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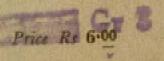
Indian Standard CODE OF PRACTICE FOR DETERMINATION OF SPECIFIC SURFACE AREA OF POWDERS BY AIR PERMEABILITY METHODS

UDC 620·168·36:666·9-492



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Indian Standard

CODE OF PRACTICE FOR DETERMINATION OF SPECIFIC SURFACE AREA OF POWDERS BY AIR PERMEABILITY METHODS

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Indian Standard CODE OF PRACTICE FOR DETERMINATION OF SPECIFIC SURFACE AREA OF POWDERS BY AIR PERMEABILITY METHODS

0. FOREWORD

- **0.1** This Indian Standard was adopted by the Indian Standards Institution on 25 April 1977, after the draft finalized by the Sieves, Sieving and Other Sizing Methods Sectional Committee had been approved by the Civil Engineering Division Council.
- 0.2 This standard lays down the procedure for finding the specific surface area of powders in the sub-sieve range. The various methods described in this standard are applicable to powders whose specific surface areas lie within the range of 500 to 40 000 cm²/g. For fine powders the method given in this standard are not valid since there will be molecular flow (known also as 'slip flow') in addition to viscous flow and it becomes significant in comparison with viscous flow. Since small quantities of material are used in these tests, accurate sampling of the material is very essential. For accurate sampling of the material see IS: 4879-1968*.
- **0.3** In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country.

1. SCOPE

- 1.1 This standard describes the following methods for the determination of specific surface area of powders. These methods are applicable to powders whose surface areas lie within the range of 500 to 40 000 cm²/g:
 - a) Constant flow type air permeability method (Lea and Nurse Type),
 - b) Variable flow type air permeability method (Blaine Type), and
 - c) Variable flow type air permeability method (Rigden Type).
- 1.2 The methods described in 1.1 differ basically in the way in which the flow resistance is measured.

^{*}Method of sub-division of gross sample of powder used for determination of particle size.

2. PRINCIPLE

2.1 The following equation is used to find the specific surface area of powders:

$$S_{\mathbf{w}} = [(e^{3} \triangle p \ g \ At)/d^{2}_{\mathbf{o}} \ (1-e)^{2} \ Lq \ K\eta]^{\frac{1}{2}}$$

where

 $S_{\rm w} = {\rm surface \ area \ of \ powder \ in \ cm^2/g},$

 $e = \text{porosity cm}^3/\text{cm}^3$,

 $\triangle p$ = pressure difference in gf/cm²,

A = cross-sectional area of the sample on which the pressure is acting in cm²,

L = length of the sample in cm,

 d_0 = true density of the sample g/cm³,

K = constant (which is generally 5 ± 0.5 for particles that are approximately spherical in shape),

 $\eta = \text{viscosity in poise,}$ $q = \text{total flow in cm}^3, \text{ and}$

 \hat{t} = time of flow for q cm³ to pass through the sample in seconds.

Note — This equation is valid in a statistical sense for the passage of an incompressible fluid through a homogeneous bed of powder. The particles of powder have been assumed to be free from pores.

2.2 Use of Compressible Fluid — In general air or gas is used as the fluid for the permeability methods. Since air is a compressible fluid some modification to the above equation is required. If p_e is the entry pressure and p_0 is the exit pressure and if q_0 is the volume rate of flow from the powder bed, the q in equation mentioned under **2.1** is given by:

$$q = q_0 (P_e - \triangle p)/(p_e - \triangle p/2)$$
 or

$$q = q_0 p_0/(p_0 + \triangle p/2).$$

Alternatively, if the flow rate *into* the bed is q_e the volume of q in equation given under **2.1** is given by:

$$q = q_e p_e/(p_e - \triangle p/2)$$
 or

$$q = q_e (p_o + \triangle p)/(p_o + \triangle p/2).$$

These corrections known as 'mass flow corrections' in many practical conditions have values very near to unity so that they can be ignored.

2.3 Non-spherical Particles — When the particles are non-spherical and possess a high specific surface, the value of the constant K may depart considerably from 5. Such a departure may also occur with powders of specific surface where particle size is extreme. 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.

3. PERMEABILITY CELL

3.1 The cell shall have a circular internal cross-sectional area which is accurately known. The compacting device should be able to produce a

uniform bed of known height with a known weight of powder. Care should be taken to avoid wall effect of the cell.

