IS 229 (1993): Ethyl acetate [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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IS 229 : 1993
(Reaffirmed 2004)

भारतीय मानक

इथाइल एसीटेट — विशिष्टि

(तीसरा पुनरीक्षण)

Indian Standard

ETHYL ACETATE — SPECIFICATION

(Third Revision)

UDC 661·731·7

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

March 1993

Price Group 4

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, coal and Related Products Division Council.

This standard was first published in 1957 and subsequently revised in 1964 and 1972. The revised version of 1964 covered two grades of product, namely, Grade 1 (99 percent purity) and Grade 2 (90 percent purity).

During the second revision, it was noted that Grade 2 material (90 percent purity) has very little application in the trade, it was, therefore, felt necessary to upgrade the existing grade 2 product to 94 percent purity. It was also felt that in the light of the latest development, Grade 1 also needed revision and updating. In view of this, the requirements for distillation range for both the grades were modified as well as limit for water content was introduced in the standard in 1972.

The present revision updates the standard with respect to quality of the material available in the market. The requirement for distillation range has been modified in consultation with the users as well as manufacturers of this product.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 '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

ETHYL ACETATE — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and testing for ethyl acetate. The material is used mainly as a solvent by the paint and lacquer industry, and also for the manufacture of drugs, cosmetics and flavouring essences.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
323 : 1959	Specification for rectified spirit (*revised*),
1070 : 1992	Reagent grade water — Specification (*third revision*),
2362 : 1973	Determination of water by the Karl Fischer method (*first revision*), and
4905 : 1968	Methods for random sampling.

3 GRADES

The material shall be of the following grades:

Grade 1 — It is generally used in the manufacture of drugs, cosmetics and flavouring essences; and

Grade 2 — It is generally used as an industrial solvent mainly in the paint and lacquer industry.

4 REQUIREMENTS

4.1 Description

The material shall be clear, colourless liquid with characteristic odour and free from visible impurities. It shall consist, essentially of the ethyl ester of acetic acid, $CH_3COOC_2H_5$.

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

5 PACKING AND MARKING

5.1 Packing

5.1.1 Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in tightly closed containers made of galvanized iron or of any other suitable material, subject to the provisions of law in force in the country.

5.1.2 All containers in which the material is stored shall be clean, dry and leak-proof. It shall be kept in cool place.

Table 1 Requirements for Ethyl Acetate

(*Clause* 4.2)

Sl No.	Characteristic	Requirements for		Method of Test, Ref to
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Relative density at 27°/27°C	0·894 to 0·898	0·874 to 0·894	Annex A
ii)	Residue on evaporation, percent by mass, *Max*	0·01	0·02	Annex B
iii)	Acidity as acetic acid, percent by mass, *Max*	0·01	0·01	Annex C
iv)	Ester content as ethyl acetate, percent by mass, *Max*	99	94	Annex D
v)	Distillation range	The difference between initial boiling point (IBP) and dry point (DP) shall not exceed 2·5°C including 77·2°C (temperature being corrected for a pressure of 760 mm Hg)	The difference between initial boiling point (IBP) and dry point (DP) shall not exceed 11°C including 77·2°C (temperature being corrected for a pressure of 760 mm Hg)	Annex E
vi)	Water content, percent by mass, *Max*	0·1	0·5	IS 2362 : 1973

5.2 Marking

5.2.1 Each container shall be marked with the following:

a) Name and grade of the material;
b) Indication of the source of manufacture;
c) Gross and net mass;
d) Month and year of manufacture; and
e) Batch or code number.

5.2.2 The containers may also be marked with the Standard Mark.

6 SAMPLING

6.1 Preparation of Test Samples

Representative samples of the material shall be drawn as prescribed in Annex F.

6.2 Number of Tests

Tests for the determination of relative density and ester content shall be conducted on individual samples. Tests for the remaining characteristics shall be carried out on the composite sample.

6.3 Criteria for Conformity

The lot shall be declared as conforming to the requirements of the specification, if all the test results on each of the individual samples and the composite sample meet the relevant specification requirements.

