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Draft Sri Lanka Standard
SPECIFICATION FOR DISTILLED LIQUOR/ SPIRIT DRINKS
PART 2: WHISKY/ WHISKEY
(DSLS..... :)

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இவ்வரைவு இலங்கைக் கட்டளையெனக் கருதப்படவோ அன்றிப் பிரயோகிக்கப்படவோ கூடாது
This draft should not be regarded or used as a Sri Lanka Standard.

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Comments to be sent to: SRI LANKA STANDARDS INSTITUTION, 17, VICTORIA PLACE,
ELVITIGALA MAWATHA, COLOMBO 08.

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XX

Introduction

This Draft Sri Lanka Standard has been prepared by the Sri Lanka Standards Institution and is now being circulated for technical comments to all interested parties.

All comments received will be considered by the SLSI and the draft if necessary, before submission to the Council of the Institution through the relevant Divisional Committee for final approval.

The Institution would appreciate any views on this draft which should be sent before the specified date. It would also be helpful if those who find the draft generally acceptable could kindly notify us accordingly.

All Communications should be addressed to:

The Director General
Sri Lanka Standards Institution,
17, Victoria Place,
Elvitigala Mawatha,
Colombo 08.

Draft Sri Lanka Standard
SPECIFICATION FOR DISTILLED LIQUOR/ SPIRIT DRINKS
PART 2: WHISKY/ WHISKEY

FOREWORD

This Sri Lanka Standard was approved by the Sectoral Committee on Food Products and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on

Whisky/ whiskey is a distilled alcoholic beverage, made from malted barley or malted cereals. The Standard specifies chemical and other quality parameters in-order to assure the quality and the safety of the product.

This Standard is subject to the restrictions imposed under the Excise Ordinance of 2009 and the regulations framed thereunder.

For the purpose of deciding whether a particular requirement of this Standard is complied with the final value, observed or calculated, expressing the results of a test or an analysis shall be rounded off in accordance with **SLS 102**. The number of significant places retained in the rounded off value shall be the same as that of the specified value in this Standard.

In the preparation of this Standard, valuable assistance derived from the following publications is gratefully acknowledged.

EEC 110/ 2008	Regulation of European Parliament and of the Council
IS 6749: 1972	Glossary of terms relating to alcohol (Ethyl) industry and trade
IS 4449: 2005	Alcoholic drinks – Whiskies – Specification
Volume 2/ Chapter 4 and 7	The Beverage Alcohol Manual, Alcohol and Tobacco Tax and Trade Bureau, U S Department of the Treasury
Title 27/ Chapter 1/ Section 5.22	Code of Federal Regulations, Federal Register, Government of United States
No 2890/ 2009	The Scotch Whisky Regulations

1 SCOPE

This Standard prescribes requirements and methods of sampling and test for whisky/ whiskey.

2 REFERENCES

SLS	102	Presentation of numerical values
SLS	143	General principles of food hygiene
SLS	290	Glass liquor bottles
SLS	428	Random sampling methods
SLS	614	Potable water
SLS	1102	Metal closures ROPP for glass bottles
Official Methods of Analysis of the Association of Official Analytical Chemists (AOAC), 21 st Edition, 2019		

3 DEFINITIONS

For the purpose of this Standard, the following definitions shall apply:

3.1 whisky/ whiskey: A spirit drink produced exclusively by one or more distillations at less than 94.8 % volume, distillation of a mash made from malted barley/ cereals with or without whole grains of other cereals, which has been saccharified by the diastase of the malt contained therein, with or without other natural enzymes and fermented only by the action of yeast and possesses specific organoleptic characteristics generally attributed to whisky/ whiskey and known to the trade as such.

3.2 maturation/ ageing: Allowing certain reactions to develop naturally in appropriate containers, thereby giving the spirit drink in question organoleptic qualities previously absent.

3.3 blending: Combining two or more spirit drinks of the same category, distinguished only by minor differences in composition due to the method of preparation and/ or the period of maturation/ ageing and/ or the geographical area of production. The spirit drink so produced shall be of the same category of spirit drink as the original spirit drinks before blending.

3.4 single malt whisky/ whiskey: Whisky/ whiskey produced at a single distillery from mash of malted barley and water without the addition of any other cereals in pot-stills.

