas containing carbon monoxide are toxic and therefore hazardous. During the following procedure, testing shall be carried out in a well ventilated area or under a ventilation hood. Precautions should be taken for the safety of the operator, according to the safety codes of each country.

Introduce the reducing gas to replace the inert gas, at a flow rate of 15 l/min ± 1 l/min, and continue reduction for 60 min.

NOTE 1 Some pellets show a higher degree of swelling within a shorter reduction time than 60 min. Therefore, a shorter reduction time of 40 min may be used as an alternative when appropriate.

Replace the reducing gas with inert gas, remove the reduction tube from the furnace and cool the test portion at a flow rate of 5 l/min.

8.4 Determination of volume after reduction

Remove the cooled test portion from the reduction tube and determine the total volume of the test portion (V_1) in accordance with one of the methods specified in annex A.

9 Expression of results

9.1 Calculation of free-swelling index

Calculate the free-swelling index (V_{FS}), as a percentage, using the following equation:

$$V_{FS} = \frac{V_1 - V_0}{V_0} \times 100$$

where

V_0 is the volume, in millilitres, of the test portion before reduction;

V_1 is the volume, in millilitres, of the test portion after reduction.

Record the free-swelling index to one decimal place.

9.2 Permissible tolerance

The difference between the two individual results of the free-swelling index shall be less than 3 % absolute. If this is not the case, the procedure described in annex B shall be followed.

9.3 Calculation of final result

The final free-swelling index, expressed as a percentage, shall be determined in accordance with the flowsheet shown in annex B and expressed to one decimal place.

10 Test report

The test report shall include the following information:

a) name and address of the testing laboratory;

b) date of issue of the test report;

c) reference to this International Standard;

d) description of the sample;

e) time of reduction, if it is not 60 min;

f) free-swelling index;

g) fractional loss of mass;

h) type of volumetric method employed.

Annex A
(normative)

Methods for determination of the volume of the test portion

This annex specifies four methods for the determination of the volume of the test portion of fired iron ore pellets.

A.1 Mercury volumetric method

A.1.1 Principle

Determination of the volume of the test portion from the volume change of mercury when the test portion is immersed in mercury.

A.1.2 Apparatus

The mercury volumetric apparatus is shown in figure A.1.

A.1.3 Preparation of the sample

Dry the test portion for 8 h at 110 °C.

A.1.4 Procedure

A.1.4.1 Set the volumetric apparatus as follows.

Clamp the empty test portion holder in the apparatus.

Raise the level of the mercury with the plunger driven by the handle so that the mercury surface is level with the zero mark of the measuring tube.

Fix the stopper so that the level of the mercury cannot be raised any more with the handle.

Check that the stopper prevents further movement of the plunger when the mercury has reached the zero mark of the tube.

Lower the mercury into the container.

A.1.4.2 After the apparatus has been set, carry out the determination as follows.

Take the test portion consisting of 18 pellets. Remove the test portion holder from the apparatus and place the test portion in the holder. Clamp the test portion holder in the volumetric apparatus and raise the mercury level until the fixed stopper prevents further movement of the plunger.

Read the volume on the measuring tube. Repeat the determination and ensure that the same value is obtained.

Ensure that no mercury remains when the mercury is allowed to flow down through the test portion for the last time.

Remove the test portion holder and carefully pour the test portion into a bowl.

Check the pellets one by one to ensure that there is no mercury on them and transfer them to another bowl. Then pour any mercury left in the first bowl back into the container.

When the determination has been carried out, carry out a careful check to ensure that there is no free mercury on or near the apparatus. Collect any mercury that has been spilled and pour it into the mercury waste collection vessel in the laboratory.

A.2 Oleate-kerosine — volumetric method

A.2.1 Principle

Determination of the volume of the test portion from the difference of the masses obtained both in air and in water, after forming a hydrophobic thin film on the surface of the pellets with sodium oleate aqueous solution and stabilizing the film with kerosine. Calculate the volume relative to the density of water.

A.2.2 Test liquids

Prepare all reagents and water freshly as required.

A.2.2.1 **Water**, distilled or ion exchange water.

A.2.2.2 **Sodium oleate aqueous solution**, $c(C_{17}H_{33}COONa) = 0{,}1$ mol/l.

A.2.2.3 **Kerosine**, of reagent grade.

