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“Step Out From the Old to the New”

IS 553 (1984): Rosin (gum rosin) [CHD 20: Paints, Varnishes and Related Products]
Indian Standard

SPECIFICATION FOR ROSIN (GUM ROSIN)

(Second Revision)

Second Reprint AUGUST 2002

UDC 665.944

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

Gr 7  December 1984
AMENDMENT NO. 2  SEPTEMBER 1998
TO
IS 553 : 1984 SPECIFICATION FOR
ROSIN (GUM ROSIN)
(Second Revision)

(Page 10, Appendix A, clause A-1.1) — Substitute the following for the existing:

'A.1.1 The colour grade of rosin is determined by comparing a representative sample of size 22 mm × 22 mm × 22 mm with the same size of Rosin Standards.'

(Page 10, Appendix A, clause A-1.2, lines 3 and 4) — Substitute the following for the existing:

'designed for the preparation of rosin sample having a size of 22 mm × 22 mm × 22 mm cut pieces of rosin.'

(PCD 18)
AMENDMENT NO. 1 MARCH 1996
TO
IS 553 : 1984 SPECIFICATION FOR ROSIN (GUM ROSIN)
(Second Revision)

(Page 3, Foreword, clause 0.3) — Add the following Note after clause 0.3:

'NOTE — International Fragrance Research Association, Geneva, Switzerland recommends that gum rosin should not be used in fragrance compounds.'

(PCD 18)

Reprography Unit, BIS, New Delhi, India
Indian Standard

SPECIFICATION FOR
ROSIN ( GUM ROSIN )

(Second Revision)

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(Continued on page 2)

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DR VINOD K. JAIN
Assistant Director (P & C), ISI
Indian Standard
SPECIFICATION FOR
ROSIN ( GUM ROSIN )
( Second Revision )

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 10 March 1984, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1955 and subsequently revised in 1969. In this second revision, original 15 colour grades as per ASTM D-509-70 (Reapproved) 1975 Methods of sampling and grading rosin as officially recognized by USDA are incorporated along with Gardner Colour Scale as per ASTM D-1544-68 (Reapproved) — 1974. Methods for colour of transparent liquids (gardner colour scale). In addition extra pale grade has also been included and saponification number requirement has been added.

0.3 There are three classes of rosins manufactured all over the world, namely: (i) Gum rosin, obtained by steam distillation of oleoresin gums of various Pinus species, (ii) Wood rosin, obtained by solvent extraction and distillation of aged pine stump wood, and (iii) Tall oil rosin, obtained by distillation of crude tall oil which is a by-product in the manufacture of kraft paper by the sulphate process. In India, rosin or colophony is obtained as the residual solid matter in the steam distillation of the oleoresin gum, exudate of Pinus roxbur ghii and other allied Pinus species grown in the country. Gum rosin thus is the main class of rosin manufactured.

0.4 The present standard prescribes requirements only for gum rosin as obtained from oleoresin gum of Indian pines. It also includes requirements of rosin for rubber, soap and other specialised industries where rosin finds potential applications.

0.4.1 Besides, this revision incorporates a major change concerning introduction of original 15 colour grades as per ASTM D-509-1970
(Reapproved 1975) Method of sampling and grading rosin as officially recognized by USDA (United States Department of Agriculture), namely, XC, XB, XA, X, WW, WG, N, M, K, I, H, G, F, E, D. The official U.S. Rosin Standards (Master Set No. 200) is available only with USDA; however, secondary sets of Lovibond U.S. Rosin Standards Set No. AF 163/15 of M/s Lovibond Tintometer contains 15 Rosin Standards as specified in ASTM D-509-1970 (Reapproved 1975). These standards are excellent duplicates of official U.S. Rosin Standards but could not be classified as identical. The Lovibond Standards do not have official recognition by USDA; however a certificate can be obtained from Naval Stores, Branch of Agriculture Marketing Service attesting to the degree with which a set compares visually with the Master Set No. 200 of the official U.S. Rosin colour standards are manufactured by M/s Tintometer Ltd, Salisbury, England.

0.4.2 This standard also prescribes additional colour scale based on Gardner Colour Scale as per ASTM D-1544-1968 (Reapproved 1974) Methods for colour of transparent liquids (Gardner Colour Scale). The Gardner Colour Values (1-18) can be directly interpreted into approximate rosin colour standards with the help of a conversion table. The Gardner colour values are used all over the world by manufacturers of rosin and its derivatives. The detailed method of colour determination is given in Appendix A. Gardner colour discs having values 1-18 can also be obtained from M/s Tintometer Ltd, Salisbury, England.

0.4.3 In preparing this revised standard, assistance regarding determination of acid number and saponification number, had been obtained from ASTM D-465-1959 (Reapproved 1975) Methods of test for acid number of rosin and ASTM D-464-1959 (Reapproved 1975) Methods of test for saponification number of rosin.

0.5 For residue on sieve, Indian Standard Sieves (conforming to IS:460-1962* ) are prescribed. Where IS Sieves are not available, other equivalent standard sieves may be used.

0.6 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.

*Specification for test sieves (revised).
†Rules for rounding off numerical values (revised).
1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for rosin (gum rosin). The material is mainly used in paper, soap, cosmetics, paint, varnish, rubber and polish industries.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS : 354-1976* and IS : 326-1968† shall apply.

3. TYPES AND GRADES

3.1 Types — The material shall be of the following four types:
   a) Extra pale,
   b) Pale,
   c) Medium, and
   d) Dark.

