# මහජන අදහස් සඳහා පුමිති කෙටුම්පත பொதுசனக் கருத்துரைக்கான கட்டளை வரைவு DRAFT STANDARD FOR PUBLIC COMMENT

(பெலக்பில் ஒவ் ஆடை திருத்தத்திற்குட்படக்கூடியது. Liable to alteration)

නිකුත් කළ දිනය බෞඛ්ඨ්රම්න ම්ෂම් Date of Issue

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Draft Sri Lanka Standard SPECIFICATION JAGGERY PART 1 : KITHUL JAGGERY (DSLS ......)

හකුරු සඳහා වන ශුී ලංකා පුමිති පිරිවිතර කෙටුම්පත කොටස 1 : කිතුල් හකුරු

මෙම කෙටුම්පත ශ්රී ලංකා පුම්තියක් ලෙස නොසැලකිය යුතු මෙන් ම භාවිතා නොකළ යුතු ද වේ. இவ்வரைவு இலங்கைக் கட்டளையெனக் கருதப்படவோ அன்றிப் பிரயோகிக்கப்படவோ கூடாது This draft should not be regarded or used as a Sri Lanka Standard.

අදහස් එවිය යුත්තේ : ශ්රී ලංකා පුමිති ආයතනය, 17, වික්ටෝරියා පෙදෙස, ඇල්ව්ටිහල මාවත, කොළඹ 08.

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

#### හැඳින්වීම

මෙම ශ් ලංකා පුමති කෙටුළුපත , ශ් ලංකා පුමති ආයතනය විසින් සකසන ලදුව, සියලුම උදෙහ්ගී අංශ වලට නාකෘණික විවේචනය සඳහා යටතු ලැබේ.

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මෙම කෙටුම්පතට අදාල යෝජනා හා විවේචන නියමිත දිනට පෙර ලැබෙන්නට සැලැස්වුවහොත් අගය කොට සලකමු, තවද, මෙම කෙටුම්පත පිළිගත හැකි බැව හැගෙන අය ඒ බව දන්වන්නේ නම් එය ආයතනයට උපකාරී වනු ඇත.

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ඩොකෂ ජනවාල් මු ලංකා පුමිති ආයතනය, 17, වික්ටෝරියා පෙදෙස, ඇල්ඩ්ටිගල මාවත, කොළඹ 08.

#### 

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 JAGGERY PART 1: KITHUL JAGGERY



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#### Draft Sri Lanka Standard SPECIFICATION FOR JAGGERY PART 1: KITHUL JAGGERY

### 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 .....

*Kithul* (*Caryota urens L*) is an indigenous plant in Sri Lanka. *Kithul* based products are unique having a long history in Sri Lanka. *Kithul* jaggery is very popular because of its unique aroma and flavour. During the recent years the exports of *kithul* products have greatly increased indicating the potential growth of the industry.

This Standard supersedes SLS 521:1981. In this Standard specific requirements including sugar profile, amino acid profile have been introduced to differentiate *Kithul* jaggery from other jaggery types.

This Standard is subject to the Food Act No. 26 of 1980 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 shall be rounded off in accordance with **SLS 102**. The number of significant figures to be retained in the rounded off value shall be the same as that of the specified value in this Standard.

### 1 SCOPE

This Standard prescribes the requirements and methods of sampling and test for *Kithul* jaggery.

# 2 **REFERENCES**

- SLS 102 Rules for rounding off numerical values
- SLS 143 Code of practice for general principles of food hygiene
- SLS 428 Random sampling methods

American Association for Cereal Chemists Approved methods (AACC) 11<sup>th</sup> edition Volume 2, 2010

Official methods of Analysis, Association of Official Analytical Chemists (AOAC) 21<sup>st</sup> edition, 2019

#### **3 DEFINITION**

For the purpose of this Standard, the following definition shall apply;

3.1 *Kithul* jaggery : It shall be the solidified product manufactured from the sap exudate collected from partially mature inflorescence of the kithul palm (Caryota urens L) (English- toddy palm, Tamil -Thipillippanai)

#### 4 **INGREDIENT**

#### 4.1 **Basic ingredients**

*Kithul* jaggery shall be made from clean, filtered, fresh sap/ juice from the inflorescence of the *Kithul* palm (*Carvota urens L*)

#### 4.2 Sap processing agents/fermentation arresting agent

Any one of the following agents may be used to arrest the fermentation.

**4.2.1**. Bark of Vateria copallifera (Retz) Alston (Sinhala/ Tamil - Hal) 2 per cent by mass (max.) jolic comme

**4.2.2** *Calcium oxide* (INS 529)

#### 4.3 **Optional ingredients**

- 4.3.1 Ginger
- **4.3.2** Spices

#### 5 REOUIREMENTS

#### 5.1 Hygiene

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

#### 5.2 Appearance

Jaggery shall be free from any plant tissues and insects, insect fragments or any other extraneous and foreign matter. Jaggery shall have a colour, of good jaggery and shall be free of added colouring matter.

