भारतीय मानक
Indian Standard

IS 286 : 2018

साबुन के लिए नमूना और परीक्षण की पद्धतियाँ

(तीसरा पुनरीक्षण)

Methods of Sampling and Test for Soaps

(Third Revision)

ICS 71.100.40

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FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Soaps and Other Surface Active Agents Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1951 and subsequently revised in 1966 and 1978. During 1978 revision the procedure for determination of matter insoluble in alcohol has been elaborated. Provision has been made for calculation of total fatty matter while determining combined alkali and total anhydrous soap. Similarly provision has also been made for calculation of neutral sodium silicate content while determining alkaline silicates. For determination of glycerol content in soaps a concentration of 6 percent of sodium periodate in place of 2 percent hitherto prescribed has been included which improves the repeatability of the method. An alternate method for estimation of rosin acids without the use of β-naphthalene sulphonic acid has been added. An additional method for determination of free carbonated alkali has been included. Amendment No. 1 has been incorporated in this revision.

This standard is intended to facilitate the introduction of uniform methods of sampling and analysis of soaps. It is a necessary adjunct to the Indian Standards on individual materials.

During this revision, Amendment No.1, 2, 3, 4 and 5 have been incorporated and also the referred standards have been updated.

The Committee responsible for the preparation of the standard is given at Annex C.

The methods given in this standard substantially correspond to the following standards of the International Organization for Standardization (ISO) :

ISO 456 : 1973	Analysis of soap. Determination of free caustic alkali.
ISO 457 : 1976	Analysis of soap. Determination of chlorides.
ISO 672 : 1978	Analysis of soaps. Determination of moisture and volatile matter.
ISO 673 : 1981	Analysis of soaps. Determination of content of ethanol — insoluble matter.
ISO 684 : 1974	Analysis of soaps. Determination of total free alkali.
ISO 685 : 1975	Analysis of soaps. Determination of total alkali.
ISO 1066 : 1975	Analysis of soaps. Determination of glycerol content — Titrimetric method
ISO 1067 : 1974	Analysis of soaps. Determination of unsaponifiable and unsaponified matter.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHODS OF SAMPLING AND TEST FOR SOAPS

(Third Revision)

1 SCOPE

1.1 This standard prescribes methods of sampling and test for soaps.

1.2 Should there be any discrepancy between the requirements of this standard and the one for an individual material specification, the latter shall prevail.

2 REFERENCES

The standards listed in Annex B contains provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. 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 standards given in Annex B.

3 TERMINOLOGY

3.1 For the purpose of this standard, the following definitions, in addition to those given in IS 7597 shall apply.

3.1.1 *Combined Alkali* — It is the alkali present in soap in combination with saponifiablc matter.

3.1.2 *Free Fatty Acid* — It is the free (uncombined) fatty acid present in soap and is expressed as percent by mass as oleic acid.

3.1.3 *Free Caustic Alkali* — It is the free (uncombined) caustic alkali present in soap.

3.1.4 *Iodine Value* (*Wijs*) — It is the number of grams of iodine absorbed per 100 grams of the mixed fatty and rosin acids obtained from soap.

3.1.5 *Matter Insoluble in Alcohol* — It comprises most of the alkaline salts, such as talc, carbonates, borates, silicates and phosphates, as well as sulphates and starch, which are insoluble in alcohol under the conditions of the test.

3.1.6 *Moisture and Volatile Matter* — It includes moisture and any other material contained in soap volatile under the conditions of the test.

3.1.7 *Titre* — It is the highest temperature reached when the mixed fatty and rosin acids obtained from soap are crystallized under the conditions of the test.

3.1.7.1 Titre is generally taken to represent the solidification point of the mixed fatty and rosin acids, although they actually solidify over a range of temperature.

3.1.8 *Total Anhydrous Soap* — It represents the fatty acids existing in soap in combination with alkali.

NOTE — Sometimes total anhydrous soap is determined by deducting the value for moisture and volatile matter from the mass of soap. This is not a precise method even for genuine soaps and is certainly not applicable to built and filled soaps.

3.1.9 *Total Fatty Matter* — It includes substances soluble in ether under the conditions of the test, such as fatty and rosin acids present in the combined state as well as unsaponified and unsaponifiable matter.

3.1.10 *Unsaponifiable Matter* — It includes substances, such as the higher aliphatic alcohols, sterols, colouring materials and hydrocarbons, which may be present in soap and which are not capable of being saponified by caustic alkali but are soluble in ordinary fat solvents.

3.1.11 *Unsaponified Matter* — It is the neutral fat (unsaponified, neutral glycerides) present in soap.

4 SAMPLING

4.1 General Precautions

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

4.1.1 Samples shall be taken in a protected place, not exposed to damp air, dust or soot.

4.1.2 The sampling instruments shall be clean and dry when used.

4.1.3 The samples, the material being sampled, the sampling instruments and the containers for samples shall be protected from adventitious contamination.

4.1.4 The samples shall be placed in clean and dry glass containers. The size of the sample containers shall be such that the latter are almost completely filled by the sample.

4.1.5 Each container shall be scaled air-tight after filling and suitably marked.

4.1.6 The samples shall be stored in such a manner that the temperature of the material does not vary unduly from the ambient temperature, and that they are protected from light.

4.2 Scale of Sampling

4.2.1 *Lot*

In a single consignment, all the packages containing

soap of the same type, grade and form, and drawn from the same batch of manufacture, shall continue a lot. If the consignment consists of packages containing soaps of different types, grades and forms, packages containing soaps of the same type, grade, form and batch of manufacture shall be grouped together and each such group shall constitute a separate lot.

4.2.2 For ascertaining the conformity of the lot to the requirements prescribed in the relevant standard, tests shall be carried out for each lot separately. The number *n* of packages to be selected for drawing the samples shall depend upon the size *N* of the lot, and shall be in accordance with Table 1.

4.2.3 The packages shall be selected at random and, to ensure randomness of selection, random number tables shall be used. In case such tables are not available, the following procedure may be adopted:

'Starting from any package, count all the packages in one order as 1, 2, 3, etc, up to *r* and so on, where *r* is the integral part of *N/n* (*N* being the lot size and *n* the number of packages to be selected). Every *r*th package thus counted shall be withdrawn to give a sample for the purpose of test.'

4.3 Preparation of Test Samples

4.3.1 *Gross Samples*

4.3.1.1 *Bars and tablets*

From each one of the packages selected as in **4.2.2** draw at random a number of bars or tablets from different parts of the package. The material so drawn from a package shall be nearly equal to thrice the quantity required for the purpose of test as indicated in **4.4**. The bars or tablets selected shall be run through a suitable chopper. The disintegrated material thus obtained from the chopper shall be mixed thoroughly to give the gross sample for the package.

4.3.1.2 *Flakes, chips and powders*

From each one of the packages selected as in **4.2.2** draw at random one or more cartons. The material in the carton so chosen shall be nearly thrice the quantity required for the purpose of test as indicated in **4.4**. This material shall then be disintegrated, if necessary, and mixed thoroughly to give the gross sample for the package.

4.3.1.3 *Liquid soaps*

From each one of the packages selected as in **4.2.2** draw at random one or more containers. The material in the containers so chosen shall be nearly thrice the quantity required for the purpose of test as indicated in **4.4**. This material shall then be mixed thoroughly to give the gross sample for the package.

4.3.2 *Test Samples*

4.3.2.1 Segregate carefully the gross samples (*see* **4.3.1**) of bars, tablets, flakes, chips, powders and liquid soaps. From the gross samples representing each form of soap, take a small but equal quantity of material and mix it thoroughly with a composite sample which should be of a size sufficient to carry out triplicate testing for all the characteristics specified under **4.4.2.** The composite samples representing each form of soap shall be divided into three equal parts — one for the purchaser, another for the supplier and the third for the referee.

4.3.2.2 The remaining portion of the material in each one of the gross samples shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *n*-packages selected shall be for the purchaser, another for the supplier and the third for the referee.

4.3.2.3 All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars.

4.3.3 *Referee Samples*

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

4.4 Number of Tests

4.4.1 Tests for the determination of important characteristics, as specified in the relevant material specification, shall be conducted on each of the individual samples separately.

4.4.2 Tests for the determination of all the remaining characteristics in the material specification shall be conducted on the composite sample.

Table 1 Scale of Sampling
(*Clause* 4.2.2)

No. of Packages in the Lot	No. of Packages to be Selected
N	*n*
(1)	(2)
4 to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
111 and above	10

NOTE — When the size of the lot is 3 packages or less, the number of packages to be selected and the criteria for judging the conformity of the lot to the specification shall be as agreed to between the purchaser and the supplier.

4.5 Criteria for Conformity

4.5.1 *For Individual Samples*

For each of those characteristics which have been determined on the individual samples, the mean ($\bar{x}$) and the range (R) of the test results shall be calculated as follows:

$$\text{Mean } (\bar{x}) = \frac{\text{the sum of test results}}{\text{number of test results}}$$

Range (R) = The difference between the maximum and the minimum value of the test results.

a) If the specification limit for the characteristic is given as a minimum, the value of the expression ($\bar{x} - KR$) shall be calculated from the relevant test results [*see also* **4.5.1**(d)]. If the value so obtained is greater than or equal to the minimum limit, the lot shall be declared as conforming to the requirement of that characteristic.

b) If the specification limit for the characteristic is given as a maximum, the value of the expression ($\bar{x} + KR$) shall be calculated from the relevant test results [*see also* **4.5.1**(d)]. If the value so obtained is less than or equal to the maximum limit, the lot shall be declared as conforming to the requirement of that characteristic.

c) If the characteristic has two-sided specification limit, the values of the expressions ($\bar{x} - KR$) and ($\bar{x} + KR$)shall be calculated from the relevant test results [*see also* **4.5.1**(d)]. If the values so obtained lie between the two specification limits, the lot shall be declared as conforming to the requirement of that characteristic.

d) The value of the factor K referred to in **4.5.1** [(a) to (c)] shall be chosen in accordance with Table 2, depending upon the acceptable quality level (that is, the percentage of non-conforming package that could reasonably be tolerated).

Table 2 Values of *K* for Achieving Different Acceptable Quality Levels
[*Clause* 4.5.1(d)]

Acceptable Quality Level	Value of *K*
Not more than 3 percent defectives	0.4
Not more than 1.5 percent defectives	0.5
Not more than 0.5 percent defectives	0.6

4.5.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics determined on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirement given in the material specification.

5 DETERMINATION OF MOISTURE AND VOLATILE MATTER

5.1 General

Two methods, namely, (a) the oven method, and (b) the distillation method, are employed. The oven method gives the moisture and volatile matter content determined by drying the material in an air-oven at a specified temperature. The distillation method gives only the moisture content determined by measuring the volume of water distilled over when boiled under reflux with xylene or toluene.

5.1.1 *Applicability*

The oven method is generally applicable to all soaps, though, for the following classes of soaps, the distillation methodis to be preferred:

a) Soaps containing appreciable amounts of sodium silicate;

b) Soaps obtained from linseed oil and other oxidizing oils which absorb oxygen and are likely to gain in mass towards the end of the test if the oven method is used. Nevertheless, if the oven method is used, the drying should be carried out either in an inert atmosphere or in vacuum;

c) Soaps containing an appreciable amount of glycerol, especially those manufactured by the cold process or by the semi-boiled process, which usually give high results by the oven method; and

d) Soaps containing sodium hydrogen carbonate, perfume, ammonia, alcohol, carbolic acid and persalts.

5.1.2 In case of dispute, moisture content shall be determined by the distillation method.

5.2 Oven Method

5.2.1 *Procedure*

Accurately weigh 5.00 ± 0.01 g of the material in a petri dish, about 6 to 8 cm in diameter and about 2 to 4 cm in depth, and dry to constant mass in an air-oven at a temperature of 105 ± 2°C. Cool in a desiccator and weigh. Constant mass shall be considered to have been attained when successive heating for 1 h period shows a difference of not more than 0.1 percent in the net loss in mass.

5.2.2 *Calculation*

Moisture and volatile matter content, percent by mass = 100

where

m = loss in mass in g of the material after drying, and

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

5.3 Distillation Method

5.3.1 *Apparatus* — The apparatus consists of a glass flask heated by suitable means and provided with a reflux condenser discharging into a trap and connected to the flask. The connections between the trap and the condenser and the condenser and the flask should be interchangeable ground-glass joints. The trap serves to collect and measure the condensed water and to return the solvent to the flask. The assembly of the apparatus is shown in Fig. 1, and the various components are described below:

a) *Flask* — a 500 to 1 000-ml flask of the shape shown in Fig. 1, made of hard resistant glass, well annealed and as free as possible from striae and similar defects.

b) *Condenser* — a water-cooled reflux-type glass condenser of the design and dimensions shown in Fig. 1A. The only mandatory dimensions for the condenser are the external diameters of the inner tube and of the jacket, which shall be 16 to 17 mm and 23 to 25 mm respectively. The joints A and B should be neatly finished as shown in Fig. 1A, particularly the bore at B shall have the minimum disturbance. The shoulder above the cone of joint D shall be elongated as shown in Fig. 1A to avoid a sharp re-entrant shape which may restrict the free flow of liquid down the inner wall. The cone shall be extended beyond the length appropriate to the joint D, and the lower end ground inclined at an angle of approximately 60 to the axis. The drainage tip shall be at the front of the condenser when the lower water connection is to the left, the finish shall be either smooth or fire-polished. When inserted into the trap, the tip of the condenser shall be 6 to 7 mm above the surface of the liquid in the trap after distillation conditions have been established. The nominal dimensions of the joint *D* are given below:

Nominal Dia of Large End of Ground Zone mm	*Nominal Dia of Small End of Ground Zone* mm	*Nominal Length of Ground Zone Measured Axially* mm
18.8	16.2	26

c) *Receiver* — also called the trap, made of hard resistant glass, well annealed and as free as possible from striae and similar defects, provided with ground-glass joints, with the shape, dimensions and tolerances given in Fig. 1B and 1C; consisting essentially of the upper chamber, together with the tube and the ground joint leading to the flask, and the graduated tube. The receivers shall be of two sizes, namely, 2 ml capacity and 10 ml capacity (*see* Fig. 1B and 1C). The mandatory dimensions and tolerances for the receivers shall be as given in Table 3.

The shoulder of the upper chamber of the receiver immediately below the conical joint shall be finished square as shown in Fig. 1B and 1C. The graduated portion of the receiver

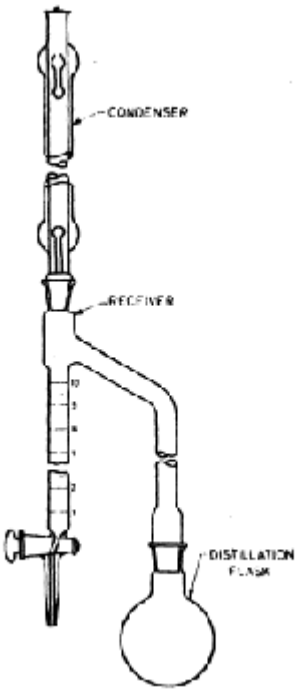
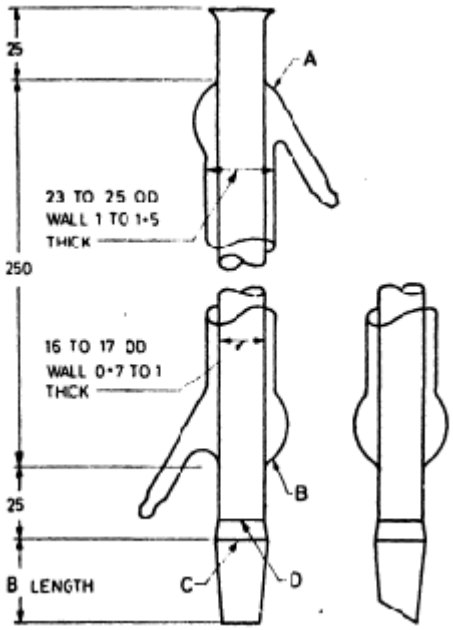


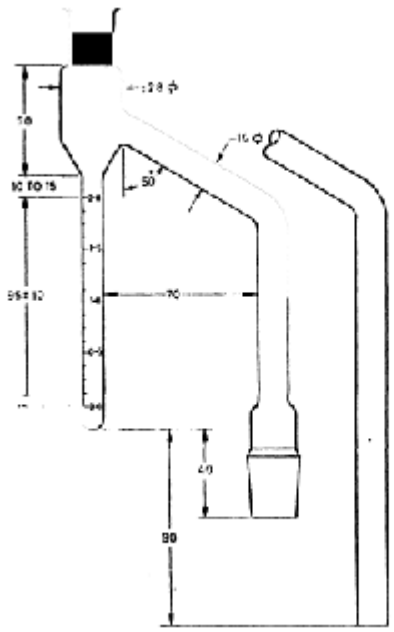
FIG. 1 TYPICAL ASSEMBLY OF DEAN AND STARK APPARATUS



All dimensions in millimetres.

