Foreword

This Bangladesh Standard was adopted by the Bangladesh Standards and Testing Institution on .... after the draft finalized by the Soap and Detergent Sectional Committee and approved by the Chemical Divisional Committee.

Glycerine is an important raw material for several industries. This specification covers crude and refined glycerine. Crude glycerine which is obtained from splitting oils and fats or from soap industry as a by-product is used for the manufacture of refined glycerine.

Refined glycerine is used for various purposes. Analytical reagent grade glycerine, as its name suggests, is used as a laboratory reagent and in biochemical work. Chemically pure glycerine is used mainly in the manufacturing of cosmetics, chemicals, pharmaceuticals and cellophane paper. Industrial white and technical grades of glycerine are used in the manufacture of paints and varnishes, printed textiles, carbon papers, typewriter ribbons, etc. Dynamite grade glycerine is used in the manufacture of explosives.

This standard was first published in 1968 for soap lye crude glycerol. In this revision, requirement parameters are included for refined glycerine. This standard also includes method for determination of volatile fatty acids even though this requirement is not considered necessary to be prescribed in the standard at present.

Suitable abbreviated symbols based on the trade practice are given for all the grades of refined glycerine for facilitating marking of the products. Such symbols are not given for the grades of crude glycerine as there is no such accepted code prevalent in trade.

Due to its growing demand the sectional committee decided to revise this standard. While revising this standard the sectional committee gave due consideration to the views of the producers, consumers and technologists and felt that it should be related to the prevailing trade and manufacturing practices followed in this field in the country.

In the preparation of this standard, assistance derived from the following publications is acknowledged with thanks:

> IS 1796:2023 Glycerine — Specification (Third Revision); Bureau of Indian Standards.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value (observed or calculated) expressing the result of a test or analysis, shall be rounded off in accordance with BDS 103.

Bangladesh Standard

Specification for Glycerine

(First Revision)

1. Scope

1.1 This standard prescribes requirements and methods of sampling and tests for glycerine.

2. Normative references

2.1 The following Bangladesh Standards are necessary adjuncts to this Standard. For undated references the latest edition of the publication referred to applies.

BDS 38	Sulphuric acid.
BDS 103	Methods of rounding off numerical values.
BDS 833	Water for laboratory use.
BDS 1242	Rectified spirit for industrial use.
BDS 1333	Methods for determination of arsenic.
ISO 760	Determination of water by karl fischer method — Test method.

3. Types and Grades

3.1 The material shall be classified into the following two types:

a) **Type 1 -** Crude glycerine; and

b) **Type 2 -** Refined glycerine.

3.1.1 Crude glycerine shall be further classified into the following two grades:

a) **Grade 1 -** Soap lye; and

b) **Grade 2 -** Saponification (hydrolyzer).

3.1.2 Refined glycerine shall be further classified into the following five grades:

a) **Grade 1 -** Analytical reagent, AR;

b) **Grade 2 -** Chemically pure, CP;

c) **Grade 3 -** Industrial white, IW;

d) **Grade 4 -** Technical, TECH; and

e) **Grade 5 -** Dynamite, DYN.

4. Requirements

4.1 Description

4.1.1 Soap Lye Crude Glycerine - The material shall be a viscous liquid prepared by evaporation of the purified lyes obtained as a by-product from the manufacture of soap by the boiling process.

4.1.2 Saponification (Hydrolyzer) Crude Glycerine - The material shall be a viscous liquid prepared by evaporation of the sweet waters obtained as a by-product from the hydrolysis of oils and fats.

4.1.3 Analytical Reagent (AR) Glycerine - The material shall be a clear, colourless, odourless, syrupy liquid, consisting essentially of glycerol. It shall have a sweet taste followed by a sensation of warmth. It shall be miscible in all proportions with water and with ethyl alcohol (90 percent (v/v), and it shall not be soluble in diethyl ether, chloroform or fixed oils. It shall be free from visible foreign matters.

4.1.4 Chemically Pure (CP) Glycerine - The material shall essentially be similar to that described under 4.1.3.

4.1.5 Industrial White (IW) Glycerine - The material shall be a clear, syrupy liquid consisting essentially of glycerol.

4.1.6 Technical (TECH) Glycerine - The material shall be a clear, syrupy liquid consisting essentially of glycerol.

4.1.7 Dynamite Grade (DYN) Glycerine - The material shall be a clear, syrupy liquid consisting essentially of glycerol.

4.1.7.1 Nitration Test - The nitration test shall be carried out, if required, by the purchaser.

4.2 Specific requirements - The material shall comply with the requirements given in Table 1 when tested in accordance with the method prescribed therein.

5. Packaging

5.1 Crude glycerine and refined glycerine analytical reagent (AR) grade, chemically pure (CP) grade, industrial white (IW) grade and technical (TECH) grade shall be supplied in suitable well closed containers as agreed to between the purchaser and the supplier.

5.2 Refined glycerine dynamite (DYN) grade shall be supplied in sound, clean and dry galvanized drums (nominal capacity of 200 litres), but galvanized by hot dipping and made of GI sheet. Alternatively, heavy duty hot dipped GI drum, 2 mm thick (nominal capacity of 200 litres) with hoops shall be used unless otherwise specified in the contract. These drums shall not be painted internally or at seam sealing/joints with any epoxy paint or any other protective compound.

6. Marking - The containers shall be securely closed and legibly marked with the following information:

a) Name, type and grade of the material;

b) Name and address of the manufacturer. In the case of imported products, name and address of the distributor/ importer including the country of origin;

c) Registered trade mark / brand name, if any;

d) Batch or lot number in code or otherwise;

e) Net content in mass;

f) Instructions for use;

g) Date of manufacture;

h) Use best before;

i) Maximum retail price; and

j) Any other requirement as prescribed by the statutory authorities.

Table 1 Specific requirements for Glycerine

Sl No.	Characteristics	Requirement							Methods of Test, Ref to Cl. No. in Annex A
		Crude Glycerine		Refined Glycerine					
		Soap Lye	Saponification (hydrolyzer)	AR	CP	IW	TECH	DYN*	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
1.	Glycerol, percent by mass, Min.	80.0	88.0	99.0	98.0	98.0	98.0	98.7	A-2
2.	Relative density at 30/30 °C, Min.	-	-	1.2578	1.2552	1.2552	1.2552	1.2571	A-3
3.	Colour reading on Lovibond scale expressed as Y + R, not deeper than (in 5½ in. cell)	-	-	0.5	1.0	3.5	8.0	5.0	A-4
4.	Ash, percent by mass, Max.	10.0	1.5	0.01	0.01	0.01	0.05	0.025	A-5
5.	Non-volatile organic residue, percent by mass, Max.	3.0	1.5	-	-	-	-	-	A-6
6.	Arsenic (as As), parts per million, Max.	2.0	1.5	1.0	2.0	-	-	-	A-7
7.	Copper	-	-	To pass the test		-	-	-	A-8
8.	Iron (as Fe), parts per million, Max.	-	-	0.5	0.5	-	2.0	-	A-9
9.	Lead (as Pb), parts per million, Max.	-	-	1.0	1.0	-	-	-	A-10
10.	Chlorides (as Cl), parts per million, Max.	-	-	2.0	10.0	60.0	60.0	30.0	A-11
11.	Sulphates (as SO_4), parts per million, Max.	-	-	10.0	10.0	-	-	34.0	A-12
12.	Alkalinity (as Na_2O), percent by mass, Max.	-	-	Nil	Nil	0.01	0.01	0.01	A-13
13.	Total alkali (free and combined), as Na_2O, percent by mass, Max.	1.0	-	-	-	-	-	-	A-14
14.	Fatty acids and esters (as Na_2O), percent by mass, Max.	-	-	0.01	0.03	-	-	0.05	A-15
15.	Acraldehyde and glucose	-	-	To pass the test		-	-	-	A-16
16.	Reducing substances	-	-	To pass the test		-	-	To pass the test	A-17
17.	Sulphuric acid test	-	-	-	-	-	-	Shall not be darker than standard	A-18
18.	Nitrogen content, percent by mass, Max.	-	-	-	-	-	-	0.03	A-19
19.	Ether soluble matter, percent by mass, Max.	-	-	-	-	-	-	0.03	A-20

NOTE – The nitration test shall be carried out, if necessary, by the purchaser.

6.1 The containers may also be marked with the BSTI Certification Mark.

NOTE - The use of the BSTI Certification Mark is governed by the provisions of the Bangladesh Standards and Testing Institution Act 2018 and the Rules and Regulations made there under. Details of conditions be under which a license for the use of the BSTI Certification Mark may granted to manufacturers or processors, may be obtained from the Bangladesh Standards and Testing Institution.

7. Sampling

7.0 General

It is very difficult to lay down detailed directions for sampling either crude glycerine or refined glycerine that will encompass all conditions and circumstances that may confront the person charged with the responsibility of taking the sample. There are many instances in which the experience and judgement of that person should prevail. There are, however, certain general rules relating to drawing, preparation, storage and handling of samples which shall always govern if the sample is to be representative. These are described under 7.1 to 7.9.

7.1 General Precautious in Sampling

7.1.1 For sampling crude glycerine, the sampling instruments may be made of copper, brass or bronze but should be nickel plated. For sampling refined glycerine, the sampling apparatus shall be either of stainless steel or glass.

7.1.2 All sampling apparatus shall be clean and dry when used. When glass apparatus is used, it is essential to ensure that the glycerine is not contaminated by glass splinters.