3.2 Grades — There shall be a total of 15 colour grades under 4 types of the material, the colour standards being approximately equivalent to Gardner Colour Values as given herein below:

<table>
<thead>
<tr>
<th>Type</th>
<th>Grade</th>
<th>Gardner Colour Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>a) Extra pale</td>
<td>XC</td>
<td>Less than 6</td>
</tr>
<tr>
<td></td>
<td>XB</td>
<td></td>
</tr>
<tr>
<td></td>
<td>XA</td>
<td></td>
</tr>
<tr>
<td></td>
<td>X</td>
<td>6</td>
</tr>
<tr>
<td>b) Pale</td>
<td>WW</td>
<td>7</td>
</tr>
<tr>
<td></td>
<td>WG</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td>N</td>
<td>9</td>
</tr>
<tr>
<td>c) Medium</td>
<td>M</td>
<td>9-10</td>
</tr>
<tr>
<td></td>
<td>K</td>
<td>10-11</td>
</tr>
<tr>
<td></td>
<td>I</td>
<td>11</td>
</tr>
<tr>
<td></td>
<td>H</td>
<td>12</td>
</tr>
<tr>
<td>d) Dark</td>
<td>G</td>
<td>13</td>
</tr>
<tr>
<td></td>
<td>F</td>
<td>14</td>
</tr>
<tr>
<td></td>
<td>E</td>
<td>16</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>18</td>
</tr>
</tbody>
</table>

---

*Methods of sampling and test for resins for paints (*first revision*).
†Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).
4. REQUIREMENTS

4.1 Description — The material shall be derived from the oleoresin gums of the pines and shall be in the form of transparent or slightly transparent brittle lumps with a glassy fracture. It shall be free from more than traces of visible particules of the dirt or other extraneous foreign matter suspended in the solid mass. Slightly specky, opaque, crystallized or cloudy rosin may be acceptable when these conditions are not of a degree to prevent the true evaluation of the grade according to the colour.

4.2 Solubility — The material shall be wholly soluble in alcohol (see IS : 321-1964*), ether (see IS : 336-1973†), petroleum hydrocarbon solvent (see IS : 1745-1978‡), and oil of turpentine (see IS : 533-1973§) under slight warming.

4.3 The material shall also comply with the requirements given in Table 1.

4.4 Special Requirements for Rubber, Soap and Other Specialized Uses — The material if required for specialized uses as in the rubber, soap and other industries in addition to the requirements prescribed under 4.1, 4.2 and Table 1, shall also comply with the requirements prescribed in Table 2.

5. PACKING AND MARKING

5.1 Packing — The material shall be packed as specified in the contract or order.

5.2 Marking — The containers shall be marked with the name, type and grade of the material, manufacturer's name and trade-mark, quantity packed and the lot or batch number.

5.2.1 The containers shall also be marked 'LEAD FREE' in black or white letters, whichever is more legible, the letters being not less than 25 mm high (see Table 2, Sl No. IV).

* Specification for absolute alcohol (revised).
† Specification for other (second revision).
‡ Specification for petroleum hydrocarbon solvents (second revision).
§ Specification for gum spirit of turpentine (oil of turpentine) (first revision).
### TABLE 1 REQUIREMENTS FOR ROSIN (GUM ROSIN)

*(Clause 4.3)*

<table>
<thead>
<tr>
<th>SL No.</th>
<th>CHARACTERISTIC</th>
<th>REQUIREMENT FOR TYPE</th>
<th>METHOD OF TEST, REF TO</th>
<th>Clause in Indian Standard</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Extra Pale</td>
<td>Pale</td>
<td>Medium</td>
</tr>
<tr>
<td>(1)</td>
<td>(2)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>i)</td>
<td>Colour</td>
<td>Equivalent to Gardner colour values (3.2) or matching colour of corresponding permanent Lovibond US rosin colour standards</td>
<td>A</td>
<td></td>
</tr>
<tr>
<td>ii)</td>
<td>Softening point, °C, Min</td>
<td>70</td>
<td>70</td>
<td>70</td>
</tr>
<tr>
<td>iii)</td>
<td>Relative density†, 27°/27°C</td>
<td>1.05 to 1.052 0</td>
<td>1.052 0 to 1.082 0</td>
<td>B</td>
</tr>
<tr>
<td>iv)</td>
<td>Acid number, Min</td>
<td>160</td>
<td>155</td>
<td>155</td>
</tr>
<tr>
<td>v)</td>
<td>Saponification number, Min</td>
<td>165</td>
<td>160</td>
<td>160</td>
</tr>
<tr>
<td>vi)</td>
<td>Volatile matter, percent by mass, Max</td>
<td>1.50</td>
<td>2.0</td>
<td>2.0</td>
</tr>
<tr>
<td>vii)</td>
<td>Ash content, percent by mass, Max</td>
<td>0.05</td>
<td>0.2</td>
<td>0.5</td>
</tr>
<tr>
<td>viii)</td>
<td>Matter insoluble in toluene, percent by mass, Max</td>
<td>0.10</td>
<td>0.40</td>
<td>1.00</td>
</tr>
<tr>
<td>ix)</td>
<td>Unsaponifiable matter, percent by mass, Max</td>
<td>6.0</td>
<td>6.0</td>
<td>6.0</td>
</tr>
</tbody>
</table>

*Methods of sampling and test for resins for paints (first revision).*

†Relative density is the term adopted for specific gravity with water as reference substance by the International Organisation for Standardization (ISO).

