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Iron ores — Determination of specific surface area — Test method using air-permeability apparatus (Blaine)

Minerais de fer — Détermination de l'aire spécifique — Méthode d'essai utilisant un perméamètre (Blaine)

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

Introduction

The international trade in pellet feeds as a merchant commodity is increasing rapidly and their specific surface is one of the most important specifications required in commercial contracts. This has led to the need for the development of an international test method to measure the specific surface area of pellet feeds.

This document concerns one of a number of test methods that have been developed to measure various characteristics of iron ores. This method was developed to provide a uniform procedure, validated by collaborative testing, to facilitate comparisons of tests made in different laboratories.

This document can 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.

NOTE Automated test methods may be used provided that preliminary test results give similar results to the manual method within the repeatability, *r*, specified in 8.2.

Iron ores — Determination of specific surface area — Test method using air-permeability apparatus (Blaine)

CAUTION — This document can involve the use of hazardous materials, operations and equipment. This document does not purport to address all of the safety issues associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices prior to its use.

1 Scope

This document specifies a method to determine the fineness of iron ores in terms of specific surface area, using the manual Blaine air-permeability apparatus.

This document is applicable to pellet feeds in the range of 400 cm^2/g to 2 500 cm^2/g of specific surface area.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3082, *Iron ores — Sampling and sample preparation procedures*

ISO 11323, *Iron ore and direct reduced iron — Vocabulary*

ISO 12154, *Determination of density by volumetric displacement — Skeleton density by gas pycnometry*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11323 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

3.1
Blaine surface area
BSA
whole area of surface of pellet feed particles per mass unit

Note 1 to entry: Generally expressed as cm^2/g.

3.2
pellet feed
iron ore fines traded for pellet production

Note 1 to entry: Usually containing at least 80 % under 0,150 mm.

3.3
skeleton density
ratio between sample mass and the volume of the sample including the volume of closed pores (if present) but excluding the volumes of open pores as well as that of void spaces between particles within the bulk sample

3.4
bed porosity
ratio of the volume of voids in the bed to the bulk volume of the bed sample

4 Principle

Ambient air is drawn through a compressed test portion of known porosity. The specific surface area of the pellet feed is determined based on the rate of airflow through the test portion.

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.

A test sample of at least 300 g, on a dry basis, of the material shall be obtained.

5.2 Preparation of test portions

Oven-dry the test sample to constant mass at 105 °C ± 5 °C. Cool it to room temperature.

NOTE 1 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.

Disaggregate the whole test sample by sieving it on a 1,0 mm sieve or the nearest aperture. Obtain about 50 g of the minus 1,0 mm material. This is the partial test sample. Disaggregate it on a 0,150 mm sieve. Mix and homogenize the material retained and passing on the 0,150 mm sieve.

NOTE 2 Manual methods of division recommended in ISO 3082, such as riffling, can be applied to obtain the test portions.

The mass of each test portion shall be calculated in accordance with 7.2.1.

At least four test portions shall be obtained from the partial test sample. Take several increments using a non-magnetic spatula and weigh to the nearest 0,01 g.

6 Apparatus

6.1 General

The test apparatus shall comprise the following:

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

b) permeability cell;

c) disk;

d) plunger;

e) filter paper;

f) manometer;

g) timer;

h) caliper rule;

i) weighing device.

Figure 1 shows an example of a manual Blaine air-permeability apparatus.

6.2 Specific

6.2.1 Permeability cell, a tube made of non-scaling metal with internal diameter of 12,70 mm ± 0,10 mm. The ending of the external tube wall shall have a width of 0,81 µm. A ledge of 0,5 mm to 1,0 mm width for supporting the perforated disc (6.2.2) shall be firmly fixed in the tube internal wall so that its upper surface is 55 mm ± 10 mm distant from the tube upper end. The lower portion of the external wall of the tube shall be standard-taper male coupling to form an airtight fit with the upper end (taper female coupling) of the manometer. The upper end of the tube shall be externally fitted with a prominent collar.