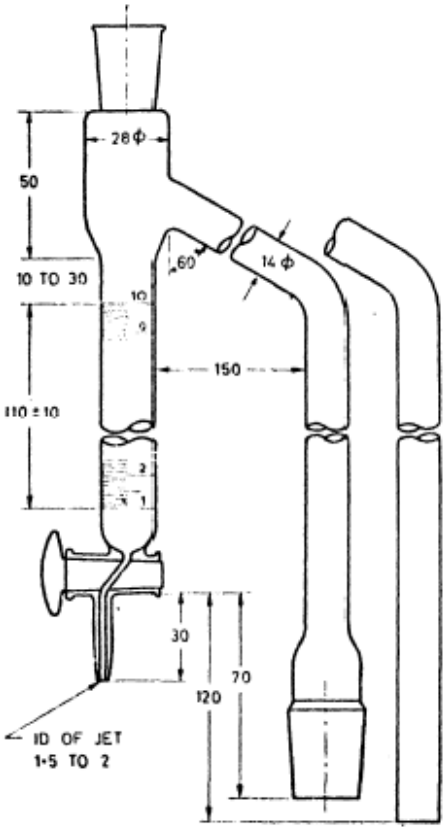
FIG. 1A CONDENSER

shall be cylindrical throughout its length. The bottom of the graduated tube of the 2-ml receiver shall be sealed, the end of the tube being approximately hemispherical in shape. The graduated scales on the receivers shall be numbered and subdivided as shown in Fig. 1B and 1C. The graduation marks shall be fine, cleanly etched permanent lines of uniform thickness lying in plane at right angles to the axis of the tube. The graduation marks shall be confined to the cylindrical portion of the tube and there shall be no evident irregularity in their spacing. In these receivers the numbered graduation marks shall be carried completely round the tube, the shortest ones half-way round it and those of intermediate length approximately two-thirds of the way round the tube, projecting



All dimensions in millimetres.

FIG. 1B 2-ml RECEIVER



All dimensions in millimetres.

FIG. 1C 10-ml RECEIVER

equally at each end beyond the shortest graduation marks.

The capacity corresponding to any graduation mark is defined as the volume of water at 27°C, expressed in millilitres, required to fill the graduated portion to that mark at 27°C, the axis of the graduated portion being vertical and the lowest point of the water meniscus being set on the graduation mark. In the case of the 10-ml receiver, the volume of the bore of the stopcock key and that of the jet below the stopcock shall not be included as part of the measured volume.

The error at any point on the receiver scale, as also the difference between the errors at any two points on the scale shall not exceed the figures given for the receivers in Table 3.

For the 10-ml receiver, the stopcock shall be of the 2-mm oblique bore having the general design shown in Fig. 1B and 1C. The rate of leakage, tested with the stopcock free from grease, the barrel and the key wetted with water, the receiver filled initially with water to the top of the scale and the key in either fully shut-off position, shall not exceed the figure given in Table 3.

Each receiver shall have permanently and legibly marked on it:

1) the abbreviation 'ml' ,
2) the inscription '27°C' to indicate that the receiver is graduated for content at 27°C, and
3) the identification number on the key.

d) *Heat Source* — The source of heat may be either an oil-bath or an electric heater provided with a sliding rheostat or other means of heat control. The temperature of oil in the bath should not be very much higher than the boiling point of xylene or toluene, whichever solvent is used.

e) *Copper Wire* — long enough to extend through the condenser, with one end twisted into a spiral. The diameter of the spiral should be such that it fits snugly within the graduated portion of the receiver and yet may be moved up and down.

Table 3 Mandatory Dimensions and Tolerances for Receiver
(*Clause* 5.3.1)

Sl No.	Characteristic	Receiver	
		2-ml	10-ml
(1)	(2)	(3)	(4)
i)	Volume, equivalent to smallest subdivision	0.05 ml	0.1 ml
ii)	Scale length	95 ± 10 mm	110 ± 10 mm
iii)	Length of cylindrical tube above upper graduation mark	10 to 15 mm	10 to 30 mm
iv)	Tolerance on capacity	± 0.02 ml	± 0.06 ml
v)	Maximum permissible leakage rate of stopcock	–	0.004 ml/min

5.3.2 *Reagents*

5.3.2.1 *Potassium dichromate-sulphuric acid cleaning solution*

5.3.2.2 *Xylene or toluene* — Saturate the xylene or toluene by shaking with a small quantity of water and distil. Use the distillate for the determination of moisture.

5.3.3 *Procedure* — Clean the entire apparatus with potassium dichromate-sulphuric acid cleaning solution to minimize the adherence of water droplets to the sides of the condenser and the receiver. Rinse thoroughly with water and dry completely before use. The quantity of material taken for the test is determined by the amount of moisture present (*m*/*m*), as indicated below:

Moisture Range m/m	*Quantity of Material (Approximately)*
Less than 1 percent	50 g
1 to 5 percent	25 g
Moisture in excess of 5 percent	Proportionately smaller quantity

Place the specified quantity of material, accurately weighed, in the distillation flask, add 200 ml of xylene or toluene and swirl to mix. Add a few porcelain pieces to regulate boiling. Assemble the apparatus and fill the receiver with the solvent by pouring it through the condenser until it begins to overflow into the distillation flask. Insert a loose cotton plug in the top of the condenser to prevent condensation of atmospheric moisture within the tube. To ensure that refluxing is under control, wrap the flask and the tube leading to the receiver with asbestos cloth. Heat the flask so that the distillation rate is about 100 drops per minute. When the greater part of the water has distilled over, increase the distillation rate to about 200 drops per minute and continue until no more water is collected. Purge the reflux condenser occasionally during the distillation with 5-ml portions of xylene or toluene to wash down any moisture adhering to the walls of condenser. The water in the receiver may be made to separate from xylene or toluene by moving the spiral copper wire up and down in the condenser and receiver occasionally, thus causing the water to settle at the bottom of the receiver. Reflux until water-level in the receiver remains unchanged for 30 min; then shutoff the source of heat.

Flush the condenser with either xylene or toluene, as required, making use of the spiral copper wire to discharge any moisture droplets. Immerse the receiver in water at about 30°C for at least 15 minutes or until the xylene or toluene layer is clear; then read the volume of water.

5.3.4 *Calculation*

$$\text{Moisture content, percent by mass} = \frac{100VD}{M}$$

where

V = volume in ml of water,
D = relative density of water at the temperature at which the volume of water is read, and
M = mass in g of the material taken for the test.

6 DETERMINATION OF MATTER INSOLUBLE IN ALCOHOL

6.0 General

This method may be used for the approximate determination of these constituents. As these salts are not completely insoluble in alcohol, separate portions of soap should be used for accurate determination, employing specific methods.

6.0.1 *Method*

It consists in digesting the material in alcohol and filtering off the residue, which is dried and weighed.

6.1 Reagents

6.1.1 *Phenolphthalein Indicator* — Dissolve 1 g in 100 ml of 95 percent rectified spirit.

6.1.2 *Ethyl Alcohol* — conforming to IS 321, or rectified spirit conforming to IS 323, freshly boiled, and neutral to phenolphthalein.

6.2 Procedure

6.2.1 Weigh accurately 2 to 10 g of the sample and reflux with 200 ml of freshly boiled ethyl alcohol fitting with a

suitable reflux condenser on a steam bath until the soap is dissolved. Filter into a filter flask through a tared, dried and counterpoised filter paper or through a tared and dried Gooch or sintered glass crucible with suction, protecting the solution from carbon dioxide and other acid fumes during the operation by covering with a watch glass. The filter paper and Gooch crucible shall be prepared as per method given under **6.2.2**. Wash it several times with ethyl alcohol at approximately 60°C till the filtrate on dilution with distilled water shows neutrality to phenolphthalein, to remove all the alcohol solubles (preserve the filtrate for **7.2)**. Dry the filter paper or the crucible with the residue at 100 ± 2°C for 3 h and cool. Weigh the total matter insoluble in alcohol.

6.2.2 Place filter paper in a weighing bottle and dry in an air oven at 105 ± 2°C with cover removed. Remove from the oven, replace cover, cool to room temperature in a desiccator and weigh. Prepare the Gooch crucible with a pad of asbestos fibre. Wash the pad with water, alcohol and ether and then dry to constant mass at 105 ± 2°C, cool to room temperature in a desiccator and weigh.

6.3 Calculation

$$\text{Matter insoluble in alcohol, percent by mass} = 100\frac{m}{M}$$

where

m = mass in g of matter insoluble in alcohol, and

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

7 DETERMINATION OF FREE CAUSTIC ALKALI OR FREE FATTY ACID

7.0 General

The method consists in dissolving the soap in alcohol and titrating the solution with sulphuric acid or alcoholic sodium hydroxide solution, as the case may be.

7.1 Reagents

7.1.1 *Phenolphthalein Indicator* — as in **6.1.1**.

7.1.2 *Ethyl Alcohol* — as in **6.1.2**.

7.1.3 *Standard Sulphuric Acid or Standard Hydrochloric Acid* — approximately 0.1 N.

7.1.4 *Standard Sodium Hydroxide Solution* — approximately 0.1 N.

7.1.5 *Barium Chloride Solution* — 10 percent (*m/v*).

7.2 Procedure for Free Caustic Alkali

7.2.1 Take the filtrate preserved in **6.2.1**. Heat it to boil. Add about 0.5 ml of phenolphthale in indicator and titrate with standard sulphuric or hydrochloric acid.

7.3 Procedure for Free Fatty Acid

Proceed as prescribed under **7.2.1** starting with a fresh sample of soap, and omitting the addition of barium chloride solution and titrate the alcoholic solution with standard sodium hydroxide solution. This procedure is applicable to pure soaps only.

7.4 Calculation

Calculate the percentage of free caustic alkali (as NaOH, KOH and K_2O) and free fatty acid (as oleic acid) as follows:

a) Free caustic alkali (as NaOH), percent by mass $= \frac{4VN}{M}$

b) Free caustic alkali (KOH), percent by mass $= \frac{5.61VN}{M}$

c) Free caustic alkali (as K_2O), percent by mass $= \frac{4.71VN}{M}$

where

V = volume in ml of standard sulphuric acid or hydrochloric acid used,

N = normality of standard sulphuric acid or hydrochloric acid, and

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

d) Free fatty acid, as oleic acid ($C_{18}H_{34}O_2$), percent by mass $= \frac{28.5V_1N_1}{M}$

where

V_1 = volume in ml of standard sodium hydroxide solution used,

N_1 = normality of standard sodium hydroxide solution, and

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

8 DETERMINATION OF MATTER INSOLUBLE IN WATER

8.0 General

To determine the matter insoluble in water, the sample is extracted with alcohol, filtered and the residue extracted with hot water.

8.1 Procedure

Starting with a fresh sample of soap, proceed as described in **6.2**, but do not dry or weigh the matter insoluble in alcohol. After filtering and washing the residue thoroughly with hot ethyl alcohol, change the receiver, extract the residue with successive portions of water at about 60°C and wash the residue thoroughly on the filter paper or in the crucible. Reserve the water solution for the determination of total alkalinity under **9.2**. Dry the filter and the residue at 100 ± 2°C for 3 h and cool. Weigh the matter insoluble in water.

8.2 Calculation

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

where

m = mass in g of matter insoluble in water, and

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

9 DETERMINATION OF TOTAL ALKALINITY OF MATTER INSOLUBLE IN ALCOHOL (ALKALINE SALTS)

9.0 General

The matter insoluble in alcohol in the soap is dissolved in water and titrated with standard acid using phenolphthalein' as indicator. Total alkalinity of matter insoluble in alcohol (alkaline salts) is expressed as NaOH.

9.1 Reagents

9.1.1 *Standard Sulphuric Acid or Hydrochloric Acid* — approximately 0.5 N.

9.1.2 *Phenolphthalein Indicator* — Dissolve 1 g in 100 ml of 95 percent of rectified spirit.

9.2 Procedure

Titrate the water solution reserved under **8.1** with standard sulphuric or hydrochloric acid, using phenolphthalein indicator.

9.3 Calculation

Unless otherwise specified or agreed to between the purchaser and the supplier, calculate the total alkalinity (as NaOH) of matter insoluble in alcohol as follows:

$$\text{Total alkalinity (as NaOH) of matter insoluble in alcohol, percent by mass} = \frac{4VN}{M}$$

where

V = volume in ml of standard sulphuric or hydrochloric acid,

N = normality of standard sulphuric acid, and

M = mass in g of the material taken for the test under **7**.

10 DETERMINATION OF COMBINED ALKALI AND TOTAL ANHYDROUS SOAP

10.0 General

Fatty acids in the sample of soap are separated by treatment with dilute sulphuric acid and are quantitatively extracted by ether. The ether extract is titrated in alcoholic medium with standard sodium hydroxide solution for determining the combined alkali. The total anhydrous soap is determined by evaporating and drying the titrated solution.

10.1 Reagents

10.1.1 *Dilute Sulphuric Acid* — 1 : 1 (*v/v*).

10.1.2 *Methyl Orange Indicator* — Dissolve 0.1 g in 100 ml of water.

10.1.3 *Sodium Chloride Solution* — saturated.

10.1.4 *Ethyl Ether — See* IS 336.

10.1.5 *Ethyl Alcohol* — as in **6.1.2**.

10.1.6 *Standard Sodium Hydroxide Solution* — approximately 0.5 N and carbonate-free.

10.1.7 *Standard Hydrochloric Acid* — approximately 0.5 N.

10.1.8 *Phenolphthalein Indicator* — as in **6.1.1**.

10.2 Procedure

10.2.1 Accurately weigh 5 to 10 g of the sample, depending upon the anhydrous soap content, and dissolve in 250-ml conical flask by warming in 100 ml of water. When dissolution is complete, add dilute sulphuric acid in slight excess (as judged by methyl orange indicator), insert a small funnel into the neck of the flask, and heat the flask to a temperature not exceeding 60°C until the fatty acids separate as a clear layer. Add 50 ml of sodium chloride solution and cool. Transfer quantitatively to a separating funnel, draw off the aqueous acid layer into a second separating funnel and shake it with three 50-ml portions of ethyl ether. Dissolve the fatty acids in the ether used for washing the aqueous liquid and extract with 10-ml portions of water until the extracts are no longer acidic to methyl orange indicator. Mix the water portions used for washing and shake with 20 ml of ether. Wash this ether until the wash water is neutral to methyl orange indicator.

10.2.2 Mix the ether solutions (if necessary, filter previously washing the paper with ether) in a suitable, weighed vessel, evaporate on a boiling water-bath to a volume not less than 50 ml, add 100 ml of ethyl alcohol and about 0.5 ml phenolphthalein indicator and titrate to exact neutrality with standard sodium hydroxide solution.

