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*Draft Indian Standard*

**SPECIFICATION FOR PETROLEUM JELLY  
FOR COSMETIC INDUSTRY**  
*(Second Revision of IS 4887)*

(ICS 71.100.70)

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Cosmetics Sectional Committee,  
PCD 19

Last date for Comments:  
**12 March 2024**

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**FOREWORD**

*(Formal Clause to be added later)*

Petroleum jelly is a soft, semi-solid material obtained from petroleum oils. It consists essentially of microcrystalline waxes in association with substantial quantities of oil.

Petroleum jelly is used in the manufacture of creams, rouges, lipsticks, lubricating creams, hair dressings, foundation creams of greasy type, etc. It is also employed as an emollient for chapped skin.

This Indian Standard was first published in 1968 and subsequently revised in 1980. In this revision, the specification table has been revised with respect to the melting range (upper limit has been increased from 56 °C to 80 °C), the Requirement of Mercury has been incorporated, and alternate test methods have been included for the determination of Arsenic. The Cross Referred standards and the Marking Clause have also been updated in this revision.

The requirement laid down in this standard covers the material for its various application in the cosmetic industry. Product-wise requirements have not been laid down.

The composition of the Committee, responsible for the formulation of this standard is given at Annex P *(to be added later)*.

For the purpose of deciding, whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (second revision)'. The number of significant places retained in the rounded off value should be the same as that of the-specified value in this standards.

## 1. SCOPE

This standard prescribes the requirements and methods of sampling and test for petroleum jelly for cosmetic industry.

## 2 REFERENCES

The standards which are necessary adjuncts to this standard are listed below. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standard:

<i>Indian/ International Standard No.</i>	<i>Title</i>
1070 : 2023	Reagent Grade Water Specification ( <i>Fourth Revision</i> )
266 : 1993	Sulphuric Acid — Specification ( <i>Third Revision</i> )
264 : 2005	Nitric Acid — Specification ( <i>Third Revision</i> )
2088 : 2023	Methods For Determination Of Arsenic ( <i>Third Revision</i> )
2316 : 1990	Methods Of Preparation Of Standard Solutions For Colorimetric And Volumetric Analysis ( <i>Second Revision</i> )
2626 : 2015/ ISO 13132 : 2011	Laboratory Glassware - Petri Dishes ( <i>Second Revision</i> )
16913 : 2018	Methods of test for cosmetics — Determination of heavy metals (Arsenic, Cadmium, Lead and Mercury) by Atomic Absorption Spectrometry (AAS)
17495 : 2021	Cosmetics - Analytical Approach for Screening and Quantification Methods for Heavy Metals in Cosmetics

## 3 REQUIREMENTS

**3.1 Description** — Petroleum jelly shall be in the form of a translucent, soft mass, unctuous to the touch and retaining these characteristics on storage and also when melted and allowed to cool without stirring. It shall be not more than slightly fluorescent by daylight, even when melted.

**3.2 Solubility** — It shall be insoluble in water and alcohol but soluble in ether and chloroform.

**3.3 Colour** — The colour of the material shall be as agreed between the purchaser and the supplier.

**3.4 Odour** — The material shall be odorless at room temperature when rubbed on the skin. Further, the material when heated to 95 to 98 °C on a boiling water-bath for 30 minutes shall give no disagreeable odour.

