

IS 323 : 2009

(Reaffirmed 2014)

(Reaffirmed 2019)

भारतीय मानक
औद्योगिक प्रयोग के लिए रेकटीफाइड स्प्रिट — विशिष्टि
(दूसरा पुनरीक्षण)

Indian Standard

RECTIFIED SPIRIT FOR INDUSTRIAL USE —
SPECIFICATION

(*Second Revision*)

ICS 71.100.80

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Price Group 6

Organic Chemicals, Alcohols and Allied Products and Dye Intermediate Sectional Committee, PCD 9

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals, Alcohols and Allied Products and Dye Intermediate Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1952. At that time, the minimum ethanol content of rectified spirit had been left to the option of the purchaser and the vendor subject to a minimum of 91.27 percent by volume [60° Overproof (OP)].

The first revision of this standard was carried out in 1959 to upgrade ethanol content and also to modify acidity, residue on evaporation, besides, potassium ferro-cyanide method was prescribed as an alternate method for the determination of copper. This standard covered two grades, Grade 1 suitable for use as a reagent for pharmaceutical/medicinal purposes and for the production of alcoholic beverages and Grade 2 was intended for other industrial purposes. Besides, additional requirement to Grade 1 were laid down to cater to the needs of the acetone industry. The computation of strength of aqueous solutions containing ethanol was made from specific gravity figures at 15.6°C (60°F) and the strength was expressed in terms of percent by volume or of proof spirit in line with prevailing practices in the country.

In this standard, grade relevant for pharmaceutical, medicinal and production of alcoholic beverages has been deleted. A separate standard for potable applications is under formulation. The rectified spirit intended for industrial purpose only is being retained in this revision. The Committee responsible for the formulation of this standard, however, felt that it would be advisable to retain the proof spirit method of expressing strength of alcoholic solutions and its co-relation with specific gravity and percentage of alcohol by volume as well as weight at 20°C as routine method for ease of testing. In this revision, GC method has also been included for determination of ethanol, methyl alcohol, *n*-propanol and *iso*-amyl alcohol. Instrumental methods have been included for determination of specific gravity as an alternative to specific gravity bottle method in this standard.

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
**RECTIFIED SPIRIT FOR INDUSTRIAL USE —
 SPECIFICATION**
(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for rectified spirit. The material is intended for industrial applications only.

2 REFERENCES

The following standards contain provisions, which through reference in this text constitute provisions of the standards. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the latest edition of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
266 : 1993	Sulphuric acid (<i>third revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
2302 : 1989	Tables for alcoholometry by hydrometer method (<i>first revision</i>)
2362 : 1993	Determination of water by Karl Fischer method — Test method (<i>second revision</i>)
6749 : 1972	Glossary of terms relating to alcohol (ethyl) industry and trade
15464 : 2004	Anhydrous ethanol for use in automotive fuel — Specification

3 TERMINOLOGY

For the purpose of this standard, the following definitions shall apply. Besides, IS 6749 may also be referred for other definitions.

3.1 Ethyl Alcohol — Ethyl alcohol (CAS No. 64-17-5) also referred to as ethanol, alcohol or grain alcohol is a flammable colourless liquid with a characteristic odour having a molecular formula as C_2H_5OH .

3.2 Rectified Spirit — Spirit purified by distillation to achieve strength of not less than 95 percent by volume of ethyl alcohol is a mixture of ethyl alcohol and water.

3.3 Proof Spirit — A measure of the absolute alcohol content of a mixture of ethanol and water, which shall, at a temperature of 10.6°C (or 51°F), weigh exactly 12/13th part of an equal volume of distilled water at the same temperature. It has a density of 0.915 5 at 20°C and contains 49.28 percent by weight of alcohol or 57.10 percent of alcohol by volume at 20°C.

3.4 Overproof Spirit (OP) — Mixture of ethanol and water containing a greater percentage of ethanol than is contained in proof spirit, especially containing more than 50 percent alcohol by volume. If an overproof strength is added to 100, the sum represents the volumes of spirit at proof strength which 100 volumes of spirit of that particular overproof strength would yield when diluted with water. For example, 100 volume of 60° OP spirit would yield 160 volumes of proof spirit.

4 REQUIREMENTS

4.1 Description

The material shall be a clear, colourless, homogenous liquid free from suspended matter and consist essentially of ethyl alcohol admixed with water.

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

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be packed in such containers as agreed to between the purchaser and the vendor under provisions of law in force from time-to-time.

5.1.2 All containers, in which the material is packed, shall be dry, clean, and leak-free.

5.1.3 Necessary safeguards against the risk arising from the storage and handling of large volume of flammable liquids shall be provided, and all due precautions shall be taken at all times to prevent accident by fire or explosion.

