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IS 7679 (1978): Hair Creams [PCD 19: Cosmetics]
Indian Standard

SPECIFICATION FOR HAIR CREAMS

(First Revision)

First Reprint JULY 2007
(Including Amendment Nos. 1, 2 & 3)

UDC 668.585.4

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

Gr 4

July 1978
AMENDMENT NO. 3  FEBRUARY 2001
TO
IS 7679 : 1978  SPECIFICATION FOR HAIR CREAMS
( First Revision )

[ Page 6, clause 4.2(e) and Amendment No. 1 ] — Substitute the following
for the existing:

e) Best use before...............(Month and year to be declared by the manufacturer).

NOTE — This is exempted in case of pack sizes of 10 g/25 ml or less and if the shelf life of
the product is more than 24 months.

f) List of key ingredients

NOTE — This is exempted in case of pack sizes of 30 g/60 ml or less.

( PCD 19 )

Reprography Unit, BIS, New Delhi, India
AMENDMENT NO. 3 FEBRUARY 2001
TO
IS 7679 : 1978 SPECIFICATION FOR HAIR CREAMS
(First Revision)

[Page 6, clause 4.2(e) and Amendment No. 1] — Substitute the following for the existing:

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   NOTE — This is exempted in case of pack sizes of 10 g/25 ml or less and if the shelf life of
   the product is more than 24 months.

f) List of key ingredients

   NOTE — This is exempted in case of pack sizes of 30 g/60 ml or less.

( PCD 19 )
AMENDMENT NO. 2 NOVEMBER 1998
TO
IS 7679 : 1978 SPECIFICATION FOR HAIR CREAMS
(First Revision)

(Page 1, Foreword, clause 0.4) — Insert the following clause after 0.4 and renumber the subsequent clauses accordingly:

'0.5 A scheme for labelling environment friendly products known as ECO Mark has been introduced at the instance of the Ministry of Environment and Forests (MEF), Government of India. The ECO Mark is being administered by the Bureau of Indian Standards (BIS) under the Bureau of Indian Standards Act, 1986 as per the Resolution No. 71 dated 21 February 1991 and No. 768 dated 24 August 1992 published in the Gazette of the Government of India. For a product to be eligible for marking with ECO logo, it shall also carry the Standard Mark of BIS besides meeting additional environment friendly requirements. For this purpose, the Standard Mark of BIS would be a single mark being a combination of the BIS monogram [ISI] and the ECO logo. Requirements for ECO friendliness will be additional, manufacturing units will be free to opt for Standard Mark alone also.

This amendment is based on the Gazette Notification No. 170 dated 18 May 1996 for hair creams environment friendly products published in the Gazette of India. This amendment is, therefore, being issued to this standard to include environment friendly requirements for hair creams.'

(Page 5, clause 3.3) — Insert the following clauses after 3.3 and renumber the subsequent clauses:

3.4 Additional Requirement for ECO Mark

3.4.1 General Requirements

3.4.1.1 The product shall conform to the requirements for quality, safety and performance prescribed under 3.1 to 3.3.

3.4.1.2 All the ingredients that go into formulation of cosmetics shall comply with the provisions of IS 4707 (Part 1) : 1988 'Classification for cosmetic raw materials and adjuncts : Part 1 Dyes, colours and pigments (first revision)' and IS 4707 (Part 2) : 1993 'Classification for cosmetic raw materials and adjuncts: Part 2 List of raw materials generally not recognized as safe (first revision)'.

1
Amend No. 2 to IS 7679 : 1978

The product shall also meet specific requirements as given in the standard.

3.4.1.3 The product package shall display a list of ingredients in descending order of quantity present.

3.4.1.4 The product shall not be manufactured from any carcinogenic ingredients.

3.4.1.5 The manufacturer shall produce to BIS the environmental consent clearance from the concerned State Pollution Control Board as per the provisions of the Water (Prevention and Control of Pollution) Cess Act, 1977 and the Air (Prevention and Control of Pollution) Act, 1981 along with the authorization, if required under the Environment (Protection) Act, 1986 and the Rules made thereunder, while applying for ECO Mark. Additionally, provisions of the Drugs and Cosmetics Act, 1940 and the Rules thereunder shall also be complied with.