7.1.3 Samples shall not be taken in an exposed place. The samples, the material being sampled, the sampling instruments and the containers for samples shall be protected from adventitious contamination. The test samples shall be placed in suitable, clean, dry airtight bottles.

7.1.4 On account of the very hygroscopic nature of glycerine, samples shall be protected at all times from moisture and moist air. For the same reason, sampling procedures shall be designed to give maximum protection to glycerine.

7.1.5 The prevailing conditions should be surveyed before the sampling procedure is decided upon, for example, by taking spot samples, by testing for the presence and condition of settled salt, evidence of stratification or other non-uniformity, etc.

7.1.6 Flow sampling should be used whenever possible.

7.1.7 To facilitate mixing, samples shall be so filled that the air space above the liquid level shall be 30 percent to 35 percent of the capacity of the sample containers.

7.2 Sampling Instruments

7.2.1 Sampling Bottle or Can (see Fig. 1)

This instrument is suitable for sampling large vessels and tanks of glycerine. It consists of a weighed glass or metal container with removable stopper to which a suitable chain, pole or cord is attached. This device is lowered to the various desired depths at which the stopper is removed, and the container is allowed to fill.

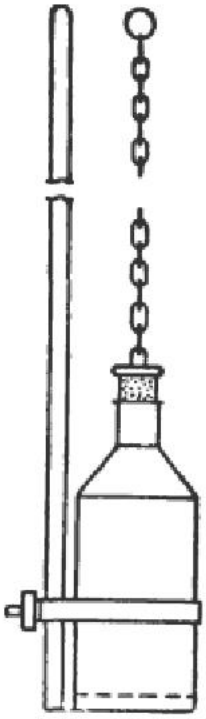

Fig. 1 Sampling Bottle or Can

7.2.2 Sampling Tubes

These instruments are suitable for sampling glycerine in drums and mobile tanks. The three recommended forms of sampling tubes are described in 7.2.2.1 to 7.2.2.3.

7.2.2.1 Closed type sampling tube, undivided (see fig. 2)

This instrument, also known as drum sampler, consists of two concentric metallic tubes closely fitted into each other throughout their entire length, so that one tube can be rotated within the other. Longitudinal openings of about one-third the circumference are cut in both tubes. In one position, the openings in the two tubes coincide; the sampling tube is open when in this position and admits the material. By turning the inner tube through an angle of 90°, it becomes a sealed container. The inner tube may have a diameter of 20 mm to 40 mm and is undivided along its length to serve as a single container.

The two concentric tubes are provided with ports at their bottom ends, so placed that the material contained in the instrument can be drained through them when the longitudinal openings coincide. The length of the instrument should be such as to enable it to reach the bottom of the container being sampled.

The instrument is inserted closed, the material is admitted by opening it, and finally it is closed and withdrawn.

7.2.2.2 Core sampler

It consists of a hollow tube of approximately 50 mm uniform internal diameter, and a length sufficient to take a sample through the entire depth of the liquid. The bottom is closed by a tight valve or cock which, when opened, allows an unrestricted opening of the full size of the tube and does not leak when closed. This valve is operated by a rod from the top and is so constructed that a sample can be taken within 1 cm of the bottom of the drum or tank.

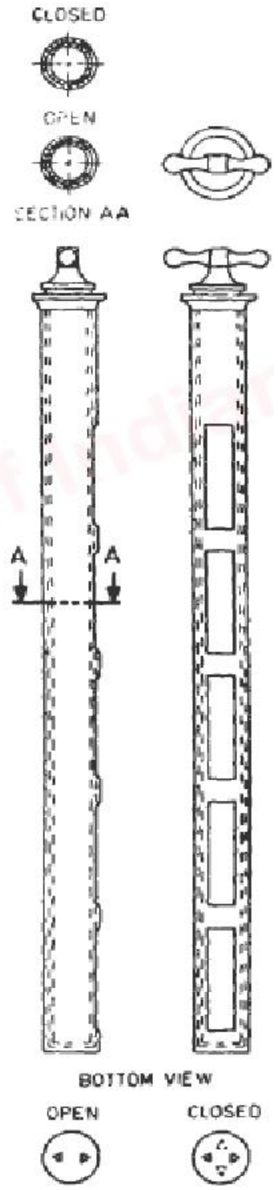

Fig. 2 Closed Type Sampling Tube

7.2.2.3 Open type sampling tube (see fig. 3)

It is made of metal or thick glass, and may be of 20 mm to 40 mm diameter and 100 mm to 800 mm length. The upper and lower ends are conical and narrow down to 10 mm to 15 mm diameter. Handling is facilitated by two rings at the upper end. For taking a sample, the instrument is first closed at the top with the thumb or a stopper and lowered until desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

7.2.2.4 Dipper Sampler (see Fig. 4)

This instrument is suitable for flow sampling. It consists of a beaker of approximately 500 mL capacity attached to a handle. This device may be used for collecting samples from the point of discharge, but great care has to be taken to avoid contamination from moisture or moist air.

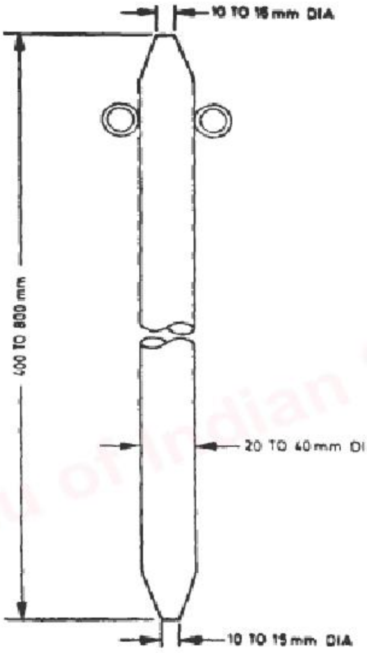

Fig. 3 Open Type Sampling Tube

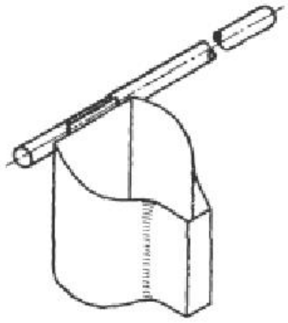

Fig. 4 Dipper Sampler

7.3 Sample Containers

7.3.1 The samples shall be packed in clean dry glass bottles provided with suitable tight stoppers. In the case of refined glycerine, glass bottles with neutral reaction shall be used. Glass stoppers or new good quality velvet corks may be used but not rubber stoppers. The corks may be suitably wrapped with material such as cellophone to prevent contact of the sample with them, and it is recommended that this should be done in the case of refined glycerine.

7.3.2 The sample container shall be sealed with wax in such a way that the contents and the label cannot be removed without breaking the imprint of the seal.

7.4 Scale of Sampling

7.4.1 Lot

All the containers in a single consignment of the type and grade of material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots. Should the consignment not be uniform in quality, the parts of the consignment which appear to be similar may be collected together and each quality treated as a separate lot.

7.4.2 Gross Sample

The general procedure for taking a gross sample is to draw a number of portions from the bulk quantity (see 7.4.2.1) or a number of portions from all or several packages (see 7.4.2.2), and mix them thoroughly. Representative portions of the gross sample shall be transferred to airtight containers of suitable size for the test samples as described under 7.7.

7.4.2.1 Gross sample from bulk quantities

Gross sample from bulk quantities shall be drawn in quantities of not less than 20 kg per 20 000 kg.

7.4.2.2 Gross sample from small packages

When sampling from drums, tins, bottles, etc, the packages from which the samples are drawn shall be selected at random from the lot. The following schedule is recommended for the number of packages to be sampled:

By agreement between the purchaser and the supplier, each package may be sampled. A minimum of 0.1 percent of the total quantity in the lot or 2 kg, whichever is greater, shall be drawn as the gross sample.

Number of Packages in the Lot	Number of Packages to be Sampled
1 to 4	All packages
5 to 100	At least 20 percent, with a minimum of 4 packages
More than 100	At least 10 percent, with a minimum of 20 packages

7.5 Procedure for Crude Glycerine

7.5.1 General

The sampling of crude glycerine is sometimes made difficult by the presence of salt. The most satisfactory method of sampling is to take a flow sample during the transfer of stock, or immediately after, before sedimentation of salt has taken place.

If it is necessary to sample after such separation has taken place, the thickness of the impenetrable salt layer at the bottom of the container may be estimated by means of a flat weight on a dipper tape. This bottom layer shall, in such instances, be sampled and accounted for separately.

7.5.2 Crude Glycerine in Drums

It is recommended that drums should be sampled before the salt has settled because it is almost impossible to obtain a correct proportion of salt after it has settled in the drums. If, however, the salt has settled in the drums, endeavour to soften any congealed salt layer by storing the drums in a warm place for several hours. Roll each drum through several revolutions to mix the contents, and test the condition of the salt layer. If it has dispersed, proceed to sample by inserting the closed or open type sampling tube (see Fig. 2 and 4) through a close-fitting funnel fitted with a suitable washer so that the tube is automatically cleaned as it emerges. Discharge the samples into a receiving vessel by opening the ports

If the salt layer has not dispersed, sample the glycerine above the salt layer in the manner and with the sampler described above. In this case arrangements should be made to sample the salt after transferring glycerine from the drums.

At the conclusion of sampling, prepare a gross composite sample by rapidly mixing the individual samples by stirring thoroughly with a clean wooden or stainless steel paddle and immediately transferring to clean dry airtight bottles.

