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IS 9681 (1980): Stearic Acid For Cosmetic Industry [PCD 19: Cosmetics]

“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”
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“Knowledge is such a treasure which cannot be stolen”
Indian Standard

SPECIFICATION FOR
STEARIC ACID FOR COSMETIC INDUSTRY

(First Reprint OCTOBER 1997)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3

March 1981
AMENDMENT NO. 1 DECEMBER 1998
TO
IS 9681 : 1980 SPECIFICATION FOR STEARIC ACID
FOR COSMETIC INDUSTRY

(Page 3, clause 2.1, line 5) — Insert the following at the end of the sentence:

'No unpleasant odour shall be observed.'

(PCD 19)

Reprography Unit, BIS, New Delhi, India
Indian Standard

SPECIFICATION FOR
STEARIC ACID FOR COSMETIC INDUSTRY

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(Continued on page 2)
IS : 9681 - 1980

(Continued from page 1)

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AMENDMENT NO. 2 MAY 2002
TO
IS 9681 : 1980 SPECIFICATION FOR STEARIC ACID
FOR COSMETIC INDUSTRY

(Page 1, clause 2.2) — Insert the following new clause after 2.2:

'2.3 Colour — The colour of the oil shall not be inferior than 2.0 Y + 0.5 R
Lovibond units when measured in 18 inch cell according to the method
prescribed in IS 1448 [P:13] : 1960 or +25 Saybolt units according to the method
prescribed in IS:1448 [P:14]:1960. Alternately, the colour of the oil shall be as
agreed to between the purchaser and the supplier.'

[ Page 4, Table 1, Sl No. (viii) ] — Delete.

(PCD 19)

Reprography Unit, BIS, New Delhi, India
Indian Standard

SPECIFICATION FOR
STEARIC ACID FOR COSMETIC INDUSTRY

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 6 January 1980, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Stearic acid is used in manufacture of various products, such as rubber, textile auxiliaries, metallic stearates, plastics, leather goods and cosmetics. Requirements of different types of stearic acid for various uses have been covered in IS:1675-1971*. However, the requirements for its use in cosmetic industry were not adequately covered in this standard and, therefore, a separate standard has been formulated to assist manufacturers of cosmetics in procuring material of the requisite quality by them.

0.3 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 stearic acid for cosmetic industry.

2. REQUIREMENTS

2.1 Description — Stearic acid shall be the product obtained from the solid fatty acids by the hydrolysis of various fats and subsequent distillation. It shall be in the form of powder, flakes or slabs. It shall be free from adulterants and foreign matter. It shall be glossy and crystalline to the naked eye. When melted, it shall form a clear and transparent liquid.

2.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Appendix A and IS:548 (Part I)-1964‡.

---

*Specification for stearic acid, technical (first revision).
†Rules for rounding off numerical values (revised).
‡Methods of sampling and test for oils and fats: Part I Methods of sampling, physical and chemical tests (revised).
### TABLE 1 REQUIREMENTS FOR STEARIC ACID FOR COSMETIC INDUSTRY

(Clause 2.2)

<table>
<thead>
<tr>
<th>Sl No.</th>
<th>Characteristic</th>
<th>Requirement</th>
<th>Method of Test, Ref to Cl No. in Appendix A</th>
<th>IS:548 (Part I)-1964*</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1)</td>
<td></td>
<td>(2)</td>
<td>(3)</td>
<td>(4)</td>
</tr>
<tr>
<td>i)</td>
<td>Saponification value</td>
<td>203 to 212</td>
<td>—</td>
<td>15</td>
</tr>
<tr>
<td>ii)</td>
<td>Acid value, shall not differ from saponification value by more than</td>
<td>3</td>
<td>—</td>
<td>7†</td>
</tr>
<tr>
<td>iii)</td>
<td>Iodine value (Wijs), Max</td>
<td>3.0</td>
<td>—</td>
<td>14</td>
</tr>
<tr>
<td>iv)</td>
<td>Titré, °C</td>
<td>53 to 56</td>
<td>—</td>
<td>12</td>
</tr>
<tr>
<td>v)</td>
<td>Heavy metals (as Pb), parts per million, Max</td>
<td>20</td>
<td>A-2</td>
<td>—</td>
</tr>
<tr>
<td>vi)</td>
<td>Sulphated ash, percent by mass, Max</td>
<td>0.1</td>
<td>A-3</td>
<td>—</td>
</tr>
<tr>
<td>vii)</td>
<td>Mineral acidity</td>
<td>To pass the test</td>
<td>A-4</td>
<td>—</td>
</tr>
<tr>
<td>viii)</td>
<td>Colour reading in 5½ in cell on the Lovibond scale, Max (on melted sample)</td>
<td>2.0 Y +0.5 R</td>
<td>—</td>
<td>13</td>
</tr>
</tbody>
</table>

*Methods of sampling and test for oils and fats: Part I Methods of sampling, physical and chemical test (revised).