10.2.3 Evaporate the alcohol, dry to constant mass in an oven at 105 ± 2°C as in the determination of moisture and volatile matter content under **5.1.1**.

10.3 Calculation

10.3.1 Ascertain the mass of soda soap and calculate as percentage on the mass of the material taken for the test. This mass of the soda soap includes that of any mineral oil, unsaponifiables and neutral fat which, if determined separately, should be deducted from the results to obtain the true soap. If desired, calculate the combined sodium oxide (Na_2O) and deduct from the mass of the soda soap to give the acid anhydrides. For calculating total fatty matter subtract ($\frac{22}{31}$ × combined Na_2O) from the mass of the soda soap.

10.3.2 If the original soap is a potash soap, combined alkali may be calculated as potassium oxide (K_2O).

10.3.3 If the soap shows an excess of free acid, proper corrections should be made in calculating the combined alkali in the original soap. A blank test should be made on the sodium hydroxide or potassium hydroxide solution for neutral salts and correction made, if necessary, by exactly neutralizing 20 ml of the standard sodium hydroxide solution with standard hydrochloric acid, using phenolphthalein indicator. Evaporate the neutral solution on a water-bath and dry to constant mass at 105 ± 2°C. From the mass of the residue subtract the mass of sodium chloride (NaCl) calculated from the amount of hydrochloric acid used up. This difference will be due to neutral salts which should be calculated per ml of the caustic solution.

10.3.4 To obtain the value for total anhydrous soap, deduct the neutral salt calculated from the volume in ml of sodium hydroxide used for titration from the mass of the residue obtained after titrating the fatty acids and drying.

10.3.5 Use the following formulae in making the calculations:

a) Combined alkali, percent by mass = $\frac{VNE}{M}$

where

V = volume in ml of standard sodium hydroxide solution required for the material;

N = normality of standard sodium hydroxide solution;

E = 4.00, 3.10 or 4.71, depending upon whether the result is to be expressed as sodium hydroxide (NaOH), sodium oxide (Na_2O) or potassium oxide (K_2O) respectively; and

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

b) Total anhydrous soap, percent by mass = $\frac{100\,(m - KV)}{M} - U$

where

m = mass in g of the soda soap;

K = correction, grams of neutral salts per millilitre of standard sodium hydroxide solution;

V = volume in ml of standard sodium hydroxide solution;

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

U = unsaponified and unsaponifiable matter present, percent by mass (*see* **12**).

10.4 For soaps containing a large amount of soluble silicates and other builders, and soap products containing a high percentage of finely divided material insoluble in water, the procedure under **10.2** cannot be applied as given. In such cases, the filtrate obtained in the determination of total matter insoluble in alcohol under **6.2.1** may be used. Evaporate the alcohol on a steam-bath, and proceed according to the instructions given under **10.3.1** and **10.3.2**.

11 DETERMINATION OF CHLORIDES

11.0 General

This method determines the chlorides in the sample and is applicable to all soaps and soap products, including those containing synthetic detergents. From an aqueous solution of soap, the fatty acids are precipitated as insoluble calcium soaps by the addition of calcium nitrate solution. This precipitate is washed free of chlorides and the chlorides in the washings and filtrate estimated by titration with standard silver nitrate solution, using potassium chromate as indicator.

11.1 Reagents

11.1.1 *Calcium Nitrate Solution* — neutral, chloride-free and containing 20 percent (*m/v*) of calcium nitrate crystals [$Ca(NO_3)_2, 4H_2O$].

11.1.2 *Methyl Orange Indicator* — Dissolve 0.1 g in 100 ml of 95 percent rectified spirit.

11.1.3 *Dilute Nitric Acid* — approximately 1 N.

11.1.4 *Potassium Chromate Indicator* — Dissolve 5 g of potassium chromate (K_2CrO_4) in 100 ml of water.

11.1.5 *Standard Silver Nitrate Solution* — approximately 0 1 N.

11.1.6 *Calcium Carbonate or Magnesium Carbonate*

11.2 Procedure

11.2.1 Weigh accurately about 10 g of the sample and dissolve in hot water in a 250-ml tall form beaker, add 20 ml of the calcium nitrate solution, mix thoroughly, cool and filter into a one-mark 250-ml volumetric flask. Wash the filter free from chlorides using water allowing the washing to run into the flask. Shake the flask and the contents and dilute to the mark.

11.2.2 Take 100 ml of the solution, neutralize to methyl orange indicator with dilute nitric acid, add pitch of calcium or magnesium carbonate and titrate with silver nitrate solution, using potassium chromate solution as indicator. Carry out a blank determination using the same quantity of all reagents but except the sample.

11.3 Calculation

a) Chlorides (as NaCl), percent by mass = $\frac{14.6(S - B)N}{M}$

b) Chlorides (as KCl), percent by mass $= \frac{18.65(S-B)N}{M}$

where

S = volume in ml of standard silver nitrate solution required for the material,

B = volume in ml of standard silver nitrate solution required for the blank,

N = normality of standard silver nitrate solution, and

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

12 DETERMINATION OF UNSAPONIFIED AND UNSAPONIFIABLE MATTER

12.0 General

The soap sample is dissolved in 50 percent alcohol and extracted with petroleum ether. The ether extract is washed free from soap and the residue obtained after evaporating the solvent is weighed. This will contain unsaponifiable and unsaponified matter. To make a correction for any fatty acids which may have been formed due to the hydrolysis of soap, the residue is dissolved in alcohol and titrated with standard alkali, and acidity, calculated as oleic acid, is subtracted from the mass of the residue.

12.1 Reagents

12.1.1 *Sodium Bicarbonate* — solid.

12.1.2 *Ethyl Alcohol* — conforming to IS 321, 95 percent (by volume) or rectified spirit conforming to IS 323, redistilled.

12.1.3 *Ethyl Alcohol* — or rectified spirit, redistilled and diluted with water to give 50 percent (*v/v) s*olution.

12.1.4 *Ethyl Alcohol* — or rectified spirit, redistilled and Muted with water to give 10 percent (*v/v)* solution.

12.1.5 *Sodium Hydroxide Solution* — approximately 0.1 N.

12.1.6 *Standard Sodium Hydroxide Solution* — approximately 0.02 N.

12.1.7 *Petroleum Ether* — The solvent shall be of the pentane type, containing a minimum amount of *iso*pentane, and *iso*hexane. The nonvolatile evaporation residue shall be not more than 0.001 percent (*m/v*) and the material shall comply with the following additional requirements:

a)	Distillation test	Initial boiling point	35° to 38°C
		Dry flask end point	52° to 60°C
		Distilling under 54°C, *Min*	95 percent
		Distilling under 40°C, *Max*	60 percent
b)	Relative density at 15.5°/15.5°C	0.630 to 0.660	
c)	Colour	Water white	
d)	Doctor test	Sweet	
e)	Copper strip corrosion test	Non-corrosive	
f)	Unsaturated compounds	Only traces permitted	
g)	Residue in distilling flask	Neutral to methyl orange	
h)	Blotter strip odour test	Odourless within 12 minutes	
j)	Aromatic compounds	No nitrobenzene odour	
k)	Saponification value, Min	1.0 mg of potassium hydroxide per 100 m	

12.2 Procedure

12.2.1 Accurately weigh about 5 g of the sample, place it in a 250-ml conical flask or beaker containing approximately 0.1 g of sodium bicarbonate and dissolve in 100 ml of 50 percent ethyl alcohol. Warm and shake to effect solution, keeping the temperature under 60°C. Transfer any undissolved residue to a Gooch crucible with an asbestos pad or to a funnel using an asbestos pad deposited on a perforated porcelain disc. Wash three times with hot 50 percent ethyl alcohol. Wash with a small amount of petroleum ether to remove any traces of unsaponified and unsaponifiable matter. Transfer the entire filtrate and washings to a 500- ml separating funnel, washing with about 50 mi of 50 percent ethyl alcohol followed by two 10-ml portions of petroleum ether. Add 50 ml of petroleum ether, shake vigorously for one minute and allow to settle until both the layers are clear. Draw off completely the lower aqueous layer into another separating funnel of 500 ml capacity.

12.2.2 Repeat the extraction at least six times, using 50 ml of petroleum ether each time. Wash the combined petroleum ether extracts, first with a mixture of 15 ml of sodium hydroxide solution and 15 ml of 95 percent ethyl alcohol and then at least thrice with 25-ml portions of 10 percent ethyl alcohol, shaking vigorously each

time, until the wash water does not show any pink colour to phenolphthalein. Transfer the petroleum ether extracts to a beaker and evaporate the petroleum ether on a steam-bath with the help of a current of air.

12.2.3 Test the residue for solubility by treating with 50 ml of petroleum ether at room temperature. Filter the insoluble residue, if any, and collect the filtrate and washings into a tared flask. Evaporate and dry in the same manner on a steam-bath and, finally, in an air-oven at 100° to 10l°C for 30 minutes. Weigh and return to the oven, reweighing at 15-minutes intervals until constant mass is reached. Take up the residue in 50 ml of 95 percent ethyl alcohol neutralized to phenolphthalcin indicator and titrate to the same colour as that of the original neutral alcohol with 0.02 N standard sodium hydroxide solution.

12.2.4 Make a blank test on the petroleum ether by evaporating 250 ml of the ether with about 0.25 g of stearin or some other hard fat previously brought to constant mass by heating and drying as in the actual determination. The blank should not exceed a few milligrams. Deduct the value for the blank on the petroleum ether from the mass before calculating the unsaponified and unsaponifiable matter.

12.3 Calculation

Calculate the fatty acids as oleic acid ($C_{18}H_{34}O_2$) equivalent of the 0.02 N standard sodium hydroxide solution used in the titration under **12.2.3,** deduct this from the mass of the residue and express as percentage of the mass of the material taken for the test.

12.3.1 Mass of fatty acids in the extract, as oleic acid = 0.282 5 *VN*

where

V = volume in ml of 0.02 N standard sodium hydroxide solution, and

N = normality of 0.02 N standard sodium hydroxide solution.

12.3.2 Unsaponified and unsaponifiable matter, percent by mass = $\frac{100(M_1 - M_2)}{M_3}$

where

M_1 = mass in g of the residue,

M_2 = mass in g of the fatty acids (*see* **11.3.1**), and

M_3 = mass in g of the material taken for the test.

13 DETERMINATION OF UNSAPONIFIABLE MATTER

13.0 General

The alcoholic solution of the sample is refluxed with potassium hydroxide solution and extracted with petroleum ether, when cold. The petroleum ether extract is then dried and weighed.

13.1 Reagents — Besides all the reagents listed under **12.1,** the following solution is required.

13.1.1 *Potassium Hydroxide Solution* — 50 percent (*m/v*).

13.2 Procedure

Accurately weigh 5 g of the sample and place in a 200-ml conical flask. Add 30 ml of 95 percent ethyl alcohol and 5 ml of potassium hydroxide solution and boil for one hour under a reflux condenser. Transfer to a separating funnel and wash with 95 percent ethyl alcohol. Complete the transfer, first with warm and then with cold water, until the total volume is about 80 ml. Repeat the process with a small quantity of petroleum ether. Cool to room temperature and add 50 ml of petroleum ether. Then proceed with the extraction as outlined under **11.2**. Weigh the residue.

13.3 Calculation

Unsaponifiable matter, percent by mass $= \frac{100(M_1 - M_2)}{M_3}$

where

M_1 = mass in g of the residue,

M_2 = mass in g of the fatty acids (*see* **11.3.1**), and

M_3 = mass in g of the material taken for the test.

13.3.1 Correct for fatty acids and express the result as percentage of the mass of the material taken for the test.

14 DETERMINATION OF UNSAPONIFIED MATTER

14.1 Calculation

From the percentage of total unsaponified and unsaponifiable matter determined under **12**, deduct the percentage of unsaponifiable matter obtained under **13** and report the difference as percentage of unsaponified matter.

15 DETERMINATION OF ROSIN

15.0 General

Two methods for estimation of rosin have been prescribed. The method given in **15.1** shall be used as referee method and that given in **15.2** shall be used for routine analysis. The latter method is applicable when rosin acids content is not more than 15 percent of total fatty matter.

15.1 Referee Method

15.1.1 *Reagents*

15.1.1.1 *Dilute sulphuric acid* — 30 percent (*m/v*) obtained by cautiously adding 16 ml of sulphuric acid, relative density 1.84 (conforming to IS 266), to 70 ml of water.

15.1.1.2 *Beta-naphthalene sulphonic acid solution* — Dissolve 40 g of the reagent in one litre of chemically pure, absolute methyl alcohol.

15.1.1.3 *Standard alcoholic potassium hydroxide solution* — approximately 0.2 N, in 95 percent (*m/v*) ethyl alcohol or in rectified spirit (*see* IS 323), accurately standardized. As alcohol is volatile, frequent restandardization is necessary.

15.1.1.4 *Phenolphthalein indicator* — same as in **6.1.1**.

15.1.2 *Procedure*

15.1.2.1 Dissolve 10 to 50 g of the sample in about 500 ml of hot water. Add 10 to 50 ml of dilute sulphuric acid to split the soap, keep in a steam-bath until the fatty matter separates as a clear layer and siphon off the lower aqueous acid layer. Add 300 ml of hot water, boil gently for a few minutes and siphon off the aqueous layer. Repeat the washing with hot water several times until the wash liquor is free of mineral acids. Complete the acidification and washing in as short a period as possible, keeping the beaker covered to prevent oxidation of the acids. Remove the mixture of rosin and fatty acids by means of a dry pipette, filter through one or two thicknesses of filter paper, and dry at 105 ± 2°C for 45 to 60 min.

15.1.2.2 Weigh accurately 2 g of the mixture of fatty and rosin acids into an esterification flask and add 25 ml of beta-naphthalene sulphonic acid solution. Boil gently under a reflux condenser for 30 minutes, adding a few glass beads to ensure smooth boiling. Cool the contents of the flask and titrate immediately with standard alcoholic potassium hydroxide solution, using 0.5 ml of phenolphthalein indicator. The end point is reached when pink colour persists for 30 seconds.

15.1.2.3 Conduct simultaneously a blank determination with 25 ml of the esterifying agent alone.

15.1.3 *Calculation*

$$\text{Rosin in fatty acids, percent by mass, uncorrected} = \frac{34.6(S-B)N}{M}$$

where

S = volume in ml of standard alcoholic potassium hydroxide solution required for the material,

B = volume in ml of standard alcoholic potassium hydroxide solution required for the blank,

N = normality of alcoholic potassium hydroxide, and

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

15.1.3.1 The method described in **15.1.2** gives results approximately one percent higher than the amount of rosin actually present. Consequently, the percentage of rosin acids actually present is one less than the percentage of rosin acids found experimentally.

15.1.3.2 Rosin in fatty acids, percent by mass, corrected = Rosin in fatty acids, percent by mass, uncorrected – 1.0.

NOTES

1 The mean equivalent mass of the rosin acids is taken as 346.

2 When the quantity of rosin, expressed as percent by mass, is less than 5 in the soaps, the results by this method are not so accurate as with soaps containing higher rosin content. This method is also liable to give erroneous results with certain types of carbolic soaps containing high boiling tar acids and with other germicidal soaps, for example, soaps containing hexachlorophene.

15.1.4 *Liebermann-Storch Test*

In all cases where the rosin content is found to be less than 5 percent, the actual presence or absence of rosin should be checked qualitatively by the Liebermann-Storch test, described below.

15.1.4.1 *Reagents*

a) *Acetic anhydride* — pure.

b) *Dilute sulphuric acid* — relative density 1.53.