7.5.3 Crude Glycerine in Storage Tanks and Tank Wagons

7.5.3.1 Static tanks

It is not possible to lay down a standard method applicable in all instances, but the methods given here will usually be found suitable. If there is a hard impenetrable layer of salt which cannot be sampled at all, the thickness of this shall be measured but it shall not be finally accounted for until the tank is subsequently emptied. The material above this layer shall be sampled by either of the following two methods which are also suitable if there is no settled salt:

Method 1 — Lower the core sampler vertically through the tank with the bottom valve or cock wide open, at a uniform rate, slow enough to permit the tube to fill as it is lowered, that is, the tube should not be lowered faster than the glycerine can flow in so that the levels inside and outside the sampler are nearly equal at all times. This precaution shall be carefully observed. Allow the core sampler to rest at the bottom of the tank for a short time before closing the valve or cock. Withdraw the core sampler slowly, wiping off glycerine clinging to the outside of the tube, and discharge into a mixing can. Repeat this operation, if necessary, to obtain a sample of sufficient size. Keep the mixing can covered at all times except when actually discharging samples into it or mixing the combined withdrawals. Rapidly mix the contents of the can by stirring thoroughly with a clean wooden or stainless steel paddle and immediately transfer to clean dry airtight bottles.

Method 2 — Take samples individually at uniformly spaced depths by lowering the sampling bottle B (see Fig. 1) to each chosen depth, opening it, filling and withdrawing. The individual portions so taken, including a portion as near to the tank bottom as possible, shall then be mixed in proportion to the quantities of material represented. Finally, sample the bottom layer of the glycerine by means of a core sampler or a broad, flat-bottomed, relatively shallow sampler open at the top (such as 150 mm diameter, 75 mm high). Mix this portion with that from the sampling bottle in proportion to the quantities of material represented by the samples. Mix the combined samples by stirring with a clean wooden or stainless steel paddle and immediately transfer to clean dry airtight bottles.

7.5.3.2 During loading or unloading

A dipper sampler (see Fig. 4) is recommended for sampling flowing glycerine at the discharge end of the pipe as the material enters or leaves the tank or tank wagon. The total gross sample to be accumulated should represent not less than 25 withdrawals of similar size, and spaced as uniformly as possible throughout the loading or unloading operation. Wipe the dipper sampler after each withdrawal or rinse it and dry before reusing, otherwise the remaining glycerine may absorb and thus include a significant quantity of moisture. Accumulate the withdrawals in a can large enough to permit thorough mixing. Keep the can tightly covered at all times except when actually discharging samples into it or mixing the combined withdrawals. Rapidly mix the contents of the can by stirring thoroughly with a clean wooden or stainless steel paddle and immediately transfer a portion of the glycerine to clean dry airtight bottles.

7.6 Procedure for Refined Glycerine

7.6.1 The procedure for sampling refined glycerine in storage tanks, tank wagons, drums, etc., shall be the same as that prescribed for crude glycerine (see 7.5) except that the sampling apparatus shall be of stainless steel or glass. When glass apparatus is used, it is essential to take adequate precautions to ensure that the glycerine is not contaminated by glass splinters.

7.6.2 Unlike crude glycerine, the refined grades are free from settled salts but the possibility of stratification is more pronounced particularly if the glycerine has been frozen and subsequently thawed. In such cases, the material should be mixed thoroughly before drawing samples.

7.7 Test and Referee Samples

7.7.1 Size of Test Sample

The minimum size for each test sample shall be 500 g.

7.7.2 Preparation of Test Sample

Normally, all the samples drawn as described under 7.5 or 7.6 shall be put into a clean dry airtight receptacle and mixed thoroughly. At least four uniform samples (test samples) shall be drawn therefrom. One test sample shall be sent to the purchaser and one to the supplier.

7.7.3 Referee Sample

Two of the test samples, bearing the seals of the purchaser and the supplier, shall constitute the referee samples, to be used in case of dispute between the purchaser and the supplier.

7.8 Test for Acceptance

7.8.1 General

It is advisable that all tests should be carried out in duplicate.

7.8.2 Examination and Tests

The purchaser may separately examine test samples of each of the separate quantities (see 7.4.2.2) for compliance with the requirements of the individual specification, or he may prepare, for the purpose of such examination and at any stage of the progress of the examination, a composite sample representing the whole of the consignment, by mixing the test samples.

7.8.3 Criterion for Judgement

If two or more quantities are examined from a consignment and if one or more of them do not comply with the requirements of the specification for that particular grade of crude or refined glycerine, the purchaser shall have the right to accept only that portion which complies with requirements, or accept or reject the whole of the consignment.

7.9 Marking of Sample Containers

7.9.1 Each sample container after filling shall be sealed and marked with full details of sampling, the number of packages sampled, the date of sampling, and other particulars of the consignment.

7.9.1 A label bearing the particulars given under 7.9.1 shall be attached to every sample container. A recommended form of label is given below:

Name and grade of material ……………

Manufacturer or consigner ……………

Brand name, if any ……………

Lot number ……………

Size of consignment ……………
Number of packages sampled ……………
Place of sampling ……………
Date of sampling ……………
Signature of sampling officer ……………

7.9.3 The containers may also be marked with the BSTI Certification Mark.

NOTE - The use of the BSTI Certification Mark is governed by the provisions of the Bangladesh Standards and Testing Institution Act 2018 and the Rules and Regulations made there under. Details of conditions be under which a license for the use of the BSTI Certification Mark may granted to manufacturers or processors, may be obtained from the Bangladesh Standards and Testing Institution.

8. Test Methods

8.1 Unless specified otherwise, tests shall be carried out as prescribed in Annex A. Reference to the relevant clauses of Annex A is given in column 10 of Table 1.

8.2 Test methods, not prescribed in this standard, shall be subject to agreement between the purchaser and the supplier.

Annex A

(Clause 6.1 and Table 1)

Methods of Test for Glycerine

A-1 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* BDS 833) shall be employed in tests.

NOTE - 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 Determination of Glycerol Content

A-2.0 Outline of the Method - Glycerine reacts with sodium metaperiodate ($NaIO_4$) in acid aqueous solution to produce formaldehyde and formic acid; the latter is used as a measure of glycerol. Other polyhydric alcohols with three or more adjacent hydroxyl groups also produce formic acid and, therefore, interfere in glycerol determination. The method cannot, therefore, be used directly on samples containing sugars.

A-2.1 Apparatus

A-2.1.1 Burette — 50 mL capacity, with 0.05 mL graduations.

A-2.1.2 Pipette — 50 mL capacity.

A-2.1.3 Conical Flaks — 500 mL capacity, with glass stopper.

A-2.1.4 pH Meter — With glass electrode.

A-2.1.5 Weighing Pipette or Bottle

A-2.2 Reagents

A-2.2.1 Standard Sodium Hydroxide Solution

0.1 N, accurately standardized. The solution shall be carbonate-free and should be crosschecked periodically against standard glycerol (see A-2.2.7) by the method described under A-2.3.

A-2.2.2 Sodium Hydroxide Solution — 0.05 N.

A-2.2.3 Phenol Red Indicator Solution

0.05 percent (m/v) solution. Dissolve 0.05 g of phenol red in 10 mL of ethyl alcohol and make up to 100 mL with water.

A-2.2.4 Dilute Sulphuric Acid — 0.2 N.

A-2.2.5 Sodium Metaperiodate Solution

Dissolve 70 g of sodium metaperiodate in one litre of water containing 10 mL of approximately 1 N sulphuric acid, and store the solution in an amber coloured bottle in the dark.

NOTE — Sodium metaperiodate shall be a white crystalline powder containing not less than 98 percent sodium metaperiodate. If metaperiodate does not dissolve in the solution, it is not of reagent quality, and a new supply should be obtained.

A-2.2.6 Ethylene Glycol — neutral and free from glycerol.

A-2.2.7 Standard Glycerol

An aqueous solution containing about 90 percent of chemically pure glycerine complying with 4.1.4 and the concentration of which is known from density at 30 °C (see Table 2) and moisture [Karl Fischer method] determinations.

NOTE — If necessary, density of glycerine may be calculated by multiplying its relative density (see **A-3**) by the density of water at the desired temperature.

A-2.3 Procedure

Using a weighing pipette or bottle, transfer into a conical flask a well-mixed and accurately weighed sample (see Note 1). Add 100 mL of carbon dioxide-free water and 3 drops of phenol red indicator solution, and acidify with sulphuric acid solution to a definite yellow colour.

Heat to boiling and cool to room temperature. Adjust the pH value to about 8 to 8.1 by adding sodium hydroxide solution (see A-2.2.2) drop by drop until the colour changes from yellow to just pink. If the colour of the original solution interferes with detection of the colour change of the indicator, use a pH meter.

Pipette accurately 50 mL of sodium metaperiodate solution, replace the glass stopper, swirl for intimate mixing, and allow the flask to stand in the dark for 30 minutes.

Wash down the sides of the flask with water, add 5 mL of ethylene glycol, replace the stopper, swirl gently, and allow the flask to stand in the dark for further 20 minutes.

Titrate the liberated formic acid with standard sodium hydroxide solution (see A-2.2.1) from yellow to pink end point. Carry out a blank test simultaneously under similar test conditions (see Note 2).