6.2.2 Perforated disc, made of non-scaling metal with thickness of 0,9 mm ± 0,1 mm, diameter 0,1 mm less than the cell internal diameter and with 30 to 40 holes of 1,0 mm in diameter equally distributed over its area. When placed on the cell ledge the disc shall fit the inside of the cell and its surfaces firmly. The downward side of the disc shall be marked.

6.2.3 Plunger, a compact straight cylinder made of non-scaling metal, with diameter 0,1 mm less than the cell internal diameter, with an air vent slot of a width of 3,0 mm ± 0,3 mm on one of its sides and a collar on the top end. The cylinder length shall be such that when the plunger is placed in the cell and its collar brought in contact with the top of the cell, the distance between the base of the cylinder and the top of the perforated disc shall be 15 mm ± 1 mm.

6.2.4 Manometer, a transparent glass U-tube with outside diameter of 9 mm, vertically mounted, having one of its arms with a side outlet at 250 mm to 305 mm above the bottom of the manometer provided with a positive airtight valve or clamp 50 mm distant from the manometer arm. The top end of the arm to which this side outlet is connected shall be a standard-taper female coupling to receive and form an airtight connection with standard-taper male coupling of the permeability cell base. This arm shall have four midpoint lines etched around the tube: the lowest at 125 mm to 145 mm below the side outlet tie-in, and the others at 15 mm ± 1 mm, 70 mm ± 1 mm, and 110 mm ± 1 mm above the lowest line. The manometer shall be filled to the lowest line level with a non-volatile, non-corrosive, non-hygroscopic liquid of low viscosity and density and free of debris (e.g. a light grade mineral oil). To lift the oil column the air from the manometer can be evacuated using, normally, a flexible tube with a rubber pear that shall be connected to the free end of the side outlet. Alternatively, the oil column can be lifted by pushing the air in the other branch of the manometer using a pressure device, such as a syringe.

6.2.5 Filter paper, medium-texture, circular, with smooth edges, with the same diameter as the inside of the cell.

6.2.6 Timer, capable of being read to the nearest 0,5 s or less and with the following accuracy.

Time interval, t (in seconds)	**Accuracy**
≤ 60	0,5 s
60 < t ≤ 300	1 %

6.2.7 Weighing device, capable of weighing the test portion to an accuracy of 0,001 g.

6.2.8 Thermometer, with an accuracy of 0,5 °C or less for the temperature interval.

6.2.9 Funnel, made of plastic or glass, dimensioned to fit the permeability cell and to avoid spilling of the test portion.

6.2.10 Caliper rule, able to measure the permeability cell dimensions to the nearest 0,01 mm.

7 Procedure

7.1 Calibration of apparatus

7.1.1 General

The determination of the constant, K_1 [to be used in Formula (7)] of each air permeability apparatus shall be determined using a certified reference material (CRM), which means a material with certified values of density, porosity and specific surface. This material shall be at room temperature when tested.

The first step of the calibration is to determine the volume of the cell to be occupied by the compacted test portion. The cell volume can be determined using either mercury (ACS reagent grade or better) or caliper rule.

WARNING — Mercury being harmful to health, its use shall be avoided whenever possible.

7.1.2 Measurement of cell volume

7.1.2.1 Measurement of the cell volume using mercury

Place the perforated disk on the ledge in the permeability cell with the disk marked face downwards. Place two filter paper disks in the permeability cell, pressing down the edges, using a rod with a diameter slightly smaller than that of the cell, ensuring that the filter paper is completely adhered to the perforated disc. If the cell is made of material that will amalgamate with mercury, the interior of the cell shall be protected by a very thin film of oil just prior to adding the mercury. Use tongs when handling the cell.

Fill the cell with mercury, removing any air bubbles adhering to the wall of the cell. Level the mercury with the top of the cell by lightly pressing a small glass plate against the mercury surface until the glass is flush to the surface of the mercury and rim of the cell. Be sure that no bubble or voids exist between the mercury surface and the glass plate.