7 TEST SAMPLES

7.1 Tests shall be carried out by the methods specified in col 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, ' pure chemicals ' and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

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

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF RELATIVE DENSITY

A-1 APPARATUS

A-1.1 Relative Density Bottle

50 ml capacity, of Regnault type.

A-2 PROCEDURE

A-2.1 Clean, dry and weigh accurately the relative density bottle and the stopper. Fill the bottle with water and immerse it up to the neck in a constant temperature bath at 27·0 ± 0·5°C and keep immersed at this temperature for 20 minutes. Suck off the water with a bit of filter paper till the level reaches the graduation mark and weigh again. Empty the bottle, clean and dry. Repeat the operation with the material at 27°C.

A-3 CALCULATION

$$\text{Relative density at } 27°/27°\text{C} = \frac{A - B}{C - B}$$

where

A = mass in g of the relative density bottle with the material at 27°C;
B = mass in g of the relative density bottle; and
C = mass in g of the relative density bottle with water at 27°C.

ANNEX B

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF RESIDUE ON EVAPORATION

B-1 PROCEDURE

B-1.1 Evaporate 100 ml of the material to dryness in a weighed platinum or silica or borosilicate glass basin on a boiling water-bath. Dry the residue in an oven at a temperature of 100 ± 2°C to constant mass. Cool in a desiccator and weigh.

B-2 CALCULATION

$$\text{Residue on evaporation, percent by mass} = \frac{M}{d}$$

where

M = mass in g of the residue, and
d = relative density of the sample.

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF ACIDITY

C-1 REAGENTS

C-1.1 Rectified Spirit — 95 percent (*see* IS 323 : 1959).

C-1.2 Standard Sodium Hydroxide Solution — 0·1 N.

C-1.3 Phenolphthalein Indicator

Dissolve 0·5 g of the phenolpthalein in 100 ml of 95 percent rectified spirit. Make the solution faintly pink by adding dilute sodium hydroxide solution.

C-2 PROCEDURE

C-2.1 Take 50 ml of rectified spirit, add 0·5 ml of phenolphthalein indicator and neutralize with sodium hydroxide solution. Add 50 ml of the sample. Titrate the mixture immediately with the standard sodium hydroxide solution until the first pink colour persists for at least 10 seconds.

C-3 CALCULATION

$$\text{Acidity as acetic acid, percent by mass} = \frac{6\,V\,N}{50\,d}$$

where

V = volume in ml of standard sodium hydroxide solution,

N = normality of standard sodium hydroxide solution, and

d = relative density of ethyl acetate at the test temperature.

ANNEX D

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF ESTER CONTENT

D-1 REAGENTS

D-1.1 Standard Alcoholic Sodium Hydroxide Solution — 0·5 N.

D-1.2 Standard Hydrochloric Acid — 0·5 N.

D-1.3 Phenolphthalein Indicator — same as in **C-1.3.**

D-2 PROCEDURE

D-2.1 Weigh accurately 1·0 to 1·5 g of the material in a small tared tube and transfer the tube with its contents to a flask-containing 50 ml of standard alcoholic sodium hydroxide solution. Fit a water-cooled-reflux condenser to the flask and heat it for one hour over a boiling water-bath. Withdraw the flask, still carrying its condenser, and immerse it in cold water. When cool, rinse the inside of the condenser with distilled water. Disconnect the flask and wash the ground glass joint with distilled water. Add 0·5 ml of phenolphthalein indicator and titrate the mixture immediately with the standard hydrochloric acid solution until the pink colour is just discharged.

D-2.2 Carry out a blank determination, using all the reagents excepting the material, under similar conditions and at the same time.