**3.5** The material shall also comply with requirements given in Table 1.

**TABLE REQUIREMENTS FOR PETROLIUM JELLY FOR  
COSMETICS INDUSTRY**  
(Clause 3.5)

Sl No.	Characteristics	Requirement	Method of Test, Ref to Annex/ IS	
(1)	(2)	(3)	(4)	(5)
i)	Melting range, °C	38 to 80	A	--
ii)	Saponifiable matter	Nil	B	--
iii)	Free acids and alkalis	To pass the test	C	--
iv)	Heavy metals (as Pb), parts per million, <i>Max</i>	20	D	--
v)	Arsenic (as As <sub>2</sub> O <sub>3</sub> ), parts per million, <i>Max</i>	2	E	IS 16913 or IS 17495
vi)	Mercury, parts per million, <i>Max</i>	1	--	IS 16913 or IS 17495
vii)	Iodine value (Wijs), <i>Max</i>	1.5	F	--
viii)	Light absorption (extinction of 0.1 percent solution), <i>Max</i>	1.0	G	--
ix)	Sulphated ash, percent by mass, <i>Max</i>	0.10	H	--
x)	Organic acids	To pass the test	J	--
xi)	Sulphur and sulphide	To pass the test	K	--
xii)	Consistency	100 to 275 as agreed between the purchaser and the supplier	L	--
xiii)	Volatile matter, percent by mass, <i>Max</i>	5.0	M	--

## 4 PACKING AND MARKING

**4.1 Packing**—The material shall be packed in mild steel drums lacquered from inside or as agreed between the purchaser and the supplier.

### 4.2 Marking

**4.2.1** The containers shall be marked with the following information:

- a) Name of the material;
- b) Name of the manufacturer and his registered/recognized trademark, if any;
- c) Net mass of the material;
- d) Month and year of manufacturing/packing;
- e) Batch number in a manner to enable traceability including its date of manufacture;
- f) Use before or expiry date as per statutory requirements;
- g) List of ingredients as per statutory requirements; and
- h) Any other information required by statutory authorities.

#### 4.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the Bureau of Indian Standards Act, 2016 and the Rules and Regulations framed there under, and the products may be marked with the Standard Mark.

## 5 SAMPLING

**5.1** The method of preparing representative test samples of the material and the criteria for conformity shall be as prescribed in Annex N.

## 6 TEST METHODS

**6.1** Tests shall be conducted as prescribed under **3.5** and the appropriate references specified in col 4 and 5 of Table 1.

### 6.2 QUALITY OF REAGENTS

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

NOTE — ‘pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

**ANNEX A**  
[Table 1, Sl. No. (i)]  
**DETERMINATION OF MELTING RANGE**

**A-1 Procedure**

**A-1.1** Melt a quantity of the sample slowly while stirring until it reaches a temperature of 100 °C, Remove the source of heat and allow the molten sample to cool to a temperature of 8 to 10 °C above the expected melting range. Chill the bulb of a thermometer (range 1 to 100 °C) to 5 °C, wipe it dry and while it is still cold, dip it into the molten sample so that approximately half of the bulb is submerged. Withdraw it immediately and hold it vertically away from heat until the wax surface dulls, then dip it for five minutes into a water-bath having a temperature not higher than 16 °C.

**A-1.2** Fix the thermometer prepared in **A-1.1** securely in a test-tube so that its lowest point is about 15 mm above the bottom of the test-tube. Suspend the test-tube in a water-bath adjusted at 16 °C and raise the temperature of the bath at a rate of 2 deg/min up to 30 °C, then change to a rate of rise of 1 deg/min and note the temperature at which the first drop of the melted sample leaves the thermometer. Repeat the determination twice on a freshly melted portion of the sample. If the variation in three determinations is less than one degree take the average of the three as the melting range. If the variation in the three determinations is more than one degree, make two additional determinations and take the average of the five.

**ANNEX B**  
[Table 1, Sl. No. (ii)]  
**DETERMINATION OF SAPONIFIABLE MATTER**

**B-1 Reagents**

**B-1.1** Methyl Ethyl Ketone — Technical grade, stored in amber coloured bottle.

**B-1.2** Standard Alcoholic Potassium Hydroxide Solution — 0.5 N, standardized before use.

**B-1.3** Petroleum Ether — Boiling range 80 to – 100 °C.

**B-1.4** Standard Hydrochloric Acid — 0.5 N, accurately standardized.

**B-1.5** Phenolphthalein Indicator Solution - percent solution in 95 percent rectified spirit.

**B-2 Procedure** — Accurately weigh in a flask about 5 g of the sample and add  $25 \pm 1$  ml of methyl ethyl ketone, followed by 25 ml of standard alcoholic potassium hydroxide solution from a burette, Connect the flask to a condenser and heat for 30 minutes after refluxing begins. Disconnect the condenser, add 50 ml of petroleum ether and titrate the solution while hot (without re-heating)

