

भारतीय मानक मसौदा
सिलिका जेल – विशिष्टि
)आईएस3401 का चौथा पुनरीक्षण

Draft Indian Standard
SILICA GEL — SPECIFICATION
(*Fourth Revision of IS 3401*)

(Not to be reproduced without the permission of BIS or used as an Indian Standard)

ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

Last date of comments: 17th December 2022

FOREWORD

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

Silica gel is pure silica in a highly porous state. It has high adsorption capacity due to the large surface area of its particles. Silica gel is normally prepared by the coagulation of colloidal solution of silicic acid.

Silica gel is used as a desiccant for packages. Its uses also cover prevention of corrosion in instruments, maintenance of dry atmosphere in food and pharmaceutical packings, removal of moisture from transformer breathers, protection of articles like cameras, televisions, etc. in humid atmosphere.

Silica gel is also used for adsorbing acetylene from oxygen gas and in chromatography for analytical purposes. However, this standard does not cover the material intended for these applications.

This standard was first published in 1966 and was based on IMD/SL/7061 (b) 'Desiccant silica', issued in 1957 by the Ministry of Defence, Govt of India. The standard was revised in 1970 incorporating changes in some of the requirements and the methods of test. It was again revised in 1979 incorporating changes in the requirements for moisture, chloride content for indicating type of silica gel, cobalt and sulphate content.

In third revision, changes were made in description, loss on drying and adsorption capacity. The requirement for friability were deleted and in its place a new requirement for loss on attrition was incorporated. In this revision, instrumental test methods for the determination of chlorides and sulphates have been added as alternate test methods. Also, Packing and Marking clause has been updated. Further, amendment 1 and amendment 2 have been incorporated.

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 shall be the same as that of the specified value in this standard.

1 SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for silica gel used as industrial desiccant.

1.1.1 It does not cover silica gel for chromatographic and acetylene adsorption applications.

2 REFERENCES

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

<i>IS No.</i>	<i>Title</i>
IS 460 (Part 1) : 2020	Test Sieves — Specification Part 1 Wire Cloth Test Sieves (<i>fourth revision</i>)
695 : 2020	Acetic Acid — Specification (<i>fourth revision</i>)
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
4161 : 1967	Specification for Nessler cylinders
IS 4905 : 2015	Random sampling and randomization procedures (<i>first revision</i>)
ISO 24153: 2009	

3 TYPES

The material shall be of following two types:

- a) Normal, and
- b) Indicating.

4 REQUIREMENTS

4.1 Description

The material shall be solid with grainy porous structure free from extraneous impurities. The indicating type material shall be impregnated with cobalt chloride.

4.2 Particle Size

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

4.3 Bulk Density

The bulk density of the material shall be as agreed to between the purchaser and the supplier.

4.4 Loss on Drying

The material when tested according to the method prescribed in Annex A shall not lose more than 5 percent of its mass.

4.5 Adsorption Capacity

The material when tested according to the method prescribed in Annex B shall adsorb minimum 27 percent of moisture on the basis of its mass.

4.6 pH

The pH of aqueous extract of the material when determined by the method prescribed in Annex C shall be not more than 8 and not less than 3.5.

4.7 Loss on Attrition

When subjected to the test according to the method prescribed in Annex D, not more than 2.5 percent of the material shall pass through the test sieve.

4.8 Chemical Requirements

The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods prescribed in Annex E. Reference to the relevant clauses of Annex E is given in col 4 of the table.

4.9 Keeping Quality

The material stored in original air-tight containers shall continue to satisfy all the requirements under 4 for not less than 6 months from the date of packing.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in clean, dry and air-tight containers, as agreed to between the purchaser and the supplier.