D-3 CALCULATION

$$\text{Ester content as ethyl acetate, percent by mass} = \frac{8{\cdot}81\,(V_2 - V_1)\,N}{M}$$

where

V_2 = volume in ml of standard hydrochloric acid required in the blank determination,

V_1 = volume in ml of standard hydrochloric acid required in the test with the material,

N = normality of standard hydrochloric acid, and

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

ANNEX E

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

DETERMINATION OF DISTILLATION RANGE

E-1 APPARATUS

E-1.1 Distillation Flask

Of shape and dimensions given in Fig. 1. Fix the flask in vertical position by means of a clamp at the extreme upper end of the neck.

E-1.2 Thermometer*

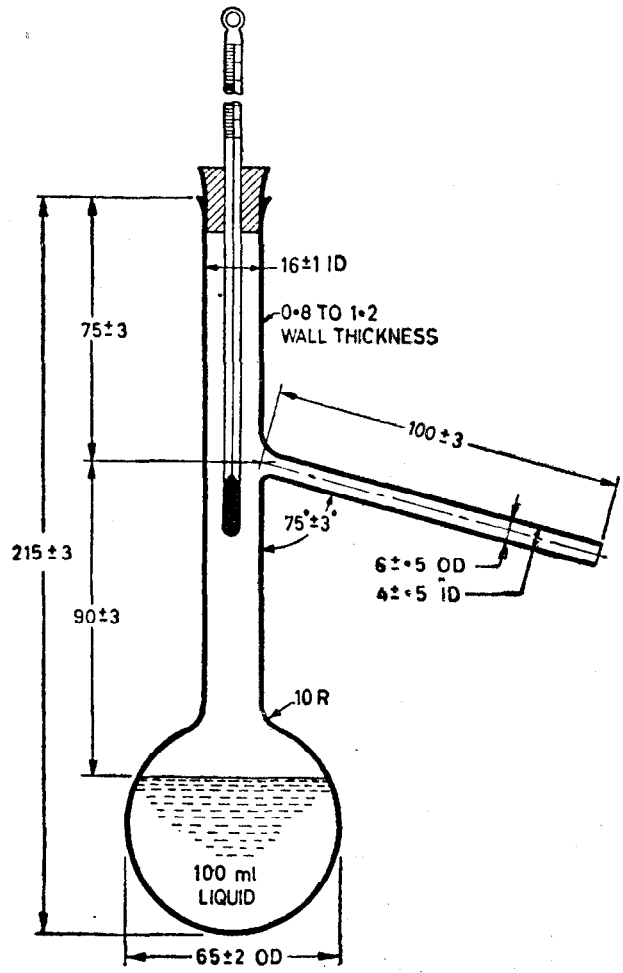
Conforming to the following requirements is recommended:

Range	30 to 200°C
Graduation	0·5°C
Immersion	Total
Overall length	390 mm
Stem diameter	5·5 to 8·0 mm
Bulb shape	Cylindrical
Bulb length	9 to 14 mm
Bulb diameter	4·5 to 5·5 mm
Length of graduated portion	245 to 280 mm
Distance from bottom of bulb to bottom of main scale	75 to 90 mm
Longer lines at each	1°C
Figured at each	5°C
Expansion chamber	To allow heating to 250°C
Top finish	Ring
Scale error not to exceed	± 0·3°C

E-1.2.1 The thermometer shall bear a certificate from the National Physical Laboratory, New Delhi, or any other institution authorized by the Government of India to issue such a certificate.