with standard hydrochloric acid, using 3 drops of phenolphthalein indicator. When the indicator colour is discharged, add 3 drops more of indicator; if this addition restores the colour, continue the titration. Proceed in this manner until the end point is reached when the indicator colour is discharged and does not immediately reappear upon the addition of 3 more drops of indicator.

**B-2.1** Run a blank under identical conditions.

**B-2.2** The sample shall be taken to have passed the requirement prescribed in Table 1 if the blank reading does not differ from the sample reading by more than 0.1 ml.

**ANNEX C**  
[Table 1, Sl. No. (iii)]  
**TEST FOR FREE ACIDS AND ALKALIS**

**C-1 Reagents**

**C-1.1** Phenolphthalein Indicator Solution — same as in **B-1.5**.

**C-1.2** Methyl Orange Indicator Solution — dissolve 0.01 g of methyl orange in 100 ml of water.

**C-2 Procedure** — Take 35 g of the sample in a 250 ml separating funnel. Add to it 100 ml of boiling water and shake vigorously for 5 minutes. Draw off the separated water layer in a beaker. Wash the sample further with two 50 ml portions of boiling water and add the washings again to the beaker. To the collective washings, add one drop of phenolphthalein indicator solution and boil. If no pink colour is produced, add 0.1 ml of methyl orange indicator and see if any red or pink colour is produced.

**C-2.1** The sample shall be taken to have passed the test if neither a red colour is produced with phenolphthalein nor a red or pink colour produced with methyl orange.

**ANNEX D**  
[Table 1, Sl. No. (iv)]  
**TEST FOR HEAVY METALS**

**D-1 Apparatus**

**Nessler Cylinders** — 50 ml capacity, matched.

**D-2 Reagents**

**D-2.1 Ammonium Acetate Solution** — 10 percent.

**D-2.2 Ammonium Citrate Solution** — Dissolve 8.75 g of citric acid in water, neutralize with ammonia and dilute with water to 100 ml.

**D-2.3 Ammonium Hydroxide** — 10 percent (m/m).

**D-2.4 Potassium Cyanide Solution** — 10 percent (m/m).

**D-2.5 Sodium sulphide Solution** — 10 percent (m/m).

**D-2.6 Standard Lead Solution** — Dissolve 1.600 g of lead nitrate in water, add 10 ml of concentrated nitric acid and dilute to 1000 ml. Pipette out 10 ml of the solution and dilute it again to 1000 ml with water. One millilitre of the final solution contains 0.01 µg of lead (as Pb). The solution should be freshly prepared.

### **D-3 Procedure**

**D-3.1 Preparation of Sample** — Treat 2.000 g of the sample as prescribed in **D-2.1**.

**D-3.2** Take the solution prepared in **E-3.1** in a Nessler Cylinder, add 10 ml of ammonium acetate solution, 5 ml of ammonium citrate solution, 5 ml of ammonium hydroxide and 1 ml of potassium cyanide solution and dilute to 50 ml with water, then add two drops of sodium sulphide solution and fix well. In another Nessler cylinder, carry out a control test using 4 ml of standard lead solution and same quantities of other reagents as used in the test with the material.

**D-3.3** The material shall be taken as fit having exceeded the limit prescribed in Table 1 if the intensity of colour produced with the material is not greater than that produced in the control test.