Table 1 Requirements for Silica Gel
(Clause 4.8 and E-5.3.1)

SI NO.	Characteristic	Requirement	Method of Test, Ref. to Cl No. in Annex E
(1)	(2)	(3)	(4)
i)	a) Chlorides (as NaCl) (for normal type), percent by mass, <i>Max</i>	0.05	E-3 or E-7
	b) Chlorides (as NaCl) (for indicating type), percent by mass. <i>Max</i>	Chlorides (as NaCl) Equivalent of cobalt actually found in the sample +0.05	E-3 or E-7
ii)	Cobalt (as COCl ₂). (for indicating type), percent by mass, <i>Min</i>	0.5	E-4
iii)	Ammonium compounds (as NH ₃), percent by mass, <i>Max</i>	0.001	E-5
iv)	Sulphates (as Na ₂ SO ₄), percent by mass. <i>Max</i>	0.5	E-6 or E-7

5.1.1 The container shall not be opened until required for use and shall not remain open for a period longer than required for taking out the material.

5.2 Marking

The containers shall be marked with the following information:

- Name and type of the material,
- Indication of the source of manufacturer,
- Mass of the material in the container,
- Batch number, and
- Date of packing.

5.2.1 BIS Certification Marking

The product may also be marked with the Standard Mark.

6.2.2 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 there under, and the products may be marked with the standard mark.

6 SAMPLING

The method of drawing representative samples of the material, the number of tests to be performed, and the criteria for conformity of the material to the requirements of the specification shall be as prescribed in Annex F.

ANNEX A
(*Clause 4.4*)
DETERMINATION OF LOSS ON DRYING

A-1 PROCEDURE

A-1.1 Weigh accurately about 30 g of the material in a flat-bottomed glass dish with ground-glass lid and keep it (after removing the lid) in an air-oven at $150 \pm 5^\circ\text{C}$ for 4 h. Cool the dish and the lid to room temperature in a desiccator and weigh. Repeat the operation till constant mass is obtained.

A-2 CALCULATION

Loss on drying, percent by mass = $100 \times \frac{M_1}{M}$

where

M_1 = loss in mass in g of the material on heating, and

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

ANNEX B
(*Clause 4.5*)
DETERMINATION OF ADSORPTION CAPACITY

B-1 APPARATUS

B-1.1 The apparatus required shall be as shown in Fig. 1. It comprises the following:

- a) Circulating pump with flowmeter.
- b) Heat exchange coil, of sufficient length and efficiency to ensure that air leaving the pump is brought to the bath temperature before entering the bubbler.
- c) Bubbler of about 250 ml capacity, fitted with a thermometer and containing a saturated solution of calcium nitrate of analytical reagent grade in the presence of excess solid calcium nitrate.
- d) Spray-catch bottle of about 250 ml capacity, containing glass wool filter.
- e) Tube with side arm, holding a thermometer for checking the temperature of air entering the adsorption tubes.
- f) Two adsorption tubes, about 15 cm long and of 14 mm internal diameter.
- g) Constant temperature bath, maintained at $27 \pm 1^\circ\text{C}$.

NOTE — The underwater connections are conveniently made by means of flexible rubber or plastics tubing or spherical joints.

B-2 PROCEDURE

B-2.1 Weigh accurately about 10 g of the dry material immediately after the determination of loss on drying (see A-1.1) into each of the two previously weighed and stoppered adsorption tubes. Assemble the apparatus as shown in Fig. 1 and adjust the pump so that air is passed at a rate of approximately 1 l per tube per minute. Allow air to circulate until no further increase in mass of the tubes is observed.