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**Table 1 Requirements for Rectified Spirit for Industrial Use
(Clause 4.2)**

Sl No.	Characteristics	Requirement	Method of Test, Ref to	
			IS No.	Annex
(1)	(2)	(3)	(4)	(5)
i)	Specific gravity at 20°C, <i>Max</i>	0.812 6	IS 15464	A
ii)	Ethanol content:			
	a) Percent by volume at 20°C, <i>Min</i>	95.0	IS 2302	B
	b) Degree overproof, <i>Min</i>	66		
iii)	Miscibility with water	Miscible	—	C
iv)	Alkalinity	Nil	—	D
v)	Total acidity (as CH ₃ COOH), mg/l, <i>Max</i>	100	—	D
vi)	Residue on evaporation percent by mass, <i>Max</i>	0.01	—	E
vii)	Aldehyde content (as CH ₃ CHO), mg/l, <i>Max</i>	100	—	F
viii)	Ester content (as CH ₃ COOC ₂ H ₅), mg/l, <i>Max</i>	200	—	G
ix)	Methyl alcohol, mg/l, <i>Max</i>	100	—	H
x)	<i>n</i> -Propanol, mg/l, <i>Max</i>	1 000	—	B
xi)	<i>Iso</i> -amyl alcohol mg/l, <i>Max</i>	300	—	B

5.1.4 Except when they are opened for the purpose of cleaning and rendering them free from alcohol vapour, all empty tanks or other containers shall be kept securely closed unless they have been thoroughly cleansed and freed from alcohol vapour.

5.2 Marking

All containers in which the material is stored or transported shall be marked as prescribed by law in force from time-to-time. The labels shall be legible and indelible with the following information:

- a) Name of the material;
- b) Manufacturer's name;
- c) Net gross and tare weight;
- d) Recognized trade-mark, if any;
- e) Date of packing;
- f) Highly flammable; and
- g) Hazardous chemical and injurious to health.

5.2.1 BIS Certification Marking

The container may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

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

7 TEST METHODS

7.1 Tests shall be conducted as prescribed in col 4 and 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) 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 SPECIFIC GRAVITY

A-1 DEFINITION

For the purpose of this standard, the specific gravity of a material shall mean the ratio of the weight of a given volume of the material at a specified temperature to the weight of an equal volume of distilled water at the same temperature.

Fill the specific gravity bottle with the material to over-flowing ensuring no entrapment of air bubbles. Insert the stopper, immerse the bottle in the water bath maintained at $20 \pm 0.2^\circ\text{C}$ and hold for 30 min. Remove the specific gravity bottle from the bath and clean and dry it thoroughly and weigh (A). Repeat the experiment with distilled water (C)

A-2 METHOD 1 — SPECIFIC GRAVITY BOTTLE METHOD

A-2.1.3 Calculation

A-2.1 The specific gravity may be determined by means of a pycnometer or a specific gravity bottle, or a special hydrometer. Specific gravity bottle method is referee method.

$$\text{Specific gravity, at } 20^\circ\text{C} = \frac{A - B}{C - B}$$

where

- A = weight of the specific gravity bottle with the material at 20°C , in g;
- B = weight of empty specific gravity bottle, in g; and
- C = weight of the specific gravity bottle with water at 20°C , in g.

A-2.1.1 Apparatus

For determination of specific gravity, two alternate specific gravity bottles shown in Fig.1A and Fig.1B are prescribed. In case of a dispute, the vacuum jacketed specific gravity bottles shown in Fig.1A shall be used.

A-3 METHOD 2 — OSCILLATING SAMPLE TUBE METHOD

A-2.1.2 Procedure

Clean the specific gravity bottle, its stopper and dry it in an oven. Cool the bottle, its stopper under a desiccator to 20°C and record the initial weight (B)

Determine the specific gravity of rectified spirit using Method 2 (see A-3 of IS 15464 or any other instrumental method such as PAAR Density Meter, etc, provided the method has been validated).

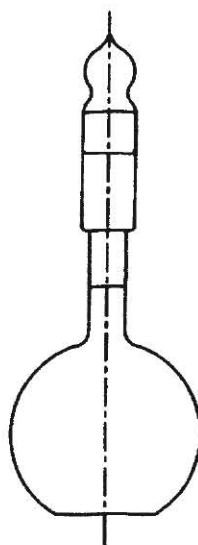
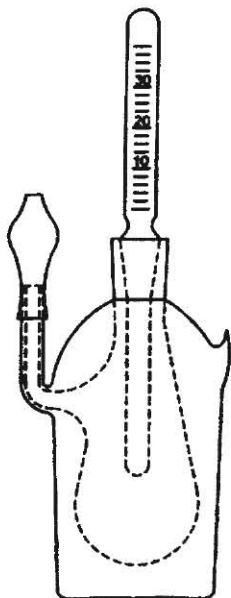


FIG. 1A VACUUM JACKETED SPECIFIC GRAVITY BOTTLE

FIG. 1B REGNAULT'S SPECIFIC GRAVITY BOTTLE

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ANNEX B

[Table 1, Sl No. (ii), (x) and (xi)]

DETERMINATION OF ETHANOL CONTENT

B-1 GENERAL

Two methods have been prescribed for determination of ethanol content in rectified spirit. Both the methods can be used for determination of ethanol content on routine basis. However, in the event of any dispute, Method 2 should be treated as a referee method.