## **ANNEX E**

[Table 1, Sl. No. (v)]

### **TEST FOR ARSENIC**

#### **E-1 Reagents**

**E-1.1 Concentrated sulphuric Acid** — *see* IS 266.

**E-1.2 Concentrated Nitric Acid** — *see* IS 264.

#### **E-2 Procedure**

**E-2.1 Preparation of Sample** — Weigh 2.000 g of the sample in a Kjeldahl flask of 500 ml capacity. Add 15 ml of concentrated sulphuric acid followed by 4 ml of concentrated nitric acid. Heat cautiously. Add drop by drop more nitric acid, if required, from a pipette to speed up the oxidation of the sample. The total amount of nitric acid shall be noted for use in the control test. When oxidation is complete, the solution is clear and faint yellow, at that stage, add 20 ml of water and again boil to fuming. Ensure removal of all nitric acid.

**E-2.2** Carry out the test for arsenic with the solution prepared in **D-2.1** as directed in IS 2088. Compare the stain obtained with that produced with 0.004 g of arsenic trioxide.

## ANNEX F

[Table 1, Sl. No. (vii)]

### DETERMINATION OF IODINE-VALUE

**F-1 Outline of the Method** — The material is treated with a known excess of iodine monochloride solution in glacial acetic acid. The excess of iodine monochloride is determined iodometrically.

#### F-1.1 Apparatus

**F-1.1.1 Thermometer** — An engraved stem thermometer, calibrated between 10 °C and 65 °C in 0.1 degree intervals and with the 0 °C point marked on the stem is recommended. The thermometer shall have an auxiliary reservoir at the upper end and length of about 370 mm and diameter of about 6 mm.

#### F-1.2 Reagents

##### F-1.2.1 Carbon Tetrachloride or Chloroform

**F-1.2.2 Acetic Acid** — glacial, 99 percent, having a melting point of 14.8°C and free from reducing impurities. Determine the melting point of the acetic acid and test it for reducing impurities as follows:

a) **Melting joint determination** — Take a 15 cm long test-tube and fill it to about two-thirds with the acetic acid insert into the-acid a thermometer satisfying the requirements specified in A-72.1 through a cork stopper fitting the test-tube. The amount of acid should be at least double the quantity required to cover the bulb of the thermometer when the bottom of the latter is 12 mm from the bottom of the test-tube. Suspend this tube within a larger test-tube through a cork. Cool the acid by immersing the assembly in ice water until the temperature is 10°C, then withdraw the assembly from the ice water and stir the acid rather vigorously for a few moment, thus causing the super cooled liquid to crystallize partially and give a mixture of liquid and solid acid. Take thermometer reading every 15 second sand record that temperature, at which the reading remains constant for at least two minutes, as the true melting point.



b) Test for reducing impurities (potassium permanganate test) — Dilute 2-ml of the acetic acid with 10 ml of water and add 0.1 ml of 0.1 N potassium permanganate solution and maintain at  $27 \pm 2^\circ\text{C}$ . The test shall be taken as having satisfied if the pink colour is not discharged at the end of two hour.

**F-1.2.3 Potassium Dichromate** — finely ground.

**F-1.2.4 Starch Solution** — Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into one litre of boiling water. Boil for 3 minutes. Allow the solution to cool and decant off the supernatant clear liquid.

**F-1.2.5 Standard Sodium Thiosulphate Solution** — 0.1 N (*see* IS 2316).

**F-1.2.6 Chlorine Gas** — dry.

**F-1.2.7 Iodine Trichloride**

**F-1.2.8 Wijs Iodine Monochloride Solution** — prepare this solution by one of the following two methods, and store in a glass stoppered bottle in a cool place, protected from light and sealed with paraffin until taken for use:

a) Dissolve 13 g of re-sublimed iodine in 1 litre of acetic acid, using gentle heat if necessary, and determine the strength by titration with standard sodium thiosulphate solution. Set aside 50 to 100 ml of the solution and introduce washed and dried chlorine gas into the remainder until the characteristic colour change occurs and the halogen content 'is nearly doubled as ascertained again by titration. If the halogen content has been more than doubled reduce it by adding the requisite quantity of the iodine acetic acid solution. A slight excess of iodine does not harm, but avoid an excess of chlorine.