Table 2 Glycerol Content and Density of Aqueous Solutions of Refined Glycerine

(Clause A-2.2.7)

Sl No. (1)	Glycerol (2)	Density At 30 °C (3)	Glycerol (4)	Density At 30 °C (5)
i)	**percent**	**g/mL**	**percent**	**g/mL**
ii)	100	1.254 95	75	1.189 00
iii)	99	1.252 35	74	1.186 35
iv)	98	1.249 75	73	1.183 65
v)	97	1.247 10	72	1.181 00
vi)	96	1.244 50	71	1.178 30
vii)	95	1.241 90	70	1.175 65
viii)	94	1.239 30	69	1.172 90
ix)	93	1.236 70	68	1.170 20
x)	92	1.234 10	67	1.167 45
xi)	91	1.231 50	66	1.164 70
xii)	90	1.228 90	65	1.161 95
xiii)	89	1.226 25	64	1.159 25
xiv)	88	1.223 60	63	1.156 50
xv)	87	1.220 95	62	1.153 75
xvi)	86	1.218 30	61	1.151 00
xvii)	85	1.215 65	60	1.148 30
xviii)	84	1.213 00	59	1.145 55
xix)	83	1.210 35	58	1.142 85
xx)	82	1.207 70	57	1.140 10
xxi)	81	1.205 05	56	1.137 40
xxii)	80	1.202 40	55	1.134 70
xxiii)	79	1.199 70	54	1.131 96
xxiv)	78	1.197 05	53	1.129 25
xxv)	77	1.194 35	52	1.126 50
xxvi)	76	1.191 70	51	1.123 80
xxvii)			50	1.121 10

*Reproduced from BOSART and SNODDY. Industrial and Engineering Chemistry, Ed 20, 1378 (1928).

NOTES -

1. The mass in grams of the sample should be within the limits given by the formula (41 ± 9)/P, where P is the expected percentage of glycerol in the sample. When the glycerol content is not known, weigh the amount specified for 100 percent glycerol, and from the results of this test, select the proper size sample taking into account that the results will tend to be high if the sample weighed is smaller than that required for the most accurate results.

2. If a pH meter is used, the end point for the blank should be pH (6.5 ± 0.1) and that for the sample (8.1±0.1).

A-2.4 Calculation

Glycerol content, percent by mass = $\frac{9.209(S-B)N}{M}$

Where,

S = volume in mL of standard sodium hydroxide solution required for the sample;

B = volume in mL of standard sodium hydroxide solution required for the blank;

N = normality of standard sodium hydroxide solution; and

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

NOTE — When the titration is carried out with phenol red solution as the indicator, add 0.3 percent (expressed on 100 percent glycerol) to the observed glycerol result. No correction is necessary if a *p*H meter is used.

A-3 Determination of Relative Density

A-3.0 General

The relative density of glycerine shall be expressed as the ratio of the mass in air of a given volume of the material at 30 °C to that of an equal volume of water at the same temperature. The relative density may be determined with a hydrostatic balance or relative density bottle. The relative density bottle method shall be adopted as the referee method in cases of dispute.

A-3.1 Hydrostatic Balance Method

A-3.1.0 General

There are several forms of hydrostatic balances; the procedure described below is applicable to a chain analytical balance.

A-3.1.1 Procedure

Take a sufficient quantity of freshly boiled and cooled water at 30 °C in a cylinder (about 125 mm high and 25 mm inside diameter), and place it on the adjustable platform inside the balance. Suspend a clean dry plummet in the cylinder and adjust the platform so that the loop of the wire by which the plummet is suspended just dips in the water. Place the rider on the beam in such a position that the pointer swings equally on either side of zero on the scale. Make the final adjustment using the Vernier scale and note the reading. Lower the platform and remove the plummet and the cylinder. Wipe the plummet dry. Fill another cylinder with the test sample at 30°C and repeat the above procedure ensuring that no air bubbles are formed in the eye hole of the plummet. Take the reading as described previously for water. As glycerine is very hygroscopic, it is essential that the test is performed as rapidly as possible.

A-3.1.2 Calculation

Apparent relative density at 30°C = $\frac{A}{B}$

Where,

A = scale reading with the material taken for the test at 30 °C; and

B = scale reading with water at 30 °C.

A-3.2 Relative Density Bottle Method

A-3.2.1 Apparatus

A-3.2.1.1 Relative density bottle

A relative density bottle of 50 mL capacity with central neck fitted with ground-glass thermometer, which serves as a stopper, graduated from 10 °C to 40 °C in 0.2 °C divisions and an overflow side tubewith ground-in glass cap, as shown in Fig. 5.

To calibrate, clean and dry the bottle thoroughly, weigh and then fill with freshly boiled and cooled water at about 25 °C after removing the cap of the side arm. Filling should be done to overflowing by holding the bottle on its side. Insert the stopper in such a way as to prevent entrapping of air bubbles. Excess water overflows through the side tube. Immerse in a water-bath maintained at (30.0 ± 0.2) °C. Keep the entire bulb completely covered with water. Allow the temperature to rise until the temperature inside the bottle and that of the bath are same. This takes about 30 minutes. Carefully remove any water which has exuded from the side opening and replace the cap on the side tube. Remove the bottle from the bath and wipe completely dry, equilibrate to room temperature and weigh. Calculate the mass of water. This is a constant for the bottle but should be checked periodically.

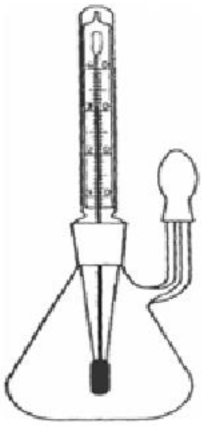

Fig. 5 Relative Density Bottle

A-3.2.1.2 Water-bath — maintained at (30.0 ± 0.2) °C.

A-3.2.1.3 Thermometer — Any convenient thermometer of a suitable range with 0.1 °C or 0.2 °C subdivisions. The thermometershould be checked against a standard thermometer which has been calibrated and certified by BSTI or any other recognized calibration laboratory.

A-3.2.2 Procedure

Fill the clean and dry bottle with the test sample previously cooled to about 25 °C, after removing the cap of the side arm, holding the bottle on its side in such a manner as to prevent entrapping of air bubbles. Insert the stopper in such a way that the capillary of the side tube is completely filled with glycerine. Immerse and hold the bottle in water-bath at (30.0 ± 0.2) °C so that the entire bulb is covered with water. Allow temperature to rise until the temperature of the water-bath and that of glycerine in the relative density bottle are same. This takes about 30 minutes. Carefully wipe off any glycerine that has come through the capillary opening and replace the cap on the side tube. Remove the bottle from the bath, clean and dry the outside thoroughly. Equilibrate to room temperature and weigh.

It is advisable to carry out the test in duplicate.

A-3.2.3 Calculation

$$\text{Relative density at 30 °C} = \frac{A - B}{C - B}$$

Where,

A = mass in g of the relative density bottle withthe glycerine taken for the test at 30 °C;

B = mass in g of the relative density bottle; and

C = mass in g of the relative density bottle withwater at 30 °C.

A-3.3 Corrections

Sometimes, especially when using the hydrostatic balance, it is not convenient to make the determination at the specified temperature, namely, 30 °C. The determination may be made at a convenient temperature, T as near to 30 °C as possible and the result corrected to 30 °C as follows:

$$\text{Relative density at 30°/30 °C} = \frac{ab + kc\,(T - 30)}{c}$$

Where,

a = relative density of glycerine at T°/T C;

b = density of water at T C;

c = density of water at 30 °C, namely, 0.995 67;

T = temperature at which relative density was determined; and

K = change in relative density per degree Celsius. The value for K shall be selected as follows:

Transposition	K
from 15°/15 °C to 20°/20 C and vice versa	0.000 40
from 20°/20 °C to 25°/25 C and vice versa	0.000 32
from 25°/25 °C to 30°/30 C and vice versa	0.000 27
from 30°/30 °C to 35°/35 C and vice versa	0.000 23

A-4 Determination of Colour

A-4.0 General - This method determines the colour of glycerine by comparison with Lovibond glasses of known colour characteristics.

A-4.1 Apparatus

A-4.1.1 Lovibond Tintometer

A-4.1.2 Glass Cells — 5¼ in. cell. The cell should be of clear, colourless glass.

A-4.2 Procedure - Select a clean dry glass cell of the prescribed size and fill it with the well-mixed sample. Put the cell in position in the Tintometer and place alongside of it such yellow, red and/or neutral Lovibond glass slides as are necessary to match the colour shade of the glycerine, observing the colours of the glycerine and of the combination of the glass slides through aneye piece.

A-4.3 Report

Report the colour of the material under test in terms of Lovibond units as follows:

Colour reading in 5¼ in. cell = Y + R

Where,

Y = the sum total of the various yellow slides used, and

R = the sum total of the various red slides used.

A-5 Determination of Ash

A-5.1 Apparatus

A-5.1.1 Platinum Dish or Crucible

A-5.1.2 Desiccator — containing concentrated sulphuric acid.

A-5.2 Procedure for Crude Glycerine

Weigh accurately about 5 g of the well-mixed sample in a platinum dish and heat it gently. Keep the temperature low to avoid volatilization of salts and formation of sulphides. When the mass is charred to a point that added water does not become coloured by soluble organic matter, lixiviate with hot water, filter through ashless filter paper, wash and ignite the residue and the filter paper in the platinum dish. Return the filtrate and washings to the dish, evaporate and carefully incinerate the residue without fusion at not more than dull red heat (Max 550 °C). Cool the dish in a desiccator and weigh the residue. Preserve the ash for test in **A-14.3**.