#### 5.3 **Odour and flavour**

The product shall have a pleasant, characteristic of good jaggery taste, odour and flavour of the Kithul jaggery. It shall be free from any objectionable odour and flavour. Free from added flavour.

# 5.4 Adulterants

The product shall be free from adulterants.

### 5.5 Other requirements

Jaggery shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Column 4 of the table.

SI	Characteristic	Limit	Method of
No			test
(1)	(2)	(3)	(4)
i)	Moisture, per cent by mass, max.	10	Appendix B
ii)	Total ash, per cent by mass, max.	3.5	Appendix C
iii)	Acid insoluble ash, per cent by mass, max.	0.5	Appendix <b>D</b>
iv)	Matter insoluble in water, per cent by mass, max.	2.0	Appendix <b>E</b>
v)	Reducing sugars, per cent by mass. max.	13	Appendix <b>F</b>
vi)	Sugars, non-reducing, per cent by mass, min.	70	Appendix G

## TABLE 1 - Requirements for Kithul jaggery

# 5.6 Characterization of different types

# 5.6.1 Sensory analysis

*Kithul* jaggery shall have the characteristic odour, flavour and colour.

### **5.6.2** Sugar profile

*Kithul* jaggery shall comply with the requirements given in Table 2, when tested according to the methods prescribed in instrumental technique using **AACC** Method 80-04: 2010 (HPLC / RI Detection).

Sl	Type of sugar	Requirement		
No				
(1)	(2)	(3)		
i)	Total reducing sugars per cent by mass max.	13.0		
ii)	Sucrose per cent by mass, min.	70.0		
iii)	Total Sugar, per cent by mass, min.	85.0		
iv)	Reducing sugar/ Total Sugar ratio	0.10 to 0.15		

### TABLE 2- Sugar composition of Kithul jaggery

#### **5.6.3** *Amino acid profile*

Analysis of the amino acid shall be carried out by HPLC method described in Appendix **H**. The chromatogram (Figure 1) shall indicate the representative and characteristic components given in Table 3.

Forpholic

DSLS: .....

SI No	Amino acid	Detectability		
(1)	(2)	(3)		
i)	Alanine (ALA)	Detected		
ii)	Arginine (ARG)	Detected		
iii)	Aspartine (ASP)	Detected		
iv)	Asperagine (ASN)	Detected		
v)	Cystine (CYS)	Detected		
vi)	Glutamine (GLU)	Detected		
vii)	Glycine (GLY)**	Not Detected		
viii)	Histidine (HIS)	Detected		
ix)	Isoleucine* (ISO)	Detected		
x)	Leucine (LEU)	Detected		
xi)	Lysine* (LYS)	Detected		
xii)	Methionine* (MET)	Detected		
xiii)	Phenylalanine (PHE)	Detected		
xiv)	Proline (PRO)	Detected		
xv)	Serine (SER)	Detected		
xvi)	Threonine (THR)	Detected		
xvii)	Tyrosine (TRY)	Detected		
xviii)	Valine (VAL)**	Not Detected		

 TABLE 3- Free amino acid detected in Kithul jaggery

\* Unique peaks for *kithul* jaggery

\*\* Detected in coconut and palmyrah jaggery

### NOTE

*The chromatographic analysis is normative, contrary to typical chromatogram* (Figure 1) *given for information in Appendix* **H**.

# 6 CONTAMINANTS

### 6.1 **Potentially toxic elements**

*Kithul* jaggery shall not exceed the limits for potentially toxic elements given in Table 4 when tested in accordance with the methods prescribed in Column 4 of the table.

SI No	Potentially toxic element	Limit	Method of test
(1)	(2)	(3)	(4)
i)	Arsenic as As, mg/ kg, max.	0.5	AOAC 986.15/AOAC 2013.06
ii)	Lead as Pb, mg/ kg, max.	1.0	AOAC 999.10/AOAC 2013.06
iii)	Cadmium as Cd, mg/ kg, max.	1.0	AOAC 999.10/AOAC 2013.06

### **TABLE 4 - Limits for potentially toxic elements**

# 7 PACKAGING

The product shall be packaged in any suitable moisture proof (preferably vacuum packed) food grade containers or / packages with barrier properties for moisture which will safeguard the hygienic, and organoleptic qualities of the product. The containers including the packaging material shall be made of substances which are safe and suitable for intended use and shall not impart any toxic substances or undesirable odour or flavour to the product.