Example:

If the titration of 20 ml of original iodine-acetic acid solution requires 22 ml of standard sodium thiosulphate solution then 20 ml of the finished Wijs solution should require between 43 and 44 ml (and not more than 44 ml) of the same sodium thiosulphate solution.

b) As an alternative method for preparing Wijs solution, dissolve 8 g of iodine trichloride in approximately 450 ml of acetic acid. Dissolve separately 9 g of iodine in 450 ml of acetic acid using heat if necessary. Add gradually the iodine solution to the iodine trichloride solution until the colour has changed to reddish brown. Add 50 ml more of iodine solution and dilute the mixture with acetic acid till 10 ml of the mixture are equivalent to 20 ml of standard sodium thiosulphate solution when the halogen content is estimated by titration in the presence of an excess of potassium iodine and water. Heat the solution at  $100^\circ\text{C}$  for 20 minutes and cool. Prevent access of water vapour in preparing the solution.

Note — The method prescribed in (a) is referable to that prescribed in (b) because of the instability of iodine trichloride.

**F-1.3 Procedure** — Melt the material and filter through the filter paper to remove any impurities and the last traces of moisture. Make sure that the glass apparatus used is absolutely clean and dry. Weigh accurately, by difference, about 10 g of the sample, into a clean dry 500 ml glass stoppered bottle to which 25 ml of carbon tetrachloride or chloroform have been added, and agitate to dissolve the contents. Add 25 ml of Wijs solution. (The quantity of Wijs solution added is 50 to 60 percent more than the quantity required) — Replace the glass stopper after wetting with potassium iodide solution, swirl for intimate mixing, and allow to stand in the dark for 45 minutes. Carry out a blank test simultaneously under similar experimental conditions. After standing, add 15 ml of potassium iodide solution and 100 ml of water, and titrate the liberated iodine with standard sodium thiosulphate solution, swirling the contents of the bottle continuously to avoid any local excess, until the colour of the solution is straw yellow. Add 0.5 ml of starch solution and continue the titration until the blue colour disappears.

#### **F-1.4 Calculation**

$$\text{Iodine value} = \frac{12.69 (B-S) N}{M}$$

Where

B = volume in ml of standard sodium thiosulphate solution required for the blank,

S = volume in ml of standard sodium thiosulphate solution required for the material,

N = normality of standard sodium thiosulphate solution, and

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

### **ANNEX G**

[Table 1, Sl. No. (viii)]

#### **DETERMINATION OF LIGHT ABSORPTION**

**G-1 General** — When radiation is passed through a layer of a solution containing an absorbing substance, part of the radiation is absorbed; the intensity of the radiation emerging from the solution is less than the intensity of the radiation entering it. The magnitude of the absorption is expressed in terms of the extinction, E which is defined by the expression,  $E = \log_{10} (I_0/I)$ , where  $I_0$  is the intensity of the radiation passing into the absorbing layer, and I is the intensity of the radiation passing out of it.

**G-1.1** The extinction depends on the concentration of the absorbing substance in the solution and thickness of the absorbing layer taken for measurement. For convenience of reference and for ease in calculations, the extinction of a 1 cm layer of a 1 percent m/v solution has been adopted.

## **G-2 Apparatus**

**G-2.1 Spectrophotometer**— comprising of a source of energy, a device for selecting the wavelength band, a cell or holder for the sample, a detector of radiant energy and a measuring device.

**G-3 Procedure** — Dissolve 0.1 g of petroleum jelly in 100 ml of trimethylpentane and measure the extinction of the solution using 1 cm cell at 290 nm ( $290 \times 10^{-7}$  cm).

## **ANNEX H**

[Table 1, Sl. No. (ix)]

### **DETERMINATION OF SULPHATED ASH**

#### **H-1 Reagents**

**H-1.1 Dilute Sulphuric Acid** — approximately 5 N.

**H-2 Procedure** — Heat a porcelain or silica dish of 30 to 100 ml capacity to redness; cool and weigh. Place about 20 g of the sample, accurately weighed, in the dish. Heat the dish gently by means of a Bunsen burner until the oil can be ignited at the surface. Remove the burner and allow the oil to burn completely, taking that all the free carbon on the sides of the dish is completely burnt. Heat the residue with a strong flame or in muffle furnace until all the carbonaceous matter has disappeared. Cool the dish, add a few drops of dilute sulphuric acid, heat gently to drive off the acid and then heat strongly. Cool the dish again in the desiccator and weigh. Repeat the heating, cooling and weighing until constant mass is obtained.