A-5.3 Procedure for Refined Glycerine

Weigh accurately about 50 g of the sample in a platinum dish and heat it gently to a point where glycerine vapours are evolved and can be ignited. Ignite the glycerine, remove the burner, and allow the glycerine to burn down to a brown or black residue. Cool for 1 minute, recommence heating and incinerate the residue at not more than dull red heat (Max 550 °C) to a white or grey white ash. Cool the dish in a desiccator and weigh the residue.

A-5.4 Calculation

$$\text{Ash, percent by mass} = \frac{100\,m}{M}$$

Where,

m = mass in g of the residue, and

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

A-6. Determination of Non-Volatile Organic Residue

A-6.0 General

This method determines any material which is not volatile at 160 °C when tested by the method described below. It includes inorganic ash and this is subtracted from the total residue to obtain the non-volatile organic residue.

A-6.1 Apparatus

A-6.1.1 Volumetric Flasks — 100 mL capacity.

A-6.1.2 Evaporating Dish — Petri or similar glass dish, 70 mm diameter and 12 mm deep.

A-6.1.3 Desiccator — containing concentrated sulphuric acid.

A-6.1.4 Air Oven — thermostatically controlled to maintain a uniform internal temperature of (160 ± 2) °C.

A-6.2 Reagents

A-6.2.1 Standard Hydrochloric Acid — 1.0 N, accurately standardized.

A-6.2.2 Standard Sodium Carbonate Solution — 10 N, accurately standardized.

A-6.3 Procedure for Total (Organic and Inorganic) Residue at 160 °C

A-6.3.1 Weigh accurately about 10 g of the well-mixed sample in a 100 mL flask, add about 75 mL of water, adjust alkalinity to 0.2 percent (as Na_2O) as described under **A-6.3.2**, and finally make up the volume to 100 mL. An excess of alkalinity beyond 0.2 percent may result in the formation of polyglycerols and should therefore be avoided.

A-6.3.2 The object of adjusting the alkalinity of the sample to 0.2 percent is to prevent loss of organic acids. After determining the acidity or alkalinity of the sample as described under **A-14**, use equations given below to determine the amount of standard hydrochloric acid or sodium carbonate solution required to be added for necessary adjustment of alkalinity:

Sl No.	Condition of Sample	Volume in mL of	
		Standard Hydrochloric Acid	Standard Sodium Carbonate Solution
i)	Alkaline (more than 0.20 percent, as Na_2O)	$\frac{(A - 0.20)M}{3.1N}$	
ii)	Alkaline (less than 0.20 percent, as Na_2O)		$\frac{(0.20 - A)M}{3.1N}$
iii)	Neutral		$\frac{0.20\,M}{3.1N}$
iv)	Acidic		$\frac{(B + 0.20)M}{3.1N}$

Where,

A = alkalinity (as Na_2O) percent by mass;

B = acidity (as Na_2O equivalent) percent by mass;

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

N = normality of standard hydrochloric acid or sodium carbonate solution as the case may be.

A-6.3.3 Transfer 10 mL of the adjusted solution from the volumetric flask into a previously weighed evaporating dish. Place the dish on a water-bath or on top of the oven until most of the water has evaporated. Transfer the dish to the air oven maintained at 160 °C and evaporate most of the glycerine at a temperature of 130 °C to 140 °C. This temperature is readily maintained by leaving the door partially open. When only slight or no vapour is seen to be coming off, remove the dish from the oven and cool. Add 0.5 mL to 1.0 mL of water and rotate the dish to bring as much of the residue into solution as possible. Allow the dish to remain on the water-bath until the excess water has evaporated and the residue is in such a condition that on returning it to the oven at 160 °C, spurting will not occur.

A-6.3.4 Allow the dish to remain inside the oven at 160 °C for exactly 1 hour. Remove it and cool. Add 0.5 mL to 1.0 mL of water, evaporate as before and bake for exactly 1 hour at 160 °C. Remove the dish, cool for a set period of time (this shall be observed in all subsequent cooling periods) in a desiccator, and weigh. Repeat the treatment with water, evaporating, baking and cooling until a constant loss of 1 mg to 1.5 mg per hour is obtained.

A-6.4 Calculation

A-6.4.1 Calculation of Total (Organic and Inorganic) Residue at 160 °C — Use the appropriate equation as given below:

Sl No.	Condition of Sample	Total Residue at 160 °C, Percent by mass
i)	Alkaline (more than 0.20 percent, as Na_2O)	$\frac{1000\,R - 1.8\,V_1\,N}{M}$
ii)	Alkaline (less than 0.20 percent, as Na_2O)	$\frac{1000\,R - 5.3\,V_2\,N}{M}$
iii)	Neutral	$\frac{1000\,R}{M} - 0.34$
iv)	Acidic	$\frac{1000\,R - 2.2\,V_3\,N}{M} - 0.20$

Where,

R = mass in g of total residue;

V_1 = volume in mL of standard hydrochloric acid added under **A-6.3.2** (i);

V_2 = volume in mL of standard sodium carbonate solution added under **A-6.3.2** (ii);

V_3 = volume in mL of standard sodium carbonate solution added under **A-6.3.2** (iv);

N = normality of standard hydrochloric acid or sodium carbonate solution used; and

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

A-6.4.2 Calculation of Non-volatile Organic Residue

Non-volatile organic residue, percent by mass = X - Y

Where,

X = total residue at 160 °C, percent by mass, as calculated under A-6.4.1; and

Y = ash, percent by mass, as determined under A-5.

NOTE — Alkaline salts of organic acids during ignition get converted into carbonates and hence are not included to the extent of carbonate produced therefrom, in the organic residue.

A-7 Determination of Arsenic

A-7.1 Procedure

Weigh accurately about 10 g of the well-mixed sample and carry out the determination as prescribed in BDS 1333.

A-8 Test for Copper

A-8.1 Reagents

The following reagents are required and shall be freefrom copper impurity.

A-8.1.1 Hydrochloric Acid — 10 percent (v/v) solution of hydrochloric acid (r.d. 1.18).

A-8.1.2 Hydrogen Sulphide Solution — aqueous, freshly prepared.

A-8.2 Procedure

Take 10 mL of the sample in a test tube and add 30 mL of water, 1 mL of hydrochloric acid and 10 mL of hydrogen sulphide solution. The non-development of any colour indicates the absence of copper.

A-9 Determination of Iron

A-9.1 Apparatus

A-9.1.1 Silica Dish

A-9.1.2 Nessler Cylinders — 50 mL capacity.

A-9.1.3 Pipette — 1 mL capacity with 0.01 mL graduations.

A-9.2 Reagents

The following reagents are required and, with the exception of **A-9.2.5**, they shall be free from iron impurity.

A-9.2.1 Potassium Hydrogen Sulphate — Fused.

A-9.2.2 Hydrochloric Acid — 10 percent (v/v)solution of hydrochloric acid (r.d. 1.18).

A-9.2.3 Nitric Acid — 10 percent (v/v) solution ofnitric, acid (r.d. 1.42).

A-9.2.4 Thioglycolic Acid Solution — 10 percent solution. Dilute 10 mL of 90 percent thioglycollic acid to 100 mL with water.

NOTE — Care shall be taken not to allow concentrated thioglycolic acid to come in contact with the skin, as it causes painful wounds which take a long time to heal.

A-9.2.5 Standard Iron Solution

Dissolve 7.03 g of ferrous ammonium sulphate [FeSO4(NH4)2SO4.6H2O] in water containing 5 mL of dilute sulphuric acid (1 : 3) and make up to 1 litre in a volumetric flask. Dilute this stock solution 100 times immediately before use. One millilitre of this solution contains 0.000 01 g of Fe.

A-9.2.6 Ammonia Solution

50 percent (v/v) aqueous solution, prepared from liquor ammonia (r.d. 0.90).

A-9.2.7 Citric Acid Solution — 50 percent (m/v), aqueous.

A-9.3 Procedure

A-9.3.1 Weigh accurately about 10 g of the sample in a silica dish and proceed to ash as described under **A-5.2**. After ashing is complete, add 0.5 g of potassium hydrogen sulphate and fuse gently until a clear melt is obtained. Allow to cool, add 5 ml of hydrochloric acid and warm, flowing the acid round the sides of the dish. Add 5 ml of nitric acid and bring to incipient boiling.

Transfer the solution quantitatively to a 100 mL volumetric flask and make up to volume with water.

A-9.3.2 Pipette out in a Nessler cylinder 25 mL of the solution obtained under **A-9.3.1**. Add 4 mL of citric acid solution and 10 mL of ammonia solution. Cool and add 5 mL of thioglycolic acid solution. Mix well after each addition. Dilute to 50 mL with distilled water. A pale purple colour is produced if iron is present.

A-9.3.3 To a second Nessler cylinder, add 0.125 g of potassium hydrogen sulphate, 25 mL of distilled water, 1.25 mL of nitric acid and 2.5 mL of hydrochloric acid. Add 4 mL of citric acid solution and 10 mL of ammonia solution. Cool and add 5 mL of thioglycolic acid solution. Mix well after each addition. Dilute to 50 mL with distilled water. From a pipette, gradually add standard iron solution so that, after shaking the cylinder, the intensity of the colour matches that produced by the material undertest. Note the volume of standard iron solution used.

A-9.4 Calculation

$$\text{Iron (as Fe), parts per million} = \frac{40\,V}{M}$$

Where,

V = volume in mL of standard iron solution used,and

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

A-10 Determination of Lead

A-10.1 Apparatus

A-10.1.1 Nessler Cylinders — 50 mL capacity.

A-10.2 Reagents — The following reagents are required and, with the exception of A-10.2.1, they shall be free from lead impurity.

