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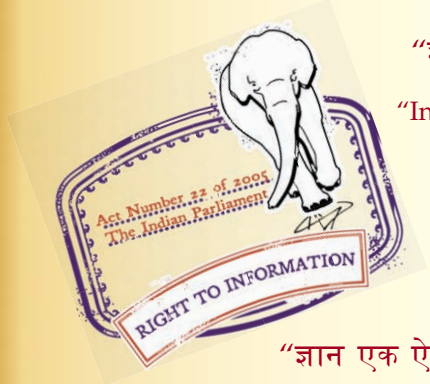

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Indian Standard

SPECIFICATION FOR SODIUM SILICATE FOR COSMETIC INDUSTRY

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SPECIFICATION FOR SODIUM SILICATE FOR COSMETIC INDUSTRY

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SPECIFICATION FOR SODIUM SILICATE FOR COSMETIC INDUSTRY

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 6October1980, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Sodium silicate is used in textile, soap, electrode, cement, adhesive and pharmaceutical industries. IS:381-1972* covers the requirements of these industries. The Cosmetics Sectional Committee felt that the requirements of cosmetic industry were not adequately covered in this standard and, therefore, decided to prepare a separate standard to assist the manufacturers of cosmetics to procure material of the requisite quality.

0.3 Initially while preparing this standard, the Committee had agreed to indicate separately the essential and optional requirements. Subsequently, this was found non-implementable for certification and therefore a single set of requirements only was decided to be stipulated.

0.4 This edition 1.1 incorporates Amendment No.1 (May1992). Side bar indicates modification of the text as the result of incorporation of the amendment.

0.5 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:2-1960†. 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 prescribes the requirements and the methods of sampling and test for sodium silicate for cosmetic industry.

1.1.1 The material shall be neutral liquid type.

*Specification for sodium silicate (*first revision*).
†Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1Description—The material shall be clear, free from dirt and other visible impurities.

2.1.1 It shall be thick, viscid, translucent mass of water-white or slightly grey colour.

2.2 The material, when tested by the methods prescribed in AppendixA, shall comply with the requirements laid down in Table1. Reference to the relevant clauses of Appendix A is given in col4 of the table.

TABLE1 REQUIREMENTS FOR SODIUM SILICATE FOR COSMETIC INDUSTRY

SL NO.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, (REF TO CL NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Total soluble silicate (as SiO_2), percent by mass, *Min*	36.0	A-3
ii)	Ratio of total alkalinity (as Na_2O) to total soluble silica (as SiO_2) (Na_2O:SiO_2)	1:3.3 ±0.2	A-3
iii)	Iron (as Fe), percent by mass, *Max*	0.1	A-4
iv)	Arsenic (as As_2O_3), parts per million, *Max*	2	A-5
v)	Heavy metals (as Pb), parts permillion, *Max*	30	A-6
vi)	Matter insoluble in water, percent by mass, *Max*	0.2	A-7
vii)	Relative density at 27°/27°C	1.35 to 1.45	A-8
viii)	Viscosity at 27°C, centipoise	120 to 200	A-9

3. PACKING AND MARKING

3.1Packing—The material shall be packed in air-tight mild steel drums of capacity as agreed to between the purchaser and the supplier.

3.2Marking—Each package shall be marked with the following information:

a)Name and description of the material;
b)Net mass of the material;
c)Manufacturer’s name and/or his recognized trade-mark, if any;
d)Year of manufacture; and
e)Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

3.3BIS Certification Marking

The product may also be marked with Standard Mark.

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

4. SAMPLING

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

A P P E N D I X A

(*Clause*2.2)

METHODS OF TEST FOR SODIUM SILICATE

A-1. QUALITY OF REAGENTS

A-1.1 Unless otherwise specified, pure chemicals and distilled water (*see*IS:1070-1977*) shall be used in tests.

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

A-2. PREPARED SAMPLE SOLUTION

A-2.1Procedure—Weigh accurately about 20g of the material and dissolve in freshly boiled water. Filter and thoroughly wash the filter paper with freshly boiled water. Transfer both the filtrate and the washings to a 500-ml volumetric flask and dilute up to the mark. Keep this prepared sample solution for test purposes.

A-3.DETERMINATION OF TOTAL ALKALINITY, TOTAL SOLUBLE SILICATES AND RATIO OF TOTAL ALKALINITY TO TOTAL SOLUBLE SILICATES

A-3.1 Reagents

A-3.1.1*Standard Hydrochloric Acid*—1N.

*Specification for water for general laboratory use (*first revision*).

