
British Standard Methods for
**Determination of specific
surface of powders**

Part 2. Recommended air permeability methods

Méthodes de détermination de la surface spécifique des poudres
Partie 2. Méthodes recommandées de perméabilité à l'air

Verfahren zur Bestimmung der spezifischen Oberfläche von Pulvern
Teil 2. Empfohlene Verfahren zur Bestimmung der Luftdurchlässigkeit

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Foreword

This British Standard, prepared under the direction of the Chemical and Petroleum Engineering Standards Committee, supersedes BS 4359 : Part 2 : 1971, which is withdrawn. This revision is based on the systematic study of the methods and the comparison of the results from various sources during the last ten years. Clause 0 details the technical changes involved in this revision.

No single method of specific surface measurement can be recommended to cover the many different types of powders encountered and the circumstances of usage, but it is possible to formulate recommended procedures that may be applied to the majority of cases. The purpose of these methods is to obtain uniformity in procedure for any one of the methods selected, in order to facilitate comparisons between determinations made in different laboratories. As with other standard methods it is advisable to check the preliminary results using standard samples.

Accurate sampling of particulate material is an essential prerequisite for the determination of the specific surface

of powders, especially when using methods in which the test portion may be relatively small in amount.

The procedures described in BS 3406 : Part 1 have been selected to prevent any alteration to the size grading of the gross sample and to avoid segregation of different sized particles when taking increments for testing purposes.

The air permeability methods described in this Part of this standard are characterized by simplicity of apparatus and rapidity of measurement. The technique may therefore be readily applied to the routine control of manufacturing and sizing processes.

Measurements wholly in the molecular diffusion zone are not dealt with in this standard but are under consideration for future inclusion in the BS 4359 series.

It has been assumed in the drafting of this standard that the execution of its provisions is entrusted to appropriately qualified and experienced people, for whose guidance it has been prepared.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Amendment No. 1
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Methods for determination of specific surface
of powders
Part 2. Recommended air permeability methods

Revised text

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Title

On the front cover and page 1 delete the generic title and substitute the following.

‘British Standard

Determination of the specific surface area of powders’.

On the front cover, in the French title, delete ‘Méthodes de détermination’ and substitute
‘Détermination’ and in the German title delete ‘Verfahren zur’.

AMD 4743
March 1985

Inside back cover

In the list of publications referred to delete the generic title of BS 3406 and substitute the
following.

‘Methods for determination of particle size distribution’.

British Standard Methods for

Determination of specific surface of powders

Part 2. Recommended air permeability methods

0. Introduction

Permeability methods measure a surface area of a solid volume of powder. The result is expressed in units of m^2/m^3 . The specific surface so obtained can be converted to a mass basis by dividing by the solid density, the result being expressed in units of m^2/kg .

The specific surface area measured by other methods is usually different from that obtained by permeability methods (e.g. see 4.2). Also, the latter methods use the effective solid density to calculate the solid volume of powder used for measurement (see 2.2). For these reasons the specific surface obtained from permeability measurements is properly called the effective permeability volume specific surface. However, for convenience, this correct terminology is implied, in this Part of this standard only, by the simpler term specific surface.

A technical change of the basic equation used to relate permeability data to specific surface area has been made in this revision. The original standard was based on the Kozeny-Carman equation and referred the user to other publications for procedures to follow in the transitional zone between wholly viscous flow and flow due to molecular diffusion. (The extra component of flow in this transitional zone is known as slip flow.) This revision is based on one of the equations that takes slip flow into account and the Carman-Arnell equation has been chosen (equation 1). For coarse powders where the effect of slip flow is small, or for some industrial purposes where the value due to viscous flow alone is sufficient for quality control, that part of the Carman-Arnell equation giving the contribution of viscous flow is identical to the Kozeny-Carman equation.

When comparing the specific surfaces of materials of different densities, the effective permeability volume specific surface, S_v , as used throughout this standard, gives a measure of the fineness of a powder, which is what is sometimes required.

