भारतीय मानक मसौदा

सक्रिय कार्बन, चूर्णित — विशिष्टि

(*IS 8366 का दूसरा पुनरीक्षण*)

Draft Indian Standard
Activated Carbons, Powdered —Specification
(*Second Revision of IS 8366*)

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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 1977 and subsequently revised in 1989. In the first revision, the requirements of moisture, ash and matter soluble in water, for Type 1, Type 2 and Type 3 material were modified. Also, *p*H of Type 3 and decolorizing power content of Type 2 and Type 3 material were modified. In addition, three new requirements namely, chloride, sulphate and cyanogen compounds were incorporated for Type 3 material.

In this revision, instrumental test methods for the determination of chloride, sulphate and iron have been added as alternate test methods. In addition to this, editorial corrections have been made wherever required. Also, Reference clause and Packing & Marking clause have been updated.

There are two types of activated carbons, namely, powdered and granular. Powdered activated carbons are used for decolorizing vegetable oils, sugar solutions, pharmaceuticals, etc. Granular activated carbons are used for absorption of obnoxious gases in industry, water purification, solvent recovery, in respirators and cigarette filters and as catalyst carrier. The specification for granular carbons is covered under a separate Indian Standard namely IS 2752 'Activated Carbons, Granular'.

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

Draft Indian Standard
ACTIVATED CARBONS, POWDERED — SPECIFICATION
(*Second Revision*)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for powdered 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 (*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*)
IS 3025	Methods of sampling and test (physical and chemical) for water and wastewater
(Part 2) : 2019/ ISO 11885 : 2007	Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP – OES) (*first revision*)
(Part 65) : 2022/ ISO 17294-2 : 2016	Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (*first revision*)

3 TYPES

There shall be three types of the material depending upon the end use:

a) *Type 1* — for decolorizing vegetable oils, fats and waxes;
b) *Type 2* — for decolorizing sugar solution, corn sugar solution; and
c) *Type 3* — for decolorizing pharmaceuticals.

4 REQUIREMENTS

4.1 Description

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

4.2 The material shall conform to the requirements given in Table 1, when tested by the methods prescribed in IS 877 Reference to the relevant clauses of IS 877 is given in Col **6** of the table.

4.3 Additional Requirements

Additional requirements for specific uses, if necessary, may be prescribed as agreed to between the purchaser and the supplier.

5 PACKING, MARKING AND STORAGE

5.1 Packing

Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in clean, sound, hermetically sealed steel drum (*see* IS 2552) or HDPE or hessian bag with polyethylene liner.

5.2 Marking

The packages shall be legibly and indelibly marked with the following information:

   a)   Name and type of the material;
   b)   Net mass of the contents;
   c)   Month and year of packing;
   d)   Manufacturer's name and/or his recognized trade-mark, if any;
   e)   Lot number to enable tile batch of manufacture to be traced from records; and
   f)   Appropriate symbol indicating the fire hazards [*see* IS 1260 (Part 1)].

5.2.1 *BIS Certification Marking*

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

5.3 Storage

The material being potentially flammable, shall be stored in building or compartments which are as nearly fire proof as possible. Other oxidizing or flammable materials shall not be stored in the same building.

6 SAMPLING

Representative samples of the material shall be draw and adjudged as prescribed in IS 877.

7 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

Characteristics which are critical for various types are as follows:

Type	Characteristics (*see* Table 1)
Type 1	– vi, vii, viii
Type 2	– vi.
Type 3	– v, vi, x, xi, xii and xiii

Table 1 Requirement for Activated Carbons, Powdered

(*Clauses* 4.2 and 7)

SI No	Characteristic	Requirement for			Method of Test (Ref.to Cl No. of IS 877)
		Type 1	Type 2	Type 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Moisture, percent by mass, *Max*	15	15	15	**4**
ii)	Ash, percent by mass, *Max*	60	40	20	**5**
iii)	Matter soluble in water, percent by mass, *Max*	—	1.5	0.5	**7**
iv)	Matter soluble in acid, percent by mass, *Max*	6	6	2.5	**8**
v)	*p*H	5 to 8	5 to 8	6.5 to 7.5	**9**
vi)	Decolorizing power, mg/g, *Min*	50	70	205	**11**
vii)	Oil retention, percent by mass, *Max*	30	—	—	**12**
viii)	Filterability, minutes, *Max*	46	—	—	**12**
ix)	Particle size:				

