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# Indian Standard

METHOD FOR DETERMINATION OF SPECIFIC SURFACE AREA OF POWDERS AND POROUS PARTICLES USING LOW TEMPERATURE GAS ADSORPTION TECHNIQUES

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# Indian Standard

# METHOD FOR DETERMINATION OF SPECIFIC SURFACE AREA OF POWDERS AND POROUS PARTICLES USING LOW TEMPERATURE GAS ADSORPTION TECHNIQUES

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# Indian Standard

# METHOD FOR DETERMINATION OF SPECIFIC SURFACE AREA OF POWDERS AND POROUS PARTICLES USING LOW TEMPERATURE GAS ADSORPTION TECHNIQUES

## **0.** FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 21 January 1986, 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** No single method of surface area determination can be recommended for different types of powders. A powder which is to be used as an adsorbent is best tested by an adsorption method. The method using low temperature gas adsorption technique is commonly used for comparing the results obtained in various laboratories. The technique, however, is not applicable to solid particles containing micropores and microporous materials. The basis and background of the technique are given in the article originally published by S. Brunauer, P.H. Emmett and E. Teller in the *Journal of American Chemical Society*, 1938, Vol 60 P 309 and have been quoted in several books. This method is commonly known as BET method.

**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. This has been met by driving assistance from BS 4359: Part 1: 1984 Determination of the specific surface area of powders: Part 1 Recommendations for gas adsorption (BET) methods, issued by the British Standards Institution.

**0.4** In reporting the results of a test or analysis, made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS :  $2-1960^*$ .

<sup>\*</sup>Rules for rounding off numerical values ( revised ).

## 1. SCOPE

1.1 This standard covers the method of determining the specific surface area of powders or porous particles by measuring the quantity of nitrogen which completely covers the surface with a single layer of adsorbed nitrogen molecules.

## 2. TERMINOLOGY

2.0 For the purpose of this standard, the definitions given in IS: 4124-1981\* together with the following definitions shall apply.

2.1 Adsorbate — The adsorbed gas.

**2.2 Adsorbed Volume** ( $V_a$ ) — The volume of gas (corrected to STP) adsorbed at the equilibrium adsorption pressure  $P_e$  (see also 2.5).

2.3 Adsorbent - The material on which the adsorption takes place.

2.4 Adsorptive — The gas to be adsorbed.

**2.5 Equilibrium Adsorption Pressure** ( $P_e$ ) — The pressure of gas in equilibrium with the adsorbate.

**2.6 Isotherm (Adsorption Isotherm )** — A curve obtained by plotting the adsorbed volume against the equilibrium pressure or relative pressure at a constant temperature.

2.7 Molecular Cross-Sectional Area ( $\sigma$ ) — The average area occupied by the adsorbate molecule in the monolayer.

**2.8 Monolayer Volume**  $(V_m)$  — The volume (corrected to STP) of gaseous adsorbate which forms a monomolecular layer over the surface of the adsorbent.

**2.9 Relative Pressure** — The ratio of the equilibrium adsorption pressure  $P_e$  to the saturation vapour pressure  $P_o$ .

**2.10 Saturation Vapour Pressure** ( $P_{\sigma}$ ) — The vapour pressure of the bulk liquefied adsorbate at the temperature of adsorption.

**2.11 Specific Surface Area** (S) — The surface area per unit mass  $(S_w)$  or volume  $(S_v)$  of the material usually expressed in  $m^2/g$  or  $m^2/cm^3$  respectively.

## 3. PRINCIPLE

**3.1** The adsorption of nitrogen at liquid nitrogen temperature is determined either volumetrically or gravimetrically. From the adsorption data, the quantity of gas that would be required to cover the whole surface with a statistical monomolecular layer is determined using the BET equation (see 3.1.2).

<sup>\*</sup>Glossary of terms relating to powders (first revision).

**3.1.1** For materials possessing specific surface area less than  $2 \text{ m}^2/\text{g}$ , krypton gas is advantageous because of its low saturation vapour pressure at liquid nitrogen temperature. The dead space corrections are reduced considerably by using this gas.

3.1.2 The BET equation is as follows:

$$\frac{P_e/P_o}{V_a (1 - P_e/P_o)} = \frac{1}{V_m C} + \frac{(C - 1)}{V_m C} \times P_e/P_o,$$

where

C = constant related to the energy of adsorption.

