भारतीय मानक मसौदा

सक्रियित कार्बन, दानेदार — विशिष्टि

(*IS 2752 का चौथा पुनरीक्षण*)

Draft Indian Standard

Activated Carbons, Granular — Specification

(*Fourth Revision of IS 2752*)

ICS 71.080.01

Inorganic Chemicals Sectional Committee, CHD 01	**Last date of comments: 9th December 2024**

Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

(formal clauses will be added later)

This standard was first published in 1963 and subsequently revised in 1978, 1989 and 1995. The second revision of this standard was brought out to incorporate 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. In the third revision, new requirements for adsorption capacity in terms of iodine number, half dechlorination value and surface area along with the relevant test methods were incorporated. A requirement for decolorizing power which was stipulated for Type 2 of the material was deleted.

In this revision, Amendment No. 1 has been incorporated. Also, Reference clause, Packing & Marking clause have been updated. The standard has been updated based on the technological advancements that may have taken place since the last publication of the Standard.

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 'Activated carbons, powdered — Specification'.

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: 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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Draft Indian Standard

ACTIVATED CARBONS, GRANULAR — SPECIFICATION

(*Fourth Revision*)

1 SCOPE

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

2 REFERENCES

The standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revisions, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
IS 877 : 2024	Activated carbons powdered and granular methods of sampling and test (*third revision*)
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision)*
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods
IS 2552 : 1989	Steel drums (galvanized and ungalvanized) — Specification (*third revision*)

3 TYPES

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. Reference to the relevant test method is given in col **5** and **6** of the Table.

Table 1 Requirements for Granular Activated Carbons

SI No	**Characteristic**	**Requirement for**		**Method of Test, Ref**	
		Type 1	**Type 2**	**Cl No in IS 877**	**Annex A**
(1)	(2)	(3)	(4)	(5)	(6)
i)	Adsorption capacity for carbon tetrachloride, percent by mass, *Min*	55	—	14	—
ii)	Moisture, percent by mass, *Max*	5	5	4	—
iii)	Ash, percent by mass, *Max*	5	0	5	—

iv)	Hardness number, *Min*	90	90	13	—
v)	Retentivity index, percent by mass, *Min*	45	—	15	—
vi)	Adsorption capacity in terms of iodine number, *Min*	900	450	—	**A-2**
vii)	Half dechlorination value, cm, *Max*	4	7	—	**A-3**
viii)	Surface area, m^2 /g, *Min*	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;

a) Name and type of the material;
b) Indication of the source of manufacture;
c) Gross and net mass;
d) Batch number;
e) Date of manufacture; and
f) Symbol indicating the fire hazards [s*ee* IS 1260 (Part 1)].

5.2.1 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

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 IS 877.

7 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

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) 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 sieve (about 1 g), dry in a preheated forced circulation oven at 145 °C 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.

A-2.2 Calculations

a) 0.1 N iodine solution
b) 0.05 N sodium thiosulphate solution,
c) 0.1 N (exact) potassium iodate solution.
   i) Standardization of 0.05 N $Na_2S_2O_3$ against 0.1 N KIO_3 using starch indicator.
   ii) Standardization of I_2 Solution against standardized $Na_2S_2O_3$ using Starch solution as indicator. Suppose, the Normality is 0.103 of Iodine
   iii) Calculate the quantity of 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

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

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)

$$\mathbf{10 \times \mathit{X} = 11.4 \times 0.05}$$

$$\boldsymbol{X} = \frac{\mathbf{11.4 \times 0.05}}{\mathbf{10}} = \mathbf{0.057\ N}\text{ of } I_2 \text{ after adsorption}$$

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

$$\mathbf{0.05\ N\ in\ 40\ cc}\ \frac{\mathbf{127 \times 0.057 \times 40}}{\mathbf{1\ 000}}$$

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 × 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/h for 30 min. Checkup initial concentration after 30 min passing through carbon bed.

A-3.2 Calculation

Calculate dechlorination half value as follows:

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

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 min to 15 min. which makes it very suitable for Q.C & process control

Specification:

1. Analysis time : 10 to 15 min/sample
2. Range : 0.2 m^2 /gm to 1 000 m^2 /gm
3. Accuracy : ± 5 percent
4. Reproducibility : ± 3 percent
5. Power : 50 Hz, 230 V, ± 10 percent

Installation

The unit is designed for 230 V operation/50 Hz with less consumption of power. A table space of 4 feet × 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 (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.

$$\frac{p/p_o}{v_s\,(1-p)} = \frac{1}{v_m\,C} + \frac{C-1}{V_m C} \times P/P_O \quad \text{.....(1)}$$

V_1 = volume adsorbed at P/Po 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 = $\frac{1}{V_m\,C}$

$$\frac{p/p_o}{V_s\,(1-p)} = \frac{1}{V_m\,C} + \frac{C-1}{V_m C} \times P/P\mathbf{o}\text{…….(1)}$$

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

$$V_m = V\text{s}\ (1 - P/P_0) \qquad \text{……..(2)}$$

Considering Avogadro number and cross-sectional area of N_2 molecule, surface area (*S. A*) expressed in m2 /gm can be calculated by following equation.