# 8 MARKING AND/ OR LABELLING

The following shall be marked and/ or labelled legibly and indelibly on each package:

- a) Name of the product as" *Kithul* jaggery";
- b) Brand name or trade mark;
- c) Net weight in grams or kg;
- d) Date of manufacture;
- e) Date of expiry;
- f) Batch number or code number or a decipherable code marking;
- g) List of ingredients if applicable; and
- h) Name and address of the manufacturer and packer/ distributor in Sri Lanka;

### 9 SAMPLING

Representative samples of the material shall be drawn as prescribed in Appendix A.

# **10 METHODS OF TEST**

Tests shall be carried out as prescribed in Appendix **B** to **G** of this Standard, American Association for Cereal Chemists Approved methods (AACC)  $11^{\text{th}}$  edition volume 2, 2010 and Methods of Analysis of the Association of Official Analytical Chemists (AOAC) 21st edition, 2019.

# 11 CRITERIA FOR CONFORMITY

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

11.1 Each container or package inspected as in A.5.1 shall satisfies the relevant requirements.

**11.2** Each individual sample tested as in **A.5.2** shall satisfies the relevant requirements.

**11.3** The composite sample tested as in **A.5.3** shall satisfies the relevant requirements.

### APPENDIX A SAMPLING

The sampling scheme given in Appendix  $\mathbf{A}$  should be applied where compliance of a lot to the requirements of this Standard is to be assessed based on statistical sampling and inspection.

Where compliance with this Standard is to be assured, appropriate schemes of sampling and inspection shall be adopted based on manufacturer's control systems coupled with type tests and testing procedures.

# A.1 LOT

In any consignment, all the containers or packages of same size containing *Kithul* jaggery drawn from a single batch of manufacture of a supply shall constitute a lot.

# A.2 GENERAL REQUIREMENTS OF SAMPLING

**A.2.1** The sampling instrument shall be clean and dry. The product being sampled, the sampling instrument and the containers or packages for samples shall be protected from adventitious contamination.

**A.2.2** To draw a representative sample, the contents of each container or package selected for sampling shall be mixed as thoroughly as possible by suitable means.

A.2.3 The samples shall be placed in clean, dry and air-tight glass or other suitable containers.

**A.2.4** Each sample container shall be sealed air-tight after filling and marked with the necessary details of sampling, the date of sampling and other important particulars of the consignment.

## A.3 SCALE OF SAMPLING

**A.3.1** Samples shall be tested from each lot for ascertaining conformity of the product to the requirements of this Standard.

**A.3.2** The number of containers or packages to be selected from the lot shall be in accordance with Table **5**.

**A.3.3** From each container or package selected as in **A.3.2**, draw number of blocks or pieces to form a sample not less than 300 g to represent that particular container or package.

**A.3.4** These samples shall be transferred to suitable containers as mentioned in **A.2.3**. They shall be sealed air-tight and marked with all details of sampling as mentioned in **A.2.4**.

A.3.5 The packages and blocks/ pieces shall be selected at random. To ensure randomness of selection, a random number table selected from **SLS 428** shall be used.

TABLE 5 – Scale of sampling

Number of packages	Number of packages to be		
in the lot	selected		
(1)	(2)		
Up to 50	3		
51 to 150	5		
151 to 500	8		
501 and above	13		

# A.4 PREPARATION OF SAMPLES

**A.4.1** From each container selected as in **A.3.3** and prepared as in **A.3.4**, draw a sufficient quantity of material using an appropriate sampling instrument to form an individual sample to represent a particular container or package in the sample. These individual samples shall be transferred to suitable containers as mentioned in **A.2.3**, sealed air-tight and marked with all details of sampling as mentioned in **A.2.4**.

**A.4.2** From all containers selected as in **A.3.3** and prepared as in **A.3.4**, a sufficient quantity of material shall be drawn using an appropriate sampling instrument and mixed together to form a composite sample to represent a homogeneous sample. The composite sample shall be transferred to a suitable container as mentioned in **A.2.3**, sealed air-tight and marked with all details of sampling as mentioned in **A.2.4**.

# A.5 NUMBER OF TESTS

**A.5.1** Each container or package selected as in **A.3.2** shall be inspected for packaging and marking and/ or labelling requirements.

A.5.2 Each individual sample drawn as in A.4.1 shall be tested for the requirements given in

Clause 5.2, 5.3, 5.4, 5.5 and 5.6.1.

A.5.3 The composite sample prepared as in A.4.2 shall be tested for requirements given in Clause 5.6.2, 5.6.3 and 6.