Carefully remove the mercury from the cell, weigh and record its mass, m_1, to the nearest 0,001 g.

Remove one of the filter disks from the cell.

Fill the cell with a trial quantity of 4,3 g of the pellet feed sample. Using a rod, place one filter disc on top of the material bed, and compress the bed in accordance with 7.1.4. This compacted bed shall be firm: increase the trial quantity of material if the plunger collar is touching the top of the cell without compression; on the other hand, diminish the trial quantity if the collar bottom is not touching the top of the cell after bed compression.

Fill the unfilled space at the top of the cell with mercury, remove entrapped air and level off the top as before.

Carefully remove the mercury from the cell, weigh and record its mass, m_2, to the nearest 0,001 g.

Calculate the bulk volume of the compacted sample to the nearest 0,005 cm^3 as shown by Formula (1):

$$V = (m_1 - m_2) / \rho_{Hg} \quad (1)$$

where

V is the bulk volume, in cubic centimetres, of the compacted sample;

m_1 is the amount of mercury, in grams, that filled the whole cell;

m_2 is the amount of mercury, in grams, that filled the cell space not occupied by the compacted sample;

ρ_{Hg} is the density of mercury, in grams per cubic centimetre, at the temperature of test (see Table 1).

Register the temperature in the vicinity of the cell and record it at the beginning and at the end of the determination.

Table 1 — Density of mercury, viscosity of air (η) at given temperatures

Room temperature °C	Density of mercury g/cm^3	Viscosity of air μPa·s (η)[a]
18	13,55	17,98
20	13,55	18,08
22	13,54	18,18
24	13,54	18,28
26	13,53	18,37
28	13,53	18,47
30	13,52	18,57
32	13,52	18,67
34	13,51	18,76

[a] For intermediate temperature values the viscosity of air may be obtained by interpolation.

Make at least two determinations of V, using separate compactions for each determination. If the two values agree within ±0,005 cm^3, take their average. If not, follow the procedure given in Annex A, using a repeatability r = 0,005 cm^3.

7.1.2.2 Measurement of the cell volume using a caliper rule

Take five different measurements of the height of the empty cell between the top of the two filter papers placed on the perforated disc and the edge of the cell, in mm, to the nearest 0,01 mm. Calculate the average, h_1.

Take five different measurements of the length of the cylinder body of the plunger, in mm, to the nearest 0,01 mm. Calculate the average, h_2. This length is equal to the height of the cell not occupied by the compacted test portion when the test is executed and the bottom of the plunger collar is in contact with the top of the cell.

Calculate the height to be occupied by the compacted test portion in the test, to the nearest 0,01 mm, as shown by Formula (2):

$$h = h_1 - h_2 \quad (2)$$

where

h is the height of the cell, in centimetres, to be occupied by the compacted test portion;

h_1 is the height of the cell, in centimetres, between the top of the two filter papers on the perforated disc and the edge of the cell;

h_2 is the length of the cylinder body of the plunger, in centimetres, which is equal to the cell height not filled with the compacted test portion.

Take five different measurements of the internal diameter of the empty cell in centimetres, to the nearest 0,01 mm. Calculate the average, d.

Calculate the bulk volume of the compacted sample to the nearest 0,005 cm^3 as shown by Formula (3):

$$V = \frac{\pi \times d^2 \times h}{4} \tag{3}$$

where

V is the bulk volume, in cubic centimetres, of the compacted bed;

π to four decimal places (3,1416);

d is the average of the internal diameter, in centimetres, of the cell;

h is the height of the cell, in centimetres, to be occupied by the compacted test portion.