	a)	passing through 150-micron IS Sieve, percent by mass, *Min*	100	100	95	
	b)	passing through 125-micron IS Sieve, percent by mass, *Min*	95	95	95	**6**
	c)	passing through 75-micron IS Sieve, percent by mass, *Min*	80	80	80	
x)	Iron (as Fe), percent by mass, *Max*		—	—	0.05	**10 or A-2 of IS 8366**
xi)	Chloride (as Cl), percent by mass, *Max*		—	—	0.35	**16 or A-1 of IS 8366**
xii)	Sulphate (as SO$_4$), percent by mass, *Max*		—	—	0.8	**17 or A-1 of IS 8366**
xiii)	cyanogen compounds		—	—	To pass the test	**18**

*This requirement by methylene blue test is for guidance only. For specific uses for decolonization of vegetable oils, fats and waxes, sugar solutions and pharmaceuticals, specific requirements as agreed to between the purchaser and the supplier may be prescribed.

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ANNEX A

(*Table* 1, *Item* x, xi, xii)

METHODS OF TEST FOR CHLORIDES, SULPHATES AND IRON

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A-1 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES

A-1.1 Principle

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides and sulphates. The technique separates ions and polar molecules based on their affinity to ion exchanger. When the method is employed for the determination of the anions as chlorides and sulphates, the identification should be made by using a matrix covering the ions of interest. In cation exchange chromatography, the stationary phase is functionalized with anions. These anions will attach cations towards it. These surface bound molecules/ionic species can then be removed by using a suitable eluent containing substituted ions to replace them or they can be removed by changing the pH of the column. Similarly, in anion exchange chromatography, the stationary phase is cationic in nature. These cations will then separate the anions.

Conductivity detector is generally used in this method. In case of suppressor ion exchange chromatography, analyte ions are separated on the ion exchange column and these ions together with the eluent move to the matrix suppressor. The eluent conductivity is lowered in the suppressor and the sample ion conductivity is increased leading to the large increase in signal to noise ratio.

A-1.2 Equipment

A-1.2.1 *Anion Guard Column* — A protector of the separator column.

A-1.2.2 *Anion Separator Column* — Suitable for selective separation of ions under analysis.

A-1.2.3 *Anion Suppressor Device* — Anion micro membrane suppressor is used to analyse the data.

A-1.2.4 *Detector* — Conductivity Detector.

A-1.2.5 *Software* — Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

A-1.2.6 Sample loop of 100 µl, 200 µl, 500 µl or 1 000 µl be used to determine ionic concentration as per instrument manual and practice.

A-1.3 Reagents

A-1.3.1 *Glass or Polyethylene Sample Bottles.*

A-1.3.2 *Distilled Water or Deionized Water free from the Anions of interest.*

A-1.3.3 *Eluent*

1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used. For preparation of these solution, 0.285 6 g of sodium bicarbonate and 0.381 6 g of sodium carbonate is dissolved in 2 litres of water.

A-1.3.4 *Micro Membrane Suppressor Solution*: (0.025 N *of sulphuric acid*) — Dilute 2.8 ml of concentrated sulphuric acid in 4 litres of water.

A-1.4 Standard Solutions

A-1.4.1 *Chloride*

Dissolve NaCl, 1.648 5 g in 1 000 ml of reagent water.

A-1.4.2 *Sulphate*

Dissolve 1.81 g of potassium sulphate in 1 000 ml of reagent water.

A-1.5 Calibration and Standardization

For each analyte of interest, prepare calibration standards at three concentration levels and a blank by adding measured stock standards and diluting with reagent water. If the concentration of the sample exceeds the calibration range, the sample may be diluted. Using 0.1 ml to 1.0 ml injections of each calibration standard, tabulate area responses or peak height against the concentration. Use these results to prepare calibration curve. Record the retention time during the procedure.

A-1.6 Procedure

Dissolve between 1g to 5 g sample in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1 ml to 1.0 ml) and flush it through an injection loop using each new sample. Use the loop of same size for the standards and samples. Record the peak in size and area units. An automated constant volume injection system may preferably be used. The width of peak for retention time of ions should be same for sample and standard. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an appropriate amount of standard and reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For solid sample of cobalt acetate, following extraction procedure may be used.

Add reagent water in an amount equal to 10 times the dry weight of the sample. The slurry is made and stirred for about 10 min using magnetic stirrer. This slurry is filtered from 0.45 µ membrane. This membrane can be directly attached to the end of the syringe. For clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

A-1.7 Data analysis and Calculations

Prepare a calibration curve for each analyte by plotting instrument response against concentration. Compare the sample response with the standard curve and compute sample concentration. Multiply the value by appropriate dilution factor.