3.4.2 Specific Requirements

3.4.2.1 Product shall be dermatologically safe when tested as prescribed in IS 4011 : 1997 ‘Methods of test for safety evaluation of cosmetics (second revision)’.

3.4.2.2 Heavy metals calculated as lead (Pb) and arsenic (As₂O₃) shall not exceed 20 and 2 ppm, respectively when tested by the respective method prescribed in Indian Standards.

( Page 6, clause 4.1 ) — Insert the following clause after 4.1 and renumber the subsequent clauses:

‘4.2 The material for produce packaging shall meet the parameters envolled under the scheme of labelling environment friendly packaging/packaging materials.’

( Page 6, clause 4.3.1 ) — Insert the following clause after 4.3.1:

‘4.4 The product package shall be suitably marked that ECO Mark label is applicable only to the contents, if the product package is not separately covered under the ECO Mark scheme.’

( PCD 19 )
AMENDMENT NO. 1 NOVEMBER 1998
TO
IS 7679 : 1978 SPECIFICATION FOR HAIR CREAMS
(First Revision)

[Page 6, clause 4.2(d)] — Insert 'e' after 'd':
e) 'Best use before ................. (Month and year to be declared by manufacturer)'.

(PCD 19)
Indian Standard
SPECIFICATION FOR HAIR CREAMS
(First Revision)

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(Continued on page 2)
(Continued from page 1)

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Indian Standard

SPECIFICATION FOR HAIR CREAMS

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 26 May 1978, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first issued in 1975. The present revision has been undertaken to incorporate a new procedure for the microbiological examination of hair creams.

0.3 Hair creams which contain ingredients that have an effect on the physiological functions of the body or the scalp or the hair, or for which therapeutic claims are made, are not covered by this standard.

0.4 Hair creams are intended to provide to the hair good grooming, lustre without greasiness, protection from wind, rain, heat, etc, and some degree of hair conditioning. The major property required for conditioning is ‘moisturizing’. For this purpose, moisture must be added, but direct application of water is of little benefit, since evaporation is equally rapid and an equilibrium is soon reached. A means has to be provided to keep the absorbed water from evaporating. The emulsions of oil and water prove useful in this respect. The emulsion must break down readily on application to hair, and not re-emulsify. Moisture in these cases is absorbed and the oil or fat forms a protective film on the hair shaft, greatly minimizing the damage by natural elements.

0.5 It is necessary that the raw materials used are such that in the concentrations in which they would be present in the hair cream, after interaction with other raw materials, they are free from any harmful effects. For determining the dermatological safety, reference may be made to IS: 4011-1967*. It shall be the responsibility of the manufacturer of the hair cream to satisfy himself of the dermatological safety of his formulation according to that standard before releasing the product for sale.

*Methods for dermatological tests for cosmetics.
0.5.1 Manufacturers of hair creams should also ensure that hair creams should be, as far as possible, free from microbiological contamination. It is recommended that skin cream on microbiological examination should not give more than 1000 micro-organism per gram when tested by the method prescribed in Appendix A.

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, 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 for hair creams and other oil-based emulsion preparations for the hair. These include water-in-oil and oil-in-water emulsions.

1.1.1 This standard does not cover hair oils, brilliantines and pomades.

2. TYPES

2.1 There shall be four types of hair creams, namely:

a) Type 1 — based on vegetable oil emulsion,

b) Type 2 — based on mineral oil emulsion,

c) Type 3 — based on vegetable-mineral oil emulsion, and

d) Type 4 — based on any combination of the above with fatty acids/fatty acid esters emulsion.

2.2 Some typical formulations of hair creams are given for information in Appendix B.

3. REQUIREMENTS

3.1 Description — The hair cream shall be in the form of a thick emulsion or unctuous mass. It shall be white or uniformly coloured, with or without perfume.

3.2 Ingredients — Unless specified otherwise, all the raw materials used in the manufacture of hair creams shall conform to the requirements prescribed in the relevant Indian Standards where such standards exist.

*Rules for rounding off numerical values (revised).
3.2.1 The dyes, if used in the manufacture of hair creams, shall comply with the provisions of IS : 4707 ( Part I )-1968* subject to the provisions of the Drugs and Cosmetics Act and Rules issued by the Government of India.

