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(□□□□ 12008 □□□□ □□□□□□□□□□□)

Draft Indian Standard
SILICA GEL, CHROMATOGRAPHIC GRADE — SPECIFICATION
(*First Revision of IS* 12008)

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ICS 71.060.50

Inorganic Chemicals Sectional Committee , CHD 01	**Last date of comments: 17ᵗʰ December 2022**

FOREWORD

This Indian Standard (First 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 prepared generally by gelling sodium silicate solution with either hydrochloric acid or sulphuric acid. However in recent practice, it is prepared by passing solution of sodium silicate through cation resin bed and gellifying pure silicic acid solution by suitable gellifying agents. Gellification is achieved by close control of operating parameters, such as pH, concentration, temperature, etc., in order to obtain silica gel of consistent quality.

In this revision, instrumental test methods for the determination of chlorides, iron and sodium have been added as alternate test methods. Also, Packing and Marking clause has been updated. Further, a new requirement for loss on drying, procedure for wet sieving and reference clause have been incorporated.

Chromatographic grade silica gel is mainly used for analytical purposes in thin layer chromatography, high performance liquid chromatography and partition column chromatography. Silica gel used as industrial desiccant is covered in IS 3401.

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.

1 SCOPE

1.1 This standard presents the requirements and the methods of sampling and test for chromatographic grade silica gel.

2 REFERENCE

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:

IS No.	*Title*
264 : 2005	Nitric acid — Specification (*third revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
3025(Part 2) : 2019/ISO 11885	Methods of sampling and test (Physical And Chemical) for water and wastewater: Part 2 determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP - OES) (*first revision*)
4905:2015	Random sampling and randomization procedures (*first revision*)
ISO 24153: 2009	

3 GRADES

3.1 Material shall be of the following two grades:

a) *Grade 1* — For thin layer chromatography,
b) *Grade 2* — For partition column chromatography, mainly 250/125 micron IS sieve or 150/75 micron IS sieve.

4 TYPES

4.1 Grade 1 of the material shall be of two types:

a) *Type 1* — shall be silica gel without binder,
b) *Type 2* — shall be silica gel with binder.

5 REQUIREMENTS

5.1 The material shall consist of silica gel in granular form of various sizes for partition column chromatography and for thin layer chromatography.

5.2 Loss on Drying — The material when tested according to the method prescribed in Annex A-10, shall not lose more than 5 percent of its mass.

5.3 The material shall also comply with the requirements given in Table 1 for thin layer chromatography and Table 2 for partition column chromatography, which tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col **5** of Table 1 and col **4** of Table 2.

6 PACKING AND MARKING

6.1 Packing

The material shall be packed in clean, dry and air-tight bottles not exceeding 250 g. The container shall not be opened until required for use.

6.2 Each container shall bear legibly and indelibly the following information:

  a) Name, grade, type and size of the material;
  b) Name of the manufacturer and/or his recognized trade-mark, if any;

c) Mass of the material in the container;
d) Date of packing; and
e) Batch number.

Table 1 Requirements for Silica Gel for Thin Layer Chromatography
(*Clause* 5.2)

Sl No.	Characteristic	Requirement		Method of Test (Ref to Cl No. in **Annex A**)
		Type 1	Type 2	
(1)	(2)	(3)	(4)	(5)
i)	Particle size passing through 50 micron IS sieve, percent by mass, *Min*	97	97*	A-2
ii)	*p*H of aqueous suspension	5.5 to 7.0	6.0 to 7.5	A-3
iii)	Sodium (as Na), ppm, *Max*	700	----	A-4 or A-13
iv)	Chloride (as Cl), ppm, *Max*	200	200	A-5 or A-11
v)	Iron (as Fe), ppm. *Max*	100	100	A-6 or A-12
vi)	Loss on ignition, percent by mass, *Max*	20	----	A-7
vii)	Gypsum, percent by mass	----	12 to 14	A-8
viii)	Suitability test	shall pass the test	shall pass the test	A-9

Table 2 Requirements for Silica Gel for Partition Column Chromatography
(*Clause* 5.2)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. In **Annex A**)
(1)	(2)	(3)	(4)
i)	Particle size distribution between any mesh (250/125 micron or 150/ 75 micron IS sieve), percent by mass, *Min*	95	A-2
ii)	*p*H of aqueous suspension	5.5 to 7.0	A-3
iii)	Sodium (as Na), ppm, *Max*	700	A-4 or A-13
iv)	Chloride (as Cl), ppm, *Max*	200	A-5 or A-11
v)	Iron (as Fe), ppm, *Max*	100	A-6 or A-12
vi)	Suitability test	To pass the test	A-9

6.2.1 *BIS Certification Marking*

The product may also be marked with the Standard Mark.