B-3 CALCULATION

$$\text{Adsorption capacity, percent by mass} = 100 \times \frac{M_1}{M}$$

where

M_1 = increase in mass in g of the material, and

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

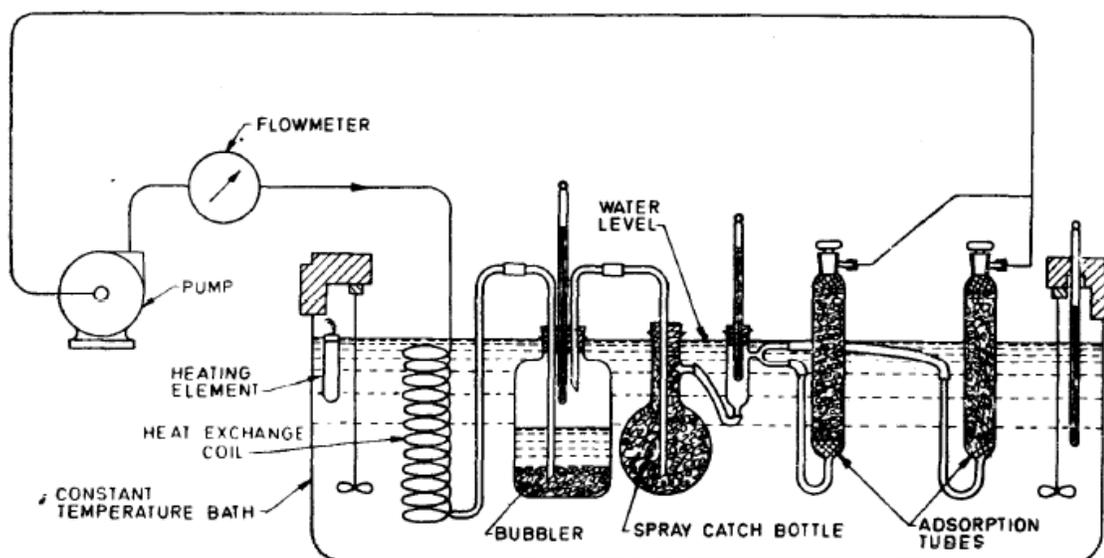


FIG. 1 APPARATUS FOR DETERMINATION OF ADSORPTION CAPACITY

ANNEX C
(*Clause 4.6*)
DETERMINATION OF pH

C-1 PROCEDURE

C-1.1 Prepare an aqueous extract of 25 g of the material by boiling for 2 h with 250 ml of water, free from ammonia and carbon dioxide, in a clean flask of chemically resistant glass fitted by means of a ground-glass joint with a reflux condenser of the same material.

C-1.1.1 Allow the flask to cool to room temperature with adequate precautions against contamination by atmospheric impurities. Decant a portion of this extract and determine the pH by a suitable pH meter using a glass electrode previously calibrated with a buffer solution of known pH.

ANNEX D
(*Clause 4.7*)
DETERMINATION OF LOSS ON ATTRITION

D-1 PROCEDURE

D-1.1 Weigh accurately 50 g of the material as received or with 95 percent relative humidity. Charge the material into two steel cylinders of 3.8 cm diameter and 30.5 cm length. Rotate the cylinders kept at a distance of 7.6 cm from the centre of rotation (to enable the material to tumble) at 30 rpm for 1 h. Sieve the material through 850 micron IS Sieve (*see* IS 460 Part 1). Weigh the material passing through 850 micron IS Sieve and report as attrition loss.

D-1.1.1 The material shall satisfy the requirement of the test if not more than 2.5 g of the material pass through the test sieve.

ANNEX E
(*Clause 4.8, and Table 1*)
METHODS OF TEST FOR CHEMICAL REQUIREMENTS OF SILICA GEL

E-1 QUALITY OF REAGENTS

E-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

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

E-2 PREPARATION OF SOLUTION**E-2.1 Procedure**

Weigh accurately about 25 g of the material and heat to boiling with 200 ml of water for 30 min. Filter and wash the residue with warm water till the washings are free from chlorides and sulphates. Mix the washings and make up to 500 ml in a volumetric flask. Use an aliquot of this prepared solution for subsequent tests under **E-3, E-4, E-5** and **E-6**.

E-3 DETERMINATION OF WATER SOLUBLE CHLORIDES

E-3.1 Reagents

E-3.1.1 *Standard Silver Nitrate Solution* — 0.1 N.

E-3.1.2 *Potassium Chromate Indicator Solution* — 5 percent (m/v).

E-3.1.3 *Dilute Acetic Acid* — 10 percent (v/v).

E-3.1.4 *Sodium Acetate Solution* — 10 percent (m/v).

E-3.2 Procedure

Take 200 ml of the prepared solution (*see E-2.1*), neutralise with sodium acetate solution in case the solution is of high acetic pH or neutralise with dilute acetic acid if the solution is of alkaline pH. Titrate it with 0.1 N silver nitrate solution using potassium chromate solution as indicator.