3.5 single grain whisky/ whiskey: The alcoholic distillate produced from mash of one type of cereal grains.

3.6 blended malt whisky/ whiskey: Whisky/ whiskey produced from blending of two or more single malt whiskies.

3.7 blended grain whisky/ whiskey: Whisky/ whiskey produced from blending of two or more single grain whiskies.

4 TYPES

4.1 Single malt whisky/ whiskey

4.2 Single grain whisky/ whiskey

4.3 Blended malt whisky/ whiskey

4.4 Blended grain whisky/ whiskey

5 REQUIREMENTS

5.1 Hygiene

The product shall be processed, packaged, stored and distributed under hygienic conditions as prescribed in **SLS 143**.

5.2 Water quality

Potable water conforming to **SLS 614** shall be used for processing activities.

5.3 Taste and aroma

The product shall possess the characteristic taste and aroma.

5.4 Colour

The product shall appear in golden brown colour.

5.5 Additives

The product shall not be sweetened or flavoured, nor contain any additives other than caramel (INS 150a, INS 150c or INS 150d) used for colouring.

5.6 Sediments, suspended and foreign matter

The product shall be free from sediments, suspended matter and foreign matter.

5.7 Maturity

The final distillate shall be matured for at least three years in wooden casks not exceeding 700 litres capacity.

5.8 Ethyl alcohol content

The Ethyl alcohol content in whisky/ whiskey shall be in the range of 40.0 to 50.0 percent by volume, when determined according to the method prescribed in Appendix B. The tolerance limits for Ethyl alcohol content shall be ± 0.2 per cent of the declared strength.

NOTE

The Ethyl alcohol content, its tolerance and method of measurement may vary according to the rules and regulations prescribed by the Excise Department of Sri Lanka.

5.9 Other requirements

The product shall comply with the other requirements prescribed in Table 1 and shall not exceed the limits given in Column 5 of the table.

Table 1 – Requirements for whisky/ whiskey

SI No (1)	Characteristic (2)	Requirement		Method of test (5)
		Single malt or grain (3)	Blended malt or grain (4)	
i)	Total solids, per cent (m/ v), max.	2.0		Appendix C
ii)	Total acids (expressed as Acetic acid, in terms of g/ 100 litres of absolute alcohol), max.	150.0	100.0	Appendix D

iii)	Esters as Ethyl acetate (expressed in terms of g/ 100 litres of absolute alcohol), max.	200.0	150.0	Appendix E
iv)	Higher alcohols as Amyl alcohol (expressed in terms of g/ 100 litres of absolute alcohol), max.	750.0		
v)	Aldehydes as Acetaldehyde (expressed in terms of g/ 100 litres of absolute alcohol), max.	30.0		Appendix G
vi)	Furfural (expressed in terms of g/ 100 litres of absolute alcohol), max.	1.0		Appendix H
vii)	Methyl alcohol (expressed in terms of mg/ 1 litre), max.	40.0		AOAC 972.11

6 CONTAMINANTS

The product shall not exceed the limits given in Table 2, when tested according to the methods given in Column 4 of the table.

TABLE 2 – Limits for potentially toxic elements

SI No (1)	Potentially toxic element (2)	Limit (3)	Method of test (4)
i)	Arsenic, as As, mg/ kg, max.	0.10	AOAC 2013.06
ii)	Copper, as Cu, mg/ kg, max.	2.00	AOAC 967.08
iii)	Lead, as Pb, mg/ kg, max.	0.10	AOAC 2013.06
iv)	Cadmium, as Cd, mg/ kg, max.	0.01	AOAC 2013.06

7 PACKAGING

7.1 The product shall be filled in glass bottles conforming to **SLS 290** or neutral or non-reactive suitable food grade containers. The bottles/ containers shall be closed with a metal closure conforming to **SLS 1102** or with any other suitable food grade closure.

7.2 All containers shall be cleaned and free from chips, cracks and any other defects and appropriately sealed. All glass bottles shall be subjected to cleansing and sanitizing process before filling.

7.3 The bottles or containers shall be packed in wooden cases, wooden crates, plastic crates, metal crates, corrugated fiber boxes or any package as agreed to between the purchaser and the supplier.