6.2.1.1 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.

7 SAMPLING

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

ANNEX A
(*Clause* 5.3 and *Tables* 1 and 2)

METHODS OF TEST FOR SILICA GEL, CHROMATOGRAPHIC GRADE

A-1 QUALITY OF REAGENTS

A-1.1 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 PARTICLE SIZE

A-2.1 For Thin Layer Chromatography

A-2.1.1 *Procedure*

Weigh accurately 50 g of the sample and place it over 50 micron IS sieve. Shake it for 15 min. The mass of material passing through-the sieve gives the particle size of the sample expressed as percentage by mass.

A-2.1.2 *Calculation*

$$\text{Material passing through 50 micron IS sieve, percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where,

M_1 = mass of material taken for sieving, and
M_2 = mass of material retained over the sieve.

A-2.1.3 *Procedure for wet sieving*

Weigh accurately 50 g of the dried sample (dried as per **Cl.5.2**) and soaked in water. The sample is stirred and left for soaking period of at least 10 min. The material is sieved through 50 micron sieve. The material is washed until the water filtered becomes clear. The sample retained on 50 micron sieve is collected and dried in oven (as per **Cl.5.2**).

A-2.1.4 *Calculation*

$$\text{Material passing through 50 micron IS sieve, percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where

M_1 = mass of the dried material taken for sieving, and

M_2 = mass of the dried material retained over the sieve.

A-2.2 For Partition Column Chromatography

A-2.2.1 *Apparatus*

A-2.2.1.1 *Sieve* — 125 micron and 250 micron IS sieve (or 75 micron and 150 micron IS sieve).

A-2.2.2 *Procedure*

Take accurately about 100.00 g of material of 250 micron/ 125 micron IS sieve size and sieved through 250 micron sieve and material passing through 250 micron sieve is sieved through 125 micron sieve, quantity retained on 125 micron sieve must not be less than 95 g. In other words, mass of material retained on 250 micron sieve plus mass of material passing through 125 micron IS sieve should not be more than 5 g.

A-3 pH VALUE

A-3.1 Apparatus

A-3.1.1 *pH Meter*

A-3.2 Procedure

Prepare an aqueous suspension of the material by shaking for 10 min, 5 g of the material with 50 ml of water (free from ammonia and carbon dioxide) in a clean polyethylene flask. Decant the supernatant liquid and measure pH by using any standard pH meter which is standardized against standard buffer solution of pH 4.0 and pH 9.2.

A-4 SODIUM AND ITS COMPOUNDS

A-4.1 Apparatus

A-4.1.1 *Flame Photometer*

A-4.2 Reagents

A-4.2.1 *Concentrated Nitric Acid — See* IS 264.

A-4.3 Procedure

Weigh 0.5 g of the sample into a 250 ml glass dish previously washed with dilute nitric acid. Add 10 ml of water and 10 ml of concentrated nitric acid and evaporate the contents to dryness on a hot plate. Add another 10 ml of concentrated nitric acid and evaporate to dryness again. Extract the residue with 20 ml of nitric acid (1 : 1) by gentle boiling. Filter through Whatman No. 40 or equivalent filter paper into a 250ml volumetric flask. Wash three times with hot water. Cool the flask and diiute to the mark with water. Take 25 ml of the solution and dilute to 100 ml in a volumetric flask. Flame the diluted solution using a flame photometer. The instrument shall be calibrated in the range of 0 to 5 mg/l of sodium.

A-4.4 Calculation

$$\text{Sodium and its compounds (as Na}_2\text{O), percent by mass } = \text{X} \times 0.2$$

Where, X = mg/l of Na_2O from the calibration curve.

A-4.5 Alternative Method

Sodium may alternatively be determined by instrumental test method as prescribed at **A-13**.

A-5 WATER SOLUBLE CHLORIDES

A-5.1 Reagents

A-5.1.1 *Standard Chloride Solution* — Prepared by adding 28.2 ml of N/10 hydrochloric acid to water to produce 100 ml.

A-5.2 Procedure

Boil 0.5 g (accurately weighed) with a mixture of 50 ml water and 1 ml of dilute nitric acid (approximately 5 N), Cool, filter and to the filtrate add 1 ml of silver nitrate solution (0.4 percent).

Any opalescence produced shall not be greater than the standard opalescence as stated in **A-5.2.1**.