1. Scope

This Part of this British Standard describes recommended methods for determining the permeability surface area of powders by measuring the resistance offered to the flow of air through a compacted bed of the powder.

The methods are applicable to powders with permeability volume specific surfaces in the range $2 \times 10^4 \text{ m}^2/\text{m}^3$ to $5 \times 10^7 \text{ m}^2/\text{m}^3$.

Four specific methods are described in appendices B to E.

Use of the permeability methods is precluded when:

- (a) agglomerates are not easily broken down (unless the specific surface area required is that of the agglomerated form);
- (b) the particles fracture or undergo deformation when the powder bed is being compacted.

NOTE. The titles of the publications referred to in this standard are listed on the inside back cover.

2. Definitions

For the purposes of this standard, the following definitions, together with those given in BS 2955, apply.

2.1 effective solid density (ρ_s). The density of powder particles as determined by a given liquid displacement method.

NOTE. See appendix A.

2.2 effective solid volume (v). The mass of the particles divided by the effective solid density.

2.3 bed porosity (ϵ). The ratio of the volume of inter-particle voids to the total bulk volume of the bed of powder used for the determination of the effective permeability specific surface. It is calculated from the mass, bulk volume and effective solid density of the powders in the manner described in 6.3.

NOTE. The term porosity has been retained in this context because of its wide usage. It should not be confused with the internal porosity of individual powder particles, much of which is not accessible to measurement by the methods of this standard.

2.4 effective permeability volume specific surface (S_v).

The permeability surface area per unit effective volume of the particles.

NOTE. For convenience, this term is referred to as 'specific surface' throughout the rest of this standard (see clause 0).

2.5 effective permeability mass specific surface (S_w).

The effective volume specific surface divided by the effective solid density, i.e. $S_w = S_v/\rho_s$.

NOTE. S_w should not be confused with the same symbol used in methods employing other principles of measurement.

2.6 viscous flow. Airflow in which adjacent layers of air do not mix except at the molecular level and when the velocity of the air at the powder interface is zero.

2.7 slip flow. Enhanced airflow caused by the air velocity at the air/powder interface not being zero as assumed for viscous flow. It becomes significant at low pressures or for very fine powders.

2.8 molecular (Knudsen) flow. At very low pressures the mean free path of the gas molecules, λ , increases and inter-molecular collisions become increasingly rare until all collisions are between the gas molecules and the void (capillary) walls. The concept of viscosity thus breaks down and molecular transfer is governed by Fick's Law of Diffusion. This regime is called the molecular flow, Knudsen flow or diffusional flow regime.

2.9 effective permeability volume surface mean diameter (d_m). The diameter of an imaginary solid sphere that has the same volume specific surface as the powder to which the term is applied. If a hypothetical powder is assumed, composed entirely of spheres of this diameter, the effective volume specific surface of the whole will be equivalent to that of the powder under test.

2.10 primary standard apparatus. Apparatus for the measurement of permeability specific surface that has been calibrated by calculation from the dimensions of the system.

2.11 secondary standard apparatus. Apparatus for the measurement of permeability specific surface that has been calibrated by the use of a powder whose specific surface has been previously determined by using a primary standard apparatus.

3. Symbols

For the purposes of this standard, the following symbols apply.