E-1.3 Draught Screen

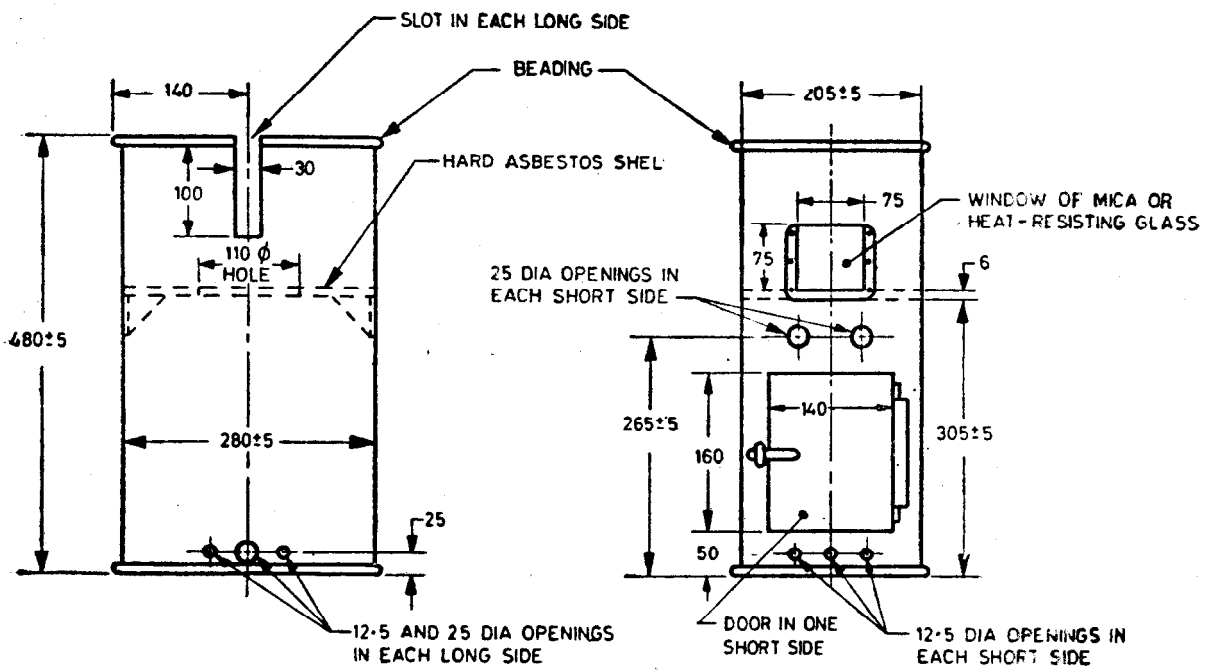
The construction and dimensions of a draught screen shall be as shown in Fig. 2. A shelf of a heat-resistant hard asbestos compound, 6 mm in thickness with a central circular hole of 110 mm in diameter, the edge of which is levelled to fit the contours of the distillation flask, shall be supported horizontally in the screen and shall fit closely to the sides of the screen to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask. The supports for this asbestos shelf may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners. In each of the longer sides of the screen there shall be one large and two small holes at the base, and a vertical slot at the top. A removable shutter conforming to the dimensions given in Fig. 3 shall be provided for closing whichever vertical slot is not in use. In each of the shorter sides of the screen there shall be two circular holes below the asbestos shelf, three circular holes at the base, and a central window of mica or heat-resisting glass, the bottom of which shall be levelled with the top of the asbestos shelf. In one of the shorter sides there shall also be a door, overlapping an opening the screen by approximately 5 mm, all round.



All dimensions in millimetres.

FIG. 1 DISTILLATION FLASK
(CAPACITY 130 ± 5 ml)

*Thermometers with the Institute of Petroleum designation IP 61C conform to these requirements. Any other thermometer of similar range and accuracy may also be used.



All dimensions in millimetres.

FIG. 2 DRAUGHT SCREEN

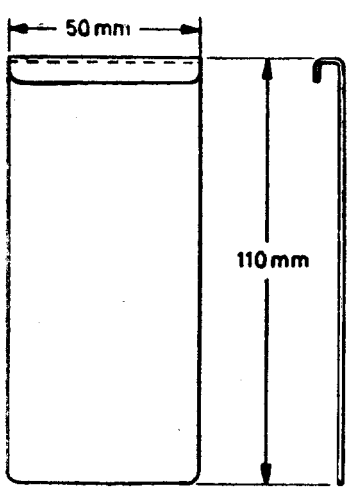


FIG. 3 REMOVABLE SHUTTER FOR DRAUGHT SCREEN

E-1.4 Liebig Condenser

With the bent and made of good quality resistant glass having a wall thickness of 1 to 1·5 mm and conforming to the shape and dimensions given in Fig. 4. Alternatively, the bent portion may be substituted by an adapter fitted externally in such a manner that the distillate does not come in contact with the cork.