#### **H-3 Calculation**

$$\text{Sulphated ash, percent by mass} = \frac{m \times 100}{M}$$

Where

m = mass in g of the residue, and

M = massing of the sample taken for the test.

## **ANNEX J**

[Table 1, Sl. No. (x)]

### **TEST FOR ORGANIC ACIDS**

## J-1 Reagents

**J-1.1 Dilute Rectified Spirit** — prepared by diluting one volume of 95 percent rectified spirit with two volumes of water, and neutralized to phenolphthalein indicator.

**J-1.2 Phenolphthalein indicator** — same as in **B-1.5**.

**J-1.3 Standard Sodium - Hydroxide Solution** — exactly 0.1 N.

**J-2 Procedure** — To 20 g of the sample, add 100 ml of dilute rectified spirit, agitate thoroughly and heat to boiling. Add one millilitre of phenolphthalein indicator and titrate rapidly with standard sodium hydroxide solution with vigorous agitation to a sharp pink end point in the alcohol water layer.

**J-2.1** The material shall be taken to have passed the test if not more than 0.4 ml of standard sodium hydroxide solution is required for the titration.

## ANNEX K

[Table 1, Sl. No. (xi)]

### TEST FOR SULPHUR AND SULPHIDES

## K-1 Reagent

**K-1.1 Copper Strips** — one centimeter, in width, and freshly, polished.

**K-2 Procedure** — Melt in a beaker about 100 g of the, sample and keep on a water-bath at a temperature of 95 °C. Then place a strip of copper in the melted sample so that it is partially immersed and allow it to remain for 10 minutes.

**K-2.1** The material shall be taken to have passed the test if the copper strip used in the test shows, no tarnishing when compared with another similar freshly polished copper strip.

## ANNEX L

[Table 1, Sl. No. (xii)]

### DETERMINATION OF CONSISTENCY

**L-1 Outline of the Method** — Determination of consistency of the material is made by measuring the penetration of a standard cone at  $25.0 \pm 0.5$  °C.

## L-1.1 Apparatus

**L-1.1.1 Penetrometer** — Any suitable penetrometer which permits cone to drop vertically without appreciable friction for at least mm and which indicates accurately the depth of penetration to the nearest 0.1 mm. The instrument shall have a table to carry the test which permits the friction for at least penetration to the sample, which may be adjusted horizontal before making the test. A mechanism for releasing the clamp of the loaded cone shall be provided.