#### **APPENDIX B DETERMINATION OF MOISTURE CONTENT**

#### **B.1 PROCEDURE**

#### **B.1.1** Preparation of sample

Mince as quickly as possible, with a sharp edged knife or grind in a dry pestle and mortar, 150 g of the sample. Mince thoroughly to secure a uniform sample. Store the minced sample immediately in an air-tight glass container and use this wherever the use of prepared sample is indicated.

B.1.2 Weigh, to the nearest milligram approximately 5 g grams of the prepared sample (see B.1.1) in a tared and covered dish having a diameter of about 40 mm and a height of about 25 mm. Distribute the material as evenly as practicable over the bottom of the dish by gently side-wise movements. Place the dish in an oven, remove the cover of the dish and dry the material for 3 hours at  $103 \pm 2^{\circ}$ C. Cool the dish in the desiccator and weigh with the lid on. Heat again at  $103 \pm 2$  °C in the oven for 30 minutes. Cool the dish in the desiccator and weigh. Repeat this process of heating for 30 minutes, cooling and weighing till the difference in mass between two successive weighings is less than one milligram. Note the lowest mass.

**NOTE** - Preserve the dish containing this dried material for the determination of total ash.

# CALCULATION **B.2**

 $\frac{100}{m} = \frac{(m-m_1)}{m} \times 100$ Moisture, per cent by mass =

where

*m* is mass, in grams, of the prepared sample taken for the test; and mass, in grams, of the material after drying  $m_1$  is

#### APPENDIX C **DETERMINATION OF TOTAL ASH**

#### **C.1** PROCEDURE

Ignite the oven-dried material (see **NOTE** under **B.1.2**) in the dish, with the flame of a burner for about one hour (Ashless filter paper may be used to prevent frothing as overflowing during initial burning). Complete the ignition by keeping in a muffle furnace at a temperature not exceeding 525  $\pm$  25 °C until grey ash results. Cool in a desiccator and weigh. Heat again at  $525 \pm 25$  °C in the muffle furnace for 30 minutes. Cool in the desiccator and weigh. Repeat the process of heating for 30 minutes.

Cooling and weighing till the difference in mass between two successive weighings is less than one milligram. Note the lowest mass.

#### NOTE

Preserve the dish containing this ash for the determination of acid insoluble ash.

### C.2 CALCULATION

Total ash per cent, by mass on dry basis =  $\frac{m_2}{m} \times 100 \times \frac{100}{100 - M}$ 

where

- *m* is mass, in grams of the prepared sample taken for the test; and
- $m_2$  is mass, in grams of the ash.
- M is the water content of the sample

# APPENDIX D DETERMINATION OF ACID INSOLUBLE ASH

#### **D.1 REAGENT**

D.1.1 Dilute hydrochloric acid, approximately 5 N.

### D.2 PROCEDURE

To the ash content in the dish (see **NOTE** under **C.1**) add 25 ml of dilute Hydrochloric acid, cover with a watch glass and heat on a water bath for 10 minutes. Allow to cool and filter the contents of the dish through ashless filter paper No.42 or its equivalent. Wash the filter-paper with hot water until the washings are free from the acid and return it to the dish. Keep it in an air-oven maintained at  $103 \pm 2$  °C for about one hour. Ignite in a muffle furnace at  $525 \pm 25$  °C for one hour. Cool the dish in a desiccator and weigh. Heat again for 30 minutes, in the muffle furnace, cool and weigh. Repeat this process of heating for 30 minutes, cooling and weighing till the difference between two successive weighings is less than one milligram. Note the lowest mass.

## **D.3** CALCULATION

**D.3.1** Acid insoluble ash, per cent, by mass on dry basis =  $\frac{m_3}{m} \times 100 \times \frac{100}{100 - M}$ 

where,

m is mass, in grams, of the prepared sample taken for the test; and

 $m_3$  is mass, in grams, of the acid insoluble ash

M is water content of the sample

#### APPENDIX E DETERMINATION OF MATTER INSOLUBLE IN WATER

#### E.1 PROCEDURE

Weigh, to the nearest milligram, approximately 40 g of the prepared sample (see **B.1.1**). Boil gently with 200 ml of hot water for 30 minutes with periodical stirring. Filter through a funnel using a No. 41 or No. 1 or equivalent filter paper which has been previously dried at oven temperature, and weighed in a covered dish. Wash the residue thoroughly with hot water till it is free of sugar. Place the filter paper in the dish and dry in an oven maintained at  $103 \pm 2$  °C to a constant mass.

### E.2 CALCULATION

**E.2.1** Matter insoluble in water, per cent, by mass = 
$$\frac{m_5}{m_4} \times 100$$

where,

 $m_4$  is mass, in grams, of the sample taken for the test; and

 $m_5$  is mass, in grams, of the matter insoluble in water.