Symbol	Quantity	Unit or value	Symbol	Quantity	Unit or value
A	cross-sectional area of a bed of powder perpendicular to the direction of the flow of air	m^2	K_a	apparatus constant, representing all the factors in a specific surface/permeability relationship that are derived solely from the apparatus dimensions	dimensionless
a	manometric cross-sectional area of a constant volume apparatus	m^2	K_b	apparatus constant for a constant volume apparatus used with a fixed volume cell and a constant bed porosity	dimensionless
B	atmospheric pressure at the time of the determination as indicated by a height of manometer liquid	m	L	linear dimension of a bed of powder parallel to the direction of the flow of air (commonly known as the height of the powder bed)	m
b	error in t (see 8.7)	%	M	mean molecular mass of air	0.029 kg
C	constant of a capillary flowmeter	dimensionless	P	atmospheric pressure at the time of the determination	Pa
c	error in either ρ_L or h (see 8.6)	%	P_o	pressure of air leaving the bed of powder	Pa
d_m	effective permeability volume surface mean diameter	μm	P_1	pressure of air entering the bed of powder	Pa
d_1	particle diameter indicated on the Fisher sub-sieve sizes chart ($d_1 = d_m$ when 1 cm^3 of powder is used in the determination)	μm	P_v	water vapour pressure at the temperature of test	Pa
g_n	local acceleration due to gravity	9.81 m/s^2	Δp	pressure difference across a bed of powder	Pa
h	pressure difference across a bed of powder as indicated by a height of manometer liquid	m	q	rate of flow of an incompressible fluid through a bed of powder	m^3/s
h_1	pressure difference across a capillary flowmeter resistance as indicated by a height of manometer liquid	m	q_o	rate of flow of air from a bed of powder	m^3/s
h'_1, h'_2	manometric heights corresponding to the first and second timing marks in a constant volume method	m	q_1	rate of flow of air into a bed of powder	m^3/s
K	Kozeny constant	dimensionless (see 4.1.2)	R	universal gas constant	$8.31\text{ J/(K}\cdot\text{mol)}$
			S_v	effective permeability volume specific surface of a powder	m^2/m^3
			S_w	effective permeability mass specific surface of a powder	m^2/kg
			S_k	effective permeability volume specific surface of a powder assuming only viscous flow occurs in the determination	m^2/m^3
			S_m	effective permeability volume specific surface of a powder assuming only slip-flow occurs in the determination	m^2/m^3
			T	temperature at the time of the determination	K
			t	time	s
			V	volume of dry air passed through a constant flow rate apparatus during the determination (or during calibration of a capillary flow meter)	m^3
			V_o	volume of a constant volume apparatus between the manometer equilibrium level and the bottom of the powder bed	m^3
			v	effective solid volume of a powder sample	

Symbol	Quantity	Unit or value
v_1	effective solid volume of a powder sample used in the Fisher sub-sieve sizer	cm ³
W	mass of a bed of powder	g
W_1	mass of pure water to fill a density bottle at the selected operating temperature	g
W_2	mass of displacement liquid to fill a density bottle at the selected operating temperature	g
W_3	W_2 plus the mass of the empty density bottle	g
W_4	mass of sample used to determine the effective solid density of a powder	g
W_5	W_4 plus the mass of the empty density bottle and the displacement liquid to fill the bottle at the selected operating temperature	g
X	(see figure 3)	m
x	error in A (see 8.3)	%
y	error in L (see 8.3)	%
z	error in ρ_s (see 8.4)	%
δk_o	constant in the Carman-Arnell equation (comprising δ and k_o)	2.25 (see 4.1.3)
ϵ	porosity of a bed of powder	dimensionless
ϵ_1	porosity of a bed of powder indicated on the Fisher sub-sieve sizer	dimensionless
η	viscosity of air at its temperature at the time of the determination	N·s/m ²
λ	mean free path of gas molecules	m
ρ_{DL}	density of the displacement liquid used in the measurement of effective solid density	kg/m ³
ρ_L	density of a manometer liquid during the determination at the selected operating temperature	kg/m ³
ρ_s	effective solid density of a powder	kg/m ³
ρ_w	density of pure water during the calibration of a density bottle at the selected operating temperature	kg/m ³

NOTE 1. All the equations given in this standard are true for SI units or any set of coherent units, except those involving v_1 , which is measured in cm³. If practical units are used, the appropriate correction factors should be made to the equations.

NOTE 2. 1 Pa = 1 N/m².