‡Methods of sampling and test for drying oil for paints (second revision).
### TABLE 2 ADDITIONAL REQUIREMENTS OF ROSIN FOR RUBBER, SOAP AND OTHER SPECIALIZED USES

(Clause 4.4)

<table>
<thead>
<tr>
<th>SL No.</th>
<th>CHARACTERISTIC</th>
<th>REQUIREMENT</th>
<th>METHOD OF TEST, REF TO Appendix Clause of Indian Standard</th>
</tr>
</thead>
<tbody>
<tr>
<td>i)</td>
<td>Copper content (as Cu), ppm, \textit{Max}</td>
<td>8</td>
<td>F</td>
</tr>
<tr>
<td>ii)</td>
<td>Manganese content (as Mn), ppm, \textit{Max}</td>
<td>10</td>
<td>G</td>
</tr>
<tr>
<td>iii)</td>
<td>Iron content (as Fe), percent by mass, \textit{Max}</td>
<td>0.01</td>
<td>H</td>
</tr>
<tr>
<td>iv)</td>
<td>Freedom from lead or compounds of lead [calculated as metallic load (Pb) in ppm], \textit{Max}</td>
<td>0.03</td>
<td>19 of IS : 33-1976*</td>
</tr>
<tr>
<td>v)</td>
<td>Acidity (of aqueous extract)</td>
<td>Not acidic to methyl orange</td>
<td>—</td>
</tr>
<tr>
<td>vi)</td>
<td>Freedom from grit (tested on ash) [see also Table 1, Item (vii)]</td>
<td>To pass 63-micron IS Sieve</td>
<td>—</td>
</tr>
</tbody>
</table>

*Methods of sampling and test for inorganic pigments and extenders for paints (second revision).

5.2.2 The product may also be marked with Standard mark.

5.2.2.1 The use of the Standard Mark is governed by the provisions of the \textit{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.
6. SAMPLING

6.1 Representative samples shall be taken from the lot according to the procedure given under 3 of IS : 354-1976*.

6.2 Preparation of Test Samples and Referee Samples

6.2.1 From each of the selected containers, remove the top surface to a depth of 10 cm with the help of a suitable sampling instrument. From the exposed surface, draw a sample of 600 g of resin at different depths. The collected material shall be broken to small lumps and kept in separate sample containers.

6.2.2 Out of the material drawn from individual containers, a small but approximately equal quantity of material shall be taken and mixed together to give a composite sample weighing about 150 g. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

6.2.3 The remaining portion of the material from each container shall be divided into three equal parts, each conforming an individual sample. One set of individual samples representing various containers shall be for the purchaser, another for the supplier and the third for the referee.

6.2.4 All the individual and composite samples shall be transferred to separate sample containers. These containers shall then be sealed airtight with stoppers and labelled with full identification particulars.

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

6.3 Number of Tests

6.3.1 Tests for colour, softening point and special requirements for rubber, soap and other specialized uses (as given in Table 2) shall be conducted on each of the individual samples separately.

6.3.2 Tests for the determination of all the remaining characteristics shall be conducted on the composite sample.

6.4 Criteria for Conformity

6.4.1 The lot shall be declared as conforming to the requirements of the specification if 6.4.1.1 and 6.4.1.2 are satisfied.

*Methods of sampling and test for resins for paints (first revision).
6.4.1.1 For the characteristics tested on individual samples (see 6.3.1), all the test results on each of the individual samples satisfy the corresponding specification requirements.

6.4.1.2 All the test results on the composite sample meet the other relevant requirements (see 6.3.2) as given in the specification.

7. TEST METHODS

7.1 Tests shall be conducted as prescribed in appendices and various Indian Standards given in col 6 and 7 of Table 1 and col 4 and 5 of Table 2.

7.2 Quality of Reagents — 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.

APPENDIX A

[ Clause 0.4.2 and Table 1, Item (i) ]

DETERMINATION OF COLOUR

A-1. PROCEDURE

A-1.1 The colour grade of rosin is determined by comparing a representative similar shaped sample with the appropriate Lovibond US Rosin Standards.

A-1.1.1 The colour comparison should be made visually with open direct north sky light, or in a direction which excludes direct rays from the sun from passing through the rosin sample to the eye. A suitable colour grading cabinet can be designed to hold sample and the colour standard.

A-1.2 Preparation of Sample — A PVC cube mold having identical dimensions as compared to Lovibond US Rosin Standards can be designed for the preparation of rosin sample having a similar shape or similar shaped cut pieces of rosin.

It sometimes happens that a sample of rosin is received in lumps too small to allow the cutting of an identical piece as per standard. In these cases, melt 15 to 30 g of small lumps with freshly prepared surfaces in a small beaker to a consistency at which it freely flows, 140 to

*Specification for water for general laboratory use (second revision).
160°C and pour into the colour mold. The sample should be protected from oxidation by heating under vacuum or in an inert atmosphere of carbon dioxide or nitrogen and care should be taken not to heat rosin sample to too high a temperature, nor to hold at the elevated temperature an unnecessary length of time because the colour of rosin is affected by heating at high temperature due to oxidation. It is advisable to stir the rosin with a thermometer during the heating.

Allow the rosin mold to come to room temperature and determine the Lovibond US Rosin Colour equivalent as above.

**A-1.2.1** The report of results should always state whether a cut cube (sample cut from a large lump of rosin) or a poured cube (a cube prepared by melting up small lumps of the rosin) was used for the determination.

**A-1.2.2** Designate the colour of the sample by the letter of the colour standard which it equals. If it is even slightly darker than one of the standards, designate it by the letter of the next darker standard*. Should the colour or appearance of the rosin be such that there is a doubt whether the rosin is as light or bright as the standard, view the sample and the standard in a suitable colorimeter. Then in case of uncertainty persisting, give the rosin the benefit of doubt. For example, if a rosin sample is definitely darker than the WW standard, but lighter than the WG standard, it is WG Grade. If, on the other hand, the grade is undecided, that is, there is a reasonable doubt or uncertainty whether the total colour of the rosin is 'as good as' or equals, or is darker than that of the WW standard, the grade is WW.