15.1.4.2 *Procedure*

Transfer 1 to 2 ml of the sample of fatty acids to a test-tube, treat with 5 to 10 ml of acetic anhydride and warm on a steam-bath. After cooling, pour 1 to 2 ml into a white porcelain dish and allow a drop or two of sulphuric acid to run down the side of the vessel. If rosin is present, a fugitive violet colouration changing to a brownish tinge is immediately produced at the margin of contact of the reagents. Check the test with a sample of fatty acids to which a small amount of rosin has been added.

15.2 Method for Routine Analysis of Rosin Acids

15.2.0 *General*

Applicable to fatty matter containing not more than 15 percent rosin acids.

15.2.1 *Apparatus*

15.2.1.1 Conical flask, 250 ml, of chemically resistant glass with a standard taper 24/40neck.

15.2.1.2 Condenser, water-cooled with joint fitting the flask given in **15.2.1.1.**

15.2.1.3 Separatory funnel, 500 ml, pear-shape, fitted with a glass stopper.

15.2.1.4 *Burette*

50 ml capacity with 0.1 ml divisions.

15.2.2 *Reagents*

15.2.2.1 *Dilute sulphuric acid* — 30 percent (*m*/*v*)

obtained by cautiously adding 16 ml of sulphuric acid, relative density 1.84 (*see* IS 266), to 70 ml water.

15.2.2.2 *Methanol*, 99.5 percent**.**

15.2.2.3 *Sulphuric acid*, relative density 1.84 (*see* IS 266 LR Grade).

15.2.2.4 *Sodium sulphate solution*, 10 percent. Dissolve 100 g of anhydrous sodium sulphate (Na_2SO_4) in water.

15.2.2.5 *Petroleum ether*, 40-60.

15.2.2.6 *Methyl orange indicator* — Dissolve 0.1 g of methyl orange in 100 ml of water.

15.2.2.7 *Ethyl alcohol*, neutral 95 percent ethyl alcohol, or neutral denatured alcohol**.**

15.2.2.8 *Phenolphthalein indicator*— Dissolve 1 g of phenolphthalein in 100 ml of methanol.

15.2.2.9 *Standard alcoholic potassium hydroxide solution* (0.2 N or 0.5 N) — Dissolve 11.2 g of potassium hydroxide (preferably in pellet form) for a 0.2 N solution or 28.0 g for 0.5 N solution in methanol. Dilute to 1 litre with methanol. Standardize to ± 0.001 N. The standardized solution should be protected against evaporation and absorption of carbon dioxide from air, and should be restandardized frequently.

15.2.3 Procedure

15.2.3.1 Weigh 40 ± 0.1 g soap sample and dissolve in 500 ml of hot water. Add 40 ml of dilute sulphuric acid to split the soap, keep in a steam bath until the fatty matter separates as a clear layer and siphon off the lower aqueous acid layer. Add 300 ml of hot water. Boil gently for a few minutes and siphon off the aqueous layer. Repeat the washing with hot water several times until the wash liquor is free of mineral acids. Complete the acidification and washing in as short period as possible, keeping the beaker covered to prevent oxidation of the acids. Remove the mixture of rosin and fatty acids by means of a dry pipette, filter through two thicknesses of filter paper of size 40 micron, and dry at 105 ± 2°C for 45-60 min. Dissolve the entire mass in 100 ml methanol in a 250 ml conical flask. Swirl the flask to dissolve the oil and add a clean boiling chip.

15.2.3.2 Add slowly 5 ml of sulphuric acid while swirling the flask vigorously. Connect the flask to the condenser, apply heat, and reflux the contents for 10 minutes.

15.2.3.3 Cool the flask to room temperature with cold water. Add 250 ml of sodium sulphate solution to a 500 ml separatory funnel. Pour the contents of the flask into the funnel and complete quantitative transfer of the contents of the flask with 100 ml of petroleum ether.

15.2.3.4 Thoroughly shake the mixture in the funnel. Allow to settle, draw off the salt layer and discard. Wash contents of the funnel twice again with 250 ml portions of sodium sulphate solution. The last washing should not react pink to methyl orange indicator. After removing the last wash, drain the contents separatory funnel into a 500-ml conical flask. Rinse the funnel with 20 ml of ether and add the rinsings to the flask.

15.2.3.5 Add 20 ml of ethyl alcohol and 1 ml of phenolphthalein indicator. Titrate to the appearance of a pink-red colour using 0.2 N alcoholic potassium hydroxide solution if the rosin acid content is less than 5 percent and 0.5 N if more than 5 percent.

NOTE — For highly coloured samples it will be difficult to identify the accurate end point using phenolphthalein as indicator. In this case, to the ether extract of **15.2.3.4**, add 5 to 6 g of activated charcoal and swirl the flask for 2 to 3 min. Filter the solution through a filter paper. Add mixed indicator for sharper end point, the colour change is from yellow to blue. Mixed indicator is the mixture of 6 parts of thymol blue and 1 part of cresol red. This mixture is violet at pH 8.4, blue at *p*H 8.3 and rose at *p*H 8.2.

15.2.4 *Calculation*

Calculate the percentage of rosin acids as follows:

$$\text{Rosin acids, percent by mass of soap} = \frac{1.031 \times V \times N \times 30.2}{M} - 0.74$$

where

V = volume of standard potassium hydroxide solution used in titration; in ml;

N = normality of potassium hydroxide solution used;

M = mass of the soap sample taken for analysis in g; and

0.74= concentration factor.

NOTE — If the calculated value of rosin acids comes out in negative, it shall be reported as nil.

15.2.4.1 Further to above, the rosin acids content based on Total Fatty Matter (TFM) may be calculated using the following formula:

$$\text{Rosin acids, percent by mass of TFM} = \frac{\text{Percentage of rosin acid based on soap} \times 100}{\text{TFM percentage}}$$

16 DETERMINATION OF TOTAL FATTY MATTER

16.0 General

he soap split by dilute sulphuric acid is extracted by ethyl ether as in the determination of combined alkali and the ether extract evaporated. The residue is treated with acetone, evaporated and estimated.

16.1 Procedure

Follow the method given in **10.2.1** until the combined

ether extract is free from acid or the wash water is neutral to methyl orange indicator. Mix the ether solution (if necessary, filter previously washing the paper with ether) in a suitable, weighed vessel. Distil off the ether slowly on a steam-bath, and, to the residue, add 5 ml of acetone. (In order to minimize the risk of loss during distillation, the flask should not be more than half full of ether at any stage.) Warm the flask on the steam-bath for about one minute, remove it from the bath and then, while imparting a rotatory motion to the flask hold it at an angle of 45°, direct a current of dry air into its mouth for about one min, thereby removing the bulk of acetone. Place the flask in a air oven at about 90°C for 10 min, remove it from the oven and blow with air as before for about 15 s. Allow the flask to cool and weigh. Return the flask to the steam-oven for another 10 min and blow for 15 s. Allow to cool and reweigh. Repeat the process until the difference between two consecutive weighings is less than 0.005 g.

16.2 Calculation

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

where

M_1 = mass in g of the fatty matter, and

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

NOTE — With soaps containing fatty acids, having more than about 20 percent of fatty acids of molecular mass 200 or below, there is some risk of loss by volatilization if heating in the oven is unduly prolonged. In such cases, it may be preferable to convert the total fatty acids into sodium soaps by titrating with ethanolic sodium hydroxide as in **10.2.2** and **10.2.3**, drying the soaps and calculating the total fatty matter. Allowance must be made for any trace of neutral salts found to be present in the sodium hydroxide solution.

17 DETERMINATION OF TITRE OF TOTAL FATTY ACIDS

17.0 General

The sample of soap is decomposed with dilute sulphuric acid and the fatty acid layer separated. The solidification point of this material is determined under prescribed conditions.

17.1 Apparatus

17.1.1 *Low Form Beaker* — of 2-litre capacity, to serve as a water-bath.

17.1.2 *Wide Mouth Bottle* — of 450-ml capacity, height 190 mm and inside diameter of neck 38 mm.

17.1.3 *Test-Tube* — 100 mm in length and 25 mm in diameter, with an etched mark extending around the tube at a distance of 57 mm from the bottom.

17.1.4 *Stirrer* — made of stainless steel or Monel metal with one end bent in the form of a loop of 19 mm outside diameter. The upper end may be formed to suit stirring with hand or attached to a mechanical stirrer.

17.1.5 *Laboratory Thermometer* — range up to 150°C.

17.1.6 *Titre Thermometer* — with the following characteristics:

a) *Type* — etched stem glass.
b) *Liquid* — mercury.
c) *Filling above liquid* — evacuated or nitrogen gas.
d) *Temperature range* — –2°C to 68°C.
e) *Subdivisions* — 0.2°C.
f) *Total length* — 385 to 390 mm.
g) *Stem diameter* — 6 to 7 mm.
h) *Stem construction* — Plain or lens front. The cross-section of the lens front type shall be such that it will pass through an 8-mm ring gauge but will not enter a 5-mm slot gauge.
j) *Bulb diameter* — from 5.5 mm to not greater than the diameter of the stem.
k) *Bulb length* — 15 to 25 mm.
m) *Distance from the bottom of the bulb to –2°C mark* — 50 to 60 mm.
n) *Distance from 68°C mark to the top of the thermometer* — 20 to 35 mm.
p) *Length of unchanged capillary between the highest graduation mark and the expansion chamber* — 10 mm.
q) *Expansion chamber* — to permit heating to at least 85°C.
r) *Top finish* — Glass ring.
s) *Longer graduation lines* — at each 1°C mark.
t) *Graduations numbered* — at zero and at each multiple of 2°C.
u) *Immersion* — 10 mm. A line shall be attached around the stem, 45 mm from the bottom of the bulb.
v) *Maximum scale error permitted at any point* — 0.2°C.
w) *Standardization* — The thermometer shall be standardized at the ice point and at intervals of approximately 20 deg, for the condition of 45 mm immersion, and for an average stem temperature of the emergent mercury column of 25 deg.

17.2 Reagents

17.2.1 *Dilute Sulphuric Acid* — 30 percent (*m/v*) obtained by cautiously adding 16 ml of concentrated sulphuric acid, relative density 1.84 (*see* IS 266), to 70 ml of water.

17.2.2 *Acetone* — pure (*see* IS 170).

17.3 Procedure

17.3.1 *Preparation of Fatty Acids*

Dissolve approximately 50 g of the sample in 500 ml of hot water contained in a 1 000-ml beaker. Add sufficient dilute sulphuric acid until the solution is distinctly acidic to methyl orange and place in a boiling water-bath until the fatty acids collect as a clear layer at the top. Remove the aqueous acid (lower) layer with a siphon, add 300 ml of hot water, place in the boiling water-bath for a few minutes, and again remove the queous acid layer with a siphon.

Wash the fatty acids thrice in this manner. Complete the acidification and washing in as short a period as possible, keeping the beaker covered to prevent oxidation of fatty acids. After the last wash, allow the fatty acids to settle for a few minutes and then decant them carefully. Filter through one or two thicknesses of filter paper, introduce into a conical flask and add 10 ml of acetone. Close the flask with an air-tight cork, carrying a glass tube. Immerse the flask in boiling water and apply suction from a water pump until bubbling ceases. Remove the cork and dry the flask at 105 ± 2°C for at least half an hour.

17.3.2 *Determination of Titre*

Adjust the temperature of the water-bath to 20 ± 1°C for all samples having titres of 35°C or higher, and to 15 to 20°C below the titre point for all samples with titres below 35°C. Place the fatty acids, prepared as prescribed in **17.3.1**, in the test-tube up to the etched mark and insert the titre thermometer in the centre of the sample. Suspend it at such a height that the immersion mark coincides with the top of the sample of fatty acids. When the titre test thermometer reads about 10°C above the expected titre value, set the stirrer moving in a vertical manner at the rate of about 100 complete up-and-down motions per minute. Continue stirring until the temperature remains constant for 30 s. Stop stirring when the temperature begins to rise, remove the stirrer or raise it out of the sample and observe the increase in temperature. Titre point is the highest temperature indicated by the thermometer during this rise. Duplicate determinations should agree within 0.2°.

18 DETERMINATION OF IODINE VALUE (WIJS)

18.0 General

A known quantity of fatty acids prepared as for the titre test is treated in carbon tetrachloride or chloroform medium with a known excess of iodine monochloride solution in glacial acetic acid (Wijs solution). The excess iodine monochloridc is treated with potassium iodide and the liberated iodine estimated by titration with sodium thiosulphate solution.

18.1 Apparatus

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

18.2 Reagents

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

a) *Melting point determination* — Fill a test-tube, 15 cm long, about two-thirds with acetic acid, and insert the thermometer described in **18.1** 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 that 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, withdraw the assembly from the ice water, stir the acid vigorously for a few moments, thereby causing the super-cooled liquid to crystallize partially and giving a mixture of liquid and solid acid. Take thermometer readings every 15 seconds and consider as true melting point that temperature at which the reading remains constant for at least 2 min.

b) *Test for reducing impurities (potassium permanganate test*) — Dilute 2 ml of acetic acid with 10 ml of water, add 0.1 ml of 0.1 N potassium permanganate solution and maintain at 27 ± 2°C. The test shall be taken as having been satisfactory if pink colour is not discharged at the end of two hours.

18.2.2 *Carbon Tetrachloride or Chloroform* — The reagent shall be inert to Wijs solution.

18.2.3 *Standard Sodium Thiosulphate Solution* — Dissolve pure sodium thiosulphate crystals ($Na_2S_2O_3.5H_2O$) in water, which has been well boiled to free it from carbon dioxide, in proportion so that 24.83 g of sodium thiosulphate is contained in one litre of the solution. Let this solution stand for about two weeks before standardizing. Standardize with pure re-sublimed iodine or potassium iodate. The solution will be approximately 0.1 N and it is better to leave it as it is after determining its exact normality instead of attempting to adjust it to exactly 0.1 N strength. Preserve in a dark-coloured stock bottle with a guard

tube filled with soda lime. The strength of the solution should be checked occasionally. A few drops of chloroform may be added to the solution for better preservation.

18.2.4 *Starch Solution* — Make a paste of 0.5 g of soluble starch in cold water, pour it into 100 ml of boiling water, boil for 5 min, cool and bottle. The solution should be prepared afresh every two or three days.

18.2.5 *Potassium Iodide Solution* — Prepare fresh solution by dissolving 10 g of potassium iodide, free from potassium iodate, in 90 ml of distilled water.

18.2.6 *Wijs Iodine Monochloride Solution* — Prepare this solution by one of the following three methods, and store in a glass-stoppered bottle in cool place, protected from light:

a) *From iodine* — Dissolve 13 g of iodine in one 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 dry 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 iodine-acetic acid solution. A slight excess of iodine does no 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, 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) *From iodine trichloride* — Dissolve 8 g of iodine trichloride in approximately 450 ml of glacial acetic acid. Dissolve separately 9 g of iodine in 450 ml of glacial acetic acid, heating if necessary. Add gradually the iodine solution to iodine trichloride solution until the colour has changed to reddish brown. Add 50 ml more of iodine solution and dilute the mixture with glacial acetic acid till 10 ml of the mixture is 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 iodide and water. Heat the solution to 100°C for 20 minutes and cool. Prevent access of water vapour in preparing the solution.

c) *From iodine monochloride* — Dissolve 10 ml of iodine monochloride in about 800 ml of glacial acetic acid and shake vigorously. Pipette 5 ml of this, add 10 ml potassium iodide solution (10 percent) and titrate with 0.1 N sodium thiosulphate solution, using starch solution as indicator. Adjust the volume of the solution till it is 0 2 N.

18.3 Procedure

18.3.1 It is essential that all the glass apparatus used in this experiment should be perfectly clean and dry.

18.3.2 Prepare the fatty acids as prescribed under **17.3.1.**

18.3.3 Weigh in a small glass tube an appropriate quantity of the fatty acids as indicated in Table 4. Drop the tube into a clean, dry 500-ml glass-stoppered bottle, to which 25 ml of carbon tetrachloride have been added, and agitate to dissolve the contents. Add 25 ml of Wijs solution and replace the glass stopper after wetting with potassium iodide solution.