4. Principles

4.1 Carman-Arnell equation

4.1.1 Basic equation. The rate of flow of a fluid moving under the influence of a constant pressure difference through a compacted bed of powder of uniform cross-sectional area is a function of the magnitude of the surface area that the walls of the channels through the bed present to the moving fluid. Because there is normally a great variation in, and a lack of precise knowledge of,

the shape and dimensions of such channels, rigorous mathematical treatment is impracticable. However, by making a number of simplifying assumptions, a relationship enabling the specific surface area of many powders to be estimated from fluid permeability data has been derived by Carman and Arnell. The equation is written in the basic form.

$$q = \frac{A\epsilon\Delta p}{K\eta L} \left\{ \frac{\epsilon^2}{S_v^2(1-\epsilon)^2} + \frac{8\delta k_o\eta\epsilon\sqrt{(2RT/\pi M)}}{3PS_v(1-\epsilon)} \right\} \quad (1)$$

Rather than attempting to solve this equation (a quadratic in S_v) directly, the calculation can be carried out by evaluating two separate terms and combining them to give S_v using equation 5.

4.1.2 The Kozeny-Carman term, S_k

$$S_k = \sqrt{\left(\frac{A\epsilon^3\Delta p}{K(1-\epsilon)^2L\eta q} \right)} \quad (2)$$

This term is identical with the Kozeny-Carman equation and gives the contribution to the surface area of the powder due to viscous flow. K , the Kozeny constant, is a complex dimensionless number not amenable to theoretical treatment. Its numerical value depends on the pore size distribution within the compacted bed. Where there is sufficient reason to believe that it is advantageous to adopt some other value for K , this is permissible but the value adopted should be stated when the results are reported. Otherwise K should be taken as 5.0.

4.1.3 The slip flow term, S_m

$$S_m = \frac{8A\Delta p\delta k_o\epsilon^2\sqrt{(2RT/\pi M)}}{3KLqP(1-\epsilon)} \quad (3)$$

δ is a complex dimensionless function of the mean pressure and k_o is a dimensionless number in the range 2 to 3. Neither of these factors has proved amenable to theoretical treatment. For the purposes of this standard the generally accepted value for the product δk_o of 2.25 should be used.

The constants in equation 3 can be combined to give a direct relationship between S_m and S_k .

When air ($M = 0.029$ kg) is used,

$$S_m = \frac{81.0S_k^2\eta(1-\epsilon)\sqrt{T}}{P\epsilon} \quad (4)$$

4.1.4 The combined term, S_v

$$S_v = (S_m/2) + \sqrt{\{(S_m^2/4) + S_k^2\}} \quad (5)$$

4.2 Effect of pores within the particles. Many difficulties can arise when the particles that comprise a powder are themselves porous. Permeability measurements indicate only the surface area that contributes to the pressure drop and this includes little of the area making up the walls of capillary cracks and cul-de-sac pores within the body of a particle. Usually, the surface areas obtained by the use of gas adsorption methods are significantly higher since this procedure does include the contributions resulting from the particle internal pore system.

The possibility that a significant fraction of the particles might contain pores make it essential that the bed porosity should not be estimated using a value of solid density extracted from a reference book. The effective solid density should therefore be determined by the method given in appendix A.

4.3 Range of measurements of the methods given in this standard. The methods given in this standard are applicable to powders with specific surface areas between the limits $2 \times 10^4 \text{ m}^2/\text{m}^3$ and $5 \times 10^7 \text{ m}^2/\text{m}^3$. For air at atmospheric pressure ($\approx 10^5 \text{ Pa}$), a temperature of 20°C (293 K) and with a bed porosity of 0.5, the surface area calculated considering only viscous flow, S_k , will be more than 90 % of S_v when S_v is less than $1 \times 10^6 \text{ m}^2/\text{m}^3$. For the same conditions, the surface area calculated considering only slip flow, S_m , will be more than 90 % of S_v when S_v is more than $36 \times 10^6 \text{ m}^2/\text{m}^3$.

Modifications to the apparatus may be necessary before testing very fine or very coarse powders.

4.4 Apparent dependence of specific surface area on bed porosity. As stated in 4.1.2, the Kozeny constant K is dependent on the pore size distribution within the bed. The pore size distribution will depend not only on the nature of the particles of the powder but also on their packing in the bed, i.e. the bed porosity.