#### APPENDIX F DETERMINATION OF REDUCING SUGARS

### F.1 REAGENTS

#### **F.1.1** Standard dextrose solution

Weigh accurately 10 g of anhydrous dextrose into a one-litre graduated flask, dissolve it in water, and make up the volume to the mark with water.

Dilute a known aliquot of this solution of dextrose with water to such a concentration that more than 15 ml, but less than 50 ml of it will be required to reduce all the copper in the Fehling's solution taken for titration. Note the concentration of anhydrous dextrose in this solution as milligrams per 100 ml (see **NOTE**). Prepare this solution afresh every day.

## NOTE

When 10 ml of Fehling's solution are taken for titration, a standard dextrose solution containing 0.11 per cent to 0.30 per cent (m/v) of anhydrous dextrose is convenient for use.

**F.1.2** *Methylene blue indicator solution* 

Dissolve 0.2 gram of methylene blue in water and dilute to 100 ml.

# **F.1.3** *Fehling's solution*

Prepared by mixing immediately before use, equal volumes of Solution A and Solution B.

### F.1.3.1 Solution A

Dissolve 34.639 g of Copper sulphate (CuSO<sub>4</sub>.5H<sub>2</sub>O) in water, add 0.5 ml of concentrated Sulphuric acid of relative density 1.84 and dilute to 500-ml in a graduated flask. Filter the solution through prepared asbestos.

### **F.1.3.2** Solution B

Dissolve 173 g of Rochelle salt [Potassium sodium tartrate ( $KNaC_4H_4O_6.4H_2O$ )] and 50 g of Sodium hydroxide, analytical reagent in water, dilute to 500 ml in a graduated flask and allow the solution to stand for two days. Filter the solution through prepared asbestos.

# F.1.3.3 Standardization of Fehling's solution

Pour standard dextrose solution (see F.1.1) into a 50-ml burette (see NOTE 3 under F.2.3). Find the titre (that is the volume of standard dextrose solution required to reduce all the copper in 10 ml of Fehling's) corresponding to the concentration of standard dextrose solution from Table 4. (If, for example; the standard dextrose solution contains 167.0 mg of anhydrous dextrose per 100 ml, the corresponding titre would be 30 ml). Pipette 10 ml (see F.3.1.1) Fehling's solution into a 300- ml conical flask and run in from the burette almost the whole of the standard dextrose solution required to effect reduction of all the copper, so that not more than one milliliter will be required later to complete the titration. Heat the flask containing the mixture over a wire gauze. Gently boil the contents of the flask for 2 minutes. At the end of 2 minutes of boiling, add, without interrupting boiling, one milliliter of methylene blue indicator solution. While the contents of the flask continue to boil, begin to add standard dextrose solution (one or two drops at a time) from the burette till the blue colour of the indicator just disappears. (The titration should be completed within one minute, so that the contents of the flask boil altogether for 3 minutes without interruption (see NOTE 2 under F.2.3). Note the titre (that is the total volume in millilitres of standard dextrose solution used for the reduction of all the copper in 10 ml of Fehling's solution). Multiply the titre (obtained by direct titration) by the number of milligrams of anhydrous dextrose factor. Compare this factor with the dextrose factor given in Table 6 and determine correction, if any, to be applied to the dextrose factor derived from Table 6.

EXAMPLE :

dextrose in standard dextrose solution	=	167.0
Titre, in ml, obtained by direct titration	=	30.1
Dextrose factor for 30.1 ml of standard dextrose solution	=	Titre in ml $\times$ number of mg of anhydrous dextrose in one milliliter of standard dextrose solution
	=	$30.1 \times 1.670$ 50 2670
Dextrose factor for 30.1 ml of standard dextrose solution from Table <b>4</b> (calculated by		30.2070
interpolation)	=	50.11
Correction to be applied to the dextrose factor		XS
derived from Table 4	=	50.2670 - 50.11
		$\downarrow 0.1570 \lor $

Concentration, in mg/100ml, of anhydrous

#### F.2 **PROCEDURE**

#### F.2.1 Preparation of solution

Weigh, to the nearest milligram approximately five grams of the prepared sample (see **B.1.1**). Transfer to a standard 250 ml flask. Dissolve in water and make up to the mark with water.