Swirl for intimate mixing and allow 10 stand for 30 minutes in a dark place. Carry out a blank test simultaneously under similar experimental conditions. After 30 minutes, add 20 ml of potassium iodide solution and 100 ml of water and titrate the liberated iodine with standard sodium thiosulphate solution, swirling the system continuously to avoid any local excess until the yellow colour just disappears. Add 0.5 ml of starch solution and continue the titration until the blue colour disappears.

18.4 Calculation

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

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.

Table 4 Mass of Fatty Acids for Iodine Value Determination

(*Clause* 18.3.3)

Expected Iodine Value	Mass in g		Weighing Accuracy
	Maximum	Minimum	
(1)	(2)	(3)	(4)
< 3	—	10.00 0	± 0.001 0
5	6.346 0	5.077 0	± 0.000 5
10	3.173 0	2.538 4	± 0.000 2
60	0.528 3	0.423 1	± 0.000 1
100	0.317 3	0.253 8	± 0.000 1

19 DETERMINATION OF ALKALINE SILICATES

19.0 General

The alkaline silicates present in soaps arc determined as sodium silicate from the charred residue of the soap and its water extract.

19.1 Apparatus

19.1.1 *Platinum Evaporating Dish* — 10-ml capacity.

19.1.2 *Platinum Crucible* — 30-ml capacity.

19.1.3 *Watch Glass*

19.1.4 *Ashless Filter Papers*

19.2 Reagents

19.2.1 *Hydrochloric Acid* — concentrated, relative density 1.16 *(see* IS 265).

19.2.2 *Hydrofluoric Acid* — 48 percent (*m/v*).

19.2.3 *Sulphuric Acid* — concentrated, relative density 1.84 (*see* IS 266).

19.3 Procedure

19.3.0 The procedure varies depending upon whether the material contains mineral matter insoluble in water, or not.

19.3.1 When the material contains no mineral matter insoluble in water, ignite 1 to 5 g of it [quantity taken containing not more than 0.2 g of silica (SiO_2)], in a platinum evaporating dish over a burner at low temperature (350 to 400°C). When charred, extract the water soluble material with water, return the filter paper and the residue to the platinum dish and continue ignition to bright red heat (850 to 950°C) till all the carbonaceous material is removed. Continue the water extract and the residue, and carefully neutralize with hydrochloric acid, avoiding any loss by spray by keeping the dish covered. Finally, add 5 to 10 ml of hydrochloric acid in excess.

19.3.2 When the sample contains mineral matter insoluble in water, take a portion of the solution containing not more than 0.2 g of silica (SiO_2) after titrating the matter insoluble in alcohol but soluble in water as under **9.2** and add 5 to 10 ml of hydrochloric acid.

19.3.3 Evaporate the acidified solution, obtained as prescribed under **19.3.1** or **19.3.2** to dryness on a steam-bath. Cool the residue and moisten with hydrochloric acid. Add about 25 ml of hot water, heat for a few minutes and filter through an ashless filter paper. Wash thoroughly with water. Evaporate the filtrate to dryness and repeat the above treatment, filtering through a second ashless filter paper. Preserve this filtrate for qualitative tests under **20.1.2** and **21.1.2**. Place the two filter papers in a tared platinum crucible and ignite carefully, first at low temperature to burn off the paper and then at bright red heat (850°C) over a burner. Cool in a desiccator and weigh. Repeat heating, cooling and weighing to constant mass.

19.4 Calculation

$$\text{Sodium silicate } (Na_2O,\ 2SiO_2),\ \text{percent by mass} = 151.6\frac{A}{M}$$

where

A = mass in g of the residue, and

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

19.4.1 Neutral sodium silicate ($Na_2O,\ 3.25SiO_2$), percent by mass = $131.8\frac{A}{M}$

where A and M have the same legends as in **19-4.**

19.5 Referee Method

For accurate results, moisten the weighed contents of the crucible (obtained as prescribed under **19.3.3**) with water, add 10 ml of hydrofluoric acid and 4 drops of sulphuric acid and evaporate to dryness over a low flame. Ignite as directed previously, cool to room temperature in a desiccator and weigh. The difference between this mass and that of the residue found under **19.3.3** is the mass of the silica.

20 DETERMINATION OF BORAX

20.0 General

Conduct the qualitative test described under **20.1** and, if the result is positive, proceed with the quantitative determination described under **20.2**.

20.1 Qualitative Test

20.1.1 *Reagents*

a) *Turmeric test paper* — prepared by impregnating unglazed white paper with the clear extract obtained by macerating for one week 10 g of bruised turmeric in 60 ml of alcohol (95 percent by volume).

b) *Potassium iodide solution* — 5 percent (*m/v*).

c) *Starch solution* — perpared as under **18.2.4**.

d) *Dilute sulphuric acid* — 1 : 4 by volume.

20.1.2 *Procedure* — Wet a piece of turmeric test paper with a few milliliters of filtrate obtained under **19.3.3** after acidifying the filtrate. A borate or perborate is present if the paper, on drying in air, turns deep brick red. To determine whether the colouration is due to perborate, dissolve about 2 g of the original analysis sample in about 100 ml of potassium iodide solution containing 2 ml starch solution. Add 10 ml of dilute sulphuric acid and stir. A blue solution denotes the presence of an oxidizing agent which, with a positive turmeric test, confirms the presence of perborate.

20.2 Quantitative Test

20.2.1 *Apparatus*

a) *Platinum evaporating disk* — 100-ml capacity.
b) *Round-bottom flask* — 250-ml capacity.
c) *Water-cooled reflux condenser*

20.2.2 *Reagents*

a) *Fusion mixture* — prepared by mixing 200 g of sodium carbonate with 15 g of powdered silica.
b) *Ethyl alcohol* — 95 percent (*v/v*).
c) *Dilute hydrochloric acid* — 1 : 1 (*v/v*).
d) *Calcium carbonate* — powder.
e) *Standard sulphuric acid* — approximately 1 N.
f) *Methyl orange indicator* — Dissolve 0.1 g in 100 ml of water.
g) *Standard sodium hydroxide solution* — approximately 0.1 N, carbon dioxide-free.
h) *Glycerine* — conforming to CP Grade of IS1796
j) *Phenolphthalein indicator* — as in **6.1.1**.

20.2.3 *Procedure* — Weigh accurately about 10 g of the sample (or about 5 g if more than 5 percent borax is present) into a platinum evaporating dish and add 2.15 g of fusion mixture and 15 ml of ethyl alcohol. Mix the whole with a glass rod and, after washing the rod with a little ethyl alcohol, evaporate the mixture to dryness on a water-bath. Char the mixture thoroughly and ignite to fusion over a burner. Cool, extract the residue with boiling water into a round-bottom flask and acidify with 20 ml of dilute hydrochloric acid. An excels of acid is necessary if any phosphates are to be hydrolyzed. Boil under reflux for 2 h, add a moderate excess of calcium carbonate and continue boiling vigorously for 20 min. Add a further small amount of calcium carbonate if the precipitate is gelatinous. Filter and wash several times with small quantities of water. Transfer the filtrate and washings into a round-bottom flask, and neutralize with standard sulphuric acid in the presence of methyl orange indicator. To the neutral solution, add 0.1 ml of standard sulphuric acid, boil for 5 minutes more, cool the solution and neutralize with standard sodium hydroxide solution. Add 50 ml of glycerine to the solution and titrate it with standard sodium hydroxide solution in the presence of phenolphthalein indicator. After the end point is reached, add another 10 ml of glycerine and continue the titration until pink colour appears again. Repeat this until the addition of glycerine causes no further change in the end point.

20.2.4 *Calculation*

$$\text{Borax (as } Na_2B_4O_7.\ 10H_2O\text{), percent by mass} = 9.536\frac{VN}{M}$$

where

V = volume in ml of standard sodium hydroxide solution required for titration,
N = normality of standard sodium hydroxide solution, and
M = mass in g of the material taken for the test.

20.3 Determination of Perborates

20.3.0 *General* — Persalt content is determined from the iodine set free when the solution is reacted with acidified potassium iodide solution. The iodine liberated is titrated with standard sodium thiosulphate solution. This method is applicable even when ethylenediamine tetracetic acid or perfume is present in the soap.

20.3.1 *Reagents*

a) *Dilute sulphuric acid* — 1 : 1 (*v/v*).
b) *Potassium iodide*
c) *Ammonium molybdate solution* — 3 percent (*m/v*).
d) *Sodium thiosulphate solution* — 0.1 N.
e) *Starch indicator solution*

20.3.2 *Procedure*

Weigh accurately 2 to 5 g of the sample and transfer to a 500-ml glass-stoppered bottle. Dissolve in 100 ml water heated to 37°C and add 2 g of the potassium iodide. Acidify the solution by adding 10 ml of sulphuric acid. Add 1 ml of ammonium molybdate solution. Allow the solution to stand in a dark cupboard for 5 minutes and titrate the liberated iodine with sodium thiosulphate solution using starch solution as indicator.

20.3.3 *Calculation*

$$\text{Sodium perborate } (NaH_2BO_4,\ 3H_2O)\text{, percent by mass} = \frac{0.770T}{M}$$

where

T = volume in ml of 0.1 N sodium thiosulphate solution used, and
M = mass in g of the sample taken.

21 DETERMINATION OF PHOSPHATES

21.0 General

Conduct the qualitative test described under **21.1** and, if the result is positive, proceed with the quantitative determination described under **21.2**.

21.1 Qualitative Test

21.1.1 *Reagents*

a) *Nitric acid* — concentrated, relative density 1.42 (*see* IS 264).
b) *Ammonium nitrate solution* — 30 percent (*m/v*).
c) *Ammonium molybdate solution* — Dissolve 100 g of molybdic acid in dilute ammonium hydroxide solution prepared by adding 270 ml

of water to 144 ml of concentrated ammonium hydroxide solution. Pour the resulting solution slowly, with continuous stirring, into 489 ml of nitric acid and 1 148 ml of water. Keep the mixture in a warm place for several days. Decant the solution and make sure that no yellow precipitate is deposited on heating to 40°C.

21.1.2 *Procedure*

To 20 ml of the filtrate obtained under **19.3.3,** add 2 ml of nitric acid and 5ml of ammonium nitrate solution. Heat to boiling, add 20 ml of ammonium molybdate solution and allow the solution to stand for 20 min on a steam-bath. If a yellow precipitate forms, a phosphate is present. If metaphosphates or pyrophosphates are present, add about 10 ml of nitric acid and heat at or near boiling temperature overnight to convert them to orthophosphates before testing as described above.

21.2 Quantitative Test

21.2.1 *Reagents*

a) *Dilute hydrochloric acid* — 1 : 1 (*v/v*).
b) *Ammonium hydroxide solutions* — concentrated, relative density 0.90, and dilute 1 : 9 (*v/v*).
c) *Nitric acid* — relative density 1.43 (*see* IS 264).
d) *Ammonium nitrate* — solid or 10 percent solution (*m/v*).
e) *Ammonium molybdate solution* — prepared as described under **21.1.1** (c).
f) *Ammonium chloride* — solid.
g) *Magnesia mixture* — Dissolve 55 g of magnesium chloride ($MgCl_2, 6H_2O$) in water, and 140 g of ammonium chloride and 130.5 ml of concentrated ammonium hydroxide solution and dilute the mixture to one litre.

21.2.2 *Procedure*

Accurately weigh about 2 g of the matter insoluble in alcohol or ash and proceed as in the case of determining alkaline silicates under **19.3.** After filtering off the residue, collect the filtrate and washings and make up to 250 ml in a volumetric flask, concentrating if necessary. Take out an aliquot corresponding to 0.05 to 0.10 g of phosphorus pentoxide (P_2O_5), add a slight excess of ammonium hydroxide solution (relative density 0.90), and dissolve the precipitate with a few drops of nitric acid while stirring vigorously. Add 15 g of dry ammonium nitrate or a solution containing that amount, heat to 60°C and test for complete precipitation by the addition of 1 to 2 ml of ammonium molybdate solution. Filter and wash with ammonium nitrate solution, redissolve the precipitate on the filter paper with ammonium hydroxide solution (relative density 0.90) and wash the filter paper with hot water. The total volume of liquid should not exceed 100 ml. Collect the solution and the washings in a beaker and neutralize with dilute hydrochloric acid using litmus paper as an indicator. Cool and add slowly with constant stirring 15 ml of magnesia mixture for every decigram of phosphorus pentoxide (P_2O_5) present. Allow to stand and add 12 ml of ammonium hydroxide solution (relative density 0.90). After 2 h, filter through a filter paper and wash the precipitate with ammonium hydroxide solution (1 : 9) until the washings are free from chlorides. Dry and ignite the filter paper, starting at low heat and finishing at bright red heat (about 1 100°C) till the colour of the pyrophosphate is white. Cool to room temperature and weigh. Repeat ignition, cooling and weighing until constant mass of the precipitated magnesium pyrophosphate ($Mg_2 P_2O_7$) is obtained.

21.3 Calculation

Calculate the phosphates as phosphorus pentoxide as follows:

$$\text{Phosphorus pentoxide, percent by mass} = 63.79\frac{m_1}{m_2}$$

where

m_1 = mass in g of magnesium pyrophosphate, and
m_2 = mass in g of the material corresponding to the aliquot taken for the test.

22 DETERMINATION OF SULPHATES

22.0 General

All organic matter in the soap is thoroughly charred, the residue digested with hydrochloric acid and extracted with hot water. The solution is filtered and the sulphate determined in the filtrate gravimetrically as barium sulphate.

22.0.1 This method is applicable only to soaps and soap products free from sulphonated oils, synthetic detergents and organic compounds containing sulphur.

22.1 *Reagents*

22.1.1 *Hydrochloric Acid* — concentrated, relative density 1.16 (*see* IS 265).

22.1.2 *Barium Chloride Solution* — 10 percent.

22.2 Procedure

Proceed as for the estimation of alkaline silicates under **19.3**, igniting and charring either the original sample or the residue left after the removal of alcohol soluble matter if highly accurate results are not required. Collect the filtrates and washings after removal of any residue, if present, and make up to approximately 200 ml. Add 10 ml of hydrochloric acid, boil the solution and add hot barium chloride solution drop by drop by means of a pipette. Boil for 2 min to coagulate the precipitate and allow to stand and cool for about 4 hours. Filter through a tared Gooch crucible or a sintered glass crucible (G No. 4), wash thoroughly with hot water and heat at 105 to 110°C to constant mass.

22.2.1 The addition of 5 ml of a saturated solution of picric acid after adding 10 ml of hydrochloric acid accelerates the precipitation of barium sulphate and reduces the standing time from 4 h to about 30 min.

22.2.2 Excess of barium chloride is necessary to reduce the solubility of barium sulphate. Precipitation in hot solution by the addition of barium chloride in a slow stream, with stirring, minimizes mechanical occlusion of barium chloride and gives a course precipitate, which is less soluble in acids.

22.3 Calculation

Sulphates (as Na_2SO_4), percent by mass = $60.86 \frac{m}{M}$

where

m = mass in g of barium sulphate, and

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

23 DETERMINATION OF GLYCEROL

23.0 General

The glycerol content is estimated from the formic acid produced by its oxidation by a solution of sodium metaperiodate.

23.1 Apparatus

23.1.1 *p*H *Meter* — with glass electrodes.

23.2 Reagents

23.2.1 *Dilute Sulphuric Acid* — 10 percent (*m/v*).

23.2.2 *Beeswax*

23.2.3 *Sodium Hydroxide Solution* — approximately 1 N.

23.2.4 *Dilute Hydrochloric Acid* — approximately 1 N.