8 MARKING AND/ OR LABELING

The following shall be marked and/ or labeled legibly and indelibly on each package/ container destined to the final consumer:

- a) Common name and the type of product;
- b) Name(s) of the cereal(s) used;
- c) Name and address of the manufacture and/ or distributor;
- d) Brand name or trade mark, if any;
- e) Alcohol content per cent (v/ v);
- f) Batch number or code number or a decipherable code marking;
- g) Net content in “ml”, “cl” or “l”;
- h) Year of bottling;
- j) Maturation period or the age of the product; and
- j) Country of origin.

9 METHODS OF TEST

Tests shall be carried out as given in Appendix **B** to **H** of this Standard and Official methods of Analysis, Association of Official Analytical Chemists (**AOAC**) official methods of analysis, 21st edition, 2019.

10 CRITERIA FOR CONFORMITY

A lot shall be declared as conforming to the requirements of this Standard if the following conditions are satisfied.

10.1 All the bottles or containers examined as in **A.5.2.1** satisfy packaging and marking and/ or labelling requirements.

10.2 The volume of each container measured as in **A.5.2.2** does not vary by more than 1 per cent of declared volume and the total volume of 12 bottles or containers does not vary by more than ± 0.3 per cent of the total declared volume.

10.3 The test results on individual samples tested as in **A.5.2.3** satisfy the relevant requirements.

10.4 The composite sample tested as in **A.5.2.4** satisfies the relevant requirements.

APPENDIX A SAMPLING

A.1 LOT

In any consignment all the containers or bottles of the same size containing whisky/ whiskey of the same type from one batch of manufacture shall constitute a lot.

A.2 General requirements

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

A.2.1 Samples shall be taken in a protected place not exposed to damp air, dust and soot.

A.2.2 The sampling instruments used shall be clean and dry.

A.2.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from extraneous contamination.

A.2.4 Samples shall be placed in suitable clean, dry and air-tight glass containers.

A.2.5 The sample containers shall be of such a size that sufficient head space to allow for expansion of the liquid is left after pouring in the sample.

A.2.6 Each sample container shall be sealed air-tight with a suitable stopper after filling and marked with the following information.

- a) Sample number or other identification marks;
- b) Name of the product;
- c) Batch or code number;
- d) Year of bottling;
- e) Date of sampling;
- f) Place of sampling;
- g) Name and signature of the person drawing the sample; and
- h) Signature of the person or his representative on whose premises the sample was taken.

A.2.7 Samples shall be stored in a cool, dark and dry place.

A.3 Sampling instruments

A.3.1 The following forms of sampling instrument may be used.

- a) Weighed sampling can; and
- b) Sampling tube.

A.3.2 All the material used for fabricating the sampling instrument shall be such as not to contaminate or chemically affect the sample or the material being sampled.

A.4 Sampling from bulk container or vat

A.4.1 One sample shall be taken from each bulk container or vat using an appropriate sampling instrument and transferred to the sample container.

A.4.2 Before drawing the sample, the material shall be thoroughly mixed by stirring.

A.4.3 Each sample shall be individually tested for all the relevant requirements of this Standard.

A.5 Sampling from retail bottles or containers

A.5.1 *Scale of sampling*

A.5.1.1 The samples shall be selected and tested from each lot separately for ascertaining their conformity to the requirements of this Standard.

A.5.1.2 The number of bottles or containers to be selected from a lot shall be in accordance with Table 3.

TABLE 3 - scale of sampling

Number of bottles or containers in the lot (1)		Number of bottles or containers to be selected (2)
up to	1 000	8
1 001 to	3 000	10
3 001 to	10 000	13
10 001 and	above	15

A.5.1.3 In addition to the bottles or containers drawn as in **A.5.1.2** another 12 bottles or containers shall be drawn from each lot to determine the volume of the contents of the bottles or containers.

A.5.1.4 The bottles or containers shall be selected at random, in order to ensure randomness of selection, random number tables as given in **SLS 428** shall be used.

A.5.1.5 If a reference sample is required, the scale of sampling shall be correspondingly increased and one third of the sample retained by the purchaser, one third by the testing authority (or referee) for future reference and one third handed over to the supplier.

A.5.2 *Number of tests*

A.5.2.1 All the bottles or containers in the sample selected as in Clause **A.5.1.2** shall be examined for packaging and marking and/ or labelling requirements.