B-2 METHOD 1 — ALCOHOLOMETRIC METHOD USING HYDROMETER

Determine the ethanol content of anhydrous ethanol using method given in IS 2302.

B-3 METHOD 2 — GAS CHROMATOGRAPHIC METHOD

B-3.1 General

This test method covers the determination of the ethanol content and other components such as aldehyde, ester (as ethyl acetate), methyl alcohol, *n*-propanol and *iso*-amyl alcohol of denatured rectified spirit by gas chromatography.

B-3.1.1 The chromatographic analysis given here is for information and guidance only.

B-3.1.2 Water cannot be determined by this test method and shall be measured by a procedure as given in IS 2362 and the result used to correct the chromatographic values.

B-3.1.3 This test method is inappropriate for impurities that boil at temperatures higher than 225°C or for impurities that cause poor or no response in a flame ionization detector, such as water.

B-3.2 Summary of Test Method

B-3.2.1 A representative aliquot of the ethanol sample is introduced into a gas chromatograph equipped with a methyl silicone bonded phase fused silica capillary column. Suitable carrier gas transports the vapourized aliquot through the column where the components are separated by the chromatographic process. Components are sensed by a flame ionization detector as they elute from the column. The detector signal is processed by an electronic data acquisition system. The ethanol, methanol and other components are identified by comparing their retention, times to the ones identified by analyzing standards under identical conditions. The

concentration of all components are determined in mass percent area by normalization of the peak areas.

B-3.3 Apparatus

B-3.3.1 Gas Chromatograph, capable of operating at the conditions listed below. A heated flash vaporizing injector designed to provide a linear sample split injection (for example, 2:1) is required for proper sample introduction. Carrier gas controls shall be of adequate precision to provide reproducible column flows and split ratios in order to maintain analytical integrity. Pressure control devices and gauges shall be designed to attain the linear velocity required in the column used. A hydrogen flame ionization detector with associated gas controls and electronics, designed for optimum response with open tubular columns, is required.

B-3.3.2 Sample Introduction — Manual or automatic liquid syringe sample injection to the splitting injector is employed. Devices capable of 0.1 to 0.5 µl injections are suitable. It should be noted that inadequate splitter design, poor injection technique, and overloading the column can result in poor resolution. Avoid overloading, particularly of the ethanol peak and eliminate this condition during analysis.

B-3.3.3 Column — This test method utilizes a fused silica open tubular column with non-polar methyl silicone bonded (cross-linked) phase internal coating. Any column with equivalent or better chromatographic efficiency and selectivity to those described in B-3.3.3.1 can be used.

B-3.3.3.1 Column temperature programme

Column	Capillary column coated with 6 percent cyanopropylphenyl, 94 percent dimethylpoly-siloxane
Column length	: 30 m
Internal diameter	: 0.53 mm
Film thickness	: 3 µm
Initial temperature	: 40°C
Initial hold time	: 5 min
Programme rate	: 30°C/min
Final temperature	: 230°C
Final hold time	: 2 min
Injector	
Temperature	: 140°C
Split ratio	: 2:1
Sample size	: 0.1 to 0.5 µl

Detector
 Type : Flame ionization
 Temperature : 240°C
 Carrier gas : Nitrogen (3 ml/min)

Sl No.	Name of the Component	Retention Times min
ix)	<i>n</i> -Butanol	15.50
x)	Acetal	17.78
xi)	<i>Iso</i> -amyl alcohol	20.88

NOTE — Retention time may change slightly as per actual GC conditions.

B-3.4 Approximate Retention Time

Sl No.	Name of the Component	Retention Times min
i)	Acetaldehyde	3.96
ii)	Methyl alcohol	4.20
iii)	Ethyl alcohol	5.65
iv)	<i>n</i> -Propanol	9.12
v)	Ethyl acetate	10.53
vi)	<i>iso</i> -Butanol	12.75
vii)	Benzene	13.46
viii)	Crotonaldehyde	14.83

B-3.5 Procedure

Separately inject 0.5 µl each of standard and sample and record the chromatogram. Calculate the acetaldehyde, methanol, ethyl acetate, *n*-propanol and *iso*-amyl alcohol in the sample by area normalisation method. Reference may be made to IS 15464 for detailed analysis. A typical gas chromatogram using FID showing retention time is given in Fig. 2.