7.1.3 Determination of the mass of CRM

The mass of the CRM sample used for the test calibration shall be that required to produce a bed of CRM of the specified porosity (0,500), and shall be calculated as shown by Formula (4):

$$m_{CRM} = \rho_{CRM}\, V\left(1-\varepsilon_s\right) \tag{4}$$

where

m_{CRM} is the required amount, in grams, of CRM;

ρ_{CRM} is the density, in grams per cubic centimetre, of the CRM sample;

V is the bulk volume of the sample compacted bed, in cubic centimetres, as measured in 7.1.2;

ε_s is the specified porosity of the bed of CRM (0,500).

7.1.4 Preparation of the compacted bed of CRM

Place the perforated disk on the ledge in the permeability cell with the marked face downwards. Place a filter paper on the metal disk and press the edges down with a rod having a diameter slightly smaller than that of the cell. Weigh the CRM sample to the nearest 0,001 g to obtain the mass according to 7.1.3 and place it into the cell. Tap the side of the cell lightly in order to level the bed of CRM. Place a filter paper disk on top of the CRM bed and compress the CRM with the plunger until the plunger collar is in contact with the top of the cell. This compacted bed shall be firm (as in 7.1.2.1). Slowly withdraw the plunger a short distance, rotate about 90°, repress, and then slowly withdraw. The use of fresh paper filter disks is required for each determination.

7.1.5 Determination of the equipment constant, K_1

Attach the permeability cell to the manometer tube, making certain that an airtight connection is obtained and taking care not to jar or disturb the prepared bed of CRM.

Using the rubber pear slowly evacuate the air in one arm of the manometer U-tube until the liquid reaches the 110 mm ± 1 mm line, and then close the valve tightly, enabling the liquid column to go down.

In case a syringe is used to lift the oil column in the arm, with the valve opened, slowly inject air into the other arm until the liquid reaches the top mark, and then close the valve tightly. Wait 30 s to allow the liquid to run down the walls and withdraw the pressure device, enabling the liquid column to go down.

Start the timer when the bottom of the meniscus of the manometer liquid reaches the 70 mm ± 1 mm line and stop when the bottom of the meniscus of liquid reaches the 15 mm ± 1 mm line. Record this time interval and the temperature of the test in degrees Celsius.

Make at least three determinations of the time interval (keeping the same compacted bed) and compute their arithmetic mean. Repeat the procedures in 7.1.4 and 7.1.5 for two more samples. Compute the time average of the three results.

NOTE 1 The efficiency of the connection can be determined by attaching the cell to the manometer, stoppering it, partially evacuating one arm of the manometer, then closing the valve. Any continuous drop in pressure indicates a leak in the system.

The equipment constant, K_1, is calculated from Formula (5):

$$K_1 = \frac{S_S \times \rho_S \times (1 - \varepsilon_S) \times \sqrt{\eta_S}}{\sqrt{\varepsilon_S^3} \times \sqrt{T_S}} \quad (5)$$

where

K_1 is a constant relating a particular Blaine apparatus to the CRM;

S_S is the certified specific surface, in square centimetres per gram, of the CRM;

ρ_S is the certified density, in grams per cubic centimetre, of the CRM;

ε_S is the porosity of prepared bed of the CRM;

η_S is the viscosity of air, in micro pascal seconds (μPa-s), at the temperature of test (see Table 1);

T_S is the measured time interval, in seconds, of the liquid column drop in the manometer arm.

Record the result to three decimal places.

The apparatus shall be recalibrated:

— at periodic intervals, to correct for possible wear on the plunger or permeability cell;

— if any loss in the manometer fluid occurs; or

— if a change is made in the type or quality of the filter paper used for the tests.

NOTE 2 It is suggested that a secondary sample be prepared and used as a fineness reference for the check determinations of the instrument between regular calibrations with CRM.

7.2 Permeability test with the pellet feed sample

7.2.1 Mass of the test portion

The mass of the test portion used for the test shall be that required to produce a bed of pellet feed with a porosity of approximately 0,500, and shall be calculated as shown by Formula (6). The skeleton density shall be calculated in accordance with ISO 12154.

$$m_{\mathrm{Fe}} = \rho_{\mathrm{Fe}}\, V\left(1-\varepsilon\right) \tag{6}$$

where

m_{Fe} is the amount, in grams, of test portion required;

ρ_{Fe} is the skeleton density of the pellet feed sample;

V is the bulk volume of the compacted test portion bed, in cubic centimetres, prepared in accordance with 7.1.2;

ε is the specified porosity of the bed of the test portion (in this case 0,500).