3.2.2 Ingredients other than dyes shall comply with the provisions of IS : 4707 ( Part II )-1973†.

3.2.2.1 For Types 1, 3 and 4 — The oil or oils used as the base shall be of the quality specified below:

a) Castor Oil — conforming to Grade First Special of IS : 435-1973‡,

b) Coconut oil — conforming to Refined Grade of IS : 542-1968§,

c) Groundnut Oil — conforming to Refined Grade of IS : 544-1968||,

and

d) Sesame Oil — conforming to Refined Grade of IS : 547-1968¶.

3.2.2.2 For Types 2, 3 and 4 — The base oil shall conform to IS : 7299-1974**.

3.2.3 A list of ingredients conventionally used in the formulation of hair creams is given, for guidance, in Appendix C.

3.3 The hair cream shall comply with the requirements given in Table 1 when tested as given in col 4 of the table.

### TABLE 1 REQUIREMENTS FOR HAIR CREAMS

<table>
<thead>
<tr>
<th>SL No.</th>
<th>CHARACTERISTIC</th>
<th>REQUIREMENT</th>
<th>METHOD OF TEST (REF TO CL NO, IN APPENDIX D)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>i)</td>
<td>Thermal stability</td>
<td>To pass the test</td>
<td>D-1</td>
</tr>
<tr>
<td>ii)</td>
<td>pH</td>
<td>5.0 to 9.0</td>
<td>D-2</td>
</tr>
<tr>
<td>iii)</td>
<td>Total fatty substance content, percent by mass, Min</td>
<td>15</td>
<td>D-3</td>
</tr>
<tr>
<td>iv)</td>
<td>Water content, percent by mass, Max</td>
<td>85</td>
<td>D-4</td>
</tr>
<tr>
<td>v)</td>
<td>Test for rancidity</td>
<td>Shall be free from rancidity</td>
<td>D-5</td>
</tr>
</tbody>
</table>

*Classification of cosmetic raw materials and adjuncts, Part I.
†Classification of cosmetic raw materials and adjuncts, Part II.
‡Specification for castor oil (second revision).
§Specification for coconut oil (second revision).
||Specification for groundnut oil (second revision).
¶Specification for sesame oil (second revision).
**Specification for mineral oil for cosmetic industry.
4. PACKING AND MARKING

4.1 The cream shall be packed in suitable well-closed containers.

4.2 The containers shall be legibly marked with the following information:
   a) Name of the material;
   b) Manufacturer’s name and/or recognized trade-mark, if any;
   c) Net mass of the material; and
   d) Batch number, in code or otherwise, to enable the lot of manufacture to be traced back from records.

4.2.1 The product may also be marked with Standard Mark.

4.2.2 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 manufactures or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in IS : 3958-1966*.

5.2 Tests for all characteristics shall be carried out on the composite sample.

5.3 The material shall be taken to have conformed to the specification if the composite sample passes all the tests.

6. TEST METHODS

6.1 Tests shall be carried out as prescribed in Appendices A and D.

6.2 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.

†Specification for water for general laboratory use ( second revision ).
APPENDIX A
( Clauses 0.5.1 and 6.1 )

MICROBIOLOGICAL EXAMINATION OF HAIR CREAMS

A-1. OUTLINE OF THE METHOD
A-1.1 The test consists of plating a known dilution of the sample on soyabean casein digest agar medium suitable for the total count of aerobic bacteria and fungi after incubating them for a specified period to permit the development of visual colonies for counting.

A-2. APPARATUS
A-2.1 Tubes — of resistant glass, provided with closely fitting metal caps.

A-2.2 Autoclaves — of sufficient size. They shall keep uniform temperature within the chamber up to and including the sterilizing temperature of 120°C. They shall be equipped with an accurate thermometer, located so as to register the minimum temperature within the sterilizing chamber, a pressure gauge and properly adjusted safety valves.

A-2.3 Petri Dishes — of 100 mm diameter and 15 mm depth. The bottom of the dishes shall be free from bubbles and scratches and shall be flat so that the medium is of uniform thickness throughout the plate.

A-2.4 Colony Counter — an approved counting aid, such as Quebec colony counter. If such counter is not available, counting may be done with a lens giving a magnification of 1.5 diameter. In order to ensure uniformity of conditions during counting, illumination equivalent to that provided by the Quebec colony counter shall be employed.