4. CONSTANT FLOW TYPE AIR PERMEABILITY METHOD (LEA AND NURSE TYPE)

- 4.1 The details of the method are the same as given in IS: 5536-1969*.
- 4.2 A worked out example of this method is given in Appendix A.

5. VARIABLE FLOW TYPE AIR PERMEABILITY METHODS

5.1 Blaine Type

- **5.1.1** Apparatus The apparatus shall be the same as specified in IS: 5516-1969†.
- **5.1.2** Procedure The cell is joined to the coupling to fit the bottom of the cell. The liquid level in the U-tube initially at zero level is displaced by reducing pressure in the liquid arm fixed to the permeameter cell to a height of 11 cm above zero level. The suction pressure is applied via side tube having valve or clamp. When the valve is closed after raising the liquid level, the liquid will start to fall. The time taken for the liquid level to fall from 7 cm to 1.5 cm above zero level is noted.
- **5.1.3** Calculations The specific surface area of the powder shall be calculated from the following equation. The equation is simplified form of equation under **2.1**.

 $S_{\rm w} = (14/d_{\rm o}) [e^3/(1-e)^2 K_1]^{\frac{1}{2}}$

where

 $S_{\rm w} = {\rm specific \ surface \ area \ (cm^2/g)},$

 d_0 = true density of the material in g/cm³,

 $e = porosity (cm^3/cm^3),$

 K_1 = permeability constant which can be calculated from the following formula:

 $K_1 = \eta A_{\rm m} L \ln (h_1/h_2)/(d A_{\rm c} t)$

 η = viscosity of air in poise,

L = depth of powder bed in cm,

 $A_{\rm m} = {\rm Average\ cross\text{-}sectional\ area}$ of the manometer tube in cm²,

 h_1 = height of the liquid level in manometer above zero level at zero time in cm,

 h_2 = height of the liquid level in manometer above zero level in cm at time t seconds,

 $d = \text{density of manometric liquid in g/cm}^3$,

 $A_c = \text{cross-sectional}$ area of powder bed (that is area of cell) in cm².

t =time taken for the manometric liquid level to fall from height h_1 to h_2 in seconds.

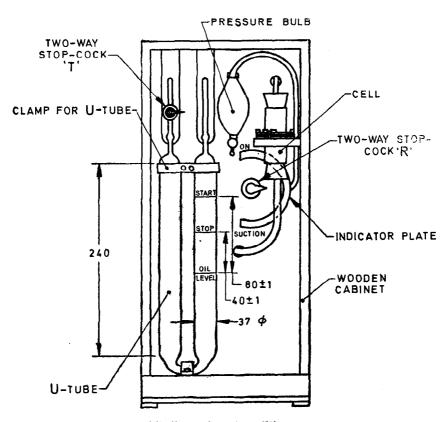
^{*}Specification for constant flow type air-permeability apparatus (Lea and Nurse type). †Specification for variable flow type air-permeability apparatus (Blaine type).

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5.1.4 A worked out example using this method is given in Appendix B.

5.2 Rigden Type

5.2.1 Apparatus — Apparatus is as shown in Fig. 1.



All dimensions in millimetres.

Fig. 1A Typical Variable Flow Type Air Permeability Apparatus (Rigden Type) (Front View)

5.2.1.1 Permeability cell — The permeability cell shall consist of two flanged cylindrical units of brass or stainless steel which can be bolted together. A rubber or other suitable gasket shall be used between the flanges to render the joint airtight. The bottom half of the cell shall have a suitable recess for supporting the perforated plate and the gasket. Dimensions and tolerances shall be as detailed in Fig. 2.

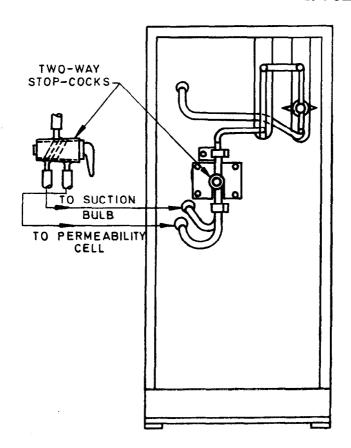
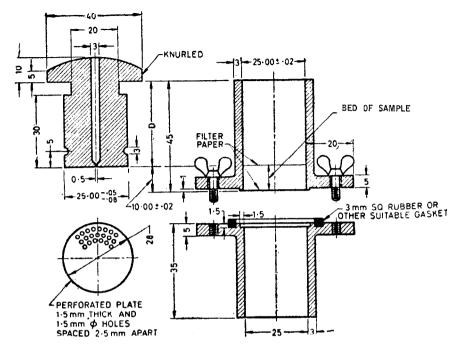