E-2 PROCEDURE

Assemble the apparatus as shown in Fig. 5. Measure 100 ml of the sample into the distillation flask from a graduated measuring cylinder and add a few anti-bumping granules. Place the flask, thermometer and a suitable receiver in position and ensure that the condenser has a steady supply of water. Adjust the rate of heating so that the first drop of distillate falls from the end of the condenser in 7 to 12 minutes. Read the temperature at the instant the first drop falls from the end of the condenser and record as the observed initial boiling point.

Further adjust the rate of heating so that the distillate is collected at the rate of 3 to 4 ml per minute. Read the temperature indicated at the instant the last drop of liquid evaporates from the lowest point in the distillation flask and record as the observed dry point. Disregard any liquid on the side of the flask.

E-3 CORRECTIONS

E-3.1 Error of Thermometer Scale

In all thermometer readings make the corrections as indicated on the certificate of the instrument.

E-3.2 Correction for Barometric Pressure

When the corrected barometer pressure deviates from 760 mm Hg, apply further corrections to the observed temperature by subtracting 0·041°C for every millimeter above 760 mm or adding 0·041°C for every millimetre below 760 mm.

NOTE — These corrections are valid only for pressure above 760 mm Hg.

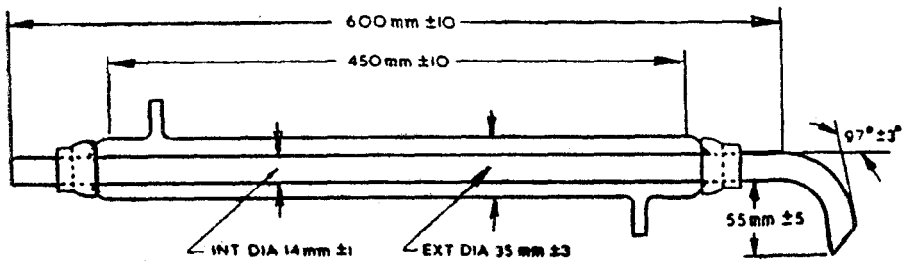


FIG. 4 LIEBIG CONDENSER

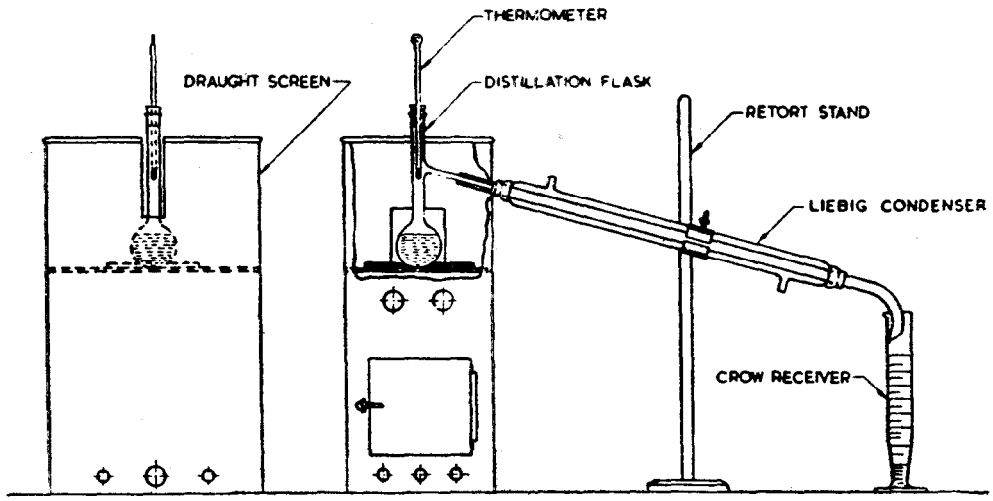


FIG. 5 ASSEMBLY OF APPARATUS

ANNEX F

(*Clause* 6.1)

SAMPLING OF ETHYL ACETATE

F-1 GENERAL REQUIREMENTS OF SAMPLING

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

F-1.1 Samples shall be taken in a protected area with good ventilation.