$$S.A = 4.38\ Vm \qquad \text{…….. (3)}$$

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:

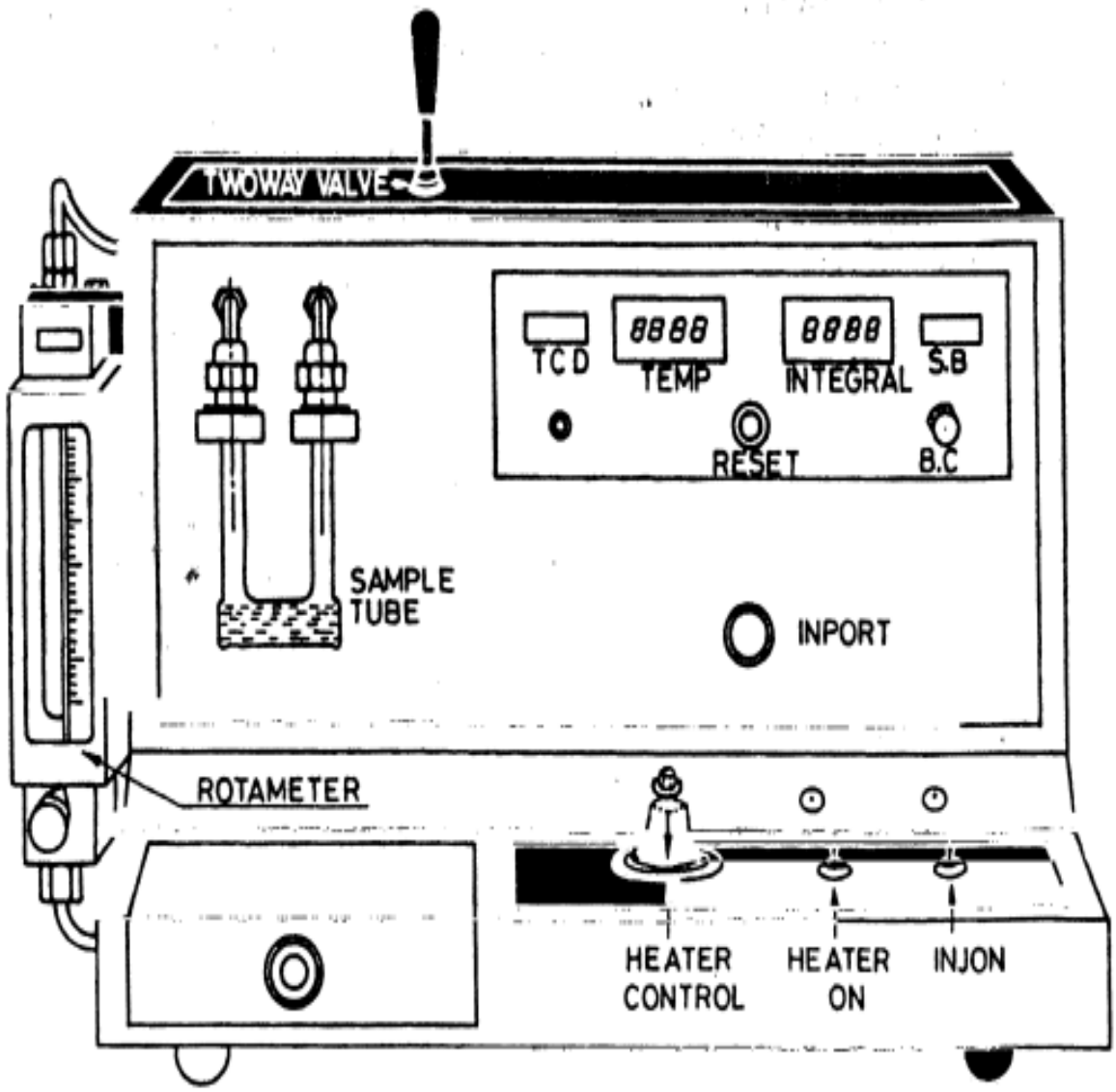

FIG. 1 SURFACE AREA TESTING EQIUPMENT

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_2 25 percent) to gas inlet. The pressure of the mixture should be around 5 to 7 psi at the outlet indicator of the cylinder. Adjust the gas flow at 60 to 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 min to 10 min 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 setup 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.
6. 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

1. Volume of gas absorbed by sample at STP,

$$V_S - \frac{ns}{nc} V_1$$

where,

n_s = number of counts for the sample (Desorption count)
n_c = number of counts for injected known volume of nitrogen gas (N_2 Count)
V_1 = volume of nitrogen gas injected

To convert Vs to normal conditions

$$\frac{V_S \times 273}{(273 + t)} = V'_S$$

2. Calculation of monolayer volume (Vm) for single point surface area is given by the following formula:

$$V_m = V_S' (1 - P/p_o)$$

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

3. Specific Surface Area (Sp) 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

a) Do not switch on the unit and integrator before starting the gas flow. Check the cylinder pressure regularly
b) Do not allow air to pass over TCD for long time. This will oxidize the element and shorten the life of TCD.
c) Switch off the integrator supply before stopping the flow of gas
d) 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.