A-3. MEDIA AND BUFFER
A-3.1 Soyabean Casein Digest Agar Medium — Dissolve 15 g of pancreatic digest of casein, 5 g of papaic digest of soyabean meal, and 5 g of sodium chloride in 1000 ml of distilled water contained in a 2-litre beaker by heating in a water-bath. Add 15 g of powdered agar and continue boiling until the agar is completely digested. Adjust the pH to 7.5 with sodium hydroxide solution. Distribute in 20 ml quantities, close the tubes with metal caps and autoclave at 122°C for 20 minutes. After autoclaving, store the tubes in a cool place and use them within 3 weeks.
A-3.2 Stock Solution pH 7.2 Phosphate Buffer — Dissolve 34 g of monobasic potassium phosphate in about 500 ml of water contained in a 1000-ml volumetric flask. Adjust the pH to 7.2 ± 0.1 by the addition of sodium hydroxide solution (4 percent). Add water to volume and mix. Sterilize at 122°C for 20 minutes, store under refrigeration.

A-3.3 Diluted Phosphate Buffer Solution pH 7.2 — Dilute 1 ml of stock solution with distilled water in the ratio of 1:800. Fill 50 ml each in conical flasks of 100 ml capacity. Plug the flasks with cotton and sterilize at 122°C for 20 minutes.

A-4. STERILIZATION OF APPARATUS

A-4.1 Tubes — These shall be sterilized in the autoclave at a temperature of 122°C and 1.05 kg/cm² pressure for 20 minutes or in the hot air oven at 160°C for one hour.

A-4.2 Petri Dishes — These shall be packed in drums and autoclaved at 122°C and 1.05 kg/cm² pressure for 20 minutes or individually wrapped in kraft paper and sterilized in hot air oven at 160°C for one hour.

A-4.3 Pipettes — These shall be placed in pipette cones (copper, stainless steel or aluminium) after plugging the broader end with cotton and sterilized in the autoclave at 122°C and 1.05 kg/cm² pressure for 20 minutes or at 160°C for one hour in hot air oven.

A-5. PROCEDURE

A-5.1 Melt sufficient number of soyabean casein digest agar medium tubes in a hot water-bath and transfer while hot into a constant temperature water-bath maintained at 48 ± 2°C.

A-5.2 Weigh and transfer aseptically 1 g of the sample to conical flask containing sterile 50 ml of diluted phosphate buffer at pH 7.2. Shake well. Pipette out in 1-ml portions into three sterile petri dishes. Pour melted and cooled (at 45°C) soyabean casein digest agar medium over it, and rotate the plates to mix thoroughly. Incubate the plates at 32°C for 72 hours in an inverted position.

A-5.3 Determine the average number of colonies on soyabean casein digest agar medium plates and multiply by 30, the dilution factor. This will be the number of micro-organism per g of the sample. If no colony is recovered from any of the plate it can be stated as less than micro-organisms per g.
# APPENDIX B

(Clause 2.2)

**TYPICAL FORMULATIONS OF HAIR CREAMS**

<table>
<thead>
<tr>
<th>Type of Cream</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Mineral Oil Emulsion</strong></td>
<td></td>
</tr>
<tr>
<td>Mineral oil : 49 percent</td>
<td></td>
</tr>
<tr>
<td>Stearic acid : 1 percent</td>
<td></td>
</tr>
<tr>
<td>Lime water : 50 percent</td>
<td></td>
</tr>
<tr>
<td>Perfume and preservative : Q. S.</td>
<td></td>
</tr>
<tr>
<td><strong>Vegetable Oil Emulsion</strong></td>
<td></td>
</tr>
<tr>
<td>Diglycol stearate : 8 percent</td>
<td></td>
</tr>
<tr>
<td>Water : 60 percent</td>
<td></td>
</tr>
<tr>
<td>Castor oil : 16 percent</td>
<td></td>
</tr>
<tr>
<td>Almond oil : 16 percent</td>
<td></td>
</tr>
<tr>
<td>Perfume and preservative : Q. S.</td>
<td></td>
</tr>
<tr>
<td><strong>Mixed Vegetable and Mineral Oil Emulsion</strong></td>
<td></td>
</tr>
<tr>
<td>Mineral oil : 32 percent</td>
<td></td>
</tr>
<tr>
<td>Tegen : 10 percent</td>
<td></td>
</tr>
<tr>
<td>Water : 50 percent</td>
<td></td>
</tr>
<tr>
<td>Beeswax : 3 percent</td>
<td></td>
</tr>
<tr>
<td>Castor oil : 5 percent</td>
<td></td>
</tr>
<tr>
<td>Perfume and preservative : Q. S.</td>
<td></td>
</tr>
<tr>
<td><strong>Fatty Acid and Fatty Acid Ester</strong></td>
<td></td>
</tr>
<tr>
<td>Oleic acid : 20 percent</td>
<td></td>
</tr>
<tr>
<td>Beeswax : 1 percent</td>
<td></td>
</tr>
<tr>
<td>Lanolin : 0.5 percent</td>
<td></td>
</tr>
<tr>
<td>Lime water : 33.5 percent</td>
<td></td>
</tr>
<tr>
<td>Saccharated lime water : 5 percent</td>
<td></td>
</tr>
<tr>
<td>Olive oil : 40 percent</td>
<td></td>
</tr>
<tr>
<td>Perfume and preservative : Q. S.</td>
<td></td>
</tr>
</tbody>
</table>
APPENDIX C