F-1.2 Sampling instruments shall be clean and dry.

F-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

F-1.4 To draw a representative sample the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking, stirring, rolling or by any other suitable means.

F-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass or metal containers on which the material has no action.

F-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

F-1.7 Each sample container shall be sealed air-tight with a suitable stopper after filling and marked with the information indicating the source of manufacture, grade of the material, the month and year of manufacture of the material, the batch number (if available) and other details of sampling, such as the date of sampling, sampler's name, etc.

F-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

F-2 SAMPLING INSTRUMENT

F-2.1 It is made of thick glass or metal on which the material has no action and is 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 6).

The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For drawing samples, the apparatus is first closed at the top with thumb or a stopper and lowered till a desired depth is reached. It is then opened for a short time to admit the material at the desired depth and finally closed and withdrawn.

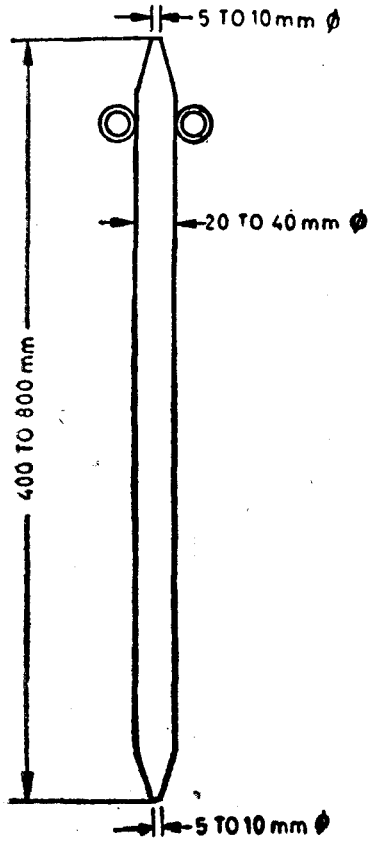


FIG. 6 SAMPLING TUBE

F-2.1.1 For small containers, the size of the sampling tube may be altered suitably.

F-3 SCALE OF SAMPLING

F-3.1 Lot

In any consignment, all the containers of the same grade, size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different grades or of different batches of manufacture or of different sizes of containers, the containers belonging to the same grade, batch and size shall be grouped together and each such group shall constitute a separate lot.

F-3.2 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose from a lot shall depend on the size of the lot and shall be in accordance with Table 2.

Table 2 Scale of Sampling

Lot Size	No. of Containers to be Selected
(1)	(2)
Up to 15	3
16 ,, 50	4
51 ,, 100	5
101 ,, 150	6
151 ,, 300	8
301 and above	10

F-3.3 These containers shall be selected at random from the lot. In order to ensure randomness of selection, procedures given in IS 4905 : 1968 may be followed.

F-4 TEST SAMPLES AND REFEREE SAMPLES

F-4.1 From each of the containers selected according to **F-3.3** a representative portion of ethyl acetate approximately 800 ml in volume (*see* Note) shall be drawn with the help of sampling data. Out of these portions equal quantity of material shall be taken and mixed thoroughly to form a composite sample not less than 1 500 ml in volume. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample. These composite samples shall be transferred to containers of 600 ml capacity and shall be sealed and marked with full identification particulars given in **F-1.7**.

NOTE — This amount may be taken out in one or more operations of sampling tube by filling it partially or completely as the occasion demands.

F-4.2 The remaining portions representing each of the individual containers shall be transferred to separate sample containers and these are termed individual samples. These sample containers, after filling, shall be sealed and marked with full identification particulars.

F-4.3 The referee sample prepared under F-4.1 shall bear the seal of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier and is to be used in the case of any dispute between the two.

Standard Mark

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act, 1986* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards Monthly Additions'. Comments on this Indian Standard may be sent to BIS giving the following reference:

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