**3.1.2.1** The value of C should be calculated. BET method is not applicable in cases where the value of C is less than 10; in cases where the value of C is between 10 and 100, there may be unacceptable errors; in all cases where the value of C is greater than 100, the method is applicable.

**3.1.3** When experimental data are plotted with  $\frac{P_o/P_o}{(1 - P_o/P_o) \Sigma V_a}$ 

on the ordinate against  $P_e/P_o$  on the abscissa, usually a straight line results for  $P_e/P_o$  values between about 0.05 and 0.35. A typical BET plot is shown in Fig. 1. The intercept and slope of this straight line plot

are respectively  $\frac{1}{V_m C}$  and  $\frac{C-1}{V_m C}$ . From these C and  $V_m$  can be

calculated as given below:

$$C \Rightarrow \frac{\text{Slope}}{\text{Intercept}} + 1$$
$$V_m = \frac{1}{(\text{Slope} + \text{Intercept})}$$

**3.1.4** The surface area of the adsorbing sample is calculated from the monolayer gas volume ( $V_m$ ), the area covered by a single layer of gas molecules ( $\sigma$ ), Avogadro's Number (N) giving the total number of gas molecules in amole of gas at standard conditions ( $6.022 \times 10^{23}$ /mol) and the molar volume of gas  $V_o$ . If the weight of sample used in the experiment is W, the specific surface area ( $S_w$ ) of the sample is calculated using the following equation:

$$S_w = \frac{V_m \ \mathcal{N} \ \sigma}{V_o \ W}$$



FIG. 1 TYPICAL BET PLOT

For nitrogen (  $\sigma = 0.162 \text{ nm}^2$  ), the relationship becomes

$$S_w = \frac{4 \cdot 35 \ V_m}{W}$$

where

 $V_o = \text{ideal gas volume} (22 \, 414 \, \text{cm}^3).$ 

3.1.5 The molecular cross-sectional areas of various adsorbates for use with BET method are given in Appendix A.

3.2 With this test method, the specific surface area would be within  $\pm 2$  percent in view of uncertainities involved in the measurement of weight, volume, pressure and temperature.

3.3 It is advisable to use Standard Reference Material (SRM) for checking the results.

#### 4. METHOD

4.1 Apparatus — The apparatus consists of the following units:

- a) High vacuum system;
- b) Vacuum manifold;

- c) Sample bulbs;
- d) Gas burette;
- e) Vacuum gauges;

f) Mercury manometer, accurate to 0<sup>1</sup> mmHg; and

g) Gas storage vessels.

A layout of the apparatus is shown in Fig. 2. The apparatus shall be able to record low equilibrium pressure precisely.

**4.1.1** High Vacuum System — The high vacuum system consists essentially of an oil or mercury diffusion pump backed by a rotary pump. The system should be capable of pumping down the manifold to a pressure of less than  $1 \times 10^{-4}$  mmHg within half an hour after putting on the diffusion pump. A cold trap is provided between the manifold and the diffusion pump to stop the oil or mercury vapours or moisture to contaminate the sample or even enter the manifold.

**4.1.2** Vacuum Manifold — The vacuum manifold consists of the glass tubes connecting the mercury manometer, sample-tubes, gas burette and the gas holders.

**4.1.3** Sample Bulbs — Sample bulbs of different sizes may be used depending on the expected surface area of the material. The volume of the sample bulb can vary from 5 to 20 cm<sup>3</sup>. The designs of such bulbs are shown in Fig. 3. The bulb shown in Fig. 3B facilitates the removal of air from the sample and reduces the tendency of the fine powder being carried away under the influence of high vacuum during outgassing and hence should be preferred.

**4.1.4** Gas Burette — The gas burette should have several calibrated bulbs of variable sizes so that a choice of volume may be there for use. In the design shown in Fig. 2, the gas burette has four bulbs with approximate volumes 2, 5, 25 and 90 cm<sup>3</sup>. The accuracy of the sample bulb calibration should be within  $\pm 0.02$  cm<sup>3</sup>. These bulbs are water jacketted so that water can be made to flow through a thermostated bath. Thus the temperature of the bulbs can be maintained constant within  $\pm 0.5^{\circ}$ C. Calibration marks are engraved between two bulbs and also at the top and bottom of the burette. The burette is connected to the vacuum manifold and the mercury reservoir as shown in Fig. 2.

**4.1.5** Rough Vacuum Line — The rough vacuum line for the mercury reservoirs may consist of a rotary vacuum pump connected to the vacuum stopcocks of the two reservoirs of about  $500 \text{ cm}^3$  capacity through a vacuum tap.