### F.2.2 Incremental method of titration

Pour the prepared solution (see F.2.1) into a 50-ml burette (see NOTE 3 below F.2.3). Pipette 10 ml of Fehling's solution into a 300 ml conical flask and run in from the burette 15 ml of the prepared solution. Without further dilution, heat the contents of the flask over a wire gauze, and boil. (After the liquid has been boiling for about 15 seconds it will be possible to judge if almost, all the copper is reduced by the bright red colour imparted to the boiling liquid by the suspended cuprous oxide). When it is judged that nearly all the copper is reduced, add one milliliter of methylene blue indicator solution (see NOTE 1). Continue boiling the contents of the flask for one to two minutes from the commencement of ebullition, and then add the prepared solution in small quantities (one milliliter or less at a time), allowing the liquid to boil for about 10 seconds between successive additions, till the blue colour of the indicator just disappears (see NOTE 2 below F.2.3). In case there still appears to be much unreduced copper, after the mixture of Fehling's solution with 15 ml of the prepared solution, has been boiling for 15 seconds, add the prepared solution from the burette in larger increments (more than one milliliter at a time according to judgement), and allow the mixture to boil for 15 seconds after each addition. Repeat the addition of the prepared solution at intervals of 15 seconds until it is considered unsafe to add a large increment of the prepared solution. At this stage, continue the boiling for an additional one or two minutes, add one milliliter of methylene blue indicator solution and complete the titration by adding the prepared solution in small quantities (less than one milliliter at a time (see also Note 2).

## NOTES

1. It is advisable not to add the indicator until the end point has been nearly reached because the indicator retains its full colour until the end point is almost reached and thus gives no warning to the operator to go slowly.

2. When the operator has had a fair amount of experience with the method, a sufficiently accurate result may often be obtained by a single estimation by the incremental method of titration. For the utmost degree of accuracy of which the method is capable, a second titration should be carried out by the standard method of titration (see F.2.3).

### F.2.3 Standard method of titration

Pipette 10 ml of Fehling's solution into a 300 ml conical flask and run in from the burette almost the whole of the prepared solution required to effect reduction of all the copper (determined under **F.2.2**), so that, if possible, not more than one milliliter will be required later to complete the titration. Gently boil the contents of the flask for two minutes.

At the end of two minutes of boiling, add without interrupting boiling, one milliliter of methylene blue indicator solution. While the contents of the flask continue to boil, start adding the prepared solution (one or two drops at a time) from the burette till the blue colour of indicator just disappears (see NOTE 1). (The titration should be completed within one minute so that the contents of the flask boil altogether for three minutes without interruption (see NOTE 2).

In case of doubt, the flame may be removed from the wire gauze for one or two seconds and the flask held against a sheet of white paper. (A hold of paper, suitably fixed around the neck of the flask is very convenient for this purpose as it can be left round the neck of the flask without the risk of overbalancing it). The top edge of the liquid would appear bluish if the indicator is not completely decolourized. It is inadvisable to interrupt the boiling for more than a few seconds as the indicator undergoes back oxidation rather rapidly when air is allowed free access into the flask, but there is no danger of this as long as a continuous stream of steam is issuing from the mouth of the flask.

# NOTES

1. The indicator is so sensitive that it is possible to determine the end point within one drop of the prepared solution in many cases. The complete decolouration of the methylene blue is usually indicated by the whole reaction liquid, in which the cuprous oxide is continuously churned up, becoming bright red or orange in colour.

2. It should he observed that with both incremental and standard methods of titration, the flask containing the reaction mixture is left on the wire gauze over the flame throughout the titration, except when it may be removed for a few seconds to ascertain if the end point is reached.

3. In adding sugar solution to the reaction mixture, the burette may be held in hand over the flask. The burette may be fitted with a small outlet tube bent twice at right angles, so that the body of the burette can be kept out of the steam while adding sugar solution. Burettes with

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glass taps are unsuitable for this work, as the taps become heated by the steam and thus are liable to jam.

# F.3 CALCULATION

**F.3.1** Refer Table 6 for the dextrose factor corresponding to the titre (determined as given under **F.2.3**) and apply the correction previously determined under **F.1.3.3**. Calculate the dextrose content of the prepared solution (see **F.2.1**) as follows:

Milligrams of anhydrous dextrose present in one milliliter of the  $m_6 = \frac{dextrose \ factor}{titre}$ 

**F. 3.1.1** Instead of using 10 ml of Fehling's solution, a 25-ml portion may also be substituted throughout the procedure (including standardization of Fehling's solution' under **F.I.3.3**).

In this case, the standard dextrose solution, used in standardizing the Fehling's solution and the prepared solution of the material (see **F.2,1**) shall contain 0.25 per cent to 0.75 per cent (m/v) of anhydrous dextrose, and Table **5** shall be used for all calculations.

**F.3.2** Reducing sugars, (as Invert) per cent by mass =  $\frac{m_6}{2} \times 25$ 

where

 $m_6$  is milligrams of reducing sugar in 1 ml of the solution of the material (see **F.3.1**); and  $m_7$  is mass, in grams, of the prepared sample used for making 250 ml of solution (see **F.2.1**).

#### APPENDIX G DETERMINATION ON NON- REDUCING SUGARS

# G.1 REAGENTS

**G.1.1** *Concentrated Hydrochloric acid* 

Relative density 1.16, of analytical reagent grade.