**A-1.2.3** A sample showing two distinct colours, usually evidenced by darker streaks through the rosin, indicates a mixture of rosins in the package, in which case the darkest part of the sample or darkest rosin to be found in the package determines the grade.

**A-1.2.4** A check sample taken from close to the bottom of a barrel or drum shall not be taken to be representative, because a natural variation in colour is sometimes found between the top and bottom rosin and also because there exists a reduced brightness of the bottom rosin due to a settling of finely-divided suspended matter usually present in normal

---

*The colour of a sample of rosin is made up of three attributes: hue, saturation and brightness. 'Hue' alludes to the characteristic described by the words 'yellow' or 'red'. 'Saturation' describes the purity or strength of the yellowness or redness of the colour. 'Brightness' depends on the relative amount of light transmitted by the rosin. The cleanliness of the rosin affects its transparency, and therefore, its brightness. The terms 'lighter than' and 'darker than' are convenient to describe the difference between a sample and a standard. The rosin may be darker than the standard in one or more of the following ways: (a) a redder hue, (b) a colour of greater saturation, and (c) a lower brightness.
gum rosin. Should such bottom sample show not more than one grade lower than the top sample, consider the latter to be the grade of the rosin in the barrel. If, however, the bottom sample is more than one grade lower than the top sample double filling or 'mixed-packing' is indicated. In that case, fix the grade of the rosin in the package according to the bottom sample.

A-2. PROCEDURE

A-2.1 Outline of the Method — A 50 percent w/w solution of rosin sample under test in pure colourless toluene is taken in a 10 ml Lovibond test tube and is visually compared with Standard Gardner Colour solution, 1 to 18 prepared from potassium dichromate and concentrated sulphuric acid also taken in an identical 10 ml Lovibond test tube in a Lovibond Comparator under standard daylight conditions, Lovibond Gardner Colour Discs 1-18 may also be used.

A-2.1.1 Preparation of Standard Gardner Colour Solutions

A-2.2 Apparatus

A-2.2.1 Erlenmeyer Flask — 250-ml capacity
A-2.2.2 Burettes — 50-ml capacity.
A-2.2.3 Test Tube — 150 × 19 mm.

NOTE — All glass apparatus is to be thoroughly cleaned with chromic acid, washed and dried before use.

A-2.3 Reagent

A-2.3.1 Concentrated Sulphuric Acid — A.R. grade (see IS : 266-1977*).
A-2.3.2 Potassium Dichromate — A.R. grade.

A-2.4 Procedure

A-2.4.1 Weigh accurately 3 g of the powdered A.R. grade potassium dichromate in the thoroughly clean Erlenmeyer flask and slowly add 100 ml of A.R. grade concentrated sulphuric acid. Occasionally shake the contents to allow dissolution of potassium dichromate (30 min). A clean dark brown stock solution of potassium dichromate in concentrated sulphuric acid 3 percent (w/w) (0.03 g/ml) is thus obtained. Transfer the solution in a thoroughly clean and dry burette* and take A.R. grade sulphuric acid in the other burette. Prepare the different Gardner Colour solutions in test tubes from the stock solution by diluting with concentrated sulphuric acid as per quantities of potassium dichro-

*Specification for sulphuric acid (second revision).
mate (K₂Cr₂O₇) per 100 ml concentrated sulphuric acid given in the following table using the formula:

\[
\text{Quantity of stocks solution (ml) required for 20 ml of standard gardner solution} = 6.666 \times X
\]

where 'X' is the mass of potassium dichromate required for any standard colour given below:

<table>
<thead>
<tr>
<th>Concentration of Potassium Dichromate, g/100 ml of Sulphuric Acid</th>
<th>Gardner Colour Standards (see Note)</th>
<th>US Rosin Standards</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.003 9</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>0.004 8</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>0.007 1</td>
<td>3</td>
<td></td>
</tr>
<tr>
<td>0.011 2</td>
<td>4</td>
<td></td>
</tr>
<tr>
<td>0.020 5</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>0.032 2</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>0.038 4</td>
<td>7</td>
<td>X</td>
</tr>
<tr>
<td>0.044 9</td>
<td>7–8</td>
<td>WW</td>
</tr>
<tr>
<td>0.051 5</td>
<td>8</td>
<td>W—WG</td>
</tr>
<tr>
<td>0.078 0</td>
<td>9</td>
<td>N</td>
</tr>
<tr>
<td>0.121 0</td>
<td>9–10</td>
<td>M</td>
</tr>
<tr>
<td>0.164 0</td>
<td>10</td>
<td>M–K</td>
</tr>
<tr>
<td>0.207 0</td>
<td>10–11</td>
<td>K</td>
</tr>
<tr>
<td>0.250 0</td>
<td>11</td>
<td>I</td>
</tr>
<tr>
<td>0.380 0</td>
<td>12</td>
<td>H</td>
</tr>
<tr>
<td>0.572 0</td>
<td>13</td>
<td>G</td>
</tr>
<tr>
<td>0.763 0</td>
<td>14</td>
<td>F</td>
</tr>
<tr>
<td>1.041 0</td>
<td>15</td>
<td>—</td>
</tr>
<tr>
<td>1.280 0</td>
<td>16</td>
<td>E</td>
</tr>
<tr>
<td>2.220</td>
<td>17</td>
<td>—</td>
</tr>
<tr>
<td>3.000</td>
<td>18</td>
<td>D</td>
</tr>
</tbody>
</table>

NOTE — Gardner colour standards should be stored in dark. They can be safely used without deterioration for about a month. Fresh standards shall be made if a greenish tinge is developed in the standards.

A-2.5 Preparation of Sample Solution

A-2.5.1 Apparatus

A-2.5.2 Beaker — 50 ml.

A-2.5.3 Lovibond Tube — 10 ml.