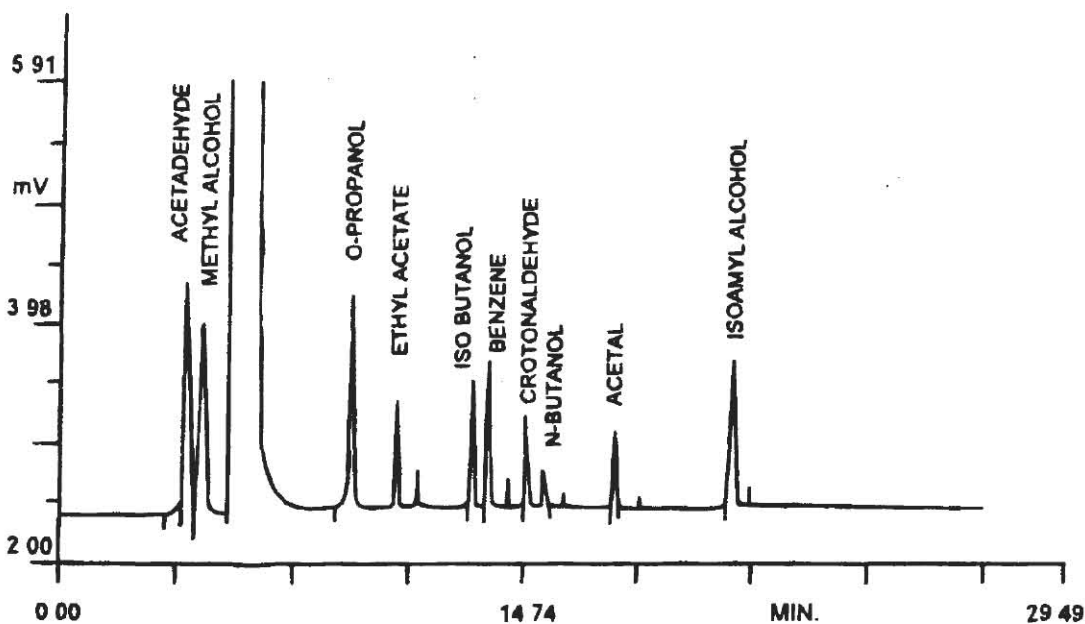


FIG. 2 TYPICAL CHROMATOGRAM FOR RECTIFIED SPIRIT

ANNEX C

[Table 1, Sl No. (iii)]

TEST FOR MISCIBILITY WITH WATER

C-1 PROCEDURE

C-1.1 Mix 10 ml of the material with 190 ml of water in a suitable glass vine and allow to stand at ambient temperature for minimum 10 min.

Compare the clarity of the mixture with that of an equal volume of water.

C-1.1.1 The material shall be taken to comply with the specified requirement, if there is no noticeable difference in clarity between the mixture and water.

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ANNEX D

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

TEST FOR ALKALINITY AND DETERMINATION OF ACIDITY

D-1 REAGENTS

D-1.1 Standard Sodium Hydroxide Solution — 0.1N.

D-1.2 Phenolphthalein Indicator — Dissolve 0.5g of phenolphthalein in 100 ml of rectified spirit and carefully add standard sodium hydroxide solution till the colour is rendered faintly pink.

D-2 PROCEDURE

D-2.1 Place 100 ml of water and a few pieces of clean porous pot in a 500 ml conical flask of resistance glass, and boil gently for 5 min to eliminate carbon dioxide. Cool slightly and add 100 ml of the material. Boil gently for a further period of 5 min. At the end of this period, close the neck of the flask with a stopper carrying a soda-lime guard tube, and allow to cool. When cool, examine for alkalinity; if not alkaline, titrate with standard sodium hydroxide solution using a micro-burette.

D-2.2 Determine the specific gravity of the material at room temperature using a specific gravity bottle or a pycnometer or a suitable hydrometer.

D-3 CALCULATION AND REPORT

D-3.1 Report whether the material is alkaline or acidic.

D-3.2 Calculate the acidity, if any, in terms of acetic acid and express as percentage by weight of the material taken for the test:

$$\text{Acidity (as CH}_3\text{COOH), mg/litre} = 600 VN$$

where

V = volume of standard sodium hydroxide solution required for the titration, in ml; and

N = normality of standard sodium hydroxide solution.