A-10.2.1 Standard Lead Solution

Dissolve 0.16 g of lead nitrate in 5 mL of concentrated nitric acid (r.d. 1.42) and make up to 100 mL with water in a volumetric flask. Dilute this stock solution 100 times immediately before use. One millilitre of this solution contains 0.00001 g of Pb.

A-10.2.2 Potassium Cyanide Solution

10 percent (m/v). Dissolve 10 g of potassium cyanide in 90 mL of water, add 2 mL of hydrogen peroxide solution(6 percent), allow to stand for 24 hours, and make upto 100 mL with water.

A-10.2.3 Sodium Sulphide Solution — 10 percent (m/v), aqueous. Filter, if necessary.

A-10.2.4 Ammonia Solution — 10 percent (m/v). Dilute 1 part of ammonia (r.d. 0.90) with 2.5 parts ofwater.

A-10.2.5 Acetic Acid Solution — 1 : 2 solution of glacial acetic acid in water.

A-10.3 Procedure

A-10.3.1 Prepare solutions from the sample by dissolving 14.5 g, 12.0 g, 9.5 g, 7.0 g, 4.5 g, and 2.0 g, respectively, in about 10 mL of hot water containing 5 mL of acetic acid solution. To the solution containing 2.0 g of the sample, add 1 mL of standard lead solution; this will serve as the control. Make all the solutions alkaline with ammonia solution, add to each 1 mL of potassium cyanide solution, and make up to 40 mL with water. If the solutions are turbid, filter them through a filter paper. Equalize any difference in colour by adding a few drops of a highly diluted solution of burnt sugar.

A-10.3.2 Dilute all the solutions, including the control, to 50 mL with water; to each add 2 drops of sodium sulphide solution, mix thoroughly and transfer to Nessler cylinders. Compare the colours by viewing the cylinders against a white tile. Note which one of the five test samples has a colour most closely matching that of the control. Repeat the test using an appropriate sample size and note the amount of glycerine required to produce a colour with an intensity exactly matching that, of the control.

A-10.4 Calculation

$$\text{Lead (as Pb), parts per million} = \frac{10}{M - 2}$$

Where,

M = mass in g of the material required to match the colour of the control sample.

A-11 Determination of Chlorides

A-11.0 General

Two methods are described, one volumetric and the other nephelometric. The former method is recommended for estimating chlorides in dynamite, Industrial white and technical grades of refined glycerine, and the latter for the analytical reagent and chemically pure grades.

A-11.1 Volumetric Method

A-11.1.1 Apparatus

A-11.1.1.1 Conical flasks — 250 mL capacity.

A-11.1.2 Reagents

The following reagents are required and shall be free from chloride impurity.

A-11.1.2.1 Nitric acid — 35 percent (v/v) solution of nitric acid (r.d. 1.42).

A-11.1.2.2 Nitrobenzene

A-11.1.2.3 Standard silver nitrate solution — 0.05 N, accurately standardized.

A-11.1.2.4 Standard ammonium thiocyanate solution — 0.05 N, accurately standardized.

A-11.1.2.5 Ferric ammonium sulphate solution — saturated, aqueous.

A-11.1.3 Procedure

Weigh accurately about 20 g of the sample in a 250 mL conical flask and add about 100 mL of water. To this solution, add 5 mL of nitric acid, a few drops of nitrobenzene and 5 mL of standard silver nitrate solution.

Swirl and titrate with standard ammonium thiocyanate solution using 1 mL of ferric ammonium sulphate solution as indicator. Carry out a blank test using the same reagents.

A-11.1.4 Calculation

$$\text{Chlorides (as Cl), parts per million} = \frac{35\,500\,(B - S)\,N}{M}$$

Where,

B = volume in mL of standard ammonium thiocyanate solution required for the blank;

S = volume in mL of standard ammonium thiocyanate solution required for the sample;

N = normality of standard ammonium thiocyanate solution; and

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

A-11.2 Nephelometric Method

A-11.2.1 Apparatus

A-11.2.1.1 Nessler cylinders — 50 mL capacity.

A-11.2.1.2 Pipette — 1 mL capacity, with 0.01 mL graduations.

A-11.2.2 Reagents

The following reagents are required and, with the exception of **A-11.2.2.3**, they shall be free from chloride impurity.

A-11.2.2.1 Nitric acid — 35 percent (v/v), solution of nitric acid (r.d. 1.42) in water.

A-11.2.2.2 Silver nitrate solution — 5 percent (m/v), aqueous.

A-11.2.2.3 Standard chloride solution

Dissolve 1.647 9 g of sodium chloride in water and make up to 1 litre in volumetric flask. Dilute this stock solution 100 times immediately before use. One millilitre of this solution contains 0.000 01 g of Cl.

A-11.2.3 Procedure

A-11.2.3.1 Weigh accurately about 15 g of the sample in a 100 mL volumetric flask and make up to volume with water. Pipette out 25 mL of the solution in a Nessler cylinder, add 1 mL of nitric acid and 10 mL of silver nitrate solution. Make up to 50 mL with water. A dull white turbidity is produced if chlorides are present.

A-11.2.3.2 To a second Nessler cylinder, add 10 mL of silver nitrate solution and 1 mL of nitric acid and make up to 50 mL with water. From a pipette, gradually add standard chloride solution drop by drop so that, after shaking the cylinder, the turbidity matches with that produced by the material under test. Note the volume of standard chloride solution used.

A-11.2.4 Calculation

$$\text{Chlorides (as Cl), parts per million} = \frac{40\ V}{M}$$

Where,

V = volume in mL of standard chloride solution used, and

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

A-12 Determination of Sulphates

A-12.1 Apparatus

A-12.1.1 Nessler Cylinders — 25 mL capacity.

A-12.1.2 Pipette — 1 mL capacity, with 0.01 mlgraduations.

A-12.2 Reagents — The following reagents are required and, with the exception of **A-12.2.3**, they shall be free from sulphate impurity.

A-12.2.1 Hydrochloric Acid — 1:1 solution of hydrochloric acid (r.d. 1.18) in water.

A-12.2.2 Barium Chloride Solution — 10 percent (m/v), aqueous.

A-12.2.3 Standard Sulphate Solution — Dissolve 1.479 g of anhydrous sodium sulphate in water and make up to 1 litre in a volumetric flask. Dilute this stock solution 10 times immediately before use. One millilitre of this solution contains 0.000 1 g of SO_4.

A-12.3 Procedure

A-12.3.1 Weigh accurately about 10 g of the sample into a Nessler cylinder, add 1 mL of hydrochloric acid and 2 mL of barium chloride solution, and make up to 25 mL with water. Allow to stand for 30 minutes. A dull white turbidity is produced if sulphates are present.

A-12.3.2 Simultaneously, introduce 0.5 mL, 0.75 mL, 1.0 mL and 1.25 mL of standard sulphate solution into 4 separate Nessler cylinders. In each, add 1 mL of hydrochloric acid and 2 mL of barium chloride solution, and make up to 25 mL with water. Allow to stand for 30 minutes. Note which one of these four solutions has a turbidity most closely matching that produced by the test material (see **A-12.3.1**). Repeat the test and note the volume of standard sulphate solution required to produce a turbidity exactly matching that of the test material.

A-12.4 Calculation

$$\text{Sulphates (as } SO_4\text{), parts per million} = \frac{40\,V}{M}$$

Where,

V = volume in mL of standard sulphate solution used, and

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

A-13 Determination of Alkalinity

A-13.0 General — Alkalinity represents the free titrable alkali present in samples of refined glycerine.

A-13.1 Apparatus

A-13.1.1 Conical Flask — 500 mL capacity.

A-13.2 Reagents

A-13.2.1 Standard Hydrochloric Acid — 0.1 N, accurately standardized.

A-13.2.2 Phenolphthalein Indicator Solution — 1 percent (m/v) in 95 percent ethyl alcohol (v/v) or rectified spirit (conforming to BDS 1242).

A-13.3 Procedure — Weigh accurately about 100 g of the well-mixed sample into a 500 mL conical flask and add 150 mL of carbon dioxide-free water. Titrate with standard solution of hydrochloric acid using phenolphthalein solution as indicator.

A-13.4 Calculation

$$\text{Alkalinity (as } Na_2O\text{), percent by mass} = \frac{3.1\,AN}{M}$$

Where,

A = volume in ml of standard hydrochloric acid used;

N = normality of standard hydrochloric acid; and

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

A-14 Determination of Total Free Alkali or Acid

A-14.0 General

This method determines total free alkali or acid in crude glycerine expressed as Na_2O. Total free alkali is the sum of free caustic alkali and carbonated alkali as percent Na_2O.

A-14.1 Apparatus

A-14.1.1 Platinum Crucible — 25 mL capacity.

A-14.1.2 Desiccator — containing concentrated sulphuric acid.

A-14.1.3 Beakers — 400 mL capacity.

A-14.1.4 Conical Flask — 250 mL capacity.

A-14.1.5 Volumetric Flask — 100 mL capacity.

A-14.2 Reagents

A-14.2.1 Standard Hydrochloric Acid — 0.1 N, accurately standardized.

A-14.2.2 Barium Chloride Solution — 10 percent (m/v), aqueous.

A-14.2.3 Standard Sodium Hydroxide Solution — 0.1 N, accurately standardized.

A-14.2.4 Phenolphthalein Indicator Solution — 1 percent (m/v), in 95 percent ethyl alcohol (v/v) or rectified spirit conforming to BDS 1242.