A-2.5.4 Lovibond Comparator
A-2.6 Reagent

A-2.6.1 Toluene — conforming to IS : 537-1967*.

A-2.7 Procedure — Powder a representative sample of rosin quickly in a pestle and mortar and prepare about 20 to 50 percent (w/w) solution in pure toluene in a beaker. Dissolve the solids in cold only agitating with the help of a glass rod, evaporation of toluene is to be made up with fresh toluene. Transfer the clear solution in a 10 ml Lovibond test tube. Take respective gardner colour solution standards also in 10 ml Lovibond test tubes and place them along with the test solution in a Lovibond comparator one by one, and visually compare the colour against standard daylight till the colour is found matching. The colour of the test solution is given the gardner colour standard value with which it matches approximately. The corresponding US Rosin standard can then be obtained from the conversion table.

APPENDIX B

[Table 1, Item (iii)]

DETERMINATION OF RELATIVE DENSITY

B-0. GENERAL

B-0.1 Outline of the Method — The relative density of the material is determined by hydrostatic method.

B-1. APPARATUS

B-1.1 Balance — equipped with a pan straddle or other stationary support.

B-2. PROCEDURE

B-2.1 Take a clean piece of rosin weighing about 1 g and having a shape suitable for hanging it securely with a piece of fine waxed silk thread sufficiently long to reach from the hook on one of the pan supports to the straddle or rest. Attach the test specimen to the thread so as to be suspended about 25 mm above the straddle from the hook on the pan support, and weigh to the nearest 0.1 mg. Again weigh the specimen, still suspended by the thread but completely immersed in freshly boiled distilled water at 27° ± 0.2°C, to the nearest 0.1 mg, adhering air bubbles being first removed with a fine wire.

*Specification for toluene, pure, nitration grade (first revision).
B-3. CALCULATION

B-3.1 Calculate the relative density of the material as follows:

\[
\text{Relative density at } 27^\circ/27^\circ C = \frac{M_1}{M_1 - M_2}
\]

where

\[M_1 = \text{mass of the sample in air (g), and} \]
\[M_2 = \text{mass of the sample in water (g).}\]

APPENDIX C

[Table 1, Item (iv)]

DETERMINATION OF ACID NUMBER

C-1. APPARATUS

C-1.1 Erlenmeyer Flask — 250-ml capacity.

C-2. REAGENTS

C-2.1 Alkali, Standard Solution (0.5 N) — Dissolve either 22 g of sodium hydroxide (NaOH) or 33 g of potassium hydroxide (KOH) (pellets or sticks) in water or in alcohol conforming to C-2.2, and dilute to 1 litre with the same solvent. Standardize to ±0.001 N.

C-2.2 Ethyl Alcohol — Neutral 95 percent ethyl alcohol conforming to IS: 323-1959* or neutral rectified denatured spirit (conforming to IS: 324-1959†).

C-2.3 Phenolphthalein Indicator Solution — Dissolve 1 g of phenolphthalein in 100 ml of alcohol conforming to C-2.2.

C-2.4 Thymol Blue Indicator Solution — Dissolve 0.1 g of thymol blue in 100 ml of alcohol conforming to C-2.2.

C-3. PROCEDURE

C-3.1 Transfer 3.95 to 4.05 g of the sample weighed to the nearest 0.0001 g to a 250-ml Erlenmeyer flask, and add 100 ml of neutral alcohol.

*Specification for rectified spirit (revised).
†Specification for ordinary denatured spirit (revised).
C-3.2 Heat, if necessary, to dissolve the rosin cool to room temperature (*see Note*), and titrate the solution with the standard alkali solution using 1 ml of either phenolphthalein or thymol blue indicator solution; with phenolphthalein, titrate to the first persistent faint pink colour and with thymol blue, titrate to a distinct blue.

**NOTE**— If the rosin does not remain in solution when it cools, use 200 ml of alcohol instead of 100 ml.

**C-4. CALCULATION**

C-4.1 Calculate the acid number of the rosin expressed as milligrams of potassium hydroxide per gram of sample, as follows and report to the nearest whole number.

\[
\text{Acid number} = \left( \frac{AN \times 56.1}{B} \right)
\]

where

- \(A\) = millilitres of alkali solution required for titration of the sample,
- \(N\) = normality of the alkali solution, and
- \(B\) = quantity of the sample used (g).

**APPENDIX D**

[Table 1, Item (v)]

**DETERMINATION OF SAPONIFICATION NUMBER**

**D-1. APPARATUS**

D-1.1 *Erlenmeyer Flask* — Capacity, 300 ml of alkali resistant glass with standard taper glass joint.

D-1.2 *Reflux Condenser* — with standard taper joint to fit the Erlenmeyer flask.

**D-2. REAGENTS**

D-2.1 *Ethyl Alcohol* — *See C-2.2.*

D-2.2 *Phenolphthalein Indicator Solution* — *See C-2.3.*

D-2.3 *Potassium Hydroxide, Alcoholic Solution (33 g/1)* — Dissolve 33 g of potassium hydroxide (KOH) pellets or sticks in alcohol conforming to C-2.1 and dilute to 1 litre with alcohol. If insoluble matter is present, filter the solution. The strength of this solution will be approximately 0.5 N and should be slightly less than that of the 0.5 N sulphuric acid (\(H_2SO_4\)).
D-2.4 Sulphuric Acid Standard (0.5 N) — Dilute 14 ml of concentrated sulphuric acid to 1 litre with water and standardize.

D-2.5 Thymol Blue Indicator Solution — See C-2.4.