ANNEX E

[Table 1, Sl No. (vi)]

DETERMINATION OF RESIDUE ON EVAPORATION

E-1 PROCEDURE

Evaporate, on a water bath, 100 ml or more of the material to dryness in a weighed, clean, dry platinum, silica or resistance glass dish. Dry the residue for 30 min in an oven at a temperature of $100 \pm 2^\circ\text{C}$. Cool in a desiccator and weigh.

E-2 CALCULATION

$$\text{Residue on evaporation, percent by mass} = \frac{B-A}{VS} \times 100$$

where

B = weight of dish after evaporation, drying and cooling, in g;

A = weight of empty dish, in g;

V = volume of the material taken for the test, in ml; and

S = specific gravity of the material determined at room temperature (see D-2).

ANNEX F

[Table 1, Sl No. (vii)]

DETERMINATION OF ALDEHYDE CONTENT

F-1 METHODS

F-1.1 For Low Aldehyde Content

A suitable colour reaction is available when the aldehyde content (as CH_3CHO) is expected not to exceed 60 mg per litre of the material. This is based on the resinification that takes place and the yellow

colour that results on treatment of acetaldehyde with sodium hydroxide. The procedure described under F-4.1 provides as satisfactory qualitative limit test, but in case of dispute, the quantitative procedure described under F-4.2 shall be adopted in the qualitative procedure, acetals are also included as aldehydes.

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F-1.2 For Higher Aldehyde Content

For materials containing 0.05 to 0.5 percent of aldehydes, only the quantitative procedure described under F-4.2 shall be adopted.

F-2 APPARATUS

F-2.1 Stopped Flasks — Two, each of 250 ml capacity and identical in shape, size and colour.

F-3 REAGENTS

F-3.1 Sodium Hydroxide Solution — Dissolve 20 g of sodium hydroxide in water and dilute to 100 ml with water.

F-3.2 Stock Solution of Hydroxylamine Hydrochloride — Dissolve 20 g of hydroxylamine hydrochloride in 100 ml of water.

F-3.3 Metaphenylenediamine Hydrochloride

F-3.4 Aldehyde Free Alcohol — Re-distill rectified spirit over solid caustic soda or caustic potash, add 2 to 3 g of metaphenylenediamine hydrochloride per litre of rectified spirit, digest at ordinary temperature for several days or under a reflux condenser on a steam bath for several hours and distil slowly, rejecting the first 100 ml and the last 200 ml of the distillate.

F-3.5 Standard Sodium Hydroxide Solution — 0.1N.

F-3.6 Bromophenol Blue Solution — Dissolve 0.1 g of bromophenol blue in 1.5 ml of standard sodium hydroxide solution and dilute with water to 250 ml.

F-3.7 Hydroxylamine Reagent (Neutral Alcohol Solution of Hydroxylamine) — Dilute 10 ml of the stock solution of hydroxylamine hydrochloride with 100 ml of aldehyde-free alcohol, add 2 ml of

bromophenol blue solution, and then add standard hydroxide solution till the characteristic dichloric yellowish green colour is obtained.

F-4 PROCEDURE

F-4.1 Qualitative Test

F-4.1.1 Mix 10 ml of the material with 5 ml of sodium hydroxide solution and set aside for 5 min.

F-4.1.2 The limit prescribed for aldehyde content (0.006 g per 100 ml) shall be taken as not have exceeded, if no yellowish colour is produced in 5 min.

F-4.2 Quantitative Test

F-4.2.1 Take 50 ml of the material in a flask, add 25 ml of hydroxylamine reagent and 25 ml of distilled water. Allow to stand for 15 min. Meanwhile prepare a blank in a similar flask by using 25 ml of hydroxylamine reagent and 75 ml of distilled water. Titrate this solution with standard sodium hydroxide solution until the characteristic dichromic yellowish green colour appears. Titrate the sample solution with standard sodium hydroxide solution until the colour matches with that of the blank solution.

F-4.2.2 Calculation

Aldehyde content (as CH_3CHO), = $880 (V - v) N$ mg/litre

where

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

v = volume of standard sodium hydroxide solution required, if any, in the blank, in ml; and

N = normality of standard sodium hydroxide solution.

ANNEX G

[Table 1, Sl No. (viii)]

DETERMINATION OF ESTER CONTENT (AS ETHYL ACETATE)

G-1 REAGENTS

G-1.1 Standard Potassium Hydroxide Solutions — Two, 0.1 N and 0.5 N.

G-1.2 Standard Sulphuric Acid (see IS 266) — 0.1 N.

G-1.3 Phenolphthalein Indicator — Dissolve 0.5 g of phenolphthalein in 100 ml of rectified spirit and carefully add standard potassium hydroxide solution (0.1 N) till the colour is rendered faintly pink.