Report results in mg/l or by suitably modifying into percentage. Only report those values that fall within the range of lowest and highest calibration standards.

A-2 DETERMINATION OF IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-2.1 Principle

The sample solution under analysis is nebulized through a nebulizer inside a spray chamber. The aerosol formed is aspirated to argon plasma torch [produced by a radio-frequency inductively coupled plasma (ICP)], where the molecules break into constituent atoms and/or molecular species and atoms are get excited. These excited atoms then return back to the lower energy state by emitting radiation of specific wavelength. These emitted radiations are characteristic of an element and are measured by the Photomultiplier tube detector and intensity of such emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

A-2.2 Recommended Wavelength, limit of quantification and important spectral interferences

Elements along with the recommended wavelengths and typical estimated limits of quantification are listed in Table 2. Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

Additionally, Table 2 lists the most important spectral interferences at the recommended wavelengths for analysis.

Table 2 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences

(*Clauses* A-2.2 and A-2.4)

Sl No.	Element	Wavelength	Approximately Achievable limits	Interfering Elements

(1)	(2)	(nm) (3)	Radial viewing (µg) (4)	Axial viewing (µg) (5)	(6)
i)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-

A-2.3 Reagents and Solutions

A-2.3.1. *Nitric Acid (65 percent) Suprapure*

A-2.3.2 *Standard Stock Solution*

Either prepare by dissolving proportionate amount of soluble compounds of elements (preferably spectroscopic grade), or use commercially available certified stock solution of 10, 100 or 1 000 µg/ml of Iron in 2 to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-2.3.3 *Standard Solution*

Pipette out 5 ml from 100 µg/ml standard stock solution into a 100 ml volumetric flask and make up volume with 2 percent nitric acid to prepare 5 µg/ml solution. From this 5 µg/ml solution, an aliquot of 1.0, 3.0 and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1, 0.3 and 0.5 µg/ml solution of respective elements under reference.

A-2.3.4 *Sample Preparation*

Weigh about 2.5 g of the sample in a 50 ml volumetric flask and add 1.0 ml nitric acid and make up the volume with water.

> NOTE — Sample should be clear before injecting to the instrument.

A-2.3.5 *Reagent Blank Solution*

Place 50 ml of nitric acid and 1000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluoroethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix and concentration of the reagent blank solution is the same as in the standard and sample solutions.

A-2.4 Instrument

Set up the instrument as per the manufacturer's instructions manual for recommended operating parameters, based on the manufacturers operating manual and evaluated by internal check analysis using of standard solution of element as well as data selected carefully from Table 2.

For analysis of mercury and arsenic, a gas (hydride)/vapour generating system is coupled with ICP, instead of using of nebulizer. The mercury vapour/arsine generated through the system is carried by the carrier gas (Ar) to plasma torch, and other instrumental conditions shall be the same as above.

> NOTE — Sensitivity, instrumental detection limit, precision, linear dynamic range and interference effects will be investigated and established for each individual analyte line on that particular instrument.

A-2.5 Procedure

A-2.5.1 *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**A-2.3.5**). The relationship between concentration and intensity is linear up to six orders of magnitude. Examine the spectra of the element and make necessary adjustments (if required) for the

exact peak positions and baselines to ensure proper measurements of the respective peak intensities. Flush the system with the reagent blank solution between each standard.

A-2.5.2 Before beginning the sample run, re-analyse the reference standard with the highest concentration as if it were a sample. Ensure that the concentration values do not deviate from the actual values by more than ± 5 percent (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Begin the sample run by flushing the system with the reagent blank solution between each sample. It is recommended to analyse a calibration check solution and the calibration blank solution every 10 samples. Analyze the sample solution and calculate the concentration in µg/ml of the Iron in the sample solution.

> NOTE — It is recommended that IS 3025 (Part 2) /ISO 11885 may be referred and practiced for ensuring precise and reproducible analysis.

A-2.6 Calculation

The mass concentrations for each element are determined with the aid of the instrument software by following steps.

i) Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot.

ii) Determine the mass concentrations of samples with the aid of the emissions and the calibration graphs and calculate the quantity in mg/kg of the constituent elemental impurities in the sample, by multiplying the value by 20 (Dilution factor).