D-3. PROCEDURE

D-3.1 Transfer 3.95 to 4.05 g of the sample weighed to the nearest 0.001 g to the Erlenmeyer flask. Dissolve the sample in 50 ml of neutral alcohol, warming the contents of the flask, if necessary. Pippete 50 ml of alcoholic potassium hydroxide solution (33 g/l) into the flask, allowing the pippet to drain in a definite time.

D-3.2 Connect the flask to a reflux condenser, place it on a hot plate, and boil the solution gently for exactly 1 hour, starting with the point of incipient boiling.

D-3.3 Blanks — Conduct two or three blank determinations simultaneously following the same procedure and using 50 ml portions of neutral alcohol and 50 ml portions of alcoholic potassium hydroxide solution (33 g/l). Use the same pippet and drain for the same length of time as for the sample.

D-3.4 Cool the solutions and titrate with the standard sulphuric acid using 1 ml of either phenolphthalein or thymol blue indicator. With thymol blue, the end-point is indicated when the colour undergoes the first change from a distinct blue to a yellowish green, just short of clear yellow.

D-4. CALCULATION

D-4.1 Calculate the saponification number, expressed as milligrams of potassium hydroxide per gram of sample as follows and report the results to the nearest whole number:

\[
\text{Saponification number} = \left( \frac{B - A}{C} \right) \times 56.1
\]

where

\[
A = \text{quantity of sulphuric acid required for titration of the sample (ml)},
\]

\[
B = \text{quantity of sulphuric acid required for titration of the blank (ml)},
\]

\[
N = \text{normality of the sulphuric acid, and}
\]

\[
C = \text{quantity of the sample used (g)}.
\]
APPENDIX E

DETERMINATION OF MATTER INSOLUBLE IN TOLUENE

E-0. GENERAL

E-0.1 Outline of the Method — A weighed quantity of the material is refluxed in toluene. The undissolved matter is filtered out, dried, weighed and reported as percentage by mass of the material taken up for test.

E-1. APPARATUS

E-1.1 Round-Bottom Flask — 100-ml capacity.

E-1.2 Reflux Condenser

E-1.3 Sintered Glass Crucible — porosity grade 3.

E-2. REAGENT

E-2.1 Toluene — conforming to IS : 537-1967*.

E-3. PROCEDURE

E-3.1 Weigh accurately about 10 g of the powdered material in the round-bottom flask and to this add 50 ml of toluene. Reflux the contents using reflux condenser for one hour. Cool, filter out the insoluble material into the tared sintered glass crucible using warm toluene. Transfer the contents from the flask to the sintered glass crucible and also wash the residue until a few drops of the filtrate yields no residue on evaporation.

E-3.2 Dry the residue in an air-oven at a temperature of 100° ± 2°C for one hour.

E-4. CALCULATION

E-4.1 Calculate and express the mass of the residue as percentage by mass of the material taken for test.

\[
\text{Matter insoluble in toluene, percent by mass} = \frac{M_1}{M_2} \times 100
\]

where

\[M_1 = \text{mass of the residue (g), and}\]
\[M_2 = \text{mass of the material taken for test (g)}\]

*Specification for toluene, pure, nitration grade (first revision).
APPENDIX F

[Table 2, Item (i)]

DETERMINATION OF COPPER CONTENT

F-0. GENERAL

F-0.1 Outline of the Method — The ash of the material is extracted with a hydrochloric/nitric acid mixture and made alkaline with ammonium hydroxide. After removing the iron complex formed in presence of ammonium citrate, the aqueous solution is shaken with solution of diethyldithiocarbamate in chloroform to form a yellow copper complex. The optical density of this solution is measured photometrically which is proportional to the concentration of copper.

F-1. APPARATUS

F-1.1 Photoelectric Absorptiometer — filter-photometer or spectro-photometer.

F-1.2 Silica Crucibles — 50-or 80-ml capacity.

F-2. REAGENTS

F-2.1 Hydrochloric Acid — Nitric Acid Mixture — Mix the following together:

1 volume of nitric acid (see IS : 264-1976*),
2 volumes of hydrochloric acid (see IS : 265-1976†), and
3 volume of water.

F-2.2 Citric Acid Solution — 50 g of solid citric acid dissolved in 100 ml of water.

F-2.3 Ammonium Hydroxide — 20 percent (m/m).

F-2.4 Concentrated Sulphuric Acid — see IS : 266-1977‡.

F-2.5 Zinc Diethyldithiocarbamate Reagent — 1 g of solid zinc diethyldithiocarbamate is dissolved in 1 litre of chloroform. If zinc diethyldithiocarbamate is not available, the reagent may be prepared as follows:

One gram of sodium diethyldithiocarbamate is dissolved in water to which 2 g of zinc sulphate are then added. The resulting zinc

---

*Specification for nitric acid (second revision).
†Specification for hydrochloric acid (second revision).
‡Specification for sulphuric acid (second revision).
diethyldithiocarbamate is extracted by shaking with 100 ml of chloroform and the chloroform solution is separated and diluted to one litre. Stored in amber-coloured bottle, this reagent is stable for at least six months.


F-2.7 Standard Copper Solution — 0.393 g of copper sulphate pentahydrate (CuSO₄·5H₂O) is weighed into a small beaker and dissolved in water. To this 3 ml of concentrated sulphuric acid are added. This is transferred to 1 000-ml volumetric flask and diluted with water to graduation mark to form the stock solution. Ten millilitres of this stock solution is pipetted into a 1 000-ml volumetric flask and diluted with water to the graduation mark. This solution contains the equivalent of 0.01 mg/ml of copper and shall be freshly prepared from stock solution when required.

F-3. PROCEDURE

F-3.1 Weigh accurately about 5 g of the material in a silica dish and heat gently over a Bunsen flame until the rosin ignites. Allow the rosin to burn away as far as possible without the application of external heat and then heat the crucible to constant mass in a muffle furnace maintained at 500 to 550°C.