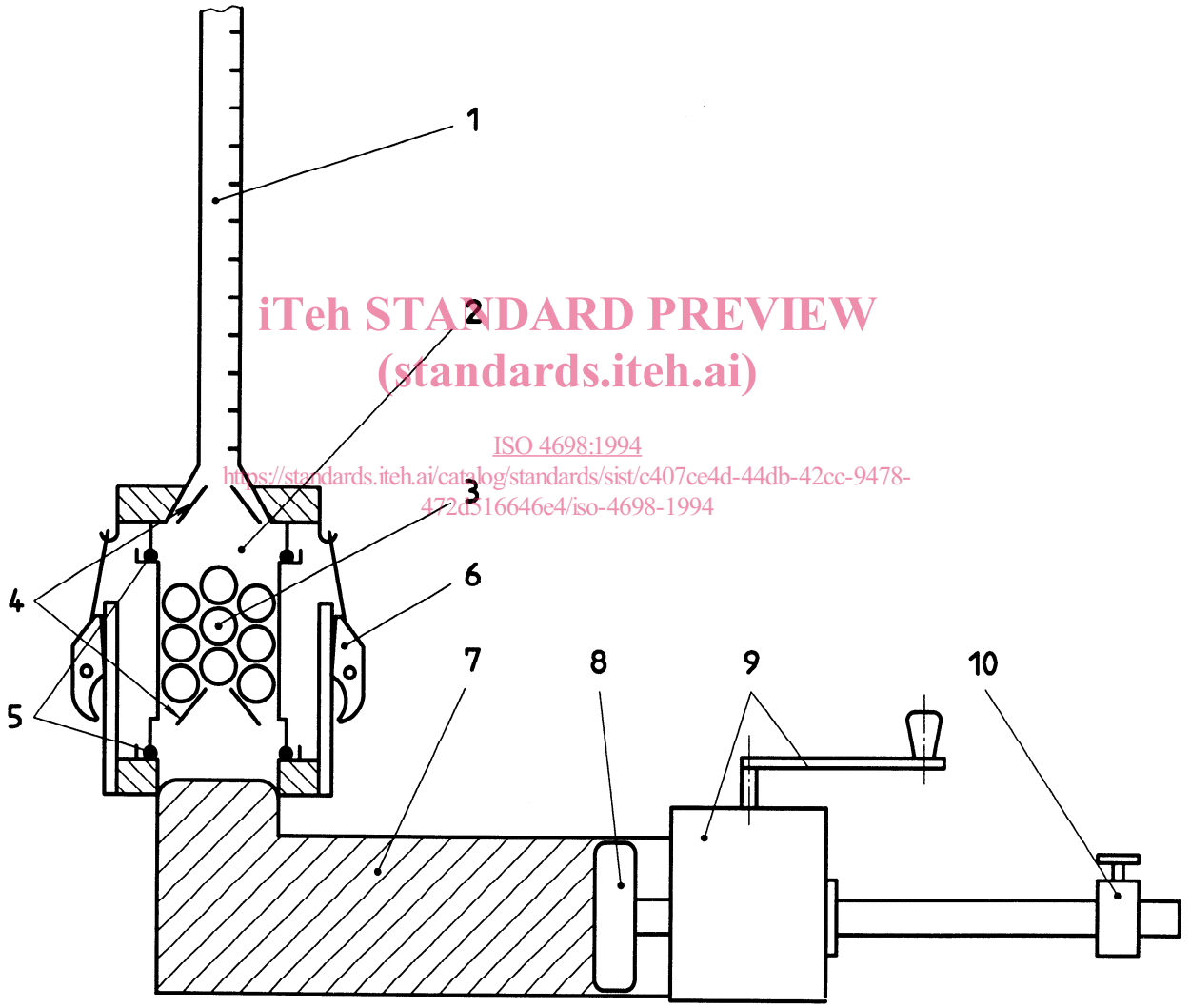
A.2.3 Apparatus

A.2.3.1 Container for test liquids (sodium oleate aqueous solution and kerosine), designed to allow free movement within it of the cage containing the pellets. It shall be made of materials, such as glass, which do not react with sodium oleate aqueous solution or kerosine.

A.2.3.2 Cage for immersion in test liquids, to hold the pellets during immersion in the reagents. It shall be made of materials which do not react with sodium oleate aqueous solution or kerosine, and shall be constructed so that the test portion can be stacked in the cage in two or three layers.

A.2.3.3 Cage for immersion in water (see figure A.2), to hold the pellets during immersion in water. It shall be made of material to which air bubbles will not adhere.

A.2.3.4 Absorbent sponges (see figure A.3), consisting of two pairs of sponges whose surfaces are covered with gauze to absorb any reagent froth on the surface of the pellets.



Key
1 Measuring tube graduated in 1/10 ml
2 Test portion holder
3 Test portion
4 Devices to retain test portion within the sample holder
5 O-rings
6 Clamp for test portion holder and measuring tube
7 Container with mercury
8 Movable plunger
9 Handle and gear box for moving plunger
10 Stopper

Figure A.1 — Example of the mercury volumetric apparatus