A.5.2.2 The volume of the contents of 12 bottles or containers selected as in Clause **A.5.1.3** shall be measured. (This may be done at the place of sampling). The volume shall be measured at $27 \pm 2^\circ\text{C}$.

A.5.2.3 After examining as in Clause A.5.1.2 each bottle or container shall be individually tested for the requirements given in Clauses 5.3, 5.4, 5.6, 5.8 and 5.9.

A.5.2.4 After testing as in A.5.1.3 an equal quantity of material shall be drawn from each bottle or container and mixed together to form a composite sample. Test for potentially toxic elements given in Clause 6 shall be done on this composite sample.

APPENDIX B DETERMINATION OF ETHYL ALCOHOL

B.1 METHOD 1 - PYCNOMETER METHOD (REFERENCE METHOD)

B.1.1 APPARATUS

B.1.1.1 Distillation Assemble as shown in Figure 1. The delivery end of the condenser is attached to a glass tube with a bulb by means of a ground glass joint. The lower part of this tube should reach the bottom of the receiver and dip into the minimum quantity of distilled.

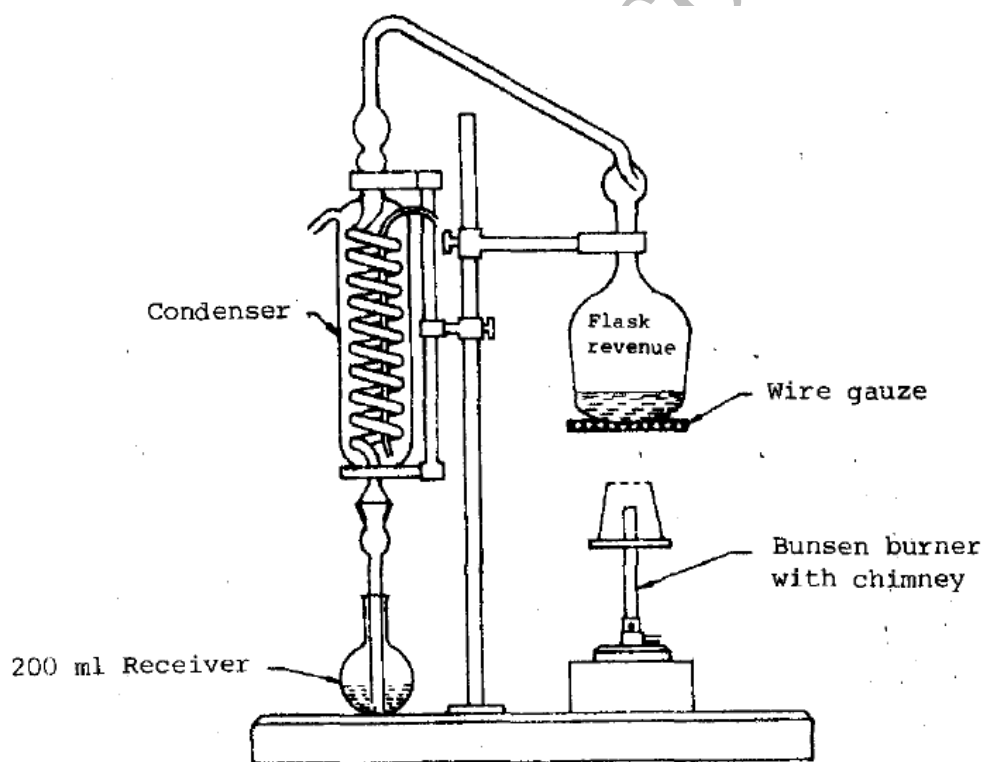


FIGURE 1 - Distillation Assemble

B.1.1.2 *Pycnometer* (specific gravity bottle), 25-ml or 50-ml

B.1.1.3 *Thermometer* 0°C to 50°C

B.1.1.4 *Standard volumetric flask*, 200-ml

B.1.2 PROCEDURE

B.1.2.1 Take, 200 ml of sample in a 500-ml distillation flask containing about 25 ml of water and a few pieces of pumice stone. Complete the distillation in about 35 minutes and collect the distillate in a 200-ml standard volumetric flask till the volume on the flask nears the mark. Allow the distillate to come to room temperature and make up the volume to 200 ml and mix thoroughly.