23.2.5 *Sodium Hydroxide Solution* — 0.05 N, accurately standardized against potassium hydrogen phthalate and free from carbonates.

23.2.6 *Sodium Periodate Solution* — 6 percent (*m/v*) solution in carbon dioxide-free water.

23.2.7 *Ethylene Glycol*

23.3 Procedure

23.3.1 Dissolve 20 g of soap in about 150 ml of water in a 250-ml beaker, precipitate the fatty acids by adding a slight excess of sulphuric acid and allow the beaker and its contents to stand either overnight at room temperature or in a steam-bath with frequent stirring until the fatty acid layer is clear. Add about 10 g of beeswax, stir occasionally to ensure homogeneous mixing of the fatty acids and the beeswax and leave the beaker with the glass rod in the bath for a few minutes until the fatty layer is clear. Remove the beaker from the bath, keep overnight and warm slightly to obtain two distinct layers. Cool quickly in running water. Remove the rod with cake attached to it, rinse with water and collect the rinsings in the beaker.

23.3.2 Transfer the aqueous phase and washings to a 250-ml graduated flask, make up to volume and filter through a filter paper (Whatman No. 540 or equivalent is suitable) into a 100-ml graduated flask. Transfer exactly 100 ml of the filtrate into a 500-ml conical flask and make just alkaline with 1 N sodium hydroxide solution. Add about 1 ml excess of hydrochloric acid, boil gently for three minutes to expel carbon dioxide, stopper the flask with a soda lime guard-tube and allow to cool in a bath of cold water. When cooled, wash down the sides of the flask with carbon dioxide- free water and adjust the *p*H of the solution to that used for final titration by adding 0.05 N sodium hydroxide solution. Add 25 ml of sodium periodate solution, insert the guard-tube, swirl and allow the reaction to proceed for 30 minutes in the dark. Wash down the sides of the flask with carbon dioxide-free water, add 5 g of ethylene glycol, swirl the flask and allow to stand in the dark for a further 20 minutes. Finally, titrate the solution with 0.05 N sodium hydroxide solution using the *p*H meter until the *p*H of the solution is 8.1. Carry out a blank test at the same time under exactly the same conditions except that while the *p*H value is adjusted to 8.1 before the addition of the periodate solution, the final titration is carried to the end point of *p*H 6.5 and not 8.1.

NOTE — If a *p*H meter is not available, use phenol red indicator (0.05 percent aqueous solution). Titrate blank and test to the colour change of the indicator (yellow to red). Add 0.3 percent (expressed on 100 percent glycerol) the observed glycerol result. This is not a reference method.

23.4 Calculation

Glycerol, percent by mass = $\frac{1.1511}{m}(T - T_1)$

where

T = volume in ml of 0.05 N sodium hydroxide solution required for test,

T_1 = volume in ml of 0.05 N sodium hydroxide solution used for blank, and

m = mass in g of the sample taken.

24 DETERMINATION OF SUGARS

24.0 General

The matter insoluble in alcohol is extracted from the soap with water to which Fehling solution is added. Sugar reduces Fehling solution to red cuprous oxide under suitable conditions. From the mass of cuprous oxide, the amount of invert sugar is obtained from the Munson and Walker table.

24.1 Reagents

24.1.1 *Ethyl Alcohol* — 95 percent (*v/v*).

24.1.2 *Dilute Sulphuric Acid* — 1 : 4 (*v/v*).

24.1.3 *Ether*

24.1.4 *Sodium Hydroxide Solution* — approximately 1 N.

24.1.5 *Modified Fehling's Solution* — Dissolve 34.639 g of copper sulphate ($CuSO_4.5H_2O$) in water and make up to 500 ml. Dissolve 173 g of Rochelle salt (sodium potassium tartrate, $NaKC_4H_4O_6$, $4H_2O$) and 50 g of sodium hydroxide in water and make up to 500 ml. Mix equal volumes of the two solutions before use.

24.2 Procedure

Proceed as in the method for the removal of alcoholinsoluble and water-insoluble matter as under **8**. Use water at 0 to 10°C if starch is to be removed. Take the water alcohol solutions, evaporate off the alcohol, concentrate to 200 ml and add 25 ml of dilute sulphuric acid. Boil gently for 20 min. Remove the cake of fatty acids after cooling and extract with 25 ml of ether to remove the ether-soluble materials. Neutralize the acids in the aqueous solution with sodium hydroxide solution and make up to 500 ml in a volumetric flask. Pipette out 50 ml of this solution (to contain less than 0.25 g of reducing sugars) and add 50 ml of the modified Fehling's solution. Heat with a flame to start boiling in exactly 4 minutes and continue boiling for exactly 2 minutes. Filter immediately through a tared Gooch crucible and wash several times with water at 60°C. Wash the filter paper finally with 10 ml of ethyl alcohol and 10 ml of ether. Dry the crucible in an air-oven at 105 ± 2°C for 30 min, cool and weigh as cuprous oxide (Cu_2O).

24.3 Calculation

Use the standard 'Munson and Walker' table (*see* Annex A) to compute the milligrams of invert sugar corresponding to the mass of the cuprous oxide (Cu_2O) formed. Calculate as follows:

$$\text{Invert sugar, percent by mass} = \frac{\text{milligrams of invert sugar} \times 0.1}{\text{mass in g of material in aliquot taken for the test}}$$

Sucrose, percent by mass = invert sugar, percent by mass × 0.95.

25 DETERMINATION OF STARCH

25.1 Reagents

25.1.1 *Dilute Hydrochloric Acid* — relative density 1.125, obtained by diluting 100 ml of concentrated hydrochloric acid, relative density 1.16 (*see* IS 265) to 130 ml.

25.1.2 *Sodium Hydroxide Solution* — approximately 0.5 N.

25.2 Procedure

Remove the alcohol-soluble matter as under 5 and wash the alcohol-insoluble residue with water at 0 to 10°C. Transfer the wet insoluble matter on the filter paper into a conical flask, add 20 ml of dilute hydrochloric acid and 200 ml of water and reflux the mixture under a water condenser for 2 to 3 h. Cool, add sodium hydroxide solution until almost neutral and make up to 250 ml in a volumetric fiask. Filter and discard the first 10 ml of the filtrate. Pipette 50 ml of the filtrate and proceed to estimate the amount of invert sugar present in the aliquot as under **24.2**.

25.3 Calculation

Use the standard 'Munson and Walker' table (*see* Annex A) to compute the milligrams of dextrose corresponding the mass of the cuprous oxide (Cu_2O) and calculate as follows:

$$\text{Starch, percent by mass} = \frac{\text{milligrams of dextrose} \times 0.093}{\text{mass in g of material in the aliquot taken for the test}}$$

26 DETERMINATION OF COMBINED SODIUM AND POTASSIUM OXIDES

26.0 General

Organic matter in the soap is removed by ashing the sample and the residue is digested with hydrochloric acid and potassium precipitated as potassium chloroplatinate. From this potassium oxide is calculated. Sodium oxide is obtained by subtracting the percentage of potassium oxide from the percentage of combined alkali.

26.1 Reagents

26.1.1 *Dilute Hydrochloric Acid* — 1 : 1 (*v/v*).

26.1.2 *Platinum Solution* — Prepare a solution containing the equivalent of 0.2 g of metallic platinum [0.42 g of chloroplatinic acid ($H_2Pt\ Cl_6$) in each 10 ml of solution].

26.1.3 *Ethyl Alcohol* — 80 percent (*v/v*). Dilute 84 ml of ethyl alcohol, 95 percent by volume, with 16 ml of water.

26.1.4 *Concentrated Hydrochloric Acid* — relative density 1.16 (*see* IS 265).

26.1.5 *Ammonium Chloride Solution* — To 500 ml of 20 percent ammonium chloride solution, add 5 to 10 g of pulverized potassium chloroplatinate (K_2PtCl_6) and shake at intervals of 6 to 8 h. Allow the mixture to settle and filter.

26.2 Procedure

Accurately weigh about 10 g of the sample in an evaporating dish and heat over a burner or in a Muffle furnace below dull red heat (350 to 450°C) until the mass is well carbonized. Cool, leach out the ash with hot water, filter into a 100 ml volumetric flask and wash the filter paper with three portions of 5 to 10 ml each of hot water. Return the filter paper and the residue to the evaporating dish and continue heating as before until all carbonaceous matter is burnt off. Add a few drops

of dilute hydrochloric acid and wash the contents of the dish through a filter paper into the 100 ml volumetric flask. Acidify the solution in the volumetric flask with dilute hydrochloric acid and dilute to 100 ml with water. Pipette 10 ml of the solution into a 100 ml beaker and acidify with a few drops of dilute hydrochloric acid. Add 10 ml of platinum solution and evaporate on a water-bath to the consistency of a thick paste. Add 5 ml of ethyl alcohol and 0.6 ml of concentrated hydrochloric acid and transfer the precipitate to a tared Gooch crucible.

Wash with ethyl alcohol until the filtrate is colourless. Then wash the residue, 5 or 6 times, with 20 ml portions of ammonium chloride solution and, finally with ethyl alcohol until the filtrate gives no test for chlorides. Dry in an air-oven at 105 to 110°C for 30 min, cool and determine the mass of potassium chloroplatinate.

26.3 Calculation

Potassium oxide (K_2O), percent by mass = $193.8\frac{B}{M}$

Sodium oxide (Na_2O), percent by mass = A — [potassium oxide (K_2O), percent by mass × 0.658 2]

where

B = mass in g of potassium chloroplatinatc (K_2PtCl_6),

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

A = combined alkali (as Na_2O), percent by mass, as found under **10.**

27 DETERMINATION OF CARBOLIC ACID AND CRESYLIC ACID

27.0 General

Insoluble metal salts of soap fatty acids are precipitated by the addition of calcium nitrate to an aqueous solution of the soap and removed by filtration. The cresylic acid in the alkaline filtrate is brominated by bromine liberated on the addition of a standard sodium bromide-sodium bromide solution and dilute mineral acid. Excess bromine is determined by adding potassium iodide and titrating the liberated iodine with standard sodium thiosulphate. From this carbolic acid and cresylic acid are calculated.

27.1 Carbolic Acid

27.1.1 *Reagents*

a) *Sodium hydroxide solution* — 10 percent (*m/v*).

b) *Calcium nitrate solution* — 20 percent (*m/v*).

c) *Sodium bromate-bromide solution* — Dilute 90 ml of sodium hydroxide solution to 400 ml with water and add liquid bromine until the solution is yellow. Boil the solution until it is clear, add 5 ml of sodium hydroxide solution and make up to 2 litres. The solution contains 5 mole of sodium bromide to one mole of sodium bromate.

d) *Dilute hydrochloric acid* — 1 : 1 (*v/v*).

e) *Potassium iodide solution* — 10 percent (*v/v*).

f) *Standard sodium thiosulphate solution* — approximately 0.1 N.

g) *Starch solution* — prepared as under **18.2.4**.

27.1.2 *Procedure*

Accurately weigh about 5 g of the sample and dissolve in 200 ml of water made alkaline with 10 ml of sodium hydroxide solution. Transfer to a 1 000 ml volumetric flask, dilute to 600 ml, add 20 ml of calcium nitrate solution, cool and make up to one litre. Filter, reject the first few millilitres of the filtrate and pipette 100 ml into a narrow-mouth stoppered bottle. Add 100 ml of water, 50 ml of sodium bromate-bromide solution and 10 ml of dilute hydrochloric acid. Allow to stand for 90 min, add 25 ml of potassium iodide solution and mix well. Titrate the liberated iodine against standard sodium thiosulphate solution using starch as indicator. Run a blank, using a material known to be free from carbolic acid.

27.1.3 *Calculation*

Carbolic acid, percent by mass = $0.156\,7\frac{V-v}{M}$

where

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

v = volume in ml of standard sodium thiosulphate solution required for the test, and

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

27.2 Cresylic Acid

27.2.1 *Reagents* — In addition to the reagents prescribed in **27.1.1** the following reagent is required:

a) *Standard cresylic acid* — a mixture of 35 percent *o*-cresol, 40 percent *m*-cresol and 25 percent *ñ*-cresol.

27.2.2 *Procedure*

Weigh quickly 0.25 g of standard cresylic acid and add immediately 10 ml of sodium hydroxide solution. Dissolve 5 g of any bar soap free from carbolic acid in water, add the cresylic acid solution and continue as described under **27.1.2**.

27.2.3 *Calculation*

Cresylic acid, percent by mass = $5\frac{V-v}{V-s}$

where

V = volume in ml of standard sodium

thiosulphate solution required for the blank,

v = volume in ml of standard sodium thiosulphate solution required with the sample, and

s = volume in ml of standard sodium thiosulphate solution required against standard cresylic acid and soap free from carbolic acid.

28 DETERMINATION OF CARBONATES

28.0 General

This method determines all of the carbonates as carbon dioxide and is applicable to all soaps and soap products.

28.1 Apparatus — The following apparatus is required.

28.1.1 *Volumetric Carbonate Apparatus* — consisting of the following and assembled as in Fig. 2.

a) *Evolution or sample flask* — round-bottom ring-neck flask of heat resistant glass, of 1 litre capacity and provided with a two-hole rubber stopper;

b) *Dropping funnel* — provided with a stopcock and having stem long enough to reach into the lowest bulb of the condenser;

c) *Conical flask* — of heat-resistant glass, 300 ml capacity and provided with a one-hole rubber stopper;

d) *Condenser* — water cooled, 3- or 4-bulb Allihn type, with a jacket about 200 mm long and a side arm for connecting to the conical flask; and

e) *Mercury manometer.*

28.1.2 *Glass Beads*

28.2 Reagents

28.2.1 *Hydrochloric Acid* — 0.5 N, accurately standardized.

28.2.2 *Dilute Hydrochloric Acid* – 1 : 2 (*v/v*).

28.2.3 *Alkaline Absorbent Solution* — Mix equal volumes of 1.0 N sodium hydroxide (carbonate-free) solution and 0.1 N barium chloride solution. Allow to settle overnight, filter and preserve in well-stoppered bottles.

28.2.4 *Magnesium Chloride Solution* — 20 percent (*m/m*).

28.2.5 *Phenolphthalein Indicator* — as in **6.1.1**.

28.2.6 *Methyl Orange Indicator* — as in **20.2.2**(f).

28.2.7 *Trichlorobenzene* — 1 : 2 : 4 isomer, boiling point 213°C and relative density 1.47.

28.3 Procedure

28.3.1 Weigh sufficient quantity of the sample into the sample flask to yield about 0.2 g of carbon dioxide. Add about 400 ml of unboiled water to which have been added 2 ml of alkaline absorbent solution to prevent the loss of

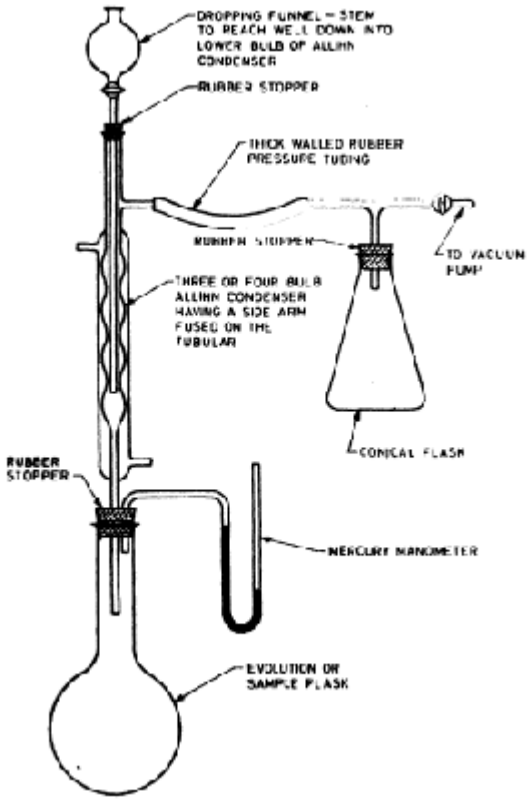


FIG. 2 TYPICAL ASSEMBLY OF VOLUMETRIC CARBONATE FOR THE DETERMINATION OF CARBONATES

carbon dioxide. Heat the flask on a steam-bath until soap is dissolved and cool until the flask is only slightly warm to the hand. Add 30 ml of magnesium chloride solution and a few glass beads to prevent bumping.