†Ensure complete dissolution and titrate while hot.

---

3. PACKING AND MARKING

3.1 Packing — The material shall be supplied in suitable containers as agreed to between the purchaser and the supplier. The containers shall be securely closed.

3.2. Marking — Each container shall be marked legibly and indelibly with the following information:

a) Name of the material;

b) Manufacturer’s name and/ his recognized trade-mark, if any;

c) Gross, tare and net mass;

d) Date of manufacture; and

e) Batch number, in code or otherwise, to enable the lot of manufacture to be traced from records.
3.2.1 The containers may also be marked with the Standard Mark.

3.2.2 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and 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 Representative samples of the material shall be drawn and conformity of the material to the requirements of the standard shall be determined according to procedure prescribed in Appendix B.

APPENDIX A

( Clause 2.2, and Table 1 )

METHODS OF TEST FOR STEARIC ACID FOR COSMETIC INDUSTRY

A-1. QUALITY OF REAGENTS

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

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

A-2. TEST FOR HEAVY METALS

A-2.1 Apparatus

A-2.1.1 Nessler Cylinders — 50-ml capacity.

A-2.2 Reagents

A-2.2.1 Concentrated Hydrochloric Acid — See IS: 265-1976†.

A-2.2.2 Concentrated Nitric Acid — See IS: 264-1976‡.

*Specification for water for general laboratory use (second revision).
†Specification for hydrochloric acid (second revision).
‡Specification for nitric acid (second revision).
A-2.2.3 Acetic Acid — 1 N.

A-2.2.4 Ammonium Chloride

A-2.2.5 Standard Lead Solution — Dissolve 1·600 g of lead nitrate in water, add 1 ml of concentrated nitric acid and make up the volume to 1 000 ml. Pipette out 10 ml of the solution and dilute again to 1 000 ml with water. One millilitre of this solution contains 0·01 mg of lead (as Pb).

A-2.2.6 Hydrogen Sulphide Solution — freshly prepared saturated solution.

A-2.3 Procedure — Weigh accurately about 10 g of the material into a platinum crucible (or sillica dish), which has been previously ignited, cooled in the desiccator and weighed. Dry the material in the crucible at 90±2°C for 2 hours. Then heat over a low flame and ignite the contents gently. Incinerate the residue in a muffle furnace at 550±10°C until free from carbon. Preserve the ash.

A-2.3.1 Extract the ash, as obtained in A-2.3 with 1 ml of concentrated hydrochloric acid and transfer to an evaporating dish after washing a number of times with water. Add 0·5 ml of concentrated nitric acid and evaporate to dryness over a steam-bath. Dissolve the residue in 20 ml of water and 8 ml of acetic acid, and dilute to 250 ml with water. Mix well and filter. Transfer 25 ml of the filtrate to the Nessler cylinder and add 1 g of ammonium chloride in it. Carry out a control test in another Nessler cylinder using 2·0 ml of standard lead solution in place of the material add 1 g of ammonium chloride and 1 ml of acetic acid. To each Nessler cylinder add 10 ml hydrogen sulphide solution, dilute to the mark and shake well. Compare the intensity of colour produced in the two cylinders.

A-2.3.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour obtained with the material is not greater than that obtained in the control test.

A-3. DETERMINATION OF SULPHATED ASH

A-3.1 Apparatus

A-3.1.1 Platinum Crucible (or Silica Dish)

A-3.1.2 Desiccator — containing an efficient desiccant such as fused calcium chloride.

A-3.2 Procedure — Weigh accurately about 2-3 g of the material in a tared crucible. Ignite gently at first until thoroughly charred then cool and moisten the residue with 1 ml of concentrated sulphuric acid. Ignite gently until the carbon is completely consumed, then heat strongly. When the carbon has completely disappeared, cool the crucible in a desiccator and weigh.
A-3.3 Calculation

Sulphated ash, percent by mass = \( \frac{m \times 100}{M} \)

where

\( m \) = mass in g of the residue obtained, and

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

A-4. TEST FOR MINERAL ACIDITY

A-4.1 Reagents

A-4.1.1 Petroleum Ether — 60°/80°C grade.

A-4.1.2 Methyl Orange Indicator — 0.05 percent (m/v) solution.

A-4.2 Procedure — Measure 10 ml of the melted sample into a separating funnel and shake intimately with three successive 10-ml portions of hot water. The temperature of the hot water should be more than the melting point of stearic acid. Combine the aqueous extracts, transfer to another separating funnel and remove traces of fatty acids in the water by extraction with petroleum ether. Test the aqueous extract with a few drops of methyl orange indicator.

A-4.2.1 The material shall be taken to have passed the test if the indicator does not show acid reaction.

