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IS 2752 (1995): Activated carbons, granular [CHD 1: Inorganic Chemicals]



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IS 2752 : 1995  
(Reaffirmed 2010)

भारतीय मानक

सक्रियित कार्बन, दानेदार — विशिष्ट

( तीसरा पुनरीक्षण )

*Indian Standard*

ACTIVATED CARBONS, GRANULAR —  
SPECIFICATION

( *Third Revision* )

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(First Reprint JANUARY 2001)

UDC 661.183.2-408.8

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**BUREAU OF INDIAN STANDARDS**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

December 1995

Price Group 4

**AMENDMENT NO. 1 OCTOBER 2004**  
**TO**  
**IS 2752 : 1995 ACTIVATED CARBON, GRANULAR —**  
**SPECIFICATION**  
*( Third Revision )*

( *Page 2, clause A-2.1, second sentence* ) — Substitute the following for the existing:

'Grind enough quantity to pass through 75 micron IS sieve (about 1 g), dry in a preheated forced circulation oven at 145 to 155°C to constant weight, cool in a desiccator to ambient temperature and weigh very accurately 0.2 g of powdered carbon and introduce it into iodine flask.'

(CHD 1)

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Reprography Unit, BIS, New Delhi, India

## FOREWORD

This Indian Standard (Third Revision ) was adopted by the Bureau of Indian Standards, after the draft finalized by the General Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

Granular activated carbons are used for absorption of obnoxious gases in industry, water purification, solvent recovery, respirators, cigarette filters and as catalyst carrier. While the material may be prepared from several sources, it has been found that the material prepared from coconut shell is most effective. Powdered activated carbons are covered under IS 8366:1989 'Activated carbons, powdered — Specification (*second revision*)'.

This standard was originally published in 1963 and then revised in 1978. The second revision of this standard was brought out in 1989 incorporating a new requirement for retentivity index for Type 1 material. The requirement for adsorption capacity for benzene was substituted by adsorption capacity for carbon tetrachloride and the requirement for number of tests and criteria for conformity were incorporated during the second revision of this standard.

In this revision, new requirements for adsorption capacity in terms of iodine number, half dechlorination value and surface area along with the relevant test methods have been incorporated. A requirement for decolourizing power which was stipulated for Type 2 (*see 3.1*) of the material has been deleted.

The composition of the committee responsible for formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding of numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

**Indian Standard**  
**ACTIVATED CARBONS, GRANULAR —**  
**SPECIFICATION**  
**( Third Revision )**

**1 SCOPE**

**1.1** This standard prescribes requirements and methods of sampling and test for granular activated carbons.

**2 REFERENCES**

**2.1** The Indian Standards listed below are necessary adjuncts to this standard:

<i>IS No.</i>	<i>Title</i>
877 : 1989	Activated carbons, powdered and granular — Methods of sampling and test ( <i>second revision</i> )
1070 : 1992	Reagent grade water ( <i>third revision</i> )
1260 (Part 1) : 1973	Pictorial markings for handling and labelling of goods : Part 1 Dangerous goods ( <i>first revision</i> )
2552 : 1979	Specification for steel drums (galvanized and ungalvanized) ( <i>second revision</i> )

**3 TYPES**

**3.1** The material shall be of the following two types depending upon the end use:

- a) Type 1 — For use as a base for respirator carbons and solvent recovery, and
- b) Type 2 — For use in water treatment.

**4 REQUIREMENTS****4.1 Description**

The material shall be in the form of fine black granules, free from foreign matter.

**4.2 Particle Size**

Particle size of the material shall be as agreed to between the purchaser and the supplier.

**4.3** The material shall comply with the requirements given in Table 1 when tested according to the methods prescribed in IS 877 : 1989. Reference to the relevant test method is given in col 5 and 6 of the Table.

**Table 1 Requirements for Granular Activated Carbons**

Sl No.	Characteristic	Requirement for		Method of Test, Ref to	
		Type 1	Type 2	Cl No. in IS 877 : 1989	Annex A
(1)	(2)	(3)	(4)	(5)	(6)
i)	Adsorption capacity for carbon tetrachloride, percent by mass, <i>Min</i>	55	—	14	—
ii)	Moisture, percent by mass, <i>Max</i>	5	5	4	—
iii)	Ash, percent by mass, <i>Max</i>	5	0	5	—
iv)	Hardness number, <i>Min</i>	90	90	13	—
v)	Retentivity index, percent by mass, <i>Min</i>	45	—	15	—
vi)	Adsorption capacity in terms of iodine number, <i>Min</i>	900	450	—	A-2
vii)	Half dechlorination value, cm, <i>Max</i>	4	7	—	A-3
viii)	Surface area, m <sup>2</sup> /g, <i>Min</i>	900	550	—	A-4

**5 PACKING AND MARKING****5.1 Packing**

The material shall be packed as agreed to between the purchaser and the supplier.