Fig. 1B Typical Variable Flow Type Air Permeability Apparatus (Rigden Type) (Rear View)

5.2.1.2 Plunger — The plunger, of brass or stainless steel, shall have a knurled head, air event and circular recess as detailed in Fig. 2. The bottom of the plunger shall have sharp square edges and shall be at right angles to the principal axis. The dimension D of the plunger in Fig. 2 shall be so adjusted that when the plunger is placed in the cell and the knurled head brought in contact with the top of the cell, a bed of powder sample of thickness 10 ± 0.05 mm is formed over the filter paper kept on top of the perforated plate. The plunger shall also fit snugly into the cell (clearance between the plunger and the cell should not be more than 0.1 mm).



All dimensions in millimetres.

Fig. 2 Details of Permeability Cell

- **5.2.1.3** Perforated plates The plate shall be of brass or stainless steel, with a number of circular perforations. The plate shall be plane and shall fit snugly in the recess in the permeability cell.
- **5.2.1.4** Manometer The manometer shall be constructed of clear borosilicate glass or equivalent material. The dimension shall generally be as shown in Fig. 1. The manometer shall be of standard wall glass tubing. The manometer shall be filled to the mid-point with a nonvolatile, nonhygroscopic liquid of low viscosity and density, such as dibutylphthalate (dibutyl 1, 2 benzene-dicarboxylate or kerosene).
- **5.2.1.5** Manometer stand The entire apparatus shall be mounted on a stand of well-seasoned teak or suitable hardwood or pressed steel sheets. For convenience in handling, the manometer and the cell shall be mounted on the front panel, with the cell assembly on one side. The mounting shall be secured by clamps. The rubber tubings and rubber corks for linking the cell and manometer shall be of good quality to resist frequent reconnections.
 - 5.2.1.6 A measuring scale may either be fixed or be etched to record

the levels of manometric liquid conveniently. All connections shall be airtight.

5.2.1.7 Filter paper — The filter paper shall be medium retentive (corresponding to Whatman No. 40). The filter paper discs shall be circular, with smooth edges, and shall have the same diameter as the inside of the cell.

Note — Filter paper discs that are too small may leave part of the sample adhering to the inner wall of the cell above the top disc. When too large in diameter, the discs have tendency to buckle and cause erratic results.

- **5.2.2** Procedure Insert the upper bung of the permeability cell. Operate the stop-cock T on the left limb of the U-tube opening it to atmosphere. Operate the stop-cock R so that the indicator line is on to pressure. Press the pressure bulb when the oil level in the right limb is well past the mark, start turn the stop-cock R to 'off'. Close the stop-cock T on the left limb. Turn the stop-cock R to 'on'. Insert the lower rubber bung taking care to avoid forcing air through the powder in the wrong direction. Measure the time of the passage of the liquid level in the U-tube between two marks.
- **5.2.3** Calculations The specific surface area of the powder shall be calculated from the following equation:

$$S_{\rm w}^2 = \frac{M t e^3}{(1-e)^2 d_{\rm o}^2 L}$$

where

 $S_{\rm w} = {\rm specific \ surface \ area \ (cm^2/g)},$

t = time of flow of the manometric liquid between two marks in seconds,

 d_0 = true density of the material in g/cm³,

e = porosity of the bed cm³/cm³, L = depth of powder bed in cm,

M = constant of the apparatus and is given by the following formula:

$$M = \frac{196 \ A_{\rm c} \ d}{\eta \ A_{\rm m} \ \ln \ h_1/h_2}$$

 η = viscosity of air in poise,

 $A_c =$ cross-sectional area of powder bed, that is, area of cell in cm²,

 $A_{\rm m}=$ average cross-sectional area of the manometer tube in ${
m cm}^2$,

 h_1 = height of the liquid level in manometer above zero level at zero time in cm,

 h_2 = height of the liquid level in manometer above zero level in cm after t seconds, and

 $d = \text{density of manometric liquid in g/cm}^3$.