**4.1.6** A Three-Way Stopcock and the Side Mercury Reservoir — In order to increase the gas pressure in the system, one three-way stopcock with a mercury reservoir is provided as shown in Fig. 2.

**4.2 Construction of the Apparatus** — Glass tubing of 7 to 8 mm internal diameter should be used for the vacuum line so that a good pumping rate may be obtained. Glass capillaries of 2 mm diameter should be used for the portion of the apparatus shown by the solid bold lines. The distances between the taps in this portion should be kept as small as possible so that the dead space is reduced to the minimum. High vacuum stopcocks should be used throughout.

**4.3 Preparation** — Stopcocks are greased with the high vacuum grease of good quality. First the apparatus is evacuated using rotary pump. When the pressure of the system falls below  $1 \times 10^{-1}$  mmHg, diffusion pump can be switched on. Provided there are no leaks, a vacuum of the order of  $10^{-4}$  mmHg is obtained within 30 minutes after starting the diffusion pump. If it takes longer time, then the system needs to be checked for leaks and repaired accordingly. The nitrogen and helium gas reservoirs are filled with these gases of ultra high purity (not less than 99.9 percent) to a pressure of about 1 000 mmHg. These gases can be filled after evacuating the reservoirs to the above vacuum.

#### 4.4 Test Procedure

**4.4.1** The procedure given here is based on the volumetric apparatus shown in Fig. 2 and nitrogen gas at liquid nitrogen temperature is to be used. The isotherm obtained should be close to Type II or IV of the BDDT (Brunauer, Deming, Deming and Teller) classification shown in Fig. 4 for which this method is applicable, even though working with the particular system, the user can get other types of isotherms, in which case this procedure should be discarded. If, methods other than nitrogen are used for standardization, the experimental results should correlate with those obtained by the standard procedure.

**4.4.2** Total Surface Area — The quantity of sample to be tested should be such that the total surface area is in the range of 20 to 100  $m^2$ , preferably in the region of 50  $m^2$ . The sample is then placed in the sample bulb. For the calculation of specific surface, the mass of the oven-dried sample should be used. This procedure avoids the error due to loss in weight during outgassing.

**4.4.3** Outgassing — A small quantity of glass wool is placed in the sample tube to avoid escape of powder while outgassing. The sample tube is connected to the apparatus. The sample is evacuated first at room temperature and then the temperature of the bulb is raised by using a small furnace. Care should be taken to avoid entrainment of powder during outgassing. The temperature and time for outgassing depends on the material under test (see Appendix B for method of outgassing).

**4.4.4** Dead Space Determination with Helium — Helium gas is generally not adsorbed on the surface of many solids even at liquid nitrogen temperature. A small quantity of helium is introduced in the gas burette keeping the sample tube stopcock shut. The gas pressure  $(H_1)$  is



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FIG. 4 Types of Isotherms According to BET Classfication

recorded to at least 0.1 mmHg with the help of a cathetometer. Now the stopcock is opened to allow the helium to expand into the sample bulb maintained at the liquid nitrogen temperature. Record the equilibrium pressure as  $(H_2)$ . Warm up the sample bulb while pumping out the helium from the system.

4.4.4.1 A reference mark on the sample tube below the ground glass at the top of the bulb portion shall be made and the liquid nitrogen level shall be maintained constant at this mark during dead space determination and adsorption measurement.

#### 4.4.5 Nitrogen Adsorption

**4.4.5.1** After the determination of the dead space, the system is evacuated till a steady vacuum better than  $10^{-4}$  mmHg is attained. Stopcock for the sample tube is shut and a thermos flask containing liquid nitrogen is placed in position so that the sample bulb is completely immersed in the bath.

4.4.5.2 A small dose of pure nitrogen from the connected flask is introduced in the system, keeping stopcock for sample bulb shut and the corresponding pressure is noted within 0.1 mmHg with the help of a cathetometer. With this value of pressure and known volumes of bulbs containing nitrogen, the dead space and the temperature of the bulbs, the volume of gas taken is easily calculated at the STP conditions using gas equation. Now stopcock for sample bulb is opened and the gas is allowed to enter the sample tube. Since the sample is already at bath temperature, that is at liquid nitrogen temperature, nitrogen gets adsorbed by the sample.