A-14.2.5 Methyl Orange Indicator Solution — percent (m/v), aqueous.

A-14.3 Procedure

A-14.3.1 Total Alkali (Free and Combined) — Dissolve the ash obtained in **A-5.2** in about 50 mL of hot water and transfer to a 400 mL beaker. Cool and titrate with standard hydrochloric acid using methyl orange as indicator.

A-14.3.2 Total Free Alkali (Caustic and Carbonate) — Weigh accurately about 10 g of the sample in a 250 mL conical flask. Dilute with 50 mL of distilled water, add a known volume of standard hydrochloric acid sufficient to neutralize the total alkali and wash down the condenser tube and sides of the flask with carbon dioxide-free distilled water. Cool rapidly and titrate the excess acid with standard sodium hydroxide solution using phenolphthalein as indicator.

A-14.3.3 Free Caustic Alkali — Weigh accurately about 20 g of the sample in a 100 mL volumetric flask, dilute with 50 mL of carbon dioxide free distilled water and add an excess of barium chloride solution with constant shaking. Make up the volumeto 100 mL with water. Allow the precipitate to settle, pipette 50 mL of the supernatant liquid into a conical flask, and titrate with standard hydrochloric acid using phenolphthalein as indicator.

A-14.3.4 Free Acid — Weigh accurately about 10 g of the sample in a 250 mL conical flask, dilute with 50 mL of carbon dioxide-free distilled water and titrate with standard sodium hydroxide solution using phenolphthalein as indicator.

A-14.4 Calculation

a) Total alkali (free and combined), as Na_2O, percent by mass = $\frac{3.1\,AN_1}{M}$

b) Total free alkali (caustic and carbonate), as Na_2O, percent by mass = $\frac{3.1(AN_1 - BN_2)}{M}$

a) Free caustic alkali, as Na_2O, percent by mass = $\frac{6.2 \times AN_1}{M}$

b) Free acid, as Na_2O equivalent, percent by mass = $\frac{3.1 \times BN_2}{M}$

Where,

N_1 = normality of standard hydrochloric acid;

N_2 = normality of standard sodium hydroxide solution;

A = volume in mL of standard hydrochloric acid used;

B = volume in mL of standard sodium hydroxide solution used; and

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

A-15 Determination of Fatty Acids and Esters

A-15.0 General

Fatty acids and esters represent the free fatty acids and the fatty acid equivalent of all the esters present in the sample.

A-15.1 Apparatus

A-15.1.1 Round-Bottomed Flask — 500 mL capacity.

A-15.2 Reagents

A-15.2.1 Standard Sodium Hydroxide Solution — 0.25 N, accurately standardized.

A-15.2.2 Standard Sulphuric Acid — 0.25 N, accurately standardized.

A-15.2.3 Phenolphthalein Indicator Solution

1 percent (m/v), in 95 percent ethyl alcohol (v/v) or rectified spirit conforming to BDS 1242.

A-15.3 Procedure

Weigh accurately about 50 g of the sample into a 500 mL round-bottomed flask. Add 100 mL of hot, carbon dioxide-free water and 1 mL of phenolphthalein indicator solution. If the solution is alkaline, neutralize it with sulphuric acid. Add exactly 15.0 mL of standard sodium hydroxide solution, connect the flask to a reflux condenser and heat to boiling. Boil for 5 minutes, allow to cool slightly and wash down the condenser with a little water. Disconnect the flask, close it with a stopper carrying a sodalime tube, and cool.

Titrate with standard sulphuric acid. Carry out a blank test simultaneously under similar test conditions but using 140 mL of water.

A-15.4 Calculation

Fatty acids and esters, as Na_2O, precent by mass = $\frac{3.1(B - S)\,N}{M}$

Where,

B = volume in mL of standard sulphuric acid required for the blank;

S = volume in mL of standard sulphuric acid;

N = normality of standard sulphuric acid; and

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

A-16 Test for the Presence of Acraldehyde and Glucose

A-16.1 Procedure

Take about 5 mL of the sample in an evaporating dish and heat strongly. Heat further until it volatilizes and burns. The development of not more than a faint yellow, but not pink, colour in the preliminary heating, and the burning of the sample with little or no charring and with no odour of burnt sugar in the subsequent heating, indicates the absence of acraldehyde and glucose.

A-17 Test for the Presence of Reducing Substances

A-17.0 General

This test is also referred to as ammonical silver nitrate test.

A-17.1 Apparatus

A-17.1.1 Nessler Cylinders — 50 mL capacity, thoroughly cleaned with concentrated nitric acid and washed with water until acid-free.

A-17.2 Reagents

A-17.2.1 Ammonia Solution — 10 percent (m/v), aqueous.

A-17.2.2 Silver Nitrate Solution — 5 percent (m/v), aqueous, and 10 percent (m/v), aqueous.

A-17.2.3 Dextrose Solution — 0.1 percent, aqueous.

A-17.3 Procedure

A-17.3.1 For Analytical Reagent and Chemically Pure Grade

Take 5 mL of the sample in a Nessler cylinder, add 5 mL of ammonia solution and heat at 60°C for 5 minutes. Add rapidly from a pipette 0.5 mL of 5 percent silver nitrate solution, keeping the tip of the pipette above the mouth of the cylinder and allowing the reagent to fall directly into the solution without touching the sides of the cylinder. Mix thoroughly and keep in the dark for 5 minutes. Simultaneously, carry out a blank test under similar test conditions. Compare the turbidities and colour of the two solutions in normal daylight, viewing from the top of the cylinder, preferably against a white background. The development of turbidity or production of darkening in the test sample is not greater than that of the blank indicates the absence of reducing substances.

A-17.3.2 For Dynamite Grade — Take 10 ml of the sample in a 100 mL volumetric flask, and make up the volume with water. Prepare solution A by taking 10 mL of the diluted solution in a Nessler cylinder, and adding 10 mL of ammonia solution and 10 mL of 10 percent silver nitrate solution. Prepare solution B by diluting 0.5 ml of dextrose solution to 10 ml with water in a Nessler cylinder, and adding 10 ml of ammonia solution and 10 mL of 10 percent silver nitrate solution. Heat solutions A and B in a water bath maintained at 62 °C to 65 °C. When the solutions attain a temperature of 60 °C, remove them from the bath and keep in the dark for 10 minutes. Compare the turbidities of the two solutions in normal daylight, viewing them from the top of cylinders against a background of black glazed paper. The turbidity of the test sample (solution A) shall not exceed that produced in solution B.

NOTE — Residues should be acidified with nitric acid and disposed of as soon as the test is completed as explosive compounds may be formed.

A-18 Test for the Presence of Substances Darkened by Sulphuric Acid

A-18.1 Reagents

A-18.1.1 Sulphuric Acid — 96 percent (m/v).

A-18.1.2 Standard Colour Solution — Take 10 mL of solution (a), 1 mL of solution (b) and 1mL of solution (c) in a 250 mL volumetric flask and make up to volume with water:

a) Dissolve 200 g of copper sulphate ($CuSO_4.5H_2O$) in 1 litre of water;

b) Dissolve 80 g of potassium dichromate ($K_2Cr_2O_7$) in 1 litre of water; and

c) Dissolve 3.16 g of potassium permanganate ($KMnO_4$) in 1 litre of water.

A-18.2 Procedure

Take 2 mL of the sample and 2 mL of sulphuric acid in separate test tubes and cool in ice water for 15 minutes. Add the acid to the sample and mix by inclining the test tube and rolling it rapidly between the fingers. Replace the test tube immediately in ice water and allow to stand for 15 minutes. In a similar test tube, take 4 mL of the standard colour solution and compare visually the intensities of the colours produced. The development of colour in the test sample not greater than that of the standard indicates the absence of substances darkened by sulphuric acid.

A-19 Determination of Nitrogen

A-19.1 Apparatus

A-19.1.1 Kjeldahl Flask — 500 mL nominal capacity, fitted with a loose stopper.

A-19.1.2 Ammonia Distillation Apparatus — consisting of a 1 litre round- bottomed flask fitted with a dropping funnel, spray trap, Liebig condenser and a 100 mL graduated receiver.

A-19.1.3 Nessler Tubes — two, 100 mL capacity each.

A-19.2 Reagents

A-19.2.1 Concentrated Sulphuric Acid — r d. 1.84 conforming to Analytical Reagent Grade of IS 266.

A-19.2.2 Potassium Sulphate — not containing more than 15 parts per million of nitrogen.

A-19.2.3 Sucrose — not containing more than 15 parts per million of nitrogen.

A-19.2.4 Selenium Powder

A-19.2.5 Ammonia-Free Water — Redistil 500 mL of distilled water in a resistant glass apparatus from a solution containing one gram of potassium permanganate and one gram of anhydrous sodium carbonate. Reject the first and last thirds of the distillate and collect the middle third.

A-19.2.6 Phenolphthalein Solution

A-19.2.7 Sodium Hydroxide Solution — 50 percent (m/v).

A-19.2.8 Standard Nitrogen Solution — containing 0.038 2 g of ammonium chloride (NH_4Cl) per litre, equivalent to 0.01 mg of nitrogen per millilitre.

A-19.2.9 Nessler Solution

Dissolve 35 g potassium iodide in 100 mL of water and add 5 percent mercuric chloride solution with stirring and shaking until a slight red precipitate persists (about 325 mL are required). Then introduce with stirring, a solution of 120 g sodium hydroxide in 250 ml and make up to 1 litre with water. Add 4 percent mercuric chloride solution drop by drop until there is a permanent turbidity. Keep overnight in the dark. Decant from sediment and store in a dark-coloured bottle.