**5.2 Marking**

Each drum shall bear legibly and indelibly the following information:

- Name and type of the material,
- Indication of the source of manufacture,
- Gross and net mass,
- Batch number,
- Date of manufacture, and
- Symbol indicating the fire hazards  
[ see IS 1260 ( Part 1 ) : 1973 ].

**5.2.1** The product may also be marked with Standard Mark.

**5.2.1.1** The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards

Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

**5.3 Storage**

This material, being potentially flammable, should be stored in buildings or compartments which are as nearly fireproof as possible. Other oxidizing or flammable materials should not be stored in the same building.

**6 SAMPLING**

Representative samples of the material shall be drawn and adjudged as prescribed in 19 of IS 877 : 1989.

**7 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY**

**7.1** All the tests are critical for individual samples and also for composite samples.

**ANNEX A**

(Table 1)

**METHOD OF TEST FOR ACTIVATED CARBONS,  
GRANULAR****A-1 QUALITY OF REAGENTS**

Unless specified otherwise, pure chemicals and distilled water ( see IS 1070 : 1992 ) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

**A-2 DETERMINATION OF IODINE  
ADSORPTION VALUE OF ACTIVATED  
CARBON (GRANULAR)****A-2.1 Procedure**

Take about 5 g of granular carbon sample. Grind enough quantity to pass through 75 micron IS sieves ( about 1 g ), weigh very accurately 0.2 g of powdered carbon and introduce it into iodine flask. Introduce 40 ml of 0.1 N iodine solution. Shake the contents for exactly four minutes. Filter through Whatman filter paper No. 1. Take 10 ml of the filtrate and titrate against standard sodium thiosulphate solution.

**A-2.2 Calculations**

Solutions required

- 0.1 N iodine solution,
- 0.05 N sodium thiosulphate solution,
- 0.1 N (exact) potassium iodate solution.

i) Standardization of 0.05 N  $\text{Na}_2\text{S}_2\text{O}_3$  against 0.1 N  $\text{KIO}_3$  using starch indicator.

ii) Standardization of  $\text{I}_2$  Solution against standardized  $\text{Na}_2\text{S}_2\text{O}_3$  using Starch solution as indicator.

Suppose, the Normality is 0.103 of Iodine.

iii) Calculate the quantity of  $\text{I}_2$  in 40 cc of 0.103 Normality. 1 N in 1 000 cc contains 127 g of Iodine.

Therefore, 0.103 N in 40 cc contains

$$\frac{127 \times 0.103 \times 40}{1000} = 0.523 \text{ g}$$

Calculate the normality of the filtrate ( after adsorption ) and calculate the quantity of Iodine in 40 cc after adsorption. Suppose the reading of thio is 11.4 cc ( Normality of Thio is 0.5 N )

$$10 \times X = 11.4 \times 0.05$$

$X = \frac{11.4 \times 0.05}{10}$  0.057 N of  $\text{I}_2$  after adsorption

Therefore, 1 N in 1 000 cc 127 g of Iodine

$$0.05 \text{ N in } 40 \text{ cc } \frac{127 \times 0.057 \times 40}{1000}$$



Quantity of Iodine (after adsorption) = 0.289 56  
 Quantity of Original Iodine is = 0.523  
 Quantity of Original Iodine (after adsorption) = 0.289 56  
 Therefore, quantity of iodine adsorbed = 0.233 g  
 = 233 mg  
 Therefore  $233 \times 5 = 1\ 165$  mg/g

### A-3 DETERMINATION OF HALF DECHLORINATION

#### A-3.1 Procedure

Prepare bleaching powder solution in water having 10 ppm available chlorine in water. Prepare a carbon column of 10 cm carbon bed depth.

Pass 10 ppm available chlorine water at a rate of 20 M/hr for 30 minutes. Check up initial concentration after 30 minutes passing through carbon bed.

#### A-3.2 Calculation

Calculate dechlorination half value as follows:

$$G_1 = \frac{0.301 \times h}{10 \text{ ga/b}}$$

### A-4 DETERMINATION OF SURFACE AREA

#### A-4.1 General

The surface area is measured by the surface area of the solids over a wide range by non-destructive method. This information is very useful in R & D, process and quality control of Catalysts & Absorbents, Ceramics and Refractories, Pigments, Adhesives, Food Stuff, Plastics, Metal Powders, etc.

The operation of the instrument is very simple. The analysis time required for sample analysis is 10-15 min. which makes it very suitable for Q. C & process control.

#### Specification:

1. Analysis time : 10-15 min/sample
2. Range :  $0.2\text{m}^2/\text{gm}-1\ 000\text{m}^2/\text{gm}$
3. Accuracy :  $\pm 5$  percent
4. Reproducibility :  $\pm 3$  percent
5. Power : 50Hz, 230V,  $\pm 10$  percent

#### Installation:

The unit is designed for 230 V operation/50 Hz with less consumption of power. A table space of 4 feet  $\times$  3 feet is sufficient for installation.