G-2 PROCEDURE

G-2.1 Transfer exactly 100 ml of the material into a heat resistant flask of about 200-ml capacity, add a

few drops of phenolphthalein indicator and neutralize, in the cold (temperature below 10°C), the free acid, if present, with standard potassium hydroxide solution (0.1 N). Add 2 ml of standard potassium hydroxide solution (0.5 N) attach the flask to a reflux condenser provided with a soda-lime guard tube and reflux the contents on a water bath for at least one hour. Cool the contents (temperature below 10°C), pour into another flask, wash the original flask with 100 ml of freshly distilled water, add the washings to the original liquor and then titrate with standard sulphuric acid adding a few drops of phenolphthalein indicator.

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G-2.2 Carry out a blank, using 100 ml of water in place of the neutralized material.

G-3 CALCULATION

Calculate the percentage of esters (as ethyl acetate) as follows:

Esters as ethyl acetate

$$(\text{CH}_3\text{COOC}_2\text{H}_5) \text{ g /100 litre} = 880 (V_2 - V_1) N$$

where

V_1 = volume of standard sulphuric acid required in the blank, in ml;

V_2 = volume of standard sulphuric acid required with the material, in ml; and

N = Normality of standard sulphuric acid.

ANNEX H

[Table 1, Sl No. (ix)]

DETERMINATION OF METHYL ALCOHOL

H-0 Two methods, namely, spectrophotometric method and gas chromatography methods are employed. The spectrophotometric method is sufficiently sensitive for routine type of analysis. The gas chromatography method is more sensitive and shall serve as a reference method.

H-1.2.5 Methanol Standard Solutions — Dilute appropriate volume of methanol stock solution (see H-2.4) to 100 ml volumetric flask with 40 percent (v/v) ethanol (methanol free) to get final concentration 20, 40, 60, 80, 100 ppm of methanol.

H-1 SPECTROPHOTOMETRIC METHOD

H-1.3 Procedure

H-1.1 Apparatus

Take 50 ml of sample in a distillation flask and distil, collecting about 40 ml of distillate. Dilute 1 ml of distillate to 5 ml with distilled water and shake well. Take 1ml of this solution, 1 ml of distilled water (for blank) and 1 ml of methanol standard solution in to 50 ml stoppered test tubes and keep them in an ice cold water bath. Add to each test tube 2 ml of potassium permanganate reagent and keep aside for 30 min. Decolorize the solution by adding a little sodium bisulphite and add 1 ml of chromotropic acid solution. Mix well and 15 ml of sulphuric acid slowly with swirling and place in hot water bath maintained at 80°C for 20 min. Observe the colour development from violet to red. Cool the reaction mixture to 20°C and measure the absorbance at 575 nm using 1 cm path length cell.

H-1.1.1 Spectrophotometer, of any make with wavelength range from 350 to 700 nm and a maximum band width of 5 nm.

H-1.2 Reagents

H-1.4 Calculation

H-1.2.1 Sodium Bisulphite, AR grade.

Calculate methanol content in g/100 litre of absolute ethanol as follows:

H-1.2.2 Potassium Permanganate Solution — Dissolve 3.0 gm of potassium permanganate and 15 ml of phosphoric acid in 100 ml distilled water. The solution shall be prepared monthly.

$$\text{Methanol content} = \frac{A_2 \times C \times D \times 1000 \times 100 \times 100}{A_1 \times S}$$

H-1.2.3 Sodium salt of chromotropic acid solution (Sodium 1, 8-dihydroxy naphthalene - 3, 6 -disulphonate), 5 percent Aqueous solution (m/v)

- a) Prepare fresh solution every week of either acid or salt and filter, if not clear.
- b) If necessary, prepare purified chromotropic acid by dissolving 10 g of chromotropic acid or its sodium salt in 25 ml water (add 2 ml sulphuric acid to aqueous solution of salt to convert it to free acid). Add 50 ml of methyl alcohol heat just to boiling and filter. Add 100 ml iso-propyl alcohol to precipitate free acid-chromotropic acid. Add more iso-propyl alcohol to complete precipitation of acid.

where

A_2 = absorbance for sample standard solution;

C = concentration of methanol standard solution, in g/ml;

D = dilution factor for sample solution;

A_1 = absorbance for methanol standard solution; and

S = ethanol content in sample, in percent (v/v).

H-1.2.4 Methanol Stock Solution — Dilute 1 g of methanol [99.9 percent (v/v)] to 100 ml with 40 percent (v/v) ethanol (methanol free). Dilute 10 ml of this solution to 100 ml with 40 percent ethanol (methanol free).

H-2 GAS CHROMATOGRAPHIC METHOD

This test method is related to determination of methyl alcohol content by gas chromatography.