A-3.1.2 *Sodium Fluoride* —solid.

A-3.1.3 *Rectified Spirit* -- *See* IS:323-1959*.

A-3.1.4 *Methyl Red Indicator Solution*—Dissolve 0.1g of methyl red in 100ml of 60percent rectified spirit.

A-3.1.5 *Methyl Red-Xylene Cyanol FF Indicator Solution*—Dissolve 0.8g of methyl red and 0.2g of xylene cyanol FF in 1000ml of rectified spirit.

A-3.1.6 *Standard Sodium Hydroxide Solution*—1N.

A-3.2 Procedure—Transfer 50ml of the prepared sample solution to a 250-ml beaker and add about 0.5ml of methyl red indicator solution. Titrate with standard hydrochloric acid to the first colour change. Then, add to it about 5g sodium fluoride and dissolve as much of it as possible by agitation. Add 25ml of ethyl alcohol and again continue titration till the colour of the solution is definitely red (an excess of about 2ml of 1N hydrochloric acid is sufficient). At this point, add about 0.5ml of methyl red-xylene cyanol FF indicator solution and titrate back with standard sodium hydroxide solution till the end point is reached. (The end point is an intermediate grey or the colour immediately after the disappearance of the pink and before the appearance of the green).

A-3.3 Calculations

Total soluble silica (SiO_2), percent by mass $= \frac{(BN_1 - CN_2) \times 1.502}{M}$

Total alkalinity (Na_2O), percent by mass $= \frac{AN_1}{M} \times 3.1$

where

B = volume in ml of standard hydrochloric acid after the addition of sodium fluoride and rectified spirit;

N_1 = normality of standard hydrochloric acid;

C = volume, in ml, of standard sodium hydroxide solution used for the back titration;

N_2 = normality of standard sodium hydroxide solution;

M = mass, in g, of the material present in the aliquot of the sample solution; and

A = volume, in ml, of standard hydrochloric acid required to first methyl red end point.

*Specification for rectified spirit (*revised*).

A-3.3.1 Determine total soluble silicates by adding the percentage of total soluble silica and total alkalinity. Find out the ratio of total alkalinity to total soluble silica as obtained in **A-3.3**

A-4. DETERMINATION OF IRON

A-4.1 Apparatus

A-4.1.1 *Nessler Cylinders*—50ml capacity.

A-4.2 Reagents

A-4.2.1 *Concentrated Nitric Acid*—*See* IS:264-1976*.

A-4.2.2 *Ammonium Persulphate*—solid.

A-4.2.3 *Butanolic Potassium Thiocyanate*—Dissolve 10g of potassium thiocyanate in 10ml of water. Add sufficient *n*-butanol to make up to 100ml and shake vigorously until the solution is clear.

A-4.2.4 *Dilute Sulphuric Acid*—approximately 10percent(*v*/*v*).

A-4.2.5 *Standard Iron Solution*—Weigh 0.702g of ferrous ammonium sulphate [$FeSO_4(NH_4)_2 SO_4.6H_2O$] and dissolve in 10ml of dilute sulphuric acid. Dilute with water to make up the volume to 1000ml. Transfer 10ml of this solution and again dilute with water to make up the volume to 100ml. One millilitre of this solution is equivalent to 0.01mg of iron (as Fe).

A-4.3 Procedure—Take about 5g of the sample accurately weighed, dissolve it in water as prescribed in **A-2** and make up the solution to exactly 100ml. Take 10ml aliquot of this solution, add 1ml of concentrated nitric acid and boil. Cool, transfer the solution to a Nessler cylinder and add 30mg of ammonium persulphate and 15ml of butanolic potassium thiocyanate solution. Shake for 30seconds and allow the liquid to separate. Carry out a control test in the other Nessler cylinder, adding slowly from a burette a quantity of the standard iron solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture until the colours of butanol layers in the two cylinders are exactly matched.

A-4.4 Calculation

$$\text{Iron (as Fe), percent by mass} = \frac{0.001V}{M}$$

where

V = volume, in ml, of standard iron solution required in the control test; and

M = mass, in g, of the material present in the aliquot of prepared sample solution.

*Specification for nitric acid (*second revision*).

A-5.DETERMINATION OF ARSENIC

A-5.1 Reagents

A-5.1.1*Dilute Hydrochloric Acid*—approximately 5N.

A-5.2Procedure—Dissolve 2.0g of the material in the minimum amount of dilute hydrochloric acid (about 4ml). Add water to make up to 10ml. Transfer quantitatively to the Gutzeit bottle and carry out the test for arsenic as prescribed in IS:2088-1971*, using 4ml of standard arsenic trioxide solution for the comparison stain.