A-5.2.1 One millilitre of the solution is equivalent to 0.1 mg of chloride (as cl). To 50 ml of water add 1 ml of standard chloride solution, 1 ml of dilute nitric acid and 1 ml of silver nitrate solution. Mix after each addition, and use as a comparison standard after 5 min.

A-5.3 Alternative Method

Chlorides may alternatively be determined by instrumental test method as prescribed at **A-11**.

A-6 1RON

A-6.1 Reagents

A-6.1.1 *Dilute Hydrochloric Acid* — 1 : 1 (*v/v*).

A-6.1.2 *Standard Potassium Permanganate* — 0.1 N.

A-6.1.3 *Ammonium Thiocyanate Solution* — 57 percent.

A-6.1.4 *Standard iron Solution*

Dissolve 8.65 g of ammonium ferric sulphate in 50 ml of concentrated nitric acid and dilute to 1000 ml. One ml of this solution is equivalent to 0.01 mg of iron (as Fe).

A-6.2 Procedure

Boil 0.1 g with a mixture of 10 ml water and 1 ml of dilute hydrochloric acid, cool and filter.

Take filtrate and add 1 drop of N/10 potassium permanganate and mix. Add 5 ml of ammonium thiocyanate solution (570 g/l) and 10 ml of mixture of equal volumes of amyl alcohol and amyl acetate: shake vigorously and allow to separate.

Take 1 ml of standard solution of iron, add 1 ml of dilute hydrochloric acid and dilute with water to the same volume as acidified solution of the test sample. Add 1 drop of N/l0 potassium permanganate and from this point follow the procedure described for sample and compare colour with standard. The colour produced by the sample is less than colour produced by standard if iron content in sample is less than 100 ppm.

A-6.3 Alternative Method

Iron may alternatively be determined by instrumental test method as prescribed at **A-12**.

A-7 LOSS ON IGNITION

A-7.1 Apparatus

A-7.1.1 *Platinum Crucible*

A-7.2 Procedure

Take 1 g of the material in a clean, dry platinum crucible. Keep the crucible and its contents on a hot plate for 10 min. Heat further at 550 °C for 5 min and finally at 1100 °C for 30 min. Cool, weigh and calculate the percent loss on ignition.

A-7.3 Calculation

$$\text{Loss on ignition, percent by mass} = \frac{M_1 \times 100}{M_2}$$

where,

M_1 = Loss in mass in g, and

M_2 = Mass in g of the sample taken for the test.

A-8 GYPSUM

A-8.1 Reagents

A-8.1.1 *Dilute Ammonia Solution* — 25 percent.

A-8.1.2 *Standard EDTA Solution* — 0.1 M.

A-8.1.3 *Eriochrome Black T Indicator*

A-8.2 Procedure

Take about 1 g of the material in a 250 ml beaker and mix with about 150 ml of water and stir well with a glass rod. Filter through a double layer filter paper No. 41 after 30 min. Wash 5 times with water. Add 2 ml of dilute ammonia solution in the filtrate and titrate against standard EDTA solution using eriochrome black T as indicator.

A-8.3 Calculation

$$\text{Gypsum (as } CaSO_4.5H_2O), \text{percent by mass} = \frac{A \times 1.45}{M}$$

where,

A = volume in ml of standard EDTA, and
M = mass in g of sample taken for the test.

A-9 SUITABILITY TEST

A-9.1 Suitability for Thin Layer Chromatography

A-9.1.1 *Reagents*

A-9.1.1.1 *Solvent mixture — See* **A-9.2.1.1**.

A-9.1.1.2 *Toluene*

A-9.1.2 *Procedure*

Make 30 g into slurry with about 60 ml water. Spread five 20×20 cm plates and allow the slurry to dry in air. The material should be smooth and free from hollows. Developed plates using toluene after spotting with test mixture should give clear separation with colour in straight lines horizontally and vertically. Spray plates with 2N (sulphuric acid) H₂SO₄ in alcohol and heat at 110°C for 30 min, Very few black particles shall be observed.

NOTE- Time taken For solvent to travel 20 cm of length of plate shall be about 30 min.

A-9.2 Suitability Test For Partition Column Chromatography

A-9.2.1 *Reagents*

A-9.2.1.1 *Solvent mixture*

Mix 10 mg of 4 methoxy-azo benzene with 10 mg each of Sudan yellow and Sudan red. Dissolve this, mix in 20 ml of the solvent prepared by mixing 4 parts of petroleum ether with 1 part of benzene.

