

भारतीय मानक मसौदा
पोटेशियम सिलिकेट, तकनीकी — विशिष्टि
(IS 8813 का दूसरा पुनरीक्षण)

Draft Indian Standard
Potassium Silicate, Technical — Specification
(*Second Revision of IS 8813*)

(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: 26th March 2025

Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

(Formal clauses will be added later)

This standard was first published in 1978 and subsequently revised in 1995. In the first revision, the methods of test for all the requirements were modified. In this second revision, instrumental test method for the determination of iron has been added as alternate test method. In addition to this, editorial corrections have been made wherever required. Also, Reference clause and Packing & Marking clause have been updated.

Potassium silicate is used in welding electrode, as corrosion inhibitor and in cement industries etc.

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***POTASSIUM SILICATE, TECHNICAL — SPECIFICATION**
(*Second Revision*)**1 SCOPE**

This standard prescribes the requirements and method of sampling for potassium silicate meant for use in welding electrode industry.

2 REFERENCES

The Indian Standards given 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.

<i>IS No.</i>	<i>Title</i>
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) (<i>first revision</i>)
(Part 65) : 2022/ ISO 17294-2 : 2016	Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (<i>first revision</i>)
IS 4905 : 2015/ISO 24153: 2009	Random sampling and randomization procedures (<i>first revision</i>)
IS 14212 : 1995	Sodium and potassium silicates — Methods of test

3 GRADES

The material shall be of two grades, namely,

- a) Grade 1
- b) Grade 2

4 REQUIREMENTS**4.1 Description**

The material shall be in the form of liquid. It shall be a thick, viscous and translucent mass. The material shall be water-white or slight grey in colour and shall be clear, free from dirt and other visible impurities.

4.2 Viscosity

The viscosity of the material shall be as agreed to between the purchaser and the supplier. The method for determination of viscosity shall be as prescribed in IS 14212.

4.3 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in IS 14212. Reference to the relevant clauses of IS 14212 is given in col 1 (5) of the table.

5 PACKING AND MARKING**5.1 Packing**

The material shall be packed in air-tight iron, mild steel drums or high density polyethylene containers.

5.2 Marking

Each package shall be legibly and indelibly marked with the following information:

- a) Name and grade of the material;
- b) Indication of the source of manufacture;
- c) Gross and net mass of the material; and
- d) Batch number.

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.

6 SAMPLING

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

Table 1 Requirements for Potassium Silicate, Technical

(Clause 4.3)

Sl. No.	Characteristic	Requirement for		Method of Test, Ref to Annex	Method of Test, Ref to Cl No. in IS 14212
		Grade 1	Grade 2		
(1)	(2)	(3)	(4)	(5)	(6)
i)	Matter insoluble in water, percent by mass, <i>Max</i>	0.20	0.20		A-3
ii)	Relative density at 27/27 °C	1.37 to 1.39	1.32 to 1.35		A-5
iii)	Total soluble silicates, percent by mass	37 to 38	32.5 to 34.5		A-6 and A-8
iv)	Ratio of total alkalinity (as K ₂ O) to total soluble silica as (SiO ₂) (Molecular ratio K ₂ O : SiO ₂)	1 : 3.1 ± 0.1	1 : 3.4 ± 0.1		A-9
v)	Iron (as Fe), percent by mass, <i>Max</i>	0.02	0.05	A	A-11

ANNEX A

(Table 1, Sl No. v)

DETERMINATION OF IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD**A-1 General**

Three methods are prescribed for determining iron, namely, Method A as prescribed at A-11 of IS 14212, ICP-OES method as prescribed at Annex A and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

A-1.1 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-1.2 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-1.2 and A-1.4)

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-1.3 Reagents and Solutions

A-1.3.1. Nitric Acid (65 percent) Suprapure

A-1.3.2 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 Lead, Iron, calcium, magnesium,

manganese, arsenic, molybdenum, aluminium and mercury in 2 to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-1.3.3 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-1.3.4 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-1.3.5 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, 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-1.4 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-1.5 Procedure

A-1.5.1 Calibration

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**A-1.3.5**). 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-1.5.2 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, arsenic) 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-1.6 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).

ANNEX B

(Clause 6)

METHOD OF SAMPLING OF POTASSIUM SILICATE

B-1 GENERAL REQUIREMENTS

B-1.1 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

B-1.2 Precautions shall be taken to protect the samples, sampling instruments and the containers for samples from contamination.

B-1.3 The material shall be thoroughly mixed before drawing the sample.

B-1.4 The samples shall be placed in suitable, clean and dry containers with proper closures.

B-1.5 Each sample container, after filling, shall be sealed air-tight and marked with full details of sampling.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers in a single consignment of the material of one grade drawn from a single- hatch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different grades or batches or manufacture, the containers shall be suitably separated and grouped to form lots as defined above.

B-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.2 The number (n) of containers to be chosen from a lot shall depend on the size of the lot (N) and shall be in accordance with col 1 and 2 of Table 3

B-2.3 The drums to be selected for sampling shall be chosen at random, preferably with the aid of random number tables. In order to ensure randomness of selection, IS 4905 may be followed.

Table 3 Number of Drums to be selected

(Clause B-2.2)

Sl. No.	Lot Size N	Number of Drums to be Selected n
(1)	(2)	(3)
i)	Up to 10	3

ii)	11 to 20	5
iii)	21 to 30	7
iv)	31 to 50	10
v)	51 and Above	15

B-3 TEST SAMPLES AND REFEREE SAMPLES

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument a small portion of material from different parts of each drum selected for sampling, after mixing thoroughly the contents of the drum and then allowing the liquid to come to rest. The total quantity of the material drawn from each selected drum shall be sufficient to make triplicate determinations for all the characteristics given in Table 1.

B-3.1.2 Thoroughly mix portions of material drawn from the same drum. Out of these portions, a small equal quantity shall be taken from each selected drum and shall be well mixed up together so as to form a composite sample. This composite sample shall be divided into three equal parts, one for the purchaser, and another for the supplier and the third to be used as referee sample.

B-3.1.3 The remaining portions of the material drawn from each drum (after small quantity needed for the preparation of composite sample has been taken) shall be divided into three equal parts. These parts shall be transferred immediately to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with the particulars of sampling given in **B-1.5**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets in such a way that each set has a test sample representing each drum selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as referee sample.

B-3.2 Referee Sample

The referee sample shall consist of the composite sample (*see B-3.1.2*) and a set of individual test samples (*see B-3.1.3*) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in the case of dispute between the two.

B-4 NUMBER OF TESTS

B-4.1 Tests for total soluble silicates and the ratio for total alkalinity to total soluble silica shall be carried out on any three individual samples, selected at random out of those prepared according to **B-3.1.3**. The same samples shall be used for both the tests.

B-4.2 Tests for all other characteristics shall be carried out on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 For those characteristics which have been tested on the individual test sample (*see B-4.1*), all the test results on each of the individual samples shall satisfy the corresponding requirements given in Table 1, if the lot is to be deemed to conform to those requirements.

B-5.2 For the remaining characteristics prescribed in Table 1, the test results on the composite sample shall meet the corresponding specified requirements.

B-5.3 The lot shall be declared as conforming to the requirements of the specification if **B-5.1** and **B-5.2** are satisfied.