B.1.2.2 Determine the specific gravity of the distillate at $27 \pm 2^\circ\text{C}$ with the help of the pycnometer. Obtain percentage of alcohol by volume from the tables (52.003) of AOAC (Association of Official Analytical Chemists) official methods of analysis, 21st edition, 2019.

B.2 METHOD 2 - HYDROMETER METHOD (Routine method)**B.2.1 PROCEDURE**

B.2.1.1 When several tests are carried out, rinse the interior of the glass cylinder, hydrometer, thermometer with the portion of the distillate to be tested after each test so that the previous liquid which has wetted the sides of the cylinder may not alter the density of the liquid under test.

B.2.1.2 Pour the sample into the glass cylinder until its level is about 50 mm below the rim of the latter. Immerse the thermometer in the liquid and stir until the mercury column becomes stationary. Then note down the temperature. If the surface of the mercury stands between any two readings of the scale, record the nearest reading above it as the temperature of the sample. Then withdraw the thermometer and immediately immerse the hydrometer very slowly and allow it to float freely without touching the sides of the glass cylinder. Keeping the eye in level of the surface of the liquid note down the reading (density), that is cut by the surface of the liquid. If the surface of the liquid is between any two readings, the reading to the nearest division below the surface of the sample (see through the liquid) is taken as the density.

Refer tables (52.003) of AOAC (Association of Official Analytical Chemists) official methods of analysis, 21st edition, 2019.

B.2.1.3 The temperature shall be recorded to the nearest 0.5°C above the surface of the mercury when it stands between any two consecutive divisions of the thermometer, and the hydrometer reading to the nearest division below the surface of the sample.

B.3 METHOD 3 – DENSITY METER METHOD (ROUTINE METHOD)**B.3.1 APPARATUS****B.3.1.1** *Density meter* DMA 4100M

The hardware of the instrument is mainly composed with Xsample 22 sample filling unit and Xsample 52 sample handling unit acting as the supportive hardware units for the alcohol determination process.

B.3.1.2 *Waste vessel*

B.3.1.3 *Sample vessel***B.3.2 PROCEDURE****B.3.2.1** Assemble the apparatus.

B.3.2.2 First, immerse the needle in the sample vessel with adequate amount of sample. Place the pump liver in the vertical position and pump the sample into the measuring cell. Avoid air bubbles at the suction process. At the end of the filling, take the constant value displayed on the screen as the percentage value of ethyl alcohol.

**APPENDIX C
DETERMINATION OF TOTAL SOLIDS****C.1 PROCEDURE**

Evaporate 50 ml of the sample in a dried, tared dish on a water bath. Dry the dish in an air-oven at 110 ± 2 °C. Cool in a desiccator and weigh the dish. Repeat till constant mass is obtained. Calculate the total solids as per cent (m/ v). Express the results to four decimal places.

C.2 CALCULATION

Total solids, expressed as in grams per 100 litre of absolute alcohol = $2 \times 10^3 \times m \times f$

where,

m is the mass, in grams, of the residue; and

f is 100/ per cent of Ethyl alcohol in the sample.

**APPENDIX D
DETERMINATION OF TOTAL ACIDITY****D.1 REAGENTS**

D.1.1 *Sodium hydroxide*, standard volumetric, 0.1 mol/ l

D.1.2 *Phenolphthalein indicator*, 1 per cent (v/ v)

D.2 PROCEDURE

Take, 50 ml of the sample and add about 100 ml of water. Titrate against Sodium hydroxide solution using Phenolphthalein as indicator.

D.3 CALCULATION

Calculation on the basis that 1 ml of 1 mol/ 1 Sodium hydroxide solution is equivalent to 0.06009 g of Acetic acid.

$$\left. \begin{array}{l} \text{Total acidity expressed as Acetic acid, grams per 100 litre} \\ \text{of absolute alcohol} \end{array} \right\} = 120 \times v \times c \times f$$

where,

v is the volume, in ml, of standard Sodium hydroxide used for titration;

c is the concentration, in mol/ 1, of the Sodium hydroxide solution; and

f is 100/ per cent of Ethyl alcohol in the sample.