E-3.3 Calculation

Water soluble chlorides (as NaCl), percent by mass =
$$\frac{V \times N \times 0.05844 \times 100}{M}$$

where

V = volume in ml of standard silver nitrate solution,

N = normality of standard silver nitrate solution, and

M = mass in g of the material contained in the aliquot.

E-3.4 Alternative Method

Chlorides may alternatively be determined by instrumental test method as prescribed in **E-7**.

E-4 DETERMINATION OF COBALT

E-4.1 Reagents

E-4.1.1 *Dilute Hydrochloric Acid* — 5 N.

E-4.1.2 *a-Nitroso β -Naphthol Solution*

Dissolve 3 g of dry *a*-nitroso β -naphthol in 45 ml of glacial acetic acid (*see IS 695*).

E-4.2 Procedure

Take 50 ml aliquot of the prepared solution (*see E-2.1*). Dilute it with water to 403 ml, acidify with 10 ml of dilute hydrochloric acid and heat to boiling. Add 33 ml of *a*-nitroso β -naphthol solution with stirring. Allow to settle for 2 h and filter through Whatman filter paper No. 41 or equivalent. Wash the residue with dilute hydrochloric acid and then thoroughly with hot water. Take the filter paper and the residue in a tared

porcelain crucible and ignite first gently on the flame of a Bunsen burner and then finally at 750°C to 800°C. Cool in a desiccator and weigh to constant mass.

E-4.3 Calculation

$$\text{Cobalt (as CoCl}_2\text{), percent by mass} = \frac{M_1 \times 1.6175 \times 100}{M}$$

where

M_1 = mass in g of the ignited residue, and

M = mass in g of the material present in the aliquot.

E-5 DETERMINATION OF AMMONIUM COMPOUNDS

E-5.1 Apparatus

E-5.1.1 Nessler Cylinders — 50 ml capacity (see IS 4161).

E-5.2 Reagents

E-5.2.1 Sodium Hydroxide Solution — approximately 5 N.

E-5.2.2 Nessler Solution

Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water and add to it slowly with stirring saturated mercuric chloride solution until a slight permanent precipitate forms. Add 30 g of potassium hydroxide and when it has dissolved, add 1 ml more of mercuric chloride solution and dilute to 200 ml with ammonia-free water. Allow to settle overnight, decant off the clear solution and keep it in a bottle closed with a well-fitting rubber stopper.

E-5.2.3 Standard Ammonium Chloride Solution

Dissolve 0.3141 g of ammonium chloride in water and make up to 1000 ml. Take 10 ml of the standard solution and dilute to exactly 100 ml with water. One millilitre of this solution is equivalent to 0.01 mg of ammonia (as NH₃).

E-5.3 Procedure

Take 20 ml of prepared solution (see E-2.1) into a Nessler cylinder. Add 5 ml of sodium hydroxide solution and 2 ml of Nessler solution. Make up the solution to 50 ml mark. Carry out a control test in another Nessler cylinder with 1 ml of standard ammonium chloride solution in place of the material and the same quantities of other reagents. Compare the colour produced in the two cylinders.

E-5.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced in the test with the material is not greater than that produced in the control test.

E-6 DETERMINATION OF WATER SOLUBLE SULPHATES

E-6.1 Reagents

E-6.1.1 Dilute Hydrochloric Acid — approximately 5 N.

E-6.1.2 Barium Chloride Solution – approximately 12 percent (*m/v*).

E-6.2 Procedure

Take 200 ml of the prepared solution (*see E-2.1*). Neutralize the excess alkali, if any, by dropwise addition of dilute hydrochloric acid and then add 2 ml more of dilute hydrochloric acid. Boil and add 10 ml of barium chloride solution slowly with constant stirring. Boil it for another 5 min and allow to settle overnight. Filter through Whatman filter paper No. 42 or equivalent and wash the residue with hot water till washings are free from chlorides. Ignite the residue in a tared crucible. Cool, add a drop of concentrated sulphuric acid and cautiously ignite again. Cool the crucible in a desiccator and weigh. Repeat the operation till constant mass is obtained.