4.4.5.3 By adjusting the mercury level in the left limb of the manometer to the reference point and noting the pressure at the equilibrium, the amount of remaining gas after adsorption can be calculated. The criteria for attainment of equilibrium shall be that the decrease in pressure shall be not more than 0.1 mmHg in 10 minutes. Thus, by substracting the remaining amount from the amount taken, the amount of gas adsorbed during the step is obtained. In this way, for the first point of isotherm, we have equilibrium pressure and the amount adsorbed corresponding to this pressure. Equilibrium pressure is divided by the saturation vapour pressure to get the relative pressure. Similarly, by adding more and more nitrogen to the system and repeating the sequence, a set of relative pressure values and the corresponding values of amount of gas adsorbed by the sample is obtained.

**4.4.5.4** A reference mark on the sample tube below the ground glass at the top of the bulb portion shall be made and the liquid nitrogen level shall be maintained constant at this mark during adsorption measurement.

**4.4.5.5** A plot of relative pressure  $(P_e/P_o)$  on X-axis and the amount adsorbed  $(\text{cm}^s/\text{g at STP})$  on Y-axis gives the isotherm for the adsorbent-adsorbate system (*see also* Fig. 4). For the determination of surface area, it is customary to have at least four points with equilibrium relative pressures  $(P_e/P_o)$  in the range 0.05 to 0.30.

4.4.5.6 When the adsorption run is completed, the sample is allowed to warm up to room temperature while desorbed gas is pumped off. Dry air is admitted and then the adsorption tube is weighed to determine the amount of sample which was used, care being taken to ensure that the joint and tube are free of grease.

4.4.5.7 Some typical examples of the weights of the samples used and volumes of nitrogen introduced into the system are given in Appendix C for guidance only. The measurement of saturation vapour pressure of nitrogen is explained in Appendix D. 4.4.6 Measurements - The following values should be measured:

- $H_1$  = initial pressure of helium in gas burette,
- $H_2$  = pressure of helium in gas burette with bulb at temperature  $T_B$ ,
  - T = water jacket temperature ( room temperature ),
- $T_B = \text{bath temperature},$
- $P_o$  = saturation vapour pressure of nitrogen at  $T_B$ ,
- $P_i$  = initial pressure of nitrogen in burette,
- $P_{e}$  = equilibrium pressure of nitrogen in burette and bulb,
- $P_{e-1}$  = equilibrium pressure of nitrogen in burette and bulb in the previous step to the step under consideration,
  - $V_b$  = volume of burette (determined) when the apparatus is being set up,
  - w = mass of sample (see Appendix E), and
  - $V_d = \text{dead space of the manifold determined by nitrogen using gas equation.}$

## 5. CALCULATIONS

5.1 Method of Calculation — Calculations shall be made to four decimal places. The final result should be suitably rounded off. The use of four significant figures must not be taken to imply accuracy of this order.

5.1.1 In the method, no correction is made for the non-ideality of nitrogen at low temperature. The correction for usual range of pressures required for the surface area determination is negligible.

5.1.2 When plotting 
$$\frac{P_e/P_o}{(1 - P_e/P_o) \sum V_a}$$
 against  $P_e/P_o$  it is advisable to

use large scale graph paper. At least four points should lie on the straight line, between the relative pressures ( $P_e/P_o$ ) of 0.05 to 0.30. If the points do not fall on a straight line, the data should be rejected and fresh determination carried out since drawing the best line through a number of points can lead to ambiguities. If a straight line is not obtained with any adsorbate, then the BET method is not applicable.

5.1.2.1 A coefficient of variation of about 2 percent is permissible in repeat determinations of the specific surface area of different samples of the same material.

## 5.2 Values Calculated

i) Volume admitted  $V_{ad} = V_b + V_d$ 

ii) Free space of the sample tube  $(V_{fs}) = \frac{T_B}{T} \left[ \frac{H_1 - H_2}{H_2} \right] \times V_{ad}$ iii) Calculated parameter  $K_1 = \frac{273 \cdot 2}{76} \times \frac{V_{ad}}{T}$ iv) Calculated parameter  $K_2 = \frac{273 \cdot 2}{76} \times \frac{V_{fs}}{T_B}$ v) Volume absorbed  $V_a = K_1 (P_i - P_e) - K_2 (P_e - P_{e-1})$ vi) Relative pressure  $= \frac{P_e}{P_o}$ vii) BET function  $= \frac{P_e/P_o}{(1 - P_e/P_o) \Sigma V_a}$ viii) Specific surface  $S_w = \frac{4 \cdot 35}{\text{slope + intercept}}$ 

5.3 Proforma for Calculation — A typical proforma of surface area computation for simplified routine calculations is shown in Appendix E.