A-9.2.2 *Procedure*

Prepare a column of 5 cm length using petroleum ether. Use column of 1.5 cm diameter. Add 5 ml of solvent mixture, wash the column with 10 ml of the same solvent. The order of the absorption of three different dyes shall be as under:

In Column	*Order of Elution*
Yellow	1) 4-Methoxy-azo benzene solution is yellow
Yellow	2) Sudan yellow-solution is orange yellow
Red	3) Sudan red-solution is red in colour.

A-10 DETERMINATION OF LOSS ON DRYING

A-10.1 Procedure

Weight accurately about 30g of the material in flat-bottomed glass dish with ground-glass lid and keep it (after removing the lid) in an air-oven at 150 ± 5 °C for 4 h. Cool the dish and the lid to room temperature in a desiccator and weight. Repeat the operation till constant mass is obtained.

A-10.2 Calculation

Loss on drying, percent by mass $= 100 \times \dfrac{M1}{M}$ 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.

A-11 ION CHROMATOGRAPHY FOR CHLORIDES

A-11.1 Principle

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides. 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.

A-11.2 Equipment

A-11.2.1 *Anion guard column* — a protector of the separator column.

A-11.2.2 *Anion Separator column* — suitable for selective separation of ions under analysis.

A-11.2.3 *Anion Suppressor device* — Anion micromembrane suppressor is used to analyse the data Detector: Conductivity detector.

A-11.2.4 *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.

A-11.3 Reagents and Standards

A-11.3.1 *Glass or polyethylene sample bottles.*

A-11.3.2 *Distilled water or deionized water free from the anions of interest.*

A-11.3.3 *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.

A-11.3.4 *Micromembrane suppressor solution* (0.025 N of sulphuric acid) — Dilute 2.8 ml of concentrated Sulphuric acid in 4 l of water

A-11.4 Standard solutions

A-11.4.1 *Chloride* — Dissolve NaCl 1.6485 g in 1 l of reagent water

A-11.5 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.

A-11.6 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.

A-11.7 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-12 DETERMINATION OF IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-12.1 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-12.2 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 3. 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 3 lists the most important spectral interferences at the recommended wavelengths for analysis.

Table 3 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences

Sl No.	Element	Wavelength (nm)	Approximately Achievable limits		Interfering Elements
			Radial viewing (µg)	Axial viewing (µg)	
(1)	(2)	(3)	(4)	(5)	(6)
ii)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-

A-12.3 Reagents and Solutions

A-12.3.1 *Nitric acid (65 percent) Suprapure*

A-12.3.2 *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 1000 µg/ml Iron, in 2-5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-12.3.3 *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-12.3.4 *Sample preparation*

Weigh about 2.5 g polyphosphoric acid 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-12.3.5 *Reagent Blank Solution*

Place 50 ml of nitric acid and 1 000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluorethylene (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-12.4 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 3.

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-12.5 Procedure

A-12.5.1 *Calibration*

 Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**A-12.5.2**). 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-12.5.2 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 lead (and/or Iron, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury) 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-12.6 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).

A-13 DETERMINATION OF SODIUM BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-13.1 The solution under analysis is passed with the help of a peristaltic pump through a nebulizer inside a spray chamber. The aerosol formed is aspirated in the form of argon plasma where the atoms are 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 wavelength/intensity of emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

A-13.2 Reagents and Solutions

A-13.2.1 *Magnesium nitrate, 6.67 percent w/v* — Add 66.7 g of magnesium nitrate hexahydrate in 1000 ml of deionized water and mix well.

A-13.2.2 *HCl solution* — Dilute 83.0 ml concentration HCl to 1 000 ml deionized water.

A-13.2.3 *50 percent Nitric Acid* — Dilute 500 ml of Nitric Acid in 500 ml of deionized water.

A-13.3 Standards

A-13.3.1 *Yttrium Internal Standard (to infuse in the instrument T junction), 2 µg/ml*

 Prepare 1 000 µg/ml of Yttrium stock standard. Transfer 2 ml of this standard stock solution in 1 l volumetric flask and dilute the solution using 1 N HCl.

A-13.3.2 *Yttrium Working internal standard*:

Pipette out 25 ml of 1 000 µg/ml Yttrium stock standard into 50 ml volumetric flask and dilute using 1N HCl. If Yttrium Internal Standard is used to infuse in the instrument, then there is no need to add Yttrium during the preparation of calibration standards.