28.3.2 Pipette 25 ml of alkaline absorbent solution into the conical flask and assemble the apparatus as shown in Fig. 2. Start the water through the condenser. Apply suction to the side tube to evacuate the system and reduce the pressure to 65 to 85 mm as indicated by the manometer.

CAUTION : Maintain a properly reduced pressure and do not allow air to enter the system at any time during the test.

28.3.3 Add dilute hydrochloric acid containing a few drops of methyl orange indicator through the dropping funnel until the mixture in the flask is acidic (*see* Note). Avoid a large excess of acid. Add trichlorobenzene through the dropping funnel in the proportion of about 1 ml to every 2 g of the sample.

NOTE — Shake the conical flask at frequent intervals from the time the acid is added until the sample flask and the condenser have been filled with water.

28.3.4 Place the small flame of a burner immediately in contact with the bottom of the sample glass and heat continuously for 30 minutes. After this, discontinue heating and pour boiled and cooled (carbon dioxidefree) water at 50°C through the condenser tube to fill the flask and the condenser to just below the side of the arm of the condenser.

28.3.5 Disconnect the conical flask, add one millilitre of phenolphthalein indicator and titrate immediately with 0.5 N hydrochloric acid with vigorous agitation until pink colour disappears. Add the acid drop by drop. If it is not possible to titrate immediately, stopper the flask tightly to guard against entrance of air.

28.3.6 Conduct a blank determination in order to establish the equivalent of the alkaline absorbent solution in terms of 0.5 N hydrochloric acid and also to correct for any carbon dioxide in the reagents.

28.4 Calculation

$$\text{Carbonate (as } CO_2\text{), percent by mass} = \frac{(B-S)\times N\times 2.2}{M}$$

where

B = volume in ml of standard hydrochloric acid used in the blank,

S = volume in ml of standard hydrochloric acid used for the sample,

N = normality of standard hydrochloric acid, and

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

29 DETERMINATION OF FREE CARBONATED ALKALI

29.0 General

The matter insoluble in alcohol in the soap is dissolved in water and filtered. The water soluble carbonates are estimated by absorbing carbon dioxide gas evolved by reacting with the acid and expressed as sodium carbonate.

29.1 Apparatus

29.1.1 *Volumetric Carbonate Apparatus* — same as in Fig. 2.

29.1.2 *Glass Beads*

29.2 Reagents

29.2.1 *Hydrochloric Acid* — 0.5 N, accurately standardized.

29.2.2 *Dilute Hydrochloric Acid* — 1 : 2 (*v/v*).

29.2.3 *Alkaline Absorbent Solution* — same as in **28.2.3.**

29.2.4 *Magnesium Chloride Solution* — 20 percent (*m/m*).

29.2.5 *Phenolphihalein Indicator* — as in **6.1.1**.

29.2.6 *Methyl Orange Indicator* — as in **20.2.2**(f).

29.2.7 *Trichlorobenzene* — same as in **28.2.7**.

29.3 Procedure

29.3.1 Weigh accurately 2 to 10 g of the sample and digest with 200 ml of freshly boiled ethyl alcohol in a covered vessel on a steam-bath until the soap is dissolved. Filter into a filter flask through a counterpoised filter paper neutral to phenolphthalein, or through a weighed Gooch or sintered crucible with suction, protecting the solution from carbon dioxide and other acid fumes during the operation by covering with a watch glass. Wash it several times with hot ethyl alcohol to remove all the alcohol solubles. After filtering and washing the residue thoroughly with hot ethyl alcohol, change the receiver, extract the residue with successive portions of water at about 60°C and wash the residue thoroughly on the filter paper or in the crucible.

29.3.2 Then proceed as prescribed in **28.3.1** to **28.3.6**.

29.4 Calculation

$$\text{Free carbonated alkali, as sodium carbonate, percent by mass} = \frac{(B-S)\times N\times 5.3}{M}$$

where

B = volume in ml of standard hydrochloric acid used for the blank,

S = volume in ml of standard hydrochloric acid used for the sample,

N = normality of standard hydrochloric acid used, and

M = mass in g of the sample taken for the determination of matter insoluble in water.

ANNEX A

(*Clauses* 24.3 *and* 25.3)

MUNSON AND WALKER TABLE FOR CALCULATING DEXTROSE, INVERT SUGAR ALONE, INVERT SUGAR IN THE PRESENCE OF SUCROSE (0.4 g AND 2 g TOTAL SUGAR), LACTOSE, LACTOSE AND SUCROSE (2 MIXTURES) AND MALTOSE (CRYSTALLIZED)*

(APPLICABLE WHEN Cu_2O IS WEIGHED DIRECTLY)

(Expressed in Milligrams)

Cuprous Oxide (Cu_2O)	*Dextrose (D-Glucose)*	*Invert Sugar*	*Invert Sugar and Sucrose*		*Lactose*	*Lactose and Sucrose*		*Maltose*	*Cuprous Oxide (Cu_2O)*
			0.4 Grams Total Sugar	2 Grams Total Sugar	($C_{12}H_{22}O_{11}+H_2O$)	1 Lactose 4 Sucrose	1 Lac-tose 12 Sucrose	($C_{12}H_{22}O_{11}+H_2O$)	
10	4.0	4.5	1.6	…	6.3	6.1	…	6.2	10
12	4.9	5.4	2.5	…	7.5	7.3	…	7.9	12
14	5.7	6.3	3.4	…	8.8	8.5	…	9.5	14
16	6.6	7.2	4.3	…	10.0	9.7	…	11.2	16
18	7.5	8.1	5.2	…	11.3	10.9	…	12.9	18
20	8.3	8.9	6.1		12.5	12.1	…	14.6	20
22	9.2	9.8	7.0	…	13.8	13.3	…	16.2	22
24	10.0	10.7	7.9	…	15.0	14.5	…	17.9	24
26	10.9	11.6	8.8	…	16.3	15.8	…	19.6	26
28	11.8	12.5	9.7	…	17.6	17.0	…	21.2	28
30	12.6	13.4	10.7	4.3	18.8	18.2	…	22.9	30
32	13.5	14.3	11.6	5.2	20.1	19.4	…	24.6	32
34	14.3	15.2	12.5	6.1	21.4	20.7	…	26.2	34
36	15.2	16.1	13.4	7.0	22.8	22.	…	27.9	36
38	16.1	16.9	14.3	7.9	24.2	23.3	…	29.6	38
40	16.9	17.8	15.2	8.8	25.5	24.7	…	31.3	40
42	17.8	18.7	16.1	9.7	26.9	26.0	…	32.9	42
44	18.7	19.6	17.0	10.7	28.3	27.3	…	34.6	44
46	19.6	20.5	17.9	11.6	29.6	28.6	…	36.3	46
48	20.4	21.4	18.8	12.5	31.0	30.0	…	37.9	48

*U.S. *Bur. Standards Circ.* 44, p. 139. The columns headed ‘Lactose’ and ‘Lactose and Sucrose’ were taken from ‘Methods of Sugar Analysis and Allied Determinations’ by Arthur Given.

(*Continued*)

MUNSON AND WALKER TABLE — (*Continued*)
(Expressed in Milligrams)

Cuprous Oxide (Cu_2O)	*Dextrose (D-Glucose)*	*Invert Sugar*	*Invert Sugar and Sucrose*		*Lactose*	*Lactose and Sucrose*		*Maltose*	*Cuprous Oxide (Cu_2O)*
			0.4 Grams Total Sugar	2 Grams Total Sugar	($C_{12}H_{22}O_{11}+H_2O$)	1 Lactose 4 Sucrose	1 Lactose 12 Sucrose	($C_{12}H_{22}O_{11}+H_2O$)	
50	21.3	22.3	19.7	13.4	32.3	31.3	…	39.6	50
52	22.2	213.2	20.7	14.3	33.7	32.6	…	41.3	52
54	23.0	24.1	21.6	15.2	35.1	34.0	…	42.9	54
56	23.9	25.0	22.5	16.2	36.4	35.3	…	44.6	56
58	24.8	25.9	23.4	17.1	37.8	36.6	…	46.3	58
60	25.6	26.8	24.3	18.0	39.2	37.9	…	48.0	60
62	26.5	27.7	25.2	18.9	40.5	39.3	…	49.6	62
64	27.4	28.6	26.2	19.8	41.9	40.6	…	51.3	64
66	28.3	29.5	27.1	20.8	43.3	41.9	…	53.0	66
68	29.2	30.4	28.0	21.7	44.7	43.3	40.7	54.6	68
70	30.0	31.3	28.9	22.6	46.0	44.6	41.9	56.3	70
72	30.9	32.3	29.8	23.5	47.4	45.9	43.1	58.0	72
74	31.8	33.2	30.8	24.5	48.8	47.3	44.2	59.6	74
76	32.7	34.1	31.7	25.4	50.1	48.6	45.4	61.3	76
78	33.6	35.0	32.6	26.3	51.5	49.9	46.6	63.0	78
80	34.4	35.9	33.5	27.3	52.9	51.3	47.8	64.6	80
82	35.3	36.8	34.5	28.2	54.1	52.6	49.0	66.3	82
84	36.2	37.7	35.4	29.1	55.6	53.9	50.1	68.0	84
86	37.1	38.6	36.3	30.0	57.0	55.3	51.3	69.7	86
88	38.0	39.5	37.2	31.0	58.4	56.6	52.5	71.3	88
90	38.9	40.4	38.2	31.9	59.7	57.9	53.7	73.0	90
92	39.8	41.4	39.1	32.8	61.1	59.3	54.9	74.7	92
94	40.6	42.3	40.0	33.8	62.5	60.6	56.0	76.3	94
96	41.5	43.2	41.0	34.7	63.8	61.9	57.2	78.0	96
98	42.4	44.1	41.9	35.6	65.2	63.3	58.4	79.7	93
100	43.3	45.0	42.8	36.6	66.6	64.6	59.6	81.3	100
102	44.2	46.0	43.8	37.5	68.0	66.0	60.8	83.0	102
104	45.1	46.9	44.7	38.5	69.3	67.3	62.0	84.7	104
106	46.0	47.8	45.6	39.4	70.7	68.6	63.2	80.3	106
108	46.9	46.7	46.6	40.3	72.1	70.0	64.4	88.0	108
110	47.8	49.6	47.5	41.3	73.5	71.3	65.6	89.7	110
112	48.7	50.6	48.4	42.2	74.8	72.6	66.7	91.3	112
114	49.6	51.5	49.4	43.2	76.2	74.0	67.9	93.0	114
116	50.5	52.4	50.3	44.1	77.6	75.3	69.1	94.7	116
118	51.4	53.3	51.2	45.0	79.0	76.7	70.3	96.4	118

(*Continued*)

MUNSON AND WALKER TABLE — (*Continued*)
(Expressed in Milligrams)

Cuprous Oxide (Cu_2O)	*Dextrose (D-Glucose)*	*Invert Sugar*	*Invert Sugar and Sucrose*		*Lactose*	*Lactose and Sucrose*		*Maltose*	*Cuprous Oxide (Cu_2O)*
			0.4 Grams Total Sugar	2 Grams Total Sugar	($C_{12}H_{22}O_{11}+H_2O$)	1 Lactose 4 Sucrose	1 Lactose 12 Sucrose	($C_{12}H_{22}O_{11}+H_2O$)	
190	84.3	87.2	85.6	79.5	128.5	125.1	113.8	156.4	190
192	85.3	88.2	86.6	80.5	129.9	126.4	115.0	158.0	192
194	86.2	89.2	87.6	81.4	131.3	127.8	116.2	159 7	194
196	87.1	90.1	88.5	82.4	132.7	129.2	117.4	161.4	196
198	88.1	91.1	89.5	83.4	134.1	130.5	118.6	163.0	198
200	89.0	92.0	90.5	84.4	135.4	131.9	119.8	164.7	200
202	89.9	93.0	91.4	85.5	136.8	133.2	121.0	166.4	202
204	90.9	94.0	92.4	86.3	138.2	134.6	122.3	168.0	204
206	91.8	94.9	93.4	87.3	139.6	135 9	123.5	169.7	206
208	92.8	95.9	94.4	88.3	141.0	137.3	124.7	171.4	208
210	93.7	96.9	95.4	89.2	142.3	138.6	126.0	173.0	210
212	94.6	97.8	96.3	90.2	143.7	140.0	127.2	174.7	212
214	95.6	98.8	97.3	91.2	145.1	141.4	128.4	176.4	214
216	96.5	99.8	98.3	92.2	146.5	142.7	129.6	178.0	216
218	97.5	100.8	99.3	93.2	147.9	144.1	130.9	179.7	218
220	98.4	101.7	100.3	94.2	149.3	145.4	132.1	181.4	220
222	99.4	102.7	101.2	95.1	105.7	146.8	133.3	183.0	222
224	100.3	103.7	102.2	96.1	152.0	148.1	134.5	184.7	224
226	101.3	104.6	103.2	97.1	153.4	149.5	135.8	186.4	226
228	102.2	105.6	104.2	98.1	154.8	150.8	137.0	188.0	228
230	103.2	106.6	105.2	99.1	156.2	152.2	138.2	189.7	230
232	104.1	107.6	106.2	100.1	157.6	153.6	139.4	191.3	232
234	105.1	108.6	107.2	101.1	159.0	154.9	140.7	193.0	234
236	106.0	109.5	108.2	102.1	160.3	156.3	141.9	194.7	236
238	107.0	110.5	109.2	103.1	161.7	157.6	143.2	196.3	238
240	108.0	111.5	110.1	104.0	163.1	159.0	144.4	198.0	240
242	108.9	112.5	111.1	105.0	164.5	160.3	145.6	199.7	242
244	109.9	113.5	112.1	106.0	165.9	161.7	146.9	201.3	244
246	110.8	114.5	113.1	107.0	167.3	163.1	148.1	203.0	246
248	111.8	115.4	114.1	108.0	168.7	164.4	149.3	204.7	248
250	112.8	116.4	115.1	109.0	170.1	165.8	150.6	206.3	250
252	113.7	117.4	116.1	110.0	171.5	167.2	151.8	208.0	252
254	114.7	118.4	117.1	111.0	172.8	168.5	153.1	209.7	254
256	115.7	119.4	118.1	112.0	174.2	169.9	154.3	211.3	256
258	116.6	120.4	119.1	113.0	175.6	171.3	155.5	213.0	258

(*Continued*)

MUNSON AND WALKER TABLE — (*Continued*)
(Expressed in Milligrams)