H-2.1 Apparatus

Gas Chromatograph — Any gas liquid chromatograph equipped with FID or TCD detector can be used with following accessories and operating conditions:

- Column** *Chromosorb 101*
- Mesh size : 80/100
- Column length : 3 m
- I.D. : 3 mm
- Column tubing : Stainless steel (tubing must be material non-reactive with substrate, sample and carrier gas)
- Carrier gas : Hydrogen for TCD and Helium or Argon or Nitrogen for FID (Purity — 99.99 mole percent, Min)
- Syringe : 10 ml
- Sample size : 0.2 ml
- Electronic : For computation of results integration

H-2.1.1 Instrument Condition

- Detector : TCD or FID
- Oven temperature : 110°C
- Injector, temperature : 280°C
- Detector, temperature : 200°C
- Carrier gas flow rate : 30 ml/min

NOTE — The above gas chromatographic conditions are suggestive. However, any GC with different column may be used provided standardization/calibration are done after setting up chromatographic conditions for the required resolution.

H-2.1.2 Identification, Calibration and Standardization

Install the column in the chromatograph. Set the conditions given above, of column temperature and carrier gas flow that gives the necessary resolution of the components in the sample being analyzed. Set the conditions as such which gives a minimum 10 percent recorder deflection for a 0.1 percent concentration at the most sensitive setting of instrument. Allow sufficient time for the instrument to reach equilibrium as indicated by stable base line. Adjust carrier gas flow rate to a constant value.

H-2.1.2.1 Identification

Determine the retention time of each component by injecting small amount either separately or in known mixture.

H-2.1.2.2 Calibration and standardization

The response of any given detector varies from one chemical to another. Therefore, calibration is must. Calibration allows the computation of response factor which express the relative response of different components to the detector.

Calibration standard is prepared containing the impurities present in anhydrous alcohol. During standard preparation the exact amounts of all the components is noted. Inject 0.2 µl of calibration standard in the chromatographic column in accordance with the specified conditions.

Calculate response factor to four decimal places for all components relative to anhydrous alcohol using following equation:

$$Rf_x = \frac{W_x \times AM}{A_x \times WM}$$

where

- Rf_x = response factor for component x relative to acetone,
- W_x = weight of component x, in the calibration standard,
- A_x = area of component x, in the calibration run,
- WM = weight of acetone in the calibration standard, and
- AM = area of acetone in the calibration standard run which is selected as reference component.

The component which is unknown in the sample is assigned the relative response factor 1.000 0. It is advisable that the determination of response factors be made on the basis of duplicate analysis.

Response factor should be re-checked after any perceptible change in column or instruments performance.

H-3 PROCEDURE

Inject 0.2 µl of sample into the chromatographic column, using the same conditions as for components identification and standardization. Record and integrate the chromatogram using appropriate attenuation setting that provide optimum peak heights. Measure the area of all the peaks.

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H-4 CALCULATION

Calculate the concentrations of sample components using the following equation:

$$C_x, \text{ percent} = \frac{f_x \times R_x}{R_x \times f_x} \times 100$$

where

C_x = component x , mass percent,

R_x = peak response of component x ,

f_x = relative response factor of component x , and

$(R_x) \times (f_x)$ = sum of the individual component peak responses (R_x) multiplied by their relative response factors (f_x).

ANNEX J

(Clause 6)

SAMPLING OF RECTIFIED SPIRIT

J-1 GENERAL REQUIREMENTS

J-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

J-1.1 Samples shall not be taken in an exposed place.

J-1.2 The sampling instrument shall be clean and dry when used.

J-1.3 The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contaminations.

J-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or both by suitable means, or by rolling so as to bring all portions into uniform distribution.

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

J-1.6 The sample containers shall be of such a size that they are almost but not completely, filled by the sample.

J-1.7 Each sample containers shall be sealed air-tight with a glass stopper after filling, and marked with full details of sampling, the date of sampling, and the year of manufacture of the material.

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

J-1.9 Samples shall be protected from light as far as possible.

J-2 SAMPLING INSTRUMENTS

J-2.0 The following forms of sampling instruments may be used:

- a) Sampling bottle or can for taking samples from various depths in large tanks, and
- b) Sampling tube for sampling homogeneous materials.

J-2.1 Sampling bottle or can consists of a weighed bottle or metal container with removable stopper or top, to which is attached a light chain. The bottle or can is fastened to a suitable pole. For taking a sample, it is lowered in the tank to the required depth, and the stopper or top is removed by means of the chain for filling the container.

J-2.2 Sampling tube for homogeneous liquids is made of metal or thick glass, of 20 to 40 mm diameter and 400 to 800 mm length. 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 taking a sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

J-2.2.1 For small containers, the size of the sampling tube may be altered suitably.

J-3 SCALE OF SAMPLING

J-3.1 From a Tank

Take two sets of samples, one from the top and another from the bottom, and test them separately.