F-3.2 Moisten the contents of the crucible with 0.5 to 1 ml of water, then add 10 ml of dilute hydrochloric acid-nitric acid mixture and cover the crucible with a glass, and heat on a steam-bath for 30 to 60 min. Wash the contents of the crucible into a small beaker or flask, add 5 ml of citric acid solution and then add ammonium hydroxide dropwise until the solution is just alkaline to litmus paper. Cool the solution by immersion in running water, or dropwise transfer to a separating funnel and add a further 2 ml of ammonium hydroxide and then dilute to about 40 ml with water. Pipette out 25 ml of zinc diethyl-dithiocarbamate reagent into the solution and shake for 2 min. After separation, draw the chloroform layer immediately into a stoppered flask containing about 0.1 g anhydrous sodium sulphate. If turbidity persists after standing for about 30 min, make further small additions of anhydrous sodium sulphate until the solution becomes clear.

F-3.3 Photometric Measurement of Colour — Decant the chloroform solution through a plug of glass wool, or a small filter paper, into the cell of a photoelectric absorptiometer and measure the optical density approximately at 435 nm. Correct the reading by subtracting the value for the optical density of the blank solution and from the corrected reading and the calibration curve of the instrument, obtain the concentration of copper in the test solution.

*Specification for sodium sulphate, anhydrous (second revision).
F-3.3.1 Prepare a calibration curve as follows:

Prepare a series of standard copper solutions ranging from 1 to 10 ml followed by 5 ml of citric acid solution and 2 ml excess of ammonium hydroxide. Dilute the solutions to 40 ml with water, extract with 25 ml zinc diethyldithiocarbamate reagent, and measure the optical density of each dried chloroform extract. Correct reading for each standard solution by subtracting the blank value and obtain the calibrated curve by plotting the relationship between copper concentration and optical density.

NOTE — The calibration curve should be checked as necessary depending on local conditions and on the type of instrument used.

APPENDIX G

[ Table 2, Item (ii) ]

DETERMINATION OF MANGANESE CONTENT

G-0. GENERAL

G-0.1 Outline of the Method — The material is incinerated to ash and treated with potassium hydrogen sulphate and sulphuric acid to convert the manganese to a soluble form. Any iron present is complexed with orthophosphoric acid and the manganese oxidized to permanganate by boiling with potassium periodate. The optical density of the solution measured photometrically is proportional to the concentration of manganese.

G-1. APPARATUS

G-1.1 Photoelectric Absorptiometer — filter photometer or spectrophotometer.

G-1.2 Silica Crucibles — nominal capacity 50 or 80 ml.

G-2. REAGENTS

G-2.1 Sulphuric Acid, Dilute — one volume of concentrated acid is mixed with 19 volumes of water.

G-2.2 Potassium Hydrogen Sulphate

G-2.3 Orthophosphoric Acid — 85 to 95 percent purity.

G-2.4 Stabilised Water — About 0.1 g potassium permanganate is dissolved in 1 litre of water to which a few drops of sulphuric acid have been added. The water is distilled through an effective spray trap discarding the first and last 50 ml of the distillate. The rest of the distillate is collected and stored in a glass-stoppered bottle.
G-2.5 Potassium Permanganate Solution — approximately 0.001 N.

G-2.6 Standard Manganese Solution — prepared as follows.

G-2.6.1 Into a small beaker, 0.720 g potassium permanganate is weighed and dissolved in water containing 2 ml of sulphuric acid. Sulphur dioxide water is added until the solution is colourless. The solution is boiled for 15 minutes, cooled, transferred to a 500-ml volumetric flask and diluted to the mark. Ten millilitres of this solution is pipetted into a second 500-ml flask and again diluted to the mark. This dilute solution contains the equivalent of 0.01 mg/ml of manganese (Mn) and shall be freshly prepared from the stock solution when required.

G-2.6.2 Into a small beaker, 0.770 g manganese sulphate (MnSO₄, 4H₂O) is weighed and dissolved in water containing 2 ml sulphuric acid. The solution is transferred to a 500-ml volumetric flask and diluted to mark. This solution shall be stable for at least a month. Ten millilitres of this solution is pipetted into a second 500-ml flask and again diluted to the mark. This dilute solution contains the equivalent of 0.01 mg/ml of manganese (as Mn) and shall be freshly prepared from the stock solution when required.

G-3. PROCEDURE

G-3.1 Weigh accurately about 5 g of the material in a silica crucible and heat gently over a Bunsen flame until the material ignites. Allow the material to burn away as far as possible without the application of external heat and then heat the crucible to constant mass in a muffle furnace maintained at 500 to 550°C.

G-3.2 Add to the ash about 20 ml of dilute sulphuric acid, and heat the crucible on a steam-bath until the materials are dissolved or loosen from the sides of the crucible. Wash the contents in a small beaker using a glass rod to dislodge undissolved solid and boil the solution gently until no more dissolves. Filter the solution through a sintered glass filter pad into a small conical flask, wash the filter and insoluble material with two or three portions of water. Add 3 ml of orthophosphoric acid to the solution in the flask making further additions of 1 ml as may be necessary to remove any yellow colour due to iron. To the solution, add 0.3 g potassium periodate and boil. Maintain at the boiling point for 10 min. Maintain the temperature above 90°C for further 10 min to ensure full development of permanganate colour. After cooling, transfer the solution to a 50 ml volumetric flask and dilute to the mark with stabilised water at 27°C. After mixing the colour should be stable for several hours; any tendency to fading indicates incomplete removal of organic matter or chloride.
G-3.3 Rinse the cells of the photoelectric absorptiometer first with potassium permanganate solution and then with stabilized water. Rinse one cell with the test solution and the other with blank and fill each cell with the appropriate solution. Measure the optical density of the test solution against the blank as reference solution at a wavelength of approximately 525 μ. Calculate the concentration of manganese from the observed optical density and the calibration graph of the instrument.