Sodium — Calibration standard 0 µg/ml- Add 0 µl of 10 000 µg/ml sodium stock solution in 50 ml *volumetric flask* — To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Sodium — Calibration standard 6 µg/ml- Add 30 µl of 10 000 µg/ml of sodium stock solution in 50 ml *volumetric flask* — To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Sodium — Calibration standard 12 µg/ml- Add 60 µl of 10000 µg/ml of sodium stock solution in 50 ml *volumetric flask* — To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Sodium —Calibration standard 24 µg/ml- Add 120 µl of 10000 µg/ml of sodium stock solution in 50 ml volumetric flask — To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Sodium —Caibration standard 48 µg/ml- Add 240 µl of 10000 µg/ml of sodium stock solution in 50 ml *volumetric flask* — To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Similarly, calibration standard solutions can be prepared for iron and calcium analysis.

A-13.4 Sample Preparation

Process the sample until homogeneous.

A-13.5 Procedure (Sample Preparation)

Take sample in a platinum crucible. Add magnesium nitrate (2 ml) to the sample and gently swirl it.

Place the sample in a cool muffle furnace with temperature less than 80 °C. Set the furnace program as:

Step 1 Ramp=3°C/min   Level=100°C   Dwell=360 min

Step 2 Ramp=3°C/min   Level=150°C   Dwell=60 min

Step 3 Ramp=3°C/min   Level=500°C   Dwell=480 min

Step 4 Ramp=end

Else, heat the sample in the furnace gradually as per the above steps by controlling the sample through the thermostat.

The sample must not be heated so rapidly that it ignites. Remove the sample and cool the furnace at room temperature. Add 2 ml 50 percent aqueous HNO_3 and wash with it the sides of the crucible. Dissolve all the ash and transfer in the beaker. Remove excess of acid by slow heating of the sample on a hot plate. Make sure that the sample is free from any black precipitate. If any precipitate is found, again transfer the dry mass of the beaker into the crucible using little water and evaporate the water and heat at 500-550°C for 1 h. Again repeat this procedure till the sample becomes free from black precipitate. Remove the sample from furnace and cool it to room temperature. Add HCl (5 ml, 1 N) and dissolve the ash/mass. Transfer the above solution to 50 ml graduated flask and make up the final volume to 50 ml using 1 N HCl. Fortify the sample with 200 µl of Yttrium working standard and mix it. This step is omitted if T junction is used on the instrument. Analyse the sample using ICP/OES

A-13.6 Instrument Settings

Set up the instrument as per the manufacturer's instructions. Relative to the internal standard concentration, the quantification of the ions is done by the system software. Calibrate the instrument using ICP reagent blank and the calibration standard. Monitor the wavelengths for the respective metal ions.

Na=589.592 nm, Pb=220 nm, Fe=238.2 nm

A-13.7 Manual Calculation

Use linear regression analysis to determine a standard curve (emission vs. concentration)

ppm (analyzed) = <u>µg/ml (evaluated from calibration curve) ×ml (final volume)</u>

                            g (sample weight)

ANNEX **B**
(*Clause* 7.1)
SAMPLING OF SILICA GEL, CHROMATOGRAPHIC GRADE

B-1 SCALE OF SAMPLING

B-1.1 Lot

All the containers in a single consignment of silica gel of the same type and for the same use (for thin layer chromatography or for partition column chromatography) drawn from a single batch of manufacture shall constitute a lot.

B-1.2

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

B-1.3

Unless otherwise agreed to between the buyer and the seller, the number of containers to be chosen from the lot shall depend upon the size of the lot and shall be as given in Table 4.

Table 4 Scale of Sampling

Lot Size	Number of Containers to be Selected
(1)	(2)
Up to 25	3
26 to 50	4
51 to 100	5
101 to 150	7
151 and above	10

B-1.4 The containers shall be selected from the lot at random and in order to ensure the randomness of selection, the method given in IS 4905.

B-2 TEST SAMPLE AND REFEREE SAMPLE

B-2.1 From each of the containers selected draw approximately 150 g of silica gel with help of suitable sampling implements, the material drawn from different containers shall be mixed thoroughly to give a composite sample weighing about 500 g.

B-2.2 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.

B-2.3 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 case of a dispute.

B-3 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

B-3.1 All the sample containers shall be visually examined for the requirements given in **5**.

B-3.2 Tests for all the characteristics given in Table 1 or Table 2 shall be conducted on composite sample.

B-3.3 The lot shall be declared as conforming to the requirements of the specification if all the containers pass the requirements of **5** and the composite sample satisfies the relevant requirements given in Table 1 nor Table 2.