5.2.4 A worked out example using this method is given in Appendix C.

APPENDIX A

(Clause 4.2)

WORKED OUT EXAMPLE USING LEA AND NURSE TYPE APPARATUS

A-1. SPECIFIC SURFACE AREA OF CEMENT

Density d_0 of cement=3.15 g/cm³

Diameter of cell=2.5 cm

Cross-sectional area of cell = $\sqrt{(2.5/2)^2}$ = 4.908 cm²

Volume of bed=4.908 cm³ (since the height of bed is 1 cm)

Porosity=0.475

Mass of powder in the bed

= 8.118 g (see Table 2, IS: 5536-1969*)

 $C = 2.23 \times 10^{-6}$ (cgs units)

 $h_1 = 16.6 \text{ cm}$

 $h_2 = 31.7 \text{ cm}$

Substituting the above values in equation given in **5.6** of IS: 5536-1969*.

 $S_{\rm w} = 2.975 \, {\rm cm}^2/{\rm g}$.

APPENDIX B

(Clause 5.1.4)

WORKED OUT EXAMPLE USING BLAINE'S TYPE APPARATUS

B-1. SPECIFIC SURFACE AREA OF LIME POWDER

Density of the powder=2.4 g/cm³

Diameter of the cell=1.27 cm

Cross-sectional area of the cell (powder bed) = $\chi (1.27/2)^2 = 1.267$ cm²

Depth of powder bed=1.5 cm

Volume of the powder bed= $1.267 \times 1.5 = 1.90$ cm³

Mass of powder used=1.377 3 g

Apparent density of powder bed=1.377 3/1.90=0.724 7 g/cm³

Porosity = 1 - (apparent density/true density)

= 1 - (0.7247/2.4) = 0.6981

Internal diameter of manometer tube=0.75 cm

Cross-sectional area of manometer tube = $\sqrt{(0.75/2)^2}$ = 0.441 5 cm²

Viscosity of air at $27^{\circ}C = 1.842 \times 10^{-4}$ poise

Density of manometer liquid=0.89 g/cm³

^{*}Constant flow type air-permeability apparatus (Lea and Nurse type).

$$h_1 = 7 \text{ cm} \quad h_2 = 1.5 \text{ cm}$$
 $t = 214 \text{ seconds}$

 K_1 is found by using equation given under **5.1.3**.

$$K_1 = (1.842 \times 10^{-4}) (0.4415) (1.5) (1n 7/1.5)/(0.89) (1.267) (214) = 7.807 \times 10^{-7}$$

Surface area of powder is found using equation given under 5.1.3.

$$S_{\rm w} = (14/2 \cdot 4) \left[(0.698 \ 1)^3 / (1 - 0.698 \ 1)^2 (7.807 \times 10^{-7}) \right]^{\frac{1}{2}}$$

= $1.276 \times 10^4 \ {\rm cm}^2/{\rm g}$.

APPENDIX C

(Clause 5.2.4)

WORKED OUT EXAMPLE USING RIGDEN TYPE APPARATUS

C-1. SPECIFIC SURFACE AREA OF FERRIC OXIDE POWDER

Density of ferric oxide=5·1 g/cm²

Diameter of the cell=2.5 cm

Cross-sectional area of the cell (powder bed)

$$= \pi \left(\frac{2.5}{2}\right)^2 = 4.908 \text{ cm}^2$$

Depth of the powder bed=1 cm

Volume of the bed=4.908 cm³ Mass of the powder=8.260 g

Apparent density of powder = $\frac{8.260}{4.908} = 1.68 \text{ g/cm}^3$

Porosity = 1 - (1.68/5.1) = 1 - 0.3294 = 0.6706

Internal area of manometer tube = $\pi \left(\frac{3.7}{2}\right)^2 = 10.75 \text{ cm}^2$

Density of manometric liquid = 0.89 g/cm³

Viscosity of air at $27^{\circ}C = 1.842 \times 10^{-4}$ poise

 $h_1 = 8 \text{ cm} \quad h_2 = 4 \text{ cm}$

t = 603 seconds

 $M = \frac{(196 \times 4.908 \times 0.89)}{(1.842 \times 10^{-4} \times 10.75 \times 1n \ 2)}$

 $= 62.4 \times 10^{+4}$

 $S_{\rm w} = [\{62.4 \times 603 \times (0.670 \ 6)^3/(1-0.670 \ 6)^2 \times (5.1)^2 \times 1\}]^{\frac{1}{2}}$ = 6 343 cm²/g.

INDIAN STANDARDS

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SIEVES, SIEVING AND SIZING METHODS

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