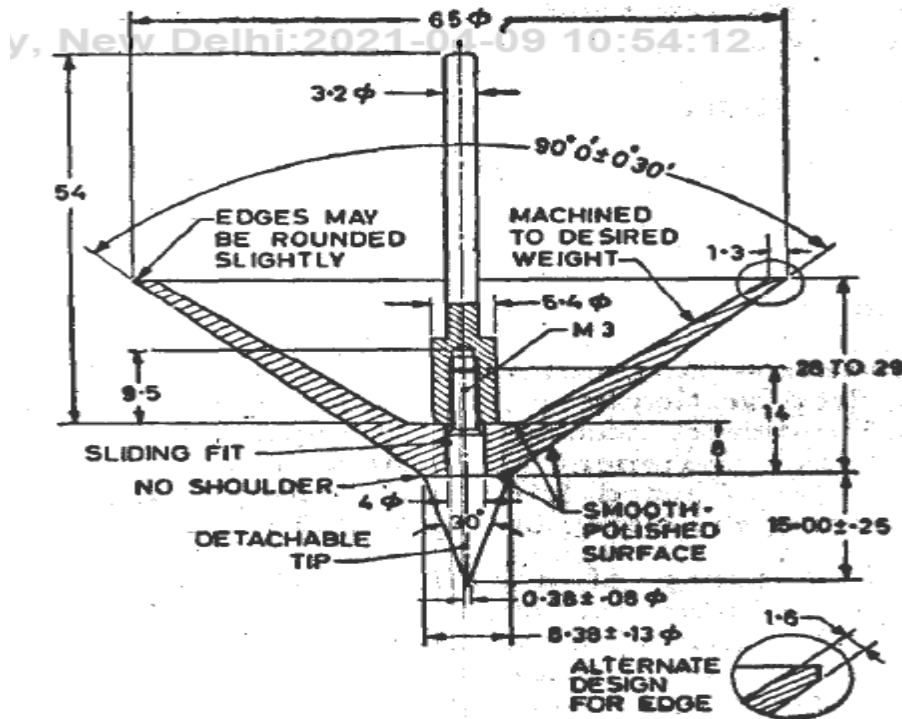
**L-1.1.2 Cone** — consisting of a conical body of brass or corrosion resistant steel with detachable hardened steel tip, constructed to conform to the dimensions and tolerances shown in Fig. 1. The total moving mass, namely, that of the cone and its movable attachments, shall be  $150.0 \pm 0.1$  g. The attachments consist of a rigid shaft having a suitable device at its lower end for engaging the cone. The outer surface should be polished to a very smooth finish.

**L-1.1.3 Constant Temperature Bath**— A water-bath capable of regulation at  $25.0 \pm 0.5$  °C and of suitable design for conveniently bringing the sample container to the test temperature. The bath should be provided with a cover to maintain the temperature of the air above the sample at 25 °C.

**L-1.1.4 Timing Device** — A stop-watch or other suitable instrument capable of measuring an interval of 5 seconds to within 0.2 second.

**L-1.1.5 Sample Container** — Flat-bottomed, metal or glass cylinders that are  $100 \pm 5$  mm in diameter and not less than 60 mm m height.

**L-1.2 Procedure** — Melt the quantity of the sample at  $85.0 \pm 2.5$  °C or higher, pour into one or more of the sample containers, filling to within 6 mm of the brim. Cool at  $25.0 \pm 2.5$  °C over a period of not less than 16 hours, protecting from draughts. Two hours before the test, place the containers in a water-bath at  $25.0 \pm 0.5$  °C. If the room temperature is below 23.5 °C, or above 26.5 °C, adjust the temperature of the cone to  $25.0 \pm 0.5$  °C by placing it in the water-bath.



All dimensions in millimetres.

FIG. 1 PENETROMETER CONE

**L-1.2.1** Without disturbing the surface of the sample, place the container on the penetrometer table, and lower the cone until the tip just touches the top surface of the sample at a spot 25 to 38 mm from the edge of the container. Adjust the zero setting and quickly release the plunger, then hold it free for 5 seconds. Secure the plunger and read the total penetration from the scale. Make 3 or more trials each so. Spaced that there is no overlapping of the areas of penetration. Where the penetration exceeds 20 mm use a separate container of the sample for each trial. Read the penetration to the nearest 0.1 mm. Calculate the average of the three or more readings and conduct further trials to a total of 10 if the individual results differ from the average by more than  $\pm 3$  percent.

### L-1.3 Calculation

$$\text{Consistency} = 10 A$$

Where

A = is the mean of all the values of penetration in mm

**ANNEX M**  
[Table 1, Sl. No. (xiii)]  
**DETERMINATION OF VOLATILE MATTER**

**M-1 Outline of the Method**

**M-1.1** The sample is weighed in petri/glass dish and kept in an oven maintained at  $105 \pm 1$  °C. The loss in mass is calculated as a percentage of the mass of the sample taken.

**M-2 APPARATUS**

**M-2.1 Petri/Glass Dishes** — made of heat-resistant glass, 90 to 100 mm in diameter and 7 to 10 mm in height (*see* IS 2626).