(Clause 3.2.3)

LIST OF RAW MATERIALS CONVENTIONALLY USED IN FORMULATION OF HAIR CREAMS

1. Emulsion Formers
   a) Mineral oil
   b) Vegetable oil, for example, castor oil, olive oil, etc

2. Emulsifiers
   a) Beeswax
   b) Carnauba wax
   c) Borax
   d) Proprietary synthetic emulsifiers
   e) Soap based on magnesium, calcium or triethanolamine

3. Chelating Agents
   a) Sodium salt of EDTA
   b) Sodium polyphosphates

4. Preservatives and Antioxidants
   a) Ethyl, propyl, methyl and butyl parahydroxy benzoates
   b) Gallic acid
   c) Octyl and dodecyl gallate
   d) Alpha tocopheryl acetate
   e) Monooctadecyl ester of carboxymethyl mercapto succinic acid

5. Inorganic Salts
   a) Magnesium sulphate

6. Emollients
   a) Lanolin, lanolin esters, lanolin oils
   b) Fatty acid esters
   c) Alkanolamides
   d) Petroleum jelly
7. **Thickening Agents**
   a) Carbopol
   b) Sodium alginate
   c) Sodium C.M.C.

8. **Hair Setters**
   a) Gum tragacanth
   b) Gum karaya
   c) Shellac (water-soluble)

9. **Other Groups of Ingredients**
   a) Perfumes
   b) Bactericides or bacteriostat
   c) Dyes

### APPENDIX D
 (*Clauses 3.3 and 6.1, and Table 1*)

**METHODS OF TEST FOR HAIR CREAMS**

**D-1. TEST FOR THERMAL STABILITY**

**D-1.1 Apparatus** — a humidity chamber controlled at 60 to 70 percent RH and 37 ± 1°C.

**D-1.2 Procedure** — Spread a 20 mm broad and 5 mm thick stripe from the material to be tested on the internal wall of a beaker of 100 ml capacity in its total height. Keep the beaker for 8 hours in the humidity chamber at 60 to 70 percent relative humidity and temperature of 37 ± 1°C.

**D-1.3** The cream shall be taken to have passed the test if on removal from the thermostat, no oil separation is observable.

**D-2. DETERMINATION OF pH**

**D-2.1 Apparatus** — a pH meter, preferably equipped with a glass electrode.
D-2.2 Procedure

D-2.2.1 For Oil-in-Water Emulsion Creams — Weigh accurately 5 ± 0.01 g of the cream in a 100-ml beaker. Add 45 ml of water and disperse the cream in it. Determine the $pH$ of the suspension at 27°C using the $pH$ meter.

D-2.2.2 For Water-in-Oil Emulsion Creams — Weigh 10 ± 0.01 g of the cream. Add 90 ml of rectified spirit previously adjusted to $pH$ 6-5 to 7.0. Warm if necessary to 45°C and stir thoroughly for 15 minutes. Filter the alcoholic layer through a filter paper and measure the $pH$ of the filtrate at 27°C using the $pH$ meter.

D-3. DETERMINATION OF TOTAL FATTY SUBSTANCE CONTENT

D-3.0 Principle of the Method — The emulsion is broken up with dilute mineral acid and the fatty matter is extracted with petroleum ether. It is weighed after removal of the solvent.