G.1.2 Fehling's solution

Prepared by mixing immediately before use, equal volumes of Solution A and Solution B.

G.1.2.1 Solution A

Prepared as in F.I.3.1

G.1.2.2 Solution B

Prepared as in F.1.3.1

# G.2 PROCEDURE

Take 25 ml of the prepared solution (see **F.2.1**) in a conical flask and add 2.5ml of the concentrated hydrochloric acid and about 10ml water. Keep the flask at 60  $^{\circ}$ C to 70  $^{\circ}$ C for 10 minutes in a water bath. Cool immediately and neutralize with 30 per cent sodium hydroxide (m/ v) and transfer quantitatively the neutralized inverted solution to a graduated flask and make up the volume to 250ml.

Determine the reducing sugars in the inverted solution as described in **F**.

# G.3 CALCULATION

**G.3.1** Sucrose, per cent, by mass = (T-R) 0.95

where,

$$\mathbf{T} = \frac{m_9}{m_8} \times 10$$

R is reducing sugars, per cent by mass (see **F.3.2**)

 $m_8$  is mass, in grams, of the original material taken for the test (see **F.2.1**); and

 $m_9$  is milligrams of total reducing sugars in 1 ml of the inverted solution.

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ml of	Dextrose	mg dextrose		ml of	Dextrose	mg dextrose
Sugar	Factor*	per 100 ml		Sugar	Factor**	per 100 ml
Solution		•		Solution		•
Required				Required		
(1)	(2)	(3)		(1)	(2)	(3)
15	49.1	237		15	120.2	801
16	49.2	307		16	120.2	751
17	49.3	289		17	120.2	707
18	49.3	274		18	120.2	448
19	49.5	260		19	120.3	633
20	49.5	247.4		20	120.3	601.5
21	49.5	235.4		21	120.3	572.9
22	49.6	225.5		22	120.4	547.6
23	49.7	216.1		23	120.4	523.6
24	49.8	207.4		24	120.5	501.9
25	49.8	199.3		25	120.5	482.0
26	49.9	191.8		26	120.6	463.7
27	49.9	184.9		27	120.6	446.8
28	50.0	178.5		28	120.7	431.1
29	50.0	172.5		29	120.7	416.4
20	50.1	167.0			100.0	400.7
30	50.1	167.0		30	120.8	402.7
31	50.2	161.8		31	120.8	389.7
32	50.2	156.9	$\sim$	32	120.8	377.6
33	50.3	152.4	Y	33	120.9	300.3
34	50.3	148.0		34	120.9	355.6
35	50.4	143.9		35	121.0	345.6
36	50.4	140.0		36	121.0	336.3
30	50.5	136.4		37	121.0	327.4
38	50.5	132.9		38	121.1	318.8
30	50.5	129.6		30	121.2	310.7
57	50.0	127.0		57	121.2	510.7
40	50.6	126.5		40	121.2	303.1
41	50.7	123.6		41	121.3	295.9
42	50.7	120.8		42	121.4	289.0
43	50.8	118.1		43	121.4	282.4
44	50.8	115.5		44	121.5	276.1
15	50.0	112.0		15	101 5	270.1
43	50.9	115.0		43	121.3	2/0.1
40 47	51.0	110.0		40	121.0	204.3
4/	51.0	106.4		4/	121.0	230.0 252.5
48	51.0	100.2		48	121./	233.3
49	51.0	104.1		49	121.7	240.4
50	51.1	102.2		50	121.8	243.6

# TABLE 6 – Dextrose table for 10 mlof Fehling's solution

# TABLE 7 – Dextrose table for 25 ml of Fehling's solution

\*mg of dextrose corresponding to 10 ml of Fehling's solution.

\*\*mg of dextrose corresponding to 25 ml of Fehling's solution.