**M-2.2 Thermometer** — Any suitable thermometer having a range /from 0 to 110 °C.

**M-2.3 Air-Oven** — an electrically or gas-heated air--oven capable of maintaining temperature at  $105 + 1$  °C.

**M-3 PROCEDURE**

**M-3.1** Weigh about 10 g of the sample, accurately to the nearest 0.1 g, in a tared petri/glass dish, distributing the sample in as uniform a layer as possible-with the help of spatula over the whole of the bottom of the dish. Keep it in the oven, maintained at  $105 \pm 1$  °C. Cool and weigh the petri/glass dish to a constant mass

**M-4 CALCULATION AND REPORT**

**M-4.1** Calculate and report the evaporation loss as follows:

$$\text{Volatile matter percent by mass, Max} = \frac{100 (M_1 - M_2)}{M_1}$$

Where

$M_1$  = mass in g of the sample taken for the test, and  
 $M_2$  = mass in g of the sample after the test.

**ANNEX N**  
(Clause 5.1)  
**SAMPLING OF PETROLEUM JELLY**

**N-1 GENER & REQUIREMENTS OF SAMPLING**

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

**N-1.1.1** Samples shall not be taken in an exposed place.

**N-1.1.2** The sampling instrument shall be clean and dry.

**N-1.1.3** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**N-1.1.4** To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

**N-1.1.5** The samples shall be placed in clean, dry, air-tight glass or other suitable means.

**N-1.1.6** The sample containers shall be of such size that they are almost completely filled by the sample.

**N-1.1.7** Each sample container shall be sealed air-tight with a suitable stopper after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

## **N-2 SCALE OF SAMPLING**

**N-2.1 Lot** — All the containers in a single consignment of the material 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 belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

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

**N-2.2** The number of containers ( $n$ ) to be chosen from the lot shall depend on the size of the lot ( $N$ ) and shall be as given in Table 2.

**N-2.3** The containers to be selected for sampling shall be chosen at random. In order to ensure randomness of selection, random number tables shall be used. For guidance and use of random number tables, IS: 4905-1968 may be referred. In the absence of random number tables, the following procedure may be adopted:

Starting from any container in the lot, count them as 1, 2, 3,... up to  $r$  and so on in one order, where  $r$  is the integral part of  $N/n$ .  $N$  being the lot size and  $n$  the sample size. Every  $r$ -th container thus counted shall be withdrawn from the lot so as to constitute the required sample size.



**TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING**  
(Clause B-2.2)

<b>LOT SIZE</b>	<b>NUMBER OF NUMBER OF CONTAINERS TO BE SELECTED</b>
<b>(N)</b>	<b>(n)</b>
<b>(1)</b>	<b>(2)</b>
Up to 50	3
51 to 100	4
101 to 300	5
301 to 500	6
501 to 1000	7
1001 and above	8

### **N-3 TEST SAMPLES AND REFEREE SAMPLES**

#### **N-3.1 Preparation of Test Samples**

**N-3.1.1** Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected (*see* Table 2). The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under 2 and shall be not less than 250 g.

**N-3.1.2** Thoroughly mix all portions of the material drawn the same container. Out Of these portions, equal quantities shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weigh not less than 500 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

**N-3.2 Referee Sample** — The referee sample shall consist of a composite sample marked for this purpose and shall bear the seals of the purchaser and the supplier. It shall be kept at a place agreed between the purchaser and the supplier and shall be used in case of dispute between the two.

### **N-4 NUMBER OF TESTS**

**N-4.1** Tests for all the characteristics given in 2 and Table 1 shall be conducted on the composite sample.

### **N-5 CRITERIA FOR CONFORMITY**

**N-5.1** A lot shall be declared as conforming to this specification if the composite sample satisfies the requirement for each of the characteristics listed in 2 and in Table 1. If the requirement for any of the characteristics are not met, the lot shall be declared to have not satisfied the requirement of the specification.