J-3.2 Material from a Single Run

Select one container at random and sample as described under **J-4.2**.

J-3.2.1 If the single sample fails to comply with the requirements of the standard, procedure for sampling of material from mixed batches, described under **J-3.3**, may be followed for re-test, if so agreed.

J-3.3 Material from Mixed Batches

J-3.3.1 Lot

All the containers in single consignment of material drawn from a single batch of manufacture shall constitute the 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.

J-3.3.2 Gross Sample

A number of containers shall be selected at random from a lot for the purpose of drawing samples for test. This number of containers shall constitute the gross sample. Size of the gross sample in relation to the size of the lot, or the scale of sampling, shall be subject to agreement between the purchaser and the vendor.

J-3.3.3 Suggested Scales of Sampling

In the absence of an agreement as to the scale of sampling and a guide to such an agreement, two scales are suggested, namely the normal scale and the low scale, as given in Table 2. The normal scale is to be used wherever possible and the low scale wherever the cost of the tests (including cost of material) is likely to be relatively high.

Table 2 Minimum Number of Containers to be Selected for Sampling from Various Sizes of Lots

Sl No.	Size or Lot	Size of Gross Sample	
		Normal Scale	Low Scale
(1)	(2)	(3)	(4)
i)	Under 25	10	5
ii)	25 to 49	15	5
iii)	50 to 99	20	10
iv)	100 to 199	30	15
v)	200 to 299	40	20
vi)	300 to 499	55	30
vii)	500 to 799	75	40
viii)	800 to 1299	115	55
ix)	1300 to 3199	150	75
x)	3200 to 8000	225	115

NOTE — The two scales of sampling suggested are based on statistical consideration. By using one of these scales, the risk of accepting a defective lot or rejecting a good lot, as judged by the inspection of the number of containers in the gross sample, remains the same, irrespective of the lot size. These risks, however, are different for the two scales of sampling. All sampling plans requiring a given percentage of the lot to be chosen as a sample are considered basically unsound for they invariably lead to relatively higher risks in case of smaller lots. These risks progressively decrease as the size of the lot increases. Furthermore, in the case of very big lots, the percentage sampling plans become uneconomical because of the unnecessarily large size of sample.

J-4 PROCEDURE

J-4.1 Sampling from a Tank

With the help of a sampling bottle or can, draw a sufficient number of samples from the top and the bottom. Keep, the top samples separate from the bottom samples, but mix each of these thoroughly and draw from each three test samples, each not less than 750 ml in volume.

J-4.2 Sampling from Containers

J-4.2.1 The procedure varies according to the size of the containers, namely whether the material is supplied in large containers of 180 litre and more, or in smaller containers.

J-4.2.2 Mixing Contents of Containers

Before drawing the test sample, thoroughly mix the contents of all containers in a gross sample, whether they are drums, bottles, cans or others, by shaking or stirring or both by suitable means, or by rolling so as to bring all portions into uniform distribution.

J-4.2.3 Drawing of Samples

Draw by inserting the sampling instrument through the bung hole or any other convenient opening, small portions of the material from different parts of each container in the gross sample.

J-4.2.4 Reduced Samples

Separately mix the samples drawn from each container obtain a composite sample for the container which shall be not less than 3 litre. Divide this composite sample in to the required number of reduced samples of not less than 750 ml in dried bottles or other containers, seal air-tight and label with all the particulars of sampling given under J-1.7.

J-4.2.5 Test Samples

The set of samples consisting of one each of the reduced samples, representatives of each of the containers in the gross sample, shall constitute the test sample.

J-4.2.6 Referee Samples

One of the test samples, bearing the seals of the purchaser and the vendor, shall constitute the referee sample, to be used in case of dispute between the purchaser and the vendor. It shall be kept at a place agreed between the purchaser and the vendor.

J-5 TESTS FOR ACCEPTANCE

J-5.1 Examination and Tests

The purchaser may examine and test each of the reduced samples constituting a test sample separately

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for compliance with the requirements of this standard, or he may prepare, for the purpose of such examination and at any stage of the progress of the examination, a composite sample representative of the whole lot, by mixing all the reduced samples constituting the test sample.

J-5.2 Criteria for Judgment

When the individual reduced samples in a test sample are separately examined and the results vary from one reduced sample to another, the criterion for

judging the quality of the lot for the purpose of acceptance on the basis of the result obtained shall be as given below, unless otherwise previously agreed between the purchaser and the vendor:

<i>Test Results</i>	<i>Mean</i>	<i>Range</i>	<i>Criterion for Acceptance</i>
1, 2, n	\bar{x}	R	($\bar{x} - 0.6R$) shall be not less than 94.85 percent by volume at 20°C or 66° OP.

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