A-19.3 Procedure

A-19.3.1 Transfer a known mass (about 2 g) of the sample to a 500 mL Kjeldahl flask fitted with a loose stopper. Add 10 g of potassium sulphate, 2 g of sucrose and 0.05 g of selenium powder and then add 30 mL of concentrated sulphuric acid which washes down the neck of the flask. Mix thoroughly by shaking.

A-19.3.2 Inside a fume cupboard with good draught, support the flask on a hole in a piece of asbestos board and incline the neck at an angle of 60°. Close the flask with the stopper and heat in a small flame till frothing ceases. Increase the flame and heat till the mixture boils gently.

Continue the heating for about half an hour till the mixture becomes clear and colourless or at the most of pale-straw colour. Allow the solution to cool. When cold, add 200 mL ammonia-free distilled water.

A-19.3.3 Steam out an ammonia distillation apparatus until free from ammonia. Run the solution to be distilled in the flask through the dropping funnel. Add a few drops of phenolphthalein. Neutralize with sodium hydroxide solution and add 10 mL in excess. Note the volume of the sodium hydroxide added for blank determination. Make up the volume in the flask to 400 mL by addition of ammonia-free water. Distil vigorously till 100 mL of distillate is collected in 30 minutes to 40 minutes in a flask previously rinsed with ammonia-free water.

A-19.3.4 Carry out a blank determination with all the reagents except the sample.

A-19.3.5 Determine the ammonia content of each ofthe above distillates separately as follows:

A-19.3.5.1 Transfer half of the distillate (50 mL) to one of the two matched Messier tubes (A). In the other Nessler tube (B), add 2 mL of standard nitrogen solution and then dilute to 50 mL. To each of these, add 2 mL of Nessler solution. Add further standard nitrogen solution to the solution in cylinder B till the colour produced matches that in the cylinder A. Record the volume of standard solution added from the burette. Repeat the determination with other half of the solution. Both the readings should agree. Calculate the mean.

A-19.3.5.2 Repeat the determination in duplicate with the distillate from the blank. Calculate the mean.

NOTE — When a solution containing ammonia is added to Nessler solution which has been diluted to 50 mL with ammonia-free water, an opalescence arises in the mixture which is sufficient to prevent accurate colour comparison. If the procedure given in the method is adhered to, no opalescence is produced. The stated order of adding the reagents shall be adhered to, if opalescence is to be avoided.

A-19.4 Calculation

$$\text{Nitrogen, percent by mass} = \frac{0.0002\,(a-b)}{M}$$

Where,

a = mean volume in mL of standard nitrogen solution required to match the sample;

b = mean volume in mL of standard nitrogen solution required to match the blank; and

M = mass in g of the sample taken.

A-20 Determination of Ether Extract

A-20.1 Apparatus

A-20.1.1 Separating Funnel — 250 mL capacity.

A-20.2 Reagents

A-20.2.1 Ether — solvent grade.

A-20.3 Procedure

Dilute 50 mL of sample with 50 mL of water in a separating funnel and add 50 mL of ether. Shake and separate the ether layer. Wash the ether extract six times with 10 mL of water each time. Transfer the ether extract to a tared 100 mL fat-extraction flask, distil off ether, dry the residue at (70 ± 1) °C for 1 hour. Cool in a desiccator and weigh.

A-20.4 Calculation

Ether extract, percent (m/v) = 2 M

Where,

M = mass in g of the ether extract.

A-21 Determination of Volatile Fatty Acids

A-21.0 General

Volatile fatty acids are expressed in terms of the volume in millilitres of 0.1 N potassium hydroxide solution required to neutralize the volatile fatty acids distilled from 100 g of glycerine when tested by the method described in **A-21.3.**

A-21.1 Apparatus

The assembly of the apparatus for distillation is shown in Fig. 6, the constituent parts being as follows.

A-21.1.1 Flat-Bottomed Boiling Flask, A — approximately 300 mL capacity.

A-21.1.2 Still-Head or Connecting Piece, B

A-21.1.3 Condenser, C

A-21.1.4 Receiver, D — a flask with two graduation marks on the neck, one at 100 mL and the other at 110 mL.

A-21.1.5 Asbestos Board, E — with a hole about 65 mm in diameter.

A-21.1.6 Bunsen Burner, F

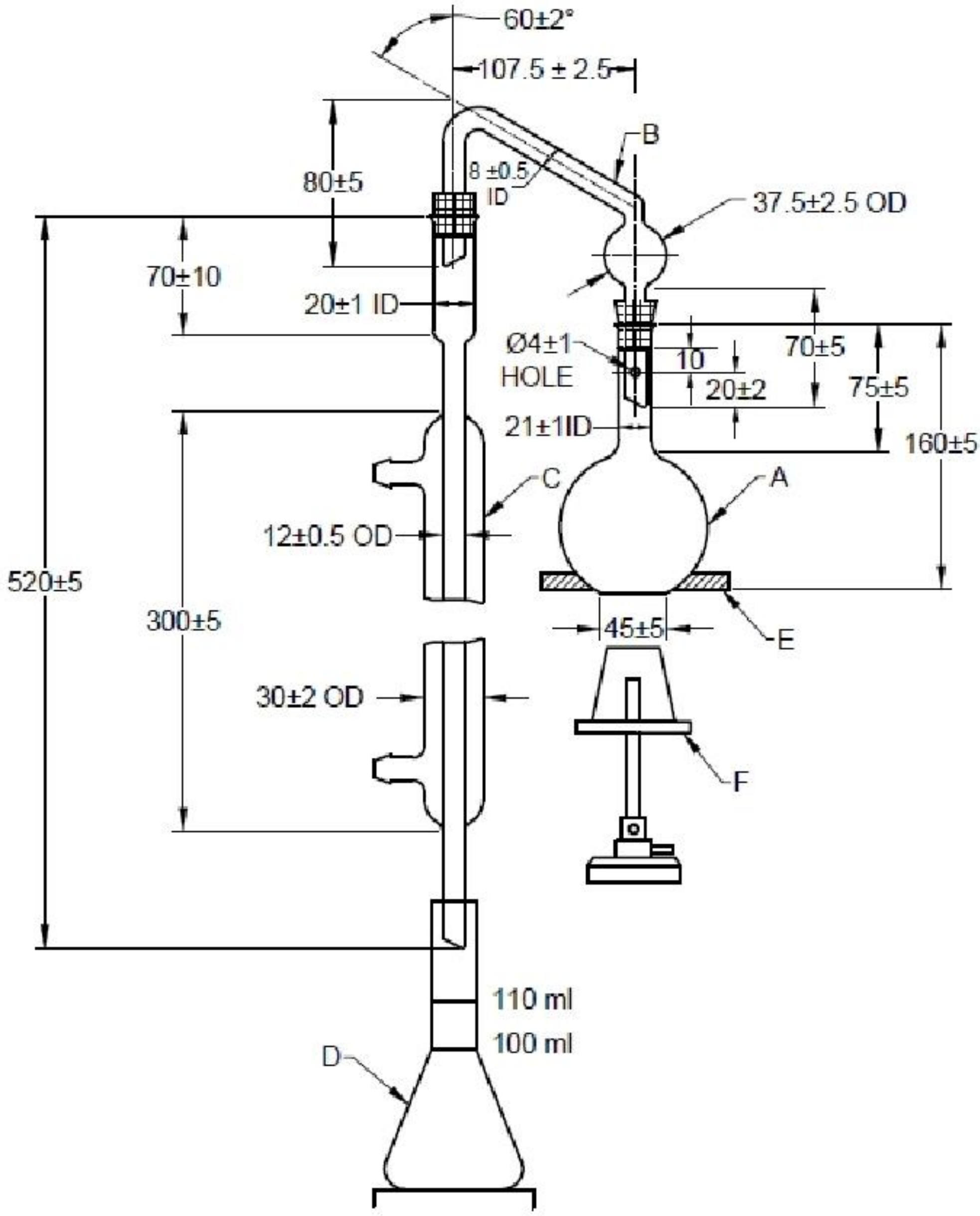

Fig. 6 Apparatus for the Determination of Volatile Fatty Acids

A-21.2 Reagents

A-21.3 Procedure

A-21.3.1 Weigh accurately about 25 g of the material into the boiling flask A. Add 90 mL of water and 2 mL of sodium hydroxide solution. Boil the mixture for 2 minutes. Cover the flask with a watch-glass and allow the flask to cool a little. Add 40 mL of boiling water which has been vigorously boiled for about 15 minutes.

A-21.3.2 Add about 0.5 g of pumice stone grains and 30 ml of sulphuric acid, and immediately connect the flask with the distilling apparatus. Heat the flask and collect the distillate in flask D. The temperature of the cooling water in the condenser should be between 15 °C and 20 °C. Adjust the flame so that 110 mL distillation is over in the course of 30 minutes. Titrate the distillate with standard potassium hydroxide solution, using phenolphthalein as indicator.

A-21.4 Calculation

$$\text{Volatile fatty acids} = \frac{1000\ VN}{M}$$

Where,

V = volume in mL of standard potassium hydroxide solution required;

N = normality of standard potassium hydroxide solution; and

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

NOTE — In some methods, 100 mL glycerine is used as the basis for the test. The method described above is based on gravimetric considerations because, as glycerine is viscous, pipetting out exact volumes of the material is virtually impossible. Also, volumetric measurements have to be made with reference to a specified temperature and density, and this is difficult to adhere to.