Following items have to be procured before installation of Smartsorb - 90

1. Helium-Nitrogen gas mixture approximately in ratio of 25 percent. It is necessary to know the exact ratio of the same.
2. Liquid Nitrogen with proper cryogenic container.
3. Gas Syringe up to 5 ml.
4. Precision Balance (0.01 gm sensitivity).
5. Nitrogen Cylinder (Iolar grade).

#### Theory of Operation:

The instrument works on the principle developed by Breunauer-Emmett-Teller ( Short form BET ) using Nitrogen adsorbed at its boiling point. Though the basic method is accurate, it takes longer time and needs special fabrication, high vacuum, mercury, etc.

Dynamic single point method was developed first by Nelson & Eggertson using Thermal Conductivity Detector (TCD) and a flow system ( $\text{N}_2$  & He mixture). It has the accuracy of the same order. (The use of Krypton for the measurement of surface area in the lower range is avoided taking advantage of high sensitivity of TCD). They have used following BET equations.

$$\frac{P/P_o}{V_s (1 - P)} = \frac{1}{V_m C} + \frac{C - 1}{V_m C} \times P/P_o \quad \dots (1)$$

$V_1$  = volume adsorbed at  $P/P_o$  under NTP conditions

$V_m$  = monolayer volume

$C = 5$  constant

$P/P_o$  = partial pressure

This is a straight line equation with slope =  $\frac{C - 1}{V_m C}$

and intercept =  $\frac{1}{V_m C}$

$$\frac{P/P_o}{V_s (1 - P)} = \frac{1}{V_m C} + \frac{C - 1}{V_m C} \times P/P_o \quad \dots (1)$$

Generally intercept is very small (due to the large value of  $C$ ) and, hence,  $\frac{1}{V_m C}$  can be neglected. then,  $V_m$  can be calculated as

$$V_m = V_s (1 - P/P_o) \quad \dots (2)$$

Considering Avogadro number and cross-sectional area of  $\text{N}_2$  molecule, surface area ( $S. A$ ) expressed in  $\text{m}^2/\text{gm}$  can be calculated by following equation.

$$S.A = 4.38 V_m \quad \dots (3)$$

#### Schematic of Operation-Set

The schematic of the operational set-up is shown in Fig. 1. The calibrated gas mixture is first passed through the Rotameter to control the flow of gas.