## 6. PRECAUTIONS

**6.1** Greasing of taps (stopcocks) and connections should always be carried out with little grease.

6.2 When the adsorption apparatus has been lying unused for some time, all taps should be regreased.

**6.3** It is advisable to make a reference mark on the sample tube, near the ground glass joint and keep the liquid nitrogen level constant at this mark during the dead space and adsorption measurement.

**6.4** Two values for the saturation vapour pressure of nitrogen, one before and one at the end of the experiment, should be obtained. The average value should be used.

**6.5** The volume of the sample bulb should be such that it is only slightly larger than the sample volume to reduce the free-space.

**6.6** The diameter of the stem of the sample tube should be decided upon depending on the size of the particles of the sample being used. This will reduce the free space to the bare minimum.

## 7. REPORTING

7.1 In the report of a specific surface area determination by the method specified above, the following should be stated clearly:

- a) The outgassing conditions,
- b) The adsorption temperature,

- c) The range of linearity of the BET-plot,
- d) The value taken for the molecular cross-sectional area and the saturation vapour pressure ( $P_o$ ) of the adsorbate, and
  - The values obtained for the monolayer volume ( $V_m$ ) and for the constant C.

## APPENDIX A

## (Clause 3.1.5)

#### VALUES OF THE MOLECULAR CROSS-SECTIONAL AREA FOR VARIOUS ADSORBATES

A-1. The values of the molecular cross-sectional area ( $\sigma$ ) of various adsorbates for use with the BET method of specific surface area measurement are listed in Table 1. In addition to the customary value, a range of values is also quoted for each adsorbate. In certain cases, particularly where the point of inflection of the adsorption isotherm is not well defined, it may be necessary to use a figure other than the customary value in order to bring measurements made using different adsorbates in agreement.

Adsorbate	TEMPERATURE	MOLECULAR CROSS-SECTIONAL AREA ( σ )				
	OF ADSORPTION	Customary Value n <b>m</b> <sup>2</sup>	Quoted Values nm <sup>2</sup>			
(1)	(2)	(3)	(4)			
Nitrogen	—196	0.162	0.136-0.20			
Argon		0.137	0.137-0.182			
Krypton	—196	0.192	0.122-0.250			
Oxygen		0.141	0.130-0.120			
Benzene	+27	0.400	0.300-0.500			

#### TABLE 1 VALUES OF THE CROSS-SECTIONAL AREA FOR VARIOUS ADSORBATES

## APPENDIX B

## (Clause 4.4.3)

#### OUTGASSING

**B-1.** The sample is outgassed at elevated temperature and under high vacuum. The purpose is to remove previously adsorbed gases and vapours from the surface of the solid sample. Usually a vacuum of the order of  $10^{-5}$  mmHg is sufficient. This vacuum should remain there for some minutes after the connection to the pumping line and the cold trap has been shut off. The time required for outgassing ranges from 15 minutes to several hours depending upon the material. In order to minimize the time required for outgassing, the temperature chosen should be the maximum compatible with avoidance of sintering, decomposition or other alteration of the nature of the surface. The insertion of a cold trap in the main pumping line and the prior drying of the sample in an oven, maintained at 110°C facilitates the outgassing process. Practical experience of the worker helps a lot in deciding the appropriate conditions of outgassing for a given sample.

#### APPENDIX C

## (Clause 4.4.5.7)

#### **TYPICAL VOLUMES OF NITROGEN USED**

**C-1.** Some typical examples of the weights of sample used and volumes of nitrogen introduced into the system for powders of various specific surface areas are given in Table 2. These values are given for guidance only.

TABLE 2	WEIGHTS OF SAMPLE A NITROGEN INTROD	AND VOLUMES OF UCED
Expected Specific Surface Area	WEIGHT OF Sample	TOTAL VOLUME OF NITROGEN INTRO- DUCED AT STP
(1)	(2)	(3)
$m^2/g$	g	$cm^3$
2	10	25
5	5	30
10	5	30
50	2	40
100	1	40
200	0.2	40
500	0.5	<b>4</b> 0
1 000	0-1	40

#### APPENDIX D

## ( Clause 4.4.5.7 )

#### MEASUREMENT OF SATURATION VAPOUR PRESSURE OF NITROGEN

**D-1.** In order to calculate the surface area, it is necessary to know the saturation vapour pressure of nitrogen at the temperature of the experiment. The saturation vapour pressure is measured most conveniently with the aid of a vapour pressure probe in the liquid nitrogen bath; this consists merely of a hollow tube containing pure condensed nitrogen and connected to a suitable manometer. A typical sketch of the vapour pressure probe and the manometer is shown in Fig. 5. This can either be used separately or attached to the apparatus assembly shown in Fig. 2. Values of the saturation pressure of nitrogen at various temperatures are shown in Fig. 6 (see also 6.4).