7.2.2 Preparation of the bed of the test portion

Place the perforated disk on the ledge in the permeability cell with the disk marked face downwards. Place a filter paper on the metal disk and press the edges down with a rod having a diameter slightly smaller than that of the cell. Weigh the pellet feed test portion to the nearest 0,001 g to obtain the mass according to 7.2.1 and place it into the cell. Tap the side of the cell lightly in order to level the bed. Place a filter paper disk on the top of the bed and compress it with the plunger until the plunger collar is in contact with the top of the cell. Slowly withdraw the plunger a short distance, rotate about 90°, repress, and then slowly withdraw. The use of fresh paper filter disks is required for each determination.

The mass and applied thumb pressure shall be adjusted to avoid either a loose bed or the plunger "rebounds" from the cell top when pressure is removed.

This compacted bed shall be firm: if the plunger collar is touching the top of the cell without compression, increase the quantity of material; on the other hand if the collar bottom is not touching the top of the cell after bed compression, diminish the quantity.

7.2.3 Permeability tests

Attach the permeability cell to the manometer tube, making certain that an airtight connection is obtained and taking care not to jar or disturb the prepared bed.

Using the rubber pear slowly evacuate the air in one arm of the manometer U-tube until the liquid reaches the 110 mm ± 1 mm line, and then close the valve tightly, enabling the liquid column to go down.

In case a syringe is used to lift the oil column in the arm, with the valve opened, slowly inject air into the other arm until the liquid reaches the top mark, and then close the valve tightly. Wait 30 s to allow the liquid to run down the walls and withdraw the pressure device, enabling the liquid column to go down.

Start the timer when the bottom of the meniscus of the manometer liquid reaches the 70 mm ± 1 mm line and stop when the bottom of the meniscus reaches the 15 mm ± 1 mm line. Record this time interval and the temperature of the test in degrees Celsius.

Make at least three determinations of the time interval (keeping the same compacted bed) and compute their arithmetic mean.

NOTE The efficiency of the connection can be determined by attaching the cell to the manometer, stoppering it, partially evacuating the one arm of the manometer, then closing the valve. Any continuous drop in pressure indicates a leak in the system.

8 Expression of results

8.1 Calculation of the Blaine specific area (BSA)

Calculate the BSA in accordance with the Formula (7). The skeleton density shall be calculated in accordance with ISO 12154.

$$\mathrm{BSA} = \frac{\mathrm{K}_1 \times \sqrt{\varepsilon^3} \times \sqrt{T}}{\rho_{\mathrm{Fe}} \times (1-\varepsilon) \times \sqrt{\eta}} \tag{7}$$

where

BSA is the Blaine specific area, in cm^2/g, of the test portion;

K_1 is the constant obtained by Formula (5);

T is the measured time interval, in seconds, of the liquid drop in the manometer arm;

η is the viscosity of air, in micro Pascal seconds (μPa-s), at the temperature of test of the test portion;

ε is the specified porosity of the bed of the test portion (in this case 0,500);

ρ_{Fe} is the skeleton density, in grams per cubic centimetre, of the pellet feed sample.

Express the result to the nearest whole number.

NOTE Values for η can be taken from Table 1.

8.2 Repeatability and acceptance of test results

The test shall be carried out at least in duplicate. Follow the procedure in Annex A, using the repeatability $r = 30\ cm^2/g$.

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

9 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 21283;

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

h) the Blaine specific area, BSA;

i) information regarding the CRM used.

10 Verification

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

The conditions of the following items shall be checked:

— cell volume;

— weighting device;

— temperature measurement devices;

— 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.