With a new material it is advisable to draw up a plot of S_v against ϵ over the range of ϵ likely to be encountered. Frequently, such a plot results in a plateau region where S_v varies little with change in ϵ . It is desirable to work in this plateau region wherever possible, provided the bulk density characteristics do not alter appreciably from sample to sample. If the bulk density characteristics do not vary, then for routine use, beds may be packed to a consistent porosity. Where the bulk density of the material does vary, it is preferable to pack the bed at constant pressure.

Where S_v changes significantly with ϵ , however carefully the bed preparation is carried out, the method is not applicable except for some industrial control purposes where the error introduced is not significant.

5. Apparatus: general

5.1 Comparison of constant flow rate and constant volume methods. The apparatus described in this standard differ according to the way in which flow resistance is measured. Two basic methods are used; constant flow rate or constant volume. For historical reasons one particular type of permeability cell (or bed holder) tends to be associated with one particular method of measuring flow resistance. In general this association is unnecessary.

Constant flow rate methods have some advantage in that the effect of flow rate variation on bed permeability and calculated specific surface can readily be studied. They may be easily used as primary standard methods, particularly if used with bubble flowmeters, which eliminate the need for frequent calibration when the apparatus is in constant use. In general, the apparatus is more cumbersome than that used in constant volume methods.

Constant volume methods do not require a separate flowmeter, the apparatus usually being more robust and often portable.

5.2 The permeability cell

5.2.1 General. The cell has a circular internal cross-sectional area that is accurately known, and it should be easy to connect to the flow resistance measuring apparatus. The compacting device should be capable of producing from a known mass of powder a uniform bed of measurable height. (The calculation of the porosity of the compacted powder bed is described in 6.3.) It is important that the effect of the cell wall should be negligible. This will generally be so if the cell diameter is not less than 12 mm^* . The flow resistance of the empty cell should be measured separately and, if necessary, allowance made for it before calculating the specific surface of any powder from permeability measurements made with the cell.

Examples of cell design are shown in figure 1. The materials indicated in the figures are generally suitable, but may not be so in all instances, for example, metals may be dangerous when used in testing explosive powders.

The cells in general use fall into two categories.

- (a) Variable volume cells in which a bed of powder is formed by some suitable compaction technique. In general, these cells produce the most satisfactory beds, but the bed height has to be measured in order to calculate the porosity of the bed. It is difficult to prepare beds of powder with a precise, predetermined porosity using this type of cell.
- (b) Fixed volume cells in which a bed of constant height is formed, the bed porosity being predetermined by the mass of powder used in the determination. Though not of such general application, this type of cell produces a compaction that is quite satisfactory for some powders, e.g. Portland cement.

5.2.2 Measurement of permeability cell dimensions.

The internal diameter in the region to be occupied by the powder bed should be measured on at least three diameters using a telescopic gauge and micrometer to an accuracy of at least 0.1 %. For fixed volume cells, the height of the powder bed L should be measured by using a test piece of hardened metal of a diameter that will easily fit into the cell and of a height $L + 0.1 \text{ mm}$ measured to an accuracy of 0.01 mm. The test pieces should be placed in the assembled cell (including the appropriate number of filter paper discs) and the plunger should be inserted. The mean gap between the shoulder of the plunger and the top of the cell should be measured with a cathetometer so that L can be calculated to an accuracy of 0.25 %.

For variable volume cells, the length between well defined fixed marks on the cell and on the plunger body should be measured to an accuracy of 0.01 mm when the plunger is inserted in the assembled cell (including the appropriate number of filter paper discs). In use, the length between the marks should be measured to a precision such that the height of the powder bed, L , can be calculated to an accuracy of 0.25 % or better.

Calculate the cross-sectional area of the powder bed, A , to an accuracy of $\pm 0.20 \text{ %}$.

*With some materials smaller cells have been used satisfactorily.