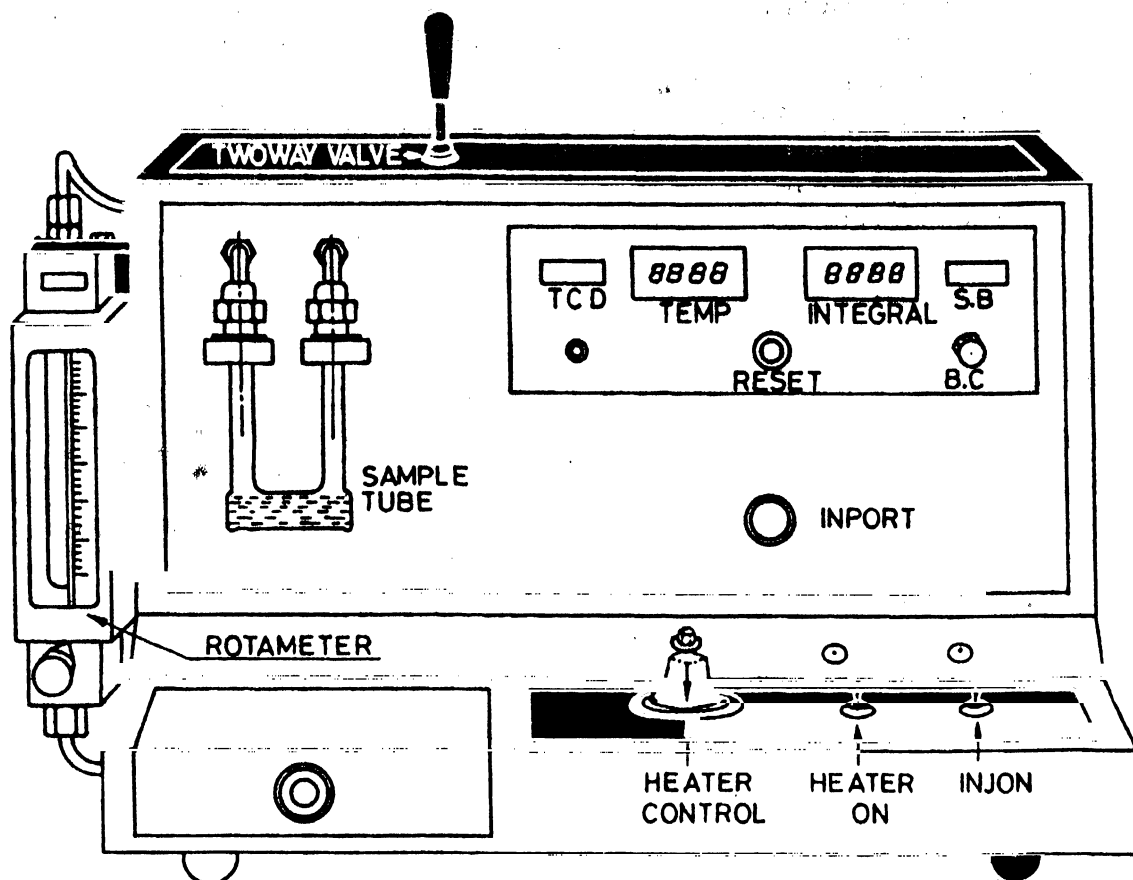


FIG. 1 SURFACE AREA TESTING EQUIPMENT

Normally it is controlled at 60 cc/min. Then it passes over the reference side of the TCD, the sample holder or bypass through a two way valve. An injection port is provided for calibration before two way valve. A known quantity of gas injected is passed over sample side and ultimately comes out through a tube where one can regenerate another sample.

#### Integrator.

It consists of regulated power required for TCD block and integrator.

Integrator is summation of the signal over a given period, which gives an area under the curve. The display in the integrator is an arbitrary number which can be calibrated by injecting known amount of nitrogen using gas tight syringe through injection port.

#### Experimental Procedure:

1. Regenerate the sample in inert atmosphere for two or three hours at appropriate temperature.
2. Connect the gas mixture (He 75 percent/N<sub>2</sub> 25 percent) to gas inlet. The pressure of the mixture should be around 5-7 psi at the outlet indicator of the cylinder. Adjust the gas flow at 60-70 cc/min using middle valve of rotameter. A gas should purge through the system for 30 minutes to drive out air inside. The two-way valve position should be on by-pass during purging.
3. Switch on the integrator unit and keep it on for 5-10 minutes stabilization. Then balance the TCD bridge with balance control potentiometer to zero.
4. Connect the sample tube with preweighed sample. Switch the valve to sample side. Allow 5 more minutes to pass. During this period the balance indicator will move to the right side indicating that the air in the sample tube is flushed off. When the indicator comes back to zero position the set-up is ready for the reading. Reset the counter.
5. Insert Dewar Flask under sample Tube slowly. At liquid nitrogen temperature nitrogen from gas mixture will start adsorbing on the sample. This phenomenon will be indicated by pointer on the right hand side of the instrument. It will return back to zero

position once adsorption is complete. Then remove the Dewar Flask slowly. The nitrogen adsorbed will start desorbing at room temperature which will be indicated on LED display as number of counts.

- Inject the known amount of nitrogen in the injection port and find out number of counts. Repeat this procedure to get counts very close to your readings of sample.

#### Calculations:

- Volume of gas absorbed by sample at STP,

$$V_s = \frac{n_s}{n_c} V_1$$

where

$n_s$  = number of counts for the sample  
(Desorbition count)

$n_c$  = number of counts for injected  
known volume of nitrogen gas  
(N<sub>2</sub> Count)

$V_1$  = volume of nitrogen gas injected.

To convert  $V_s$  to normal conditions,

$$\frac{V_s \times 273}{(273 + t)} = V'_s$$

- Calculation of monolayer volume ( $V_m$ ) for single point surface area is given by the following formula:

$$V_m = V'_s(1 - P/p_0)$$

$P/p_0$  is the fraction of Nitrogen in calibrated Nitrogen/Helium Cylinder (Valve) is given with the cylinder.

- Specific Surface Area ( $S_p$ ) of the given powder,

$$S_p = \frac{4.38 \times V_m}{M}$$

where

$M$  = mass in g of the regenerated material taken for the test.

NOTE — Following precautions should be taken while operating the apparatus:

- Do not switch on the unit and integrator before starting the gas flow. Check the cylinder pressure regularly.
- Do not allow air to pass over TCD for long time. This will oxidize the element and shorten the life of TCD.
- Switch off the integrator supply before stopping the flow of gas.
- One can use Hydrogen/Nitrogen gas mixture instead of Helium/Nitrogen. However, if your sample contains any element which will adsorb Hydrogen (for example Platinum), then it is recommended to use Helium/Nitrogen mixture only. All necessary precautions should be taken while using Hydrogen/Nitrogen gas mixture. The procedure for calculation remains the same.

**ANNEX B**  
*(Foreword)*

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This Indian Standard has been developed from Doc: No. CHD 003 ( 0987 ).

### Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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