D-3.1 Reagents

D-3.1.1 Dilute Hydrochloric Acid — 1 : 1 (v/v).

D-3.1.2 Ethyl Ether

D-3.1.3 Methyl Orange Indicator Solution — Dissolve 0.1 g of methyl orange in 100 ml of water.

D-3.1.4 Sodium Sulphate — desiccated.

D-3.2 Procedure — Weigh accurately about 2 g of the material into a conical flask, add 25 ml of dilute hydrochloric acid, fit a reflux condenser into the flask and boil the contents until the oil and water phases have separated. Pour the contents of the flask into a 300-ml separating funnel and allow it to cool to 20°C. Rinse the conical flask with 50 ml of ethyl ether in portions of 10 ml. Pour the ether rinsings into the separating funnel. Shake the separating funnel well and leave until the layers separate. Separate out the aqueous phase and shake it out with 50 ml portions of ether twice. Combine all the ether extracts and wash them with water until free of acid (when tested with methyl organge indicator solution). Filter the ether extracts through a filter paper containing sodium sulphate into a conical flask which has been previously dried at a temperature of 60 ± 2°C and then weighed. Wash the sodium sulphate on the filter with ether and dry the material remaining in the flask at a temperature of 60 ± 2°C to constant mass.
D-3.3 Calculation

Total fatty substance, percent by mass = $100 \times \frac{M_1}{M_2}$

where

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

$M_2 =$ mass in g of the material taken for the test.

D-4. DETERMINATION OF WATER

D-4.0 Methods — The toluene distillation is prescribed below. The use of Karl Fischer method (see IS: 2362-1973*) is permitted as an alternative.

D-4.1 Apparatus — The apparatus, shown in Fig. 1, consists of the following parts:

a) Flask — of 500 ml capacity, made of hard resistant glass.

b) Trap — The cylindrical portion of the receiving tube is 146 to 156 mm in length and is graduated to contain 10 ml and subdivided into 0.1 ml divisions, each 1 ml line being numbered from 10 ml at the top. The error in any indicated capacity should not be greater than 0.05 ml.

c) Condenser — This is approximately 400 mm in length and the bore of the inner tube of the condenser is 16 to 17 mm. The condenser is connected to the trap as shown in the figure.

D-4.2 Reagent

D-4.2.1 Toluene — treated with excess of water and distilled.

D-4.3 Procedure — Weigh accurately about 10 g of the material and transfer it into the flask. Add about 200 ml of toluene and a few pieces of dry pumice stone. Connect the apparatus and fill the receiving end of the trap with toluene poured through the top of the condenser. Heat the flask gently for 15 minutes, and when the toluene begins to boil reflux at a rate of 2 drops per second until most of the water has passed over. Then increase the rate to about 4 drops per second. When the water has apparently all distilled over, rinse the inside of the condenser tube with toluene while brushing down the tube with a tube brush attached to a copper wire and saturated with toluene. Continue the distillation

* Determination of water by Karl Fischer method (first revision).
Fig. 1 Toluene Distillation of Water
for 5 minutes, then remove the source of heat, and allow the receiving tube to cool to room temperature. If any droplets of water are adhering to the wall of the receiving tube, scrub them down with a brush consisting of a rubber band wrapped around a copper wire and wetted with toluene. When the water and toluene have separated, read the volume of water.

**D-4.4 Calculation**

\[
\text{Water, percent by mass} = \frac{V \times d \times 100}{M}
\]

where

- \( V \) = volume of water in ml at room temperature collected in the receiving tube,
- \( d \) = density of water at room temperature, and
- \( M \) = mass in g of the material taken for the test.

**D-5. TEST FOR RANCIDITY**

**D-5.1 Reagents**

**D-5.1.1 Concentrated Hydrochloric Acid**

**D-5.1.2 Phloroglucinol Solution** — Dissolve 0.1 g of phloroglucinol in 100 ml of diethyl ether.

**D-5.2 Procedure** — Shake 10 ml of the material, melted if necessary, with 10 ml of concentrated hydrochloric acid and 10 ml of phloroglucinol solution. Shake for 1 minute.

**D-5.2.1** The material shall be taken to have passed the test if no pink colour develops.
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