E-6.3 Calculation

Water soluble sulphates (as Na₂SO₄), percent by mass = $\frac{M_1 \times 0.6086 \times 100}{M}$

Where

M_1 = mass in g of the ignited residue, and

M = mass in g of the material contained in the aliquot.

E-6.4 Alternative Method

Sulphates may alternatively be determined by instrumental test method as prescribed in **E-7**.

E-7 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES

E-7.1 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, 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.

E-7.2 Equipment

E-7.2.1 Anion guard column — a protector of the separator column.

E-7.2.2 Anion Separator column — suitable for selective separation of ions under analysis.

E-7.2.3 Anion Suppressor device — Anion micromembrane suppressor is used to analyse the data
Detector: Conductivity detector.

E-7.2.4 Software — Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

Sample loop of 100 µl, 200 µl, 500 µl or 1000 µl be used to determine ionic concentration as per instrument manual and practice.

E-7.3 Reagents and Standards

E-7.3.1 Glass or polyethylene sample bottles.

E-7.3.2 Distilled water or deionized water free from the anions of interest.

E-7.3.3 Eluent — 1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used.

For preparation of these solution, 0.2856 g of sodium bicarbonate and 0.3816 g of sodium carbonate is dissolved in 2 l of water.

E-7.3.4 Micromembrane suppressor solution (0.025 N of sulphuric acid) — Dilute 2.8 ml of concentrated Sulphuric acid in 4 l of water

E-7.4 Standard solutions

E-7.4.1 Chloride — Dissolve NaCl, 1.6485 g in 1 l of reagent water

E-7.4.2 Sulphate — Dissolve 1.81 g of potassium sulphate in 1 l of reagent water

E-7.5 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 0.1-1.0 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.

E-7.6 Procedure

Dissolve between 1 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 (0.1-1.0 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 and deviation of retention force shall not exceed ± 10 percent of RT of calibration. 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 clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

E-7.7 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.

ANNEX F (Clause 6)

SAMPLING OF SILICA GEL

F-1 GENERAL REQUIREMENTS OF SAMPLING

F-1.1 The sampled material shall be kept in a protected place and shall not be exposed to damp air.

F-1.2 The sample shall be placed in suitable containers and each container shall be marked with full details of sampling giving the date of sampling; type of material; batch number, if any; and the name of the manufacturer.

F-2 SCALE OF SAMPLING

F-2.1 All the containers in a single consignment of silica gel of the same type from a single batch of manufacture shall constitute a lot.

F-2.2 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

F-2.3 The number of container (n) to be chosen from the lot shall depend on the size of the lot (N) and shall be as given in Table 2.

Table 2 Scale of Sampling
(Clause F-2.3)

Lot Size N (1)	No. of containers to be Selected n (2)
Up to 25	3
26 to 50	4
51 to 100	5
101 to 200	6
201 and above	8

F-2.4 The containers to be selected for sampling shall be chosen at random from the lot. For this purpose a random number table shall be used (*see* IS 4905). In case such a table is not available, the following procedure shall be adopted:

Starting from any container, count them as 1, 2, 3, , r , and so on in a systematic manner, where r is the integral part of N/n . Every r^{th} container thus counted shall be withdrawn to constitute the required sample.

F-3 TEST SAMPLE AND REFEREE SAMPLE

F-3.1 From each of the containers selected, draw approximately 1.50 g of the material with the help of suitable sampling implement. The material drawn from different containers shall be mixed thoroughly to give a composite sample weighing about 500 g.

F-3.2 The composite sample shall then be divided into three parts, one for the purchaser, another for the supplier and the third for the referee. These parts shall be transferred to separate containers which shall be suitably closed and marked with all the details of sampling.

F-3.3 The referee sample shall bear the seals of the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier and shall be used in cast of a dispute.

F-4 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

F-4.1 Tests for all the characteristics given in **4** shall be conducted on the composite sample.

F-4.2 The lot shall be declared as conforming to the requirements of the specification if all the test results on the composite sample satisfy the relevant requirements given in **4**.