A-5.2.1 The material shall be considered to have not exceeded the limit for arsenic prescribed in Table1, if the length and intensity of the stain produced with the material is not greater than in the control test.

A-6. DETERMINATION OF HEAVY METALS

A-6.1 Apparatus

A-6.1.1*Nessler Cylinders*—50-ml capacity.

A-6.2 Reagents

A-6.2.1*Dilute Hydrochloric Acid*—approximately 5N.

A-6.2.2*Dilute Acetic Acid*—approximately 1N.

A-6.2.3 *Hydrogen Sulphide Solution*—saturated.

A-6.3Procedure—Weigh 2.0g of the material into a beaker. Add 3ml of dilute hydrochloric acid. Dilute with water and make up the volume to 50ml. Filter the solution. Transfer 25ml of the filtrate into a Nessler cylinder. In the second Nessler cylinder, add 2ml of dilute acetic acid, 3.0ml of standard lead solution and make up the volume to 25ml. Add 10ml of hydrogen sulphide solution to each Nessler cylinder and make up the volume with water to 50ml. Mix, allow to stand for 10minutes and then compare the colour produced in the two Nessler cylinders.

A-6.3.1 The limit prescribed in Table1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

A-7. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-7.1Procedure—Weigh accurately about 10g of the material into a beaker and dissolve in freshly boiled water. Filter by applying suction through a tared Gooch crucible. Wash the beaker and the residue free from alkali with freshly boiled water and then dry the

*Methods of test for arsenic (*first revision*).

Gooch crucible in an oven maintained at 110±5°C. Cool in a desiccator and weigh. Repeat the operation of heating and cooling till constant mass is obtained.

A-7.2 Calculation

$$\text{Matter insoluble in water, percent by mass} = 100 \times \frac{M_1}{M}$$

where

M_1 = mass, in g, of the residue; and

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

A-8. DETERMINATION OF RELATIVE DENSITY

A-8.0 General—Two methods are prescribed, namely, the hydrometer method and the pyknometer method. The hydrometer method is recommended for routine work while pyknometer method shall be the referee method in case of dispute.

A-8.1 Hydrometer Method

A-8.1.1 *Procedure*—Pour the material to be tested into the clean hydrometer jar the diameter of which shall be at least 2.5cm greater than the diameter of the hydrometer used. Remove all air bubbles that might have formed in the liquid. Keep the jar in a vertical position and in a bath maintained at 27°C. When the sample in the jar attains the temperature of the bath, that is, 27°C, lower the hydrometer gently into the material. When it has settled, depress it about two scale divisions into the liquid. Keep the unimmersed portion of the stem dry, as any unnecessary liquid on the stem will change the effective weight of the instrument and affect the reading obtained. Allow the hydrometer to become stationary. Remove all air bubbles that might have formed during the lowering of the hydrometer. Read the point on the hydro-meter scale to which the sample rises, with the eye placed at the principal surface of the material. This reading gives the relative density of the material under test.

A-8.2 Pyknometer Method

A-8.2.0 *Principle*—The method involves: (a)measurement of the mass, at 27°C of test sample contained in a pyknometer, (b)determination of this volume by measuring the corresponding mass in water at 27°C and (c)calculation of the ratio of the mass of the sample to its volume.

A-8.2.1 *Apparatus*

A-8.2.1.1 *Wide neck pyknometer*—Capacity 50ml; the channel of the stopper not being a capillary tube.

A-8.2.1.2 *Water-bath*—maintained at 27±0.5°C.

A-8.2.2 *Procedure*

A-8.2.2.1—Clean and dry the pyknometer together with its stopper and weigh to the nearest 0.001g. Fill the pyknometer with water boiled and cooled to 27±0.5°C and place it in the water-bath controlled at 27±0.5°C. Allow at least 30minutes for the temperature to reach equilibrium and remove the pyknometer from the water-bath, holding it by the neck; stopper it, wipe externally and remove the excess water from the upper part of the stopper. Weigh the pyknometer and its stopper to the nearest 0.001g and determine, by difference, the mass of water that it contains.

A-8.2.2.2 Empty the pyknometer, rinse it with alcohol or acetone and allow it to dry. Fill it with the test sample, previously adjusted to 27±0.5°C. avoiding the formation of air bubbles, especially when the solution has a high viscosity. Place the pyknometer in the water-bath at 27±0.5°C. Allow at least 30minutes for the temperature to reach equilibrium and weigh as in **A-8.2.2.1**

NOTE —If, during the procedure, the pyknometer is externally soiled by the solution under test, it should be washed with water at a temperature slightly below 27°C and then wiped.