FIG. 5 TYPICAL SATURATION VAPOUR PRESSURE MEASUREMENT APPARATUS



FIG, 6 VARIATION OF SATURATION VAPOUR PRESSURE WITH TEMPERATURE FOR NITROGEN

## APPENDIX E

(Clauses 4.4.6 and 5.3)

### A TYPICAL PROFORMA OF SURFACE AREA COMPUTATION FOR SIMPLIFIED ROUTINE CALCULATION

Sample: Gasil Silica	
Mass of bulb:	9·808 8 g
Mass of bulb + sample:	10 <sup>.</sup> 121 3 g
Mass of sample ( w ):	0·312 5 g
Standard Reference Material (SRM):	8A/30/19
Room temperature (T):	305 K
Bath temperature ( $T_B$ ):	77·76 K
7	

$$V_{fs} = \frac{{}^{2}B}{T} \left[ \frac{H_1 - H_2}{H_2} \right] V_{ad} = 5.271, \text{ where } V_{ad} \text{ for Helium is 11.33.}$$

Date:	1977-07-26
Outgassing time:	6 hours
Outgassing temperature:	1 <b>4</b> 0°C
Outgassing pressure;	$2 \times 10^{-4} \text{ mmHg}$
Saturation vapour pressure Po:	79·7 cmHg
$H_1 = 14.248 \text{ cmHg}$	
$H_{s} = 5.044 \text{ cmHg}$	

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$$V_{ad}$$
 for Nitrogen = 32.27

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$$K_{1} = \frac{273 \cdot 2}{76} \times \frac{V_{ad}}{T} = 0.380 \ 3$$

$$K_{2} = \frac{273 \cdot 2}{76} \times \frac{V_{fs}}{T_{B}} = 0.243 \ 6$$

$$V_{a} = K_{1} ( \text{ col } 4 ) - K_{2} ( \text{ col } 5 )$$

$$S_{w} = \frac{4 \cdot 35}{\text{slope + intercept}} = 260 \ \text{m}^{2}/\text{g}$$

SI No.	$P_i$ (Initial)	P <sub>e</sub> ( Equili- brium )	$(P_i - P_e)$	$(P_e - P_{e_{-1}})$	Va for Sample	V <sub>a</sub> per Unit Mass	$\Sigma V_a$	$(Pe P_0)$	$\frac{Pe/Po}{(1 - Pe/Po) \Sigma Va}$
	cmHg	cmHg	cmHg	cmHg	$cm^3$	cm³/g	cm³/g		
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
1.	15.644	0.134	15.510	0.134	5•865	1 <b>8</b> ·768	18.768	0.002	$0.107 \times 10^{-3}$
2.	26.148	1.771	<b>24·3</b> 77	1.637	8.871	28.387	<b>4</b> 7 <b>·</b> 155	0.022	$0.477 \times 10^{-3}$
3.	22.523	7.574	<b>14·94</b> 9	5.803	<b>4</b> ·272	13.670	60 <b>·825</b>	0.092	$1.726 \times 10^{-3}$
4.	<b>20·</b> 65 <b>9</b>	11.962	8•697	4.338	2.338	7.162	6 <b>7·98</b> 7	0.120	2.596 × 10-*
5.	21-250	15.478	5•772	<b>3</b> ·516	1 <b>·3</b> 39	4.285	72.272	0.194	$3.330 \times 10^{-3}$
6.	<b>2</b> 2·092	18.148	3.944	2.670	0.849	2.717	74·989	0.228	$3.938 \times 10^{-3}$
7.	<b>24</b> · <b>4</b> 08	20.439	<b>3</b> ·696	2.291	0.951	3.043	78 <b>·03</b> 2	0.256	$4.409 \times 10^{-3}$
8.	26.456	23.011	3·445	2.572	0.648	2.189	80 <sup>.</sup> 221	0.289	$5.067 \times 10^{-3}$
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