APPENDIX E DETERMINATION OF ESTERS AS ETHYL ACETATE

E.1 REAGENTS

E.1.1 *Sodium hydroxide*, standard volumetric solution, 0.1 mol/ 1

E.1.2 *Sulfuric acid*, standard volumetric solution, 0.05 mol/ 1

E.2 PROCEDURE

E.2.1 Take 50 ml of distillate from **(B.1.2.1)** add few drops of Phenolphthalein and neutralize with Sodium hydroxide **(E.1.1)**. Add 25 ml of 0.1 mol/1 Sodium hydroxide **(E.1.1)** and reflux for 2 h. Cool and back titrate with Sulfuric acid **(E.1.2)**. One millilitre of 0.1 mol/ 1 Sodium hydroxide is equivalent to 0.0088 g of Ethyl acetate.

E.2.2 Simultaneously run a blank taking 50 ml of water in place of the distillate of the sample in the same way. The difference in titration value of the blank and the test sample in milliliters of standard acid solution gives the equivalent ester

E.3 CALCULATION

$$\left. \begin{array}{l} \text{Esters expressed as Ethyl acetate grams} \\ \text{per 100 litres of absolute alcohol} \end{array} \right\} = 352 \times v \times c \times f$$

where,

v is the difference, in ml, of standard acid used for blank and test;

c is the concentration, in mol/ 1, of the Sulphuric acid solution; and

f is 100/ per cent of Ethyl alcohol in the sample.

APPENDIX F
DETERMINATION OF HIGHER ALCOHOL - AMYL ALCOHOL

F.1 REAGENTS

F.1.1 *Sulfuric acid*, 90 per cent (v/ v)

F.1.2 *Potassium hydroxide*, 50 per cent (v/ v)

F.1.3 *Solution of higher alcohols.*

F.1.3.1 Composition

Propyl alcohol	1 Part
Isobutyl alcohol	2 Parts
Amyl alcohol	3 Parts
Capryl alcohol	1 Part

F.1.3.2 Preparation

Weigh 1 g of the mixture and dissolve in 100 ml of 50 per cent (v/ v) alcohol. Dilute 10 ml of this solution to 100 ml with 50 per cent alcohol to make 0.1 per cent solution.

F.1.4 Furfural solution

Weigh 0.4 g of furfural and dissolve in 100 ml of 50 per cent (v/ v) alcohol.

F.2 PROCEDURE

F.2.1 To 50 ml of the distillate from **B.1.2.1**, add 1 ml of 50 per cent (v/ v) Potassium hydroxide and leave overnight, distil off the alcohol to such a volume such a volume about 40 ml (v_1) that the distillate is 50 per cent (v/ v) alcohol.

F.2.2 Add a standard solution of higher alcohol (**F.1.3**) into Nessler tubes marked 0 ml, 1 ml, 2 ml, 3 ml, 4 ml, 5 ml, and 6 ml and make up to 10 ml with 50 per cent absolute alcohol (v/ v). Take 10 ml of distillate from above (v_1). Add to each tube 0.5 ml of furfural solution (**F.1.4**).

F.2.3 Place tubes one by one in an ice cold water bath and stir the content by gently shaking during thirty seconds, and afterwards allow to stand for about an hour at the ordinary temperature in the room before taking observations.

F.2.4 In the presence of an appreciable amount of higher alcohols, a reddish violet colour develops, and the proportion of the alcohols can be estimated by comparing with standards. So, compare the colour of the 10 ml of distillate with the standards solutions of higher alcohol after 30 minutes.

F.3 CALCULATION

$$\text{The amount of higher alcohol in 100 litre of distillate} = \frac{v \times 10 \times f \times v_1}{50}$$

where,

v is the volume of higher alcohol in standard solution match the sample solution;

v_1 is the volume of 50 per cent distillate; and

f is the 100/ per cent of Ethyl alcohol in the sample.

APPENDIX G DETERMINATION OF ALDEHYDES AS ACETALDEHYDE

G.1 APPARATUS

G.1.1 *Iodine flask, 500-ml*

G.2 REAGENTS

G.2.1 *Solution A, Iodine solution 0.05 mol/ l*

G.2.2 *Solution B*

Dissolve, 17 g of Sodium metabisulfite ($\text{Na}_2\text{S}_2\text{O}_5$) or Potassium metabisulfite ($\text{K}_2\text{S}_2\text{O}_5$) in water and make up to a litre. The solution must be 0.3 mol/ l when used.