Cuprous Oxide (Cu_2O)	*Dextrose (D-Glucose)*	*Invert Sugar*	*Invert Sugar and Sucrose*		*Lactose*	*Lactose and Sucrose*		*Maltose*	*Cuprous Oxide (Cu_2O)*
			0.4 Grams Total Sugar	2 Grams Total Sugar	($C_{12}H_{22}O_{11}+H_2O$)	1 Lactose 4 Sucrose	1 Lactose 12 Sucrose	($C_{12}H_{22}O_{11}+H_2O$)	
260	117.6	121.4	120.1	114.0	177.0	172.6	156.8	214.7	260
262	118.6	122.4	121.1	115.0	178.4	174.0	158.0	216.3	262
264	119.5	123.4	122.1	116.0	179.8	175.3	159.3	218.0	294
266	120.5	124.4	123.1	117.0	181.2	176.7	160.5	219.7	266
268	121.5	125.4	124.1	118.0	182.6	178.1	161.8	221.3	268
270	122.5	126.4	125.4	119.0	184.0	179.4	163.0	223.0	270
272	123.4	127.4	126.2	120.0	185.3	180.8	164.3	224.6	272
274	124.4	128.4	127.2	121.1	186.7	182.2	165.5	226.3	274
276	125.4	129.4	128.2	122.1	188.1	183.5	166.81	228.0	276
278	126.4	130.4	129.2	123.1	189.5	184.9	168.0	229.6	278
280	127.3	131.4	130.2	124.1	190.9	186.3	169.3	231.3	280
282	128.3	132.4	131.2	125.1	192.3	187.6	170.5	233.0	282
284	129.3	133.4	132.2	126.1	193.7	189.0	171.8	234.6	284
286	130.3	134.4	133.2	127.1	195.1	190.4	173.0	236.3	286
288	131.3	135.4	134.3	128.1	196.5	191.7	174.3	238.0	288
290	132.3	136.4	135.3	129.2	197.8	193.1	175.5	239.6	290
292	133.2	137.4	136.3	130.2	199.2	194.4	176.8	241.3	292
294	134.2	138.4	137.3	131.2	200.6	195.8	178.1	242.9	294
296	135.2	139.4	138.3	132.2	202.0	197.2	179.3	244.6	296
298	136.2	140.5	139.4	133.2	203.4	198.6	180.6	246.3	298
300	137.2	141.5	140.4	134.2	204.8	199.9	181.8	247.9	300
302	138.2	142.5	141.4	135.3	206.2	201.3	183.1	249.6	302
304	139.2	143.5	142.4	136.3	207.6	202.7	184.4	251.3	304
306	140.2	144.5	143.4	137.3	209.0	204.0	185.6	252.9	306
308	141.2	145.5	144.5	138.3	210.4	205.4	186.9	254.6	308
310	142.2	146.6	145.5	139.4	211.8	206.8	188.1	256 3	310
312	143.2	147.6	146.5	140.4	213.2	208.1	189.4	257.9	312
314	144.2	148.6	147.6	141.4	214.6	209.5	190.7	259.6	314
316	145.2	149.6	148.6	142.4	216.0	210.9	191.9	261.2	316
318	146.2	150.7	149.6	143.5	217.3	212.2	193.2	262.9	318
320	147.2	151.7	150.7	144.5	218.7	213.6	194.4	264.6	320
322	148.2	152.7	151.7	145.5	220.1	215.0	195.7	266.2	322
324	149.2	153.7	152.7	146.6	221.5	216.4	197.0	267.9	324
326	150.2	154.8	153.8	147.6	222.9	217.7	198.2	269.6	326
328	151.2	155.8	154.8	148.6	224.3	219.1	199.5	271.2	328

(*Continued*)

MUNSON AND WALKER TABLE — (*Continued*)
(Expressed in Milligrams)

Cuprous Oxide (Cu_2O)	*Dextrose (D-Glucose)*	*Invert Sugar*	*Invert Sugar and Sucrose*		*Lactose*	*Lactose and Sucrose*		*Maltose*	*Cuprous Oxide (Cu_2O)*
			0.4 Grams Total Sugar	2 Grams Total Sugar	($C_{12}H_{22}O_{11}+H_2O$)	1 Lactose 4 Sucrose	1 Lactose 12 Sucrose	($C_{12}H_{22}O_{11}+H_2O$)	
330	152.2	156.8	155.8	149.7	225.7	220.5	200.8	272.9	330
332	153.2	157.9	156 9	150.7	227.1	221.8	202.0	274.6	332
334	154.2	158.9	157.9	151.7	228.5	223.2	203.3	276.2	334
336	155.2	159.9	159.0	152.8	229.9	224.6	204.6	277.9	336
338	156.3	161.0	160.0	153.8	231.3	226.0	205.9	279.5	338
340	157.3	162.0	161.0	154.8	232.7	227.4	207.1	281.2	340
342	158.3	163.1	162.1	155.9	234.1	228.7	208.4	282.9	342
344	159.3	164.1	163.1	156.9	235.5	230.1	209.7	284.5	344
346	160.3	165.1	164.2	158.0	236.9	231.5	211.0	286.2	346
348	161.4	166.2	165.2	159.0	238.3	232.9	212.2	287.9	348
350	162.4	167.2	166.3	160.1	239.7	234.3	213.5	289.5	350
352	163.4	168.3	167.3	161.1	241.1	235.6	214.8	291.2	352
354	164.4	169.3	168.4	162.2	242.5	237.0	216.1	292.8	354
356	165.4	170.4	169.4	163.2	243.9	238.4	217.3	294.5	356
358	166.5	171.4	170.5	164.3	245.3	239.8	218 6	296.2	358
360	167.5	172.5	171.5	165.3	246.7	241.2	219.9	297.8	360
362	168.5	173.5	172 6	166.4	248.1	242.5	221.2	299.5	362
364	169.6	174.6	173.7	167.4	249.5	243.9	222.5	301.2	364
366	170.6	175.6	174.7	168.5	250 9	245.3	223.7	302.8	366
368	171.6	176.7	175.8	169.5	252.3	246.7	225.0	304.5	368
370	172.7	177.7	176.8	170.6	253.7	248.1	226.3	306.1	370
372	173.7	178 8	177.9	171.6	255.1	249.5	227.6	307.8	372
374	174.7	179.8	179 0	172.7	256.5	250.9	228.9	309.5	374
376	175.8	180.9	180.0	173.7	257.9	252.2	230.2	311.1	376
378	176.8	182.0	181.1	174.8	259.3	253.6	231.5	312.8	378
380	177.9	183.0	182.1	175.9	260.7	255.0	232.8	314.5	380
382	178.9	184.1	183.2	176.9	262.1	256.4	234.1	316 1	382
384	180.0	185.2	184.3	178.0	263 5	257.8	235.4	317.8	384
386	181.0	186.2	185.4	179.1	264.9	259.2	236.6	319.4	386
388	182.0	187.3	186.4	180.1	266.5	260.5	237.9	321.1	388
390	183.1	188.4	187.5	181.2	267.7	261.9	239.2	322.8	390
392	184.1	189.4	188.6	182.3	269.1	263.3	240.5	324.4	392
394	185.2	190.5	189.7	183 3	270.5	264.7	241.8	326.1	394
396	186.2	191.6	190.7	184.4	271.9	266.1	243.1	327.7	396
398	187.3	192.7	191.8	185.5	273.3	267.5	244.4	329.4	398

(*Continued*)

MUNSON AND WALKER TABLE — (*Continued*)
(Expressed in Milligrams)

Cuprous Oxide (Cu_2O)	*Dextrose (D-Glucose)*	*Invert Sugar*	*Invert Sugar and Sucrose*		*Lactose*	*Lactose and Sucrose*		*Maltose*	*Cuprous Oxide (Cu_2O)*
			0.4 Grams Total Sugar	2 Grams Total Sugar	($C_{12}H_{22}O_{11}+H_2O$)	1 Lactose 4 Sucrose	1 Lactose 12 Sucrose	($C_{12}H_{22}O_{11}+H_2O$)	
400	188.4	193.7	192.9	186.5	274.7	268.9	245.7	331.1	400
402	189.4	194.8	194.0	187.6	276.1	270.3	247.0	332.7	402
404	190.5	195.9	195.0	188.7	277.5	271.7	248.3	334.4	404
406	191.5	197.0	196.1	189.8	278.9	273.0	249.6	336.0	406
408	192.6	198.1	197.2	190.8	280.3	274.4	251.0	337.7	408
410	193.7	199.1	198.3	191.9	281.7	275.8	252.3	339.4	410
412	194.7	200.2	199.4	193.0	283.2	277.2	253.6	341.0	412
414	195.8	201.3	200.5	194.1	284.6	278.6	254.9	342.7	414
416	196.8	202.4	201.6	195.2	286.0	280.0	256.2	344.4	416
418	197.9	203.5	202.6	196.2	287.4	281.4	257.5	346.0	418
420	199.0	204.6	203.7	197.3	288.8	282.8	258.8	347.7	420
422	200.1	205.7	204.8	198.4	290.2	284.2	260.1	349.3	422
424	201.1	206.7	205.9	199.5	291.6	285.6	261.4	351.0	424
426	202.2	207.8	207.0	200.6	293.0	287.0	262.7	352.7	426
428	203.3	208.9	208.1	201.7	294.4	288.4	264.0	354.3	428
430	204.4	210.0	209.2	202.7	295.8	289.8	265.4	356.0	430
432	205.5	211.1	210.3	203.8	297.2	291.2	266.6	357.6	432
434	206.5	212.2	211.4	204.9	298.6	292.6	268.0	359.3	434
430	207.6	213.3	212.5	206.0	300.0	294.0	269.3	361.0	436
438	208.7	214.4	213.6	207.1	301.4	295.4	270.6	362.6	438
440	209.8	215.5	214.7	208.2	302.8	296.8	272.0	364.3	440
442	210.9	216.6	215.8	209.3	304.2	298.2	273.3	365.9	442
444	212.0	217.8	216.9	210.4	305.6	299.6	274.6	367.6	444
446	213.1	218.9	218.0	211.5	307.0	301.0	275.9	369.3	446
448	214.1	220.0	219.1	212.6	308.4	302.4	277.2	370.9	448
450	215.2	221.1	220.2	213.7	309.9	303.8	278.6	372.6	450
452	216.3	222.2	221.4	214.8	311.3	305.2	279.9	374.2	452
454	217.4	223.3	222.5	215.9	312.7	306.6	281.2	375.9	454
456	218.5	224.4	223.6	217.0	314.1	308.0	282.5	377.6	456
458	219.6	225.5	224.7	218.1	315.5	309.4	283.9	379.2	458
460	220.7	226.7	225.8	219.2	316.9	310.8	285.2	380.9	460
462	221.8	227.8	226.9	220.3	318.3	312.2	286.5	382.5	462
464	222.9	228.9	228.1	221.4	319.7	313.6	287.8	384.2	464
466	224.0	230.0	229.2	222.5	321.1	315.0	289.2	385.9	466
468	225.1	231.2	230.3	223.7	322.5	316.4	290.5	387.5	468

(*Continued*)

MUNSON AND WALKER TABLE — (*Concluded*)
(Expressed in Milligrams)

Cuprous Oxide (Cu_2O)	*Dextrose (D-Glucose)*	*Invert Sugar*	*Invert Sugar and Sucrose*		*Lactose*	*Lactose and Sucrose*		*Maltose*	*Cuprous Oxide (Cu_2O)*
			0.4 Grams Total Sugar	2 Grams Total Sugar	($C_{12}H_{22}O_{11}+H_2O$)	1 Lactose 4 Sucrose	1 Lactose 12 Sucrose	($C_{12}H_{22}O_{11}+H_2O$)	
470	226.2	232.3	231.4	224.8	323.9	317.7	291.8	389.2	470
472	227.4	233.4	232.5	225.9	325.3	319.1	293.2	390.8	472
474	228.3	234.5	233.7	227.0	326.8	320.5	294.5	392.5	474
476	229.6	235.7	234.8	228.1	328.2	321.9	295.8	394.2	476
478	230.7	236.8	235.9	229.2	329.6	323.3	297.1	395.8	478
480	231.8	237.9	237.1	230.3	331.0	324.7	298.5	397.5	480
482	232.9	239.1	238.2	231.5	332.4	326.1	299.8	399.1	482
484	234.1	240.2	239.3	232.6	333.8	327.5	301.1	400.8	484
486	235.2	241.4	240.5	233.7	335.2	328.9	302.5	402.4	486
488	236.3	242.5	241.6	234.8	336.6	330.3	303.8	404.1	488
490	237.4	243.6	242.7	236.0	338.0	331.7	305.1	403.8	490

ANNEX B

(*Clause* 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*	*IS No.*	*Title*
170 : 2004	Acetone — Specification (*fourth revision*)	323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
264 : 2005	Nitric acid — Specification (*third revision*).	336 : 1973	Specification for ether (*second revision*)
265 : 1993	Hydrochloric acid — Specification (*fourth revision*)	1070 : 1992	Reagent grade water (*third revision*)
266 : 1993	Sulphuric acid — Specification (*third revision*)	1796 : 1977	Specification for glycerine (*second revision*)
321 : 1964	Specification for absolute alcohol (*revised*)	7597 : 2001	Surface active agents — Glossary of terms (*first revision*)

ANNEX C

(Foreword)

COMMITTEE COMPOSITION

Soaps and Other Surface Active Agents Sectional Committee, CHD 25

Organization	*Representative(s)*
Drugs Controller General of India, New Delhi	Dr G. N. Singh (***Chairman***) Shri S. Dey (*Alternate*)
Association for Consumer's Action on Safety and Health, Mumbai	Dr Yogesh Kamdar Shri Satyadev Pandey (*Alternate*)
Central Pollution Control Board, Delhi	Dr M. Q. Ansari Dr Rekha L. Sitasawad (*Alternate*)
Central Revenue Control Laboratory, New Delhi	Shri Y. K. S. Rathore Shri S. C. Mathur (*Alternate*)
Colgate-Palmolive (India) Ltd, Mumbai	Dr Shashank Potnis Shri Vilas Tulle (*Alternate*)
Consumer Education & Research Centre, Ahmedabad	Ms Anindita Mehta Ms Dolly S. Jani (*Alternate*)
Consumer Guidance Society of India, Mumbai	Dr Sitaram Dixit Sri B. V. Desai (*Alternate*)
Department of Industrial Policy and Promotion, New Delhi	Shri Nand Lal
Directorate General of Health Services, New Delhi	Nomination Awaited
Directorate General of Supplies & Disposals, New Delhi	Sri N. K. Kaushal A. K. M. Kashyap (*Alternate*)
FASSSDMI, Delhi	Shri Santosh Kumar Shri Aseem Galhotra (*Alternate*)
Godrej Consumers Products Limited, Mumbai	Shri Jimmy Dordi Dr N. M. Sunder (*Alternate*)
Harcourt Butler Technological Institute, Kanpur	Prof Rakesh Trivedi Dr V. K. Tyagi (*Alternate*)
Hindustan Unilever Limited, Mumbai/Bangalore	Dr A. Sivakumar Ms Vrinda Rajwade (*Alternate*)
ITC Limited, Bangalore	Dr V. Krishnan Dr Suresh Ramamurthi (*Alternate*)
K. S. Krishnan Associates (P) Ltd, Noida	Shri S. Krishnan Shri Krishna Murthy (*Alternate*)
Khadi & Village Industries Commission, Mumbai	Shri Gulam Hussain
Ministry of Defence (DGQA), Kanpur	Shri A. K. Shukla Shri L. Tharachand (*Alternate*)
Ministry of Micro Small & Medium Enterprises (MSME), New Delhi	Shri H. S. Bisht
National Test House, Ghaziabad	Shri M. Chakraborty Dr (Smt) Madhurima Misra (*Alternate*)
Nirma Limited, Ahmedabad	Shri H. K. Patel Shri Ashish K. Desai (*Alternate*)
Oil Technologist Association of India, Mumbai	Shri Ashok Mahindru Shri H. P. Saxena (*Alternate*)
Procter & Gamble India, Mumbai	Ms Priyanka Bhat Shri Gaurav Nandrajog (*Alternate*)
Voluntary Organization in Interest of Consumer Education (VOICE), New Delhi	Sri M. A. U. Khan Shri B. K. Mukhopadh (*Alternate*)
In personal capacity (*8/308, Rajendra Nagar, Sector 3, Sahibabad Ghaziabad*)	Shri M. Mitra (Ex DGQS)
BIS Directorate General	Dr Rajiv K. Jha Scientist 'F' and Head (Chemical) [Representing Director General (*ex-officio*)]

Member Secretary
Shri T. Arivudaiyanambi
Scientist 'E' (Chemical), BIS

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