APPENDIX B

(Clause 4.1)

SAMPLING OF STEARIC ACID FOR COSMETIC INDUSTRY

B-1. GENERAL REQUIREMENTS OF SAMPLING

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

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.
B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in clean, dry glass-stoppered bottles.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and date of manufacture of the material.

B-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

B-2. SCALE OF SAMPLING

B-2.0 Samples to determine conformity of the material to this standard shall be selected in accordance with the procedure given below. However, the purchaser and the supplier may agree to adopt any other procedure.

B-2.1 Lot — All the containers in a single consignment of one type of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

B-2.2 The number of containers to be selected from a lot shall depend upon the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

<table>
<thead>
<tr>
<th>Lot Size</th>
<th>Number of Containers to be Selected</th>
</tr>
</thead>
<tbody>
<tr>
<td>(N)</td>
<td>(n)</td>
</tr>
<tr>
<td>(1)</td>
<td>(2)</td>
</tr>
<tr>
<td>Up to 5</td>
<td>All*</td>
</tr>
<tr>
<td>6 ,, 65</td>
<td>5</td>
</tr>
<tr>
<td>66 ,, 110</td>
<td>7</td>
</tr>
<tr>
<td>Over 110</td>
<td>10</td>
</tr>
</tbody>
</table>

*When the lot size is 5 or less, the test results of each of the samples shall meet the corresponding requirements.
B-2.3 These containers shall be selected at random from the lot. In order to ensure the randomness of selection, reference may be made to IS:4905-1968*. In case this standard is not readily available, following procedure may be adopted.

' Starting from any container, in the lot, count them as 1, 2, 3........ up to r and so on, where r is the integral part of (N/n). Every rth container thus counted shall be withdrawn till the requisite number of containers is obtained.'

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Draw with an appropriate sampling instrument small portions of the material from different parts of the selected containers, the total quantity being sufficient to carry out triplicate determination of all characteristics given in Table 1.

B-3.2 Mix thoroughly all portions of the material drawn from the same container to form an individual sample to represent the container. Equal quantities from the selected containers shall be mixed together to form a composite sample to represent the lot.

B-3.3 All the individual samples representing the selected containers and the composite sample representing the lot shall be divided into three equal parts, thus forming three sets of test samples. These parts shall be immediately transferred to thoroughly dried bottles which shall then be sealed air-tight with glass stoppers. These shall be labelled with all the particulars of sampling given in B-1.7. One set of the test samples shall be sent to the purchaser and another to the supplier.

B-3.4 Referee Sample — The third set of the test samples, bearing the seals of the purchaser and the supplier shall constitute the referee sample to be used in case of dispute between the purchaser and the supplier. It shall be kept at a place agreed to between them.

B-4. NUMBER OF TESTS

B-4.1 The tests for saponification value, acid value and titre shall be carried out on each individual sample of the set of test samples (see B-3.3).

B-4.2 The tests for the remaining characteristics shall be carried out on the composite sample of the test samples (see B-3.3).

B-5. CRITERIA FOR CONFORMITY

B-5.1 The lot shall be declared as conforming to the requirements of the standard if it satisfies the criteria given in B-5.2, B-5.3 and B-5.4.

*Methods for random sampling.
B-5.2 The test results for saponification value and titre value shall be recorded as shown in Table 3. The mean and range shall be calculated as follows and shall be recorded in col 3 and 4 of the Table 3 respectively.

\[
\text{Mean } (\bar{X}) = \frac{\text{The sum of the test results}}{\text{Number of test results}}
\]

\[
\text{Range } (R) = \text{The difference between the maximum and the minimum values of the test results.}
\]

B-5.2.1 For conformity, the value of expressions \(\bar{X} \pm 0.6 R\) shall satisfy the criteria as given under col 5 of Table 3.

<table>
<thead>
<tr>
<th>SL No.</th>
<th>Characteristic</th>
<th>Test Results</th>
<th>Criteria for Conformity</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1)</td>
<td></td>
<td>Mean</td>
<td>Range</td>
</tr>
<tr>
<td>i)</td>
<td>Saponification value</td>
<td>(\bar{X}_1)</td>
<td>(R_1)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>The value of (\bar{X}_1 \pm 0.6 R_1) shall lie between 203 and 212.</td>
</tr>
<tr>
<td>ii)</td>
<td>Titre value, °C</td>
<td>(\bar{X}_2)</td>
<td>(R_2)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>The value of (\bar{X}_2 \pm 0.6 R_2) shall lie between 53 and 56.</td>
</tr>
</tbody>
</table>

B-5.3 All the test results for acid value on each of the individual samples satisfy the corresponding requirements given in Table 1 of the standard.

B-5.4 The composite sample when tested for the remaining requirements (excluding saponification value, acid value and titre) satisfies the corresponding requirements as specified in Table 1.
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