G.2.3 *Solution C (Neutralk buffer)*

Dissolve, 24 g of Disodium hydrogen phosphate ($\text{Na}_2\text{PO}_4 \cdot 12\text{H}_2\text{O}$) in 25 ml of 0.5 mol/ l Sulfuric acid and dilute to a litre with water.

G.2.4 *Solution D*

Dilute, 250 ml Hydrochloric acid (rel. den. 1.18) with water and make up to a litre.

G.2.5 *Solution E*

Dissolve, 17.5 g of Boric acid in 800 ml of normal soda solution and make up to 2 litres with water.

G.2.6 *Solution F, starch solution*

G.3 PROCEDURE

G.3.1 Carry out the test within 24 hours of distillation.

G.3.2 Transfer 50 ml of Solution C (neutral buffer) (G.2.3) into 500-ml flask (G.1.1) with a measuring cylinder and add 10 ml of solution B (G.2.2).

G.3.3 Pipette 25 ml of distillate from B.1.2.1 and transfer it into the mixture, stopper the flask, shake and leave for 20 minutes. Add 1 ml of solution F (G.2.6) about 100 ml of water and 10 ml of solution. (G.2.4) using a measuring cylinder.

G.3.4 Add solution A (**G.2.1**) with a burette until a blue colour appears. Add 100 ml of the solution E (**G.2.5**) until the blue colour disappears. Titrate with solution A (**G.2.1**) to the same blue colour appears as before.

G.3.5 Note the volume of Iodine used in the last stage of titration. The final solution should be alkaline to Phenolphthalein. If it is not determination has to be repeated. Do a blank with 25 ml of water. The volume of Iodine corresponds to the amount of aldehyde present.

One millilitre of solution A is equivalent to 2.2 mg of Acetaldehyde.

NOTE

Second burette used should be calibrated to 0.01 ml.

G.4 CACULATION

Aldehydes expressed as Acetaldehyde in grams per 100 litre of, }
absolute alcohol } = $v \times c \times 176 \times f$

where,

v is the volume, in ml, of solution A used at the last stage;

c is the concentration in mol/l of the Iodine solution; and

f is 100/ per cent of Ethyl alcohol in the sample.

APPENDIX H DETERMINATION OF FURFURAL

H.1 REAGENTS

H.1.1 *Aniline*, distilled and colourless

H.1.2 *Hydrochloric acid*, concentrated, (rel. den. 1.18)

H.1.3 *Furfural-free alcohol*

Dissolve 5g of m-phenylene diamine hydrochloride in 1 litre of Ethyl alcohol. Allow to stand at least 24 hours with frequent shaking (previous treatment with Potassium hydroxide is not necessary). Reflux for at least 8 hours and shake if necessary. Allow to stand overnight and distill. Collect the distillate rejecting first 100 ml and last 200 ml.

NOTE

If this distillate forms a colouration with Aniline hydrochloride, repeat the treatment, Report the treatment.

H.1.4 *Furfural Stock solution*

Dissolve 1 g of colourless furfural in 100 ml of furfural free alcohol (**H.1.3**) 50 per cent (v/v).

H.1.4.1 *Furfural working solution*

Pipette 1 ml of (H.1.4) and dilute to 100 ml with furfural free alcohol.

One millilitre of this solution contents 0.0001 g of furfural.

H.2 **PROCEDURE**

H.2.1 Pipette 10 ml of the sample and dilute to 50 ml with furfural-free alcohol (G.1.3) in a Nessler tube. Take 0 ml, 0.2 ml 0.4 ml up to 1.2 ml of the furfural working solution (G.1.4) into Nessler tubes and dilute to 50 ml with furfural free alcohol. To each of the Nessler tube add 2 ml of Aniline and 0.5 ml of Hydrochloric acid (G.1.2) and shake well, till a reddish brown colour develops.

H.2.2 Compare the colour of sample solution with the standard solutions after 15 minutes, select the standard solution which matches the sample solution.

H.3 **CALCULATION**

$$\text{Furfural, in grams per 100 litres of absolute alcohol} = v \times f$$

where,

v is the volume, in ml of the standard furfural solution which matches the sample; and
 f is 100/ per cent of Ethyl alcohol in the sample.