A-8.2.3 *Calculation*

$$\text{Relative density at } 27/27°\text{C} = \frac{M_1}{M_2} \times d$$

where

M_1 = mass, in g, of the sample;

M_2 = mass, in g, of the same volume of water; and

d = density of water at 27°C.

A-9. DETERMINATION OF VISCOSITY

A-9.1 Apparatus

A-9.1.1 *Torsion Viscometer* —The instrument is based on the principle that the torque exerted by a liquid is proportional to its viscosity. It consists of two cylinders. The outer cylinder is connected to a motor and the inner one is suspended from a torsion spring, fitted with a pointer for measuring the torque.

A-9.2 Procedure—Maintain the outer water jacketed cylinder at a temperature of 27°C. Place the sample in the annular space between the two cylinders. Revolve the outer cylinder at a constant speed by the electric motor attached to it. The force transmitted through the sample exerts a torque on the inner cylinder. Read the torsion on the scale attached to the spindle which is calibrated in terms of viscosity. Report the result in centipoises at 27°C.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF SODIUM SILICATE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

B-1.1 Precautions shall be taken to protect the samples, sampling instruments and the containers for samples from adventitious contamination.

B-1.2 In case of sampling liquids from drums, the material shall be thoroughly mixed before drawing the sample. In case of solids, the material shall be sampled without breaking the lumps which are included in the sample drawn.

B-1.3 The samples shall be placed in suitable, clean and dry container with proper closures.

B-1.4 Each sample container shall be closed air-tight after filling 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 and drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of 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 drums to be chosen from a lot shall depend on the size of the lot (N) and shall be in accordance with col1 and 2of Table2.

TABLE2 NUMBER OF DURMS TO BE SELECTED

LOT SIZE	NO. OF DRUMS TO BE SELECTED
N n	
(1)	(2)
Upto 10	3
11 ,, 20	5
21 ,, 30	7
31 ,, 50	10
51and above	15

B-2.3 The drums to be selected for sampling shall be chosen from the lot at random. In order to ensure the randomness of selection, reference may be made to IS:4905-1968*. In case this standard is not readily available, the following procedure may be adopted:

Starting from any drum in the lot, count them in one order as 1,2,3,....., up to r and so on where $r$is the integral part of N/n. Every rth drum so counted, shall be withdrawn till the requisite number of drums is obtained from the lot.

B-3. TEST SAMPLES AND REFEREE SAMPLES

B-3.1 From each of the drums selected according to **B-2.3,** a small representative portion of the material shall be drawn with the help of a suitable sampling implement. The sample weighing approximately equal to thrice the quantity required for testing all the requirements given in the specification, shall be withdrawn after mixing the contents of the drum thoroughly and allowing the liquid to come to rest.

B-3.2 Out of these portions, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient for carrying out triplicate determinations for all the characteristics tested on the composite sample. From this composite sample, sample solution shall be prepared as prescribed in **A-2**. In case sieving is necessary, the portions shall be sieved separately and the composite sample shall be prepared only out of sieved material. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

*Methods for random sampling.

B-3.3 The remaining portion of the material from each container shall be divided into three equal parts each forming individual sample. One set of individual samples representing *n* containers sampled shall be marked for the purchaser another for the supplier and the third to be used as a referee sample.

B-3.4 All the individual and composite samples shall be transferred to separate sample containers which shall be sealed air-tight after sealing and marked with full identification particulars.

B-3.5 The referee test samples consisting of a composite sample and a set of *n* individual samples shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed between the purchaser and the supplier to be used in case of any dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for total soluble silicates and the ratio for total alkalinity shall be carried out on three individual samples drawn according to **B-2.3**. In case more than three individual samples are taken from a lot, three individual samples shall be randomly chosen, out of them. The same samples may be used for both the tests. The preparation of sample solution shall be done in accordance with **A-2.**

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

B-5. CRITERIA FOR CONFORMITY

B-5.1 The lot shall be declared as conforming to the requirements of the specification if all the test results on individual as well as composite sample(s) satisfy the relevant specification requirements, otherwise not.

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AMENDMENT NO.1 MAY 1992
TO
IS 9601:1980 SPECIFICATION FOR SODIUM SILICATE FOR COSMETIC INDUSTRY

[*Page* 4, *Table* 1, *Sl No.* (viii), *col* 3] — Substitute 'as agreed to between the buyer and seller' *for* '120 to 200'.

(PCD 19)

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