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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

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

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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.

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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*

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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.

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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

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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.

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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;

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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.

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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.

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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):

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

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