G-3.4 Prepare a calibration graph as follows:

Make up a series of standard solutions, each containing 20 ml dilute sulphuric acid, 3 ml orthophosphoric acid and 25 ml stabilized water. Add increasing volumes of 0, 2.0, 4.0, 6.0, 8.0 and 10.0 ml of standard manganese solutions and treat exactly as described for the test solution finally cooling and diluting to 50 ml in volumetric flasks. After rinsing the cells of the instrument first with potassium permanganate solution, then with stabilized water and finally by the appropriate test solutions, measure the optical density of each solution at about 525 μ. Correct all readings for the blank value and obtain the calibration curve by plotting the relationship between concentration of manganese and optical density.

NOTE 1 — The calibration curve shall be checked as necessary depending on conditions and on the type of instrument used.

NOTE 2 — All precautions and safeguards required for carrying out trace metal analysis shall be observed.

APPENDIX H

[ Table 2, Item (iii) ]

DETERMINATION OF IRON CONTENT

H-0. GENERAL

H-0.1 Outline of the Method — The iron content is estimated colorimetrically by developing colour with thioglycollic acid and comparing with a standard solution.

H-1. APPARATUS

H-1.1 Nessler Cylinders — 50-ml capacity, conforming to IS : 4161-1967*.

*Specification for Nessler cylinders.
H-2. REAGENTS

H-2.1 Sodium Bisulphate

H-2.2 Dilute Hydrochloric Acid — approximately 5 N.

H-2.3 Citric Acid Solution — approximately 10 percent (\textit{m/m}).

H-2.4 Thioglycollic Acid

H-2.5 Concentrated Ammonium Hydroxide — 30 percent (\textit{m/m}).

H-2.6 Standard Iron Solution — Dissolve 0.604 g of ferric ammonium sulphate [\(\text{FeNH}_4(\SO_4)_2\cdot12\text{H}_2\text{O}\)] in 10 ml of dilute sulphuric acid (10 percent) and dilute with water to 1 000 ml; 1 ml of this solution is equivalent to 0.1 mg of iron (as Fe\(_2\)O\(_3\)).

H-3. PROCEDURE

H-3.1 Weigh accurately about 5 g of the material and ignite at red heat and then fuse with requisite amount of sodium bisulphate in order to render the whole ash soluble in dilute hydrochloric acid. Dissolve this in dilute hydrochloric acid, filter and wash the residue on the filter paper thoroughly with water. Make up the volume of the filtrate to 1 000 ml with water. Transfer 10 ml of this solution to a Nessler cylinder and dilute to 50 ml. To carry out a blank test, dilute 3 ml of dilute hydrochloric acid to 50 ml; add 10 ml of this dilute acid to another Nessler cylinder, and dilute to 50 ml.

H-3.1.1 To each Nessler cylinder, add 5 ml of the citric acid solution, 3 drops of thioglycollic acid and ammonium hydroxide dropwise until the solution is alkaline. Allow to stand for 5 min for the full intensity of colour to develop in the test solution.

H-3.1.2 To get approximate match, add standard iron solution dropwise from a semi-micro burette to the blank, until the intensity of colour obtained matches with that produced in the test solution (allowing time after each addition for the complete development of colour). Note the volume of standard iron solution in one operation. If the colour obtained does not match with that of the test solution, repeat this procedure with different volumes of standard iron solution until the colours matches.
H-3.2 Calculation

H-3.2.1 Calculate the iron content expressed as iron oxide, percent by mass, as follows:

\[
\text{Iron (as Fe}_2\text{O}_3\text{), percent by mass} = \frac{V}{M}
\]

where

- \( V = \) volume of standard iron solution required in the blank (ml), and
- \( M = \) mass of the material taken for test (g).
Essential Oils and Resinoids Subcommittee, PCDC 18 : 3

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Central Laboratory:
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Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113 254 13 15
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'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001 550 13 48
Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road, BANGALORE 560058 839 49 55
Commercial-cum-Office Complex, Opp. Dushera Maidan, E-5 Arera Colony, Bittan Market, BHOPAL 462016 72 34 52
62/63, Ganga Nagar, Unit VI, BHUBANESWAR 751001 40 36 27
5th Floor, Koval Towers, 44 Bala Sundaram Road, COIMBATORE 641018 21 88 35
Plot No. 58, Neelam Bata Road, NIT, FARIDABAD 121001 542 82 61
Savitri Complex, 116 G.T. Road, GHAZIABAD 201001 471 19 98
53/5 Ward No. 29, R.G. Barua Road, 5th By-lane, Apurba Sinha Path, GUWAHATI 781003 541137
5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001 320 10 84
E-52, Chitrnanjan Marg, C- Scheme, JAIPUR 302001 37 38 79
117/418 B, Sarvodaya Nagar, KANPUR 208005 21 68 76
Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road, LUCKNOW 226001 21 89 23
NIT Building, Second Floor, Gokulpal Market, NAGPUR 440010 52 51 71
Mahabir Bhawan, 1st Floor, Ropar Road, NALAGARH 174101 2 14 51
Patliputra Industrial Estate, PATNA 800013 26 28 0???
First Floor, Plot Nos. 657-660, Market Yard, Gultekdi, PUNE 411037 426 86 59
'Sahajanand House' 3rd Floor, Bhaktinagar Circle, 80 Feet Road, RAJKOT 360002 37 82 51
T.C. No. 14/1421, University P. O. Palayam, THIRUVANANTHAPURAM 695034 32 21 04

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