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			Solutions containing besides invert sugar							
ml of sugar	No sucrose		1 g sucrose per 100 ml 5 g sucrose per 100 ml		100 ml	10 g sucrose per 100 ml		25 g sucrose per 100 ml		
solution	Invert	mg	Invert	mg	Invert	mg	Invert	mg	Invert	mg
required	Sugar	Invert	Sugar	Invert	Sugar	Invert	Sugar	Invert	Sugar	Invert
(1)	Factor	Sugar per	Factor	Sugar per	Factor	Sugar per	Factor	Sugar per	Factor	Sugar per
		100 ml		100 ml		100 ml		100 ml		100 ml
	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
15	50.5	336	49.9	333	47.6	317	46.1	307	43.4	289
16	50.6	316	50.0	312	47.6	297	46.1	288	43.4	271
17	50.7	298	50.1	295	47.6	280	46.1	271	43.4	255
18	50.8	286	50.1	278	47.6	264	46.1	256	43.3	240
19	50.8		50.2	264	47.6	250	46.1	243	43.3	227
20	50.0	254.5	50.2	251.0	17.6	228.0	16.1	220.5	12.2	216
20	51.0	234.3	50.2	231.0	47.0	236.0	40.1	230.3	43.2	210
21	51.0	242.9	50.2	239.0	47.0	220.7	40.1	219.5	43.2	200
22	51.0	231.0	50.3	220.2	47.0	210.4	40.1	209.5	43.1	190
23	51.2	212.2	50.3	210.7	47.0	108.3	40.1	102.1	43.0	170
24	51.2	213.3	50.5	209.8	47.0	190.5	40.1	192.1	42.9	1/9
25	51.2	204.8	50.4	201.6	47.6	190.4	46.0	184.0	42.8	171
26	51.3	197.4	50.4	198.8	47.6	183.1	46.0	176.9	42.8	164
27	51.4	190.4	50.4	186.7	47.6	176.4	46.0	170.4	42.7	158
28	51.4	183.7	50.5	180.2	47.7	170.3	46.0	164.5	42.7	152
29	51.5	177.6	50.5	174.1	47.7	164.5	46.0	158.6	43.6	147
30	51.5	171.7	50.5	168.3	47.7	159.0	46.0	153.3	42.5	142
31	51.6	166.3	50.6	163.1	47.7	153.9	45.9	148.1	42.5	137
32	51.6	161.2	50.6	158.1	47.7	149.1	45.9	143.4	42.4	132
33	51.7	156.6	50.6	153.3	47.7	144.5	45.9	139.1	42.3	128
34	51.7	152.2	50.6	148.9	47.7	140.3	45.8	134.9	42.2	124
35	51.8	147.9	50.7	144 7	47 7	136.3	45.8	130.9	42.2	121
36	51.8	143.9	50.7	140.7	47.7	132.5	45.8	127.1	42.1	117
37	51.9	140.2	50.7	137.0	47.7	128.9	45.7	123.5	42.0	114
38	51.9	136.6	50.7	133.5	47.7	125.5	45.7	120.3	42.0	111
39	52.0	133.3	50.8	130.2	47.7	122.3	45.7	117.1	41.9	107
40	52.0	130.1	50.8	127.0	47.7	119.2	45.6	114.1	41.8	104
41	52.1	127.1	50.8	123.9	47.7	116.3	45.6	111.2	41.8	102
42	52.1	124.2	50.8	121.0	47.7	113.5	45.6	108.5	41.7	99
43	52.2	121.4	50.8	118.2	47.7	110.9	45.5	105.8	41.6	97
44	52.2	118.7	50.9	115.6	47.7	108.4	45.5	103.4	41.5	94
45	52.3	116.1	50.9	113.1	47.7	106.0	45.4	101.0	41.4	92
46	52.3	113.7	50.9	110.6	47.7	103.7	45.4	98.7	41.4	90
47	52.4	111.4	50.9	108.2	47.7	101.5	45.3	96.4	41.3	88
48	52.4	109.2	50.9	106.0	47.7	99.4	45.3	94.3	41.2	86
49	52.5	107.1	51.0	104.0	47.7	97.4	45.2	92.3	41.1	84
50	52.5	105.1	51.0	102.0	47.7	95.4	45.2	90.4	41.0	82

ml sugar solution	Solution containing besides invert sugar									
required	No suc	rose	1 g sucrose per 100ml							
-	Invert sugar factor*	mg invert sugar	Invert sugar	mg invert sugar						
	(2)	per 100 ml	factor*	per 100 ml						
(1)		(3)	(4)	(5)						
15	123.6	824	122.6	817						
16	123.6	772	122.7	767						
17	123.6	727	122.7	721						
18	123.7	687	122.7	682						
19	123.7	651	122.8	646						
20	123.8	619.0	122.8	614.0						
21	123.8	589.5	122.8	584.8						
22	123.9	563.2	122.9	558.2						
23	124.9	538.7	122.9	534.0						
24	124.0	516.7	122.9	512.1						
25	124.0	496.0	123.0	492.0						
26	124.1	477.3	123.0	473.1						
27	124.1	459.7	123.0	455.6						
28	124.2	443.6	123.1	439.6						
29	124.2	428.3	123.1	424.4						
30	124.3	414.3	123.1	410.4						
31	124.3	401.0	123.2	397.4						
32	124.3	388.7	123.2	385.0						
33	124.3	377.0 01	123.2	373.4						
34	124.5	366.2	123.3	362.6						
		JO'								
35	124.5	355.8	123.3	352.3						
36	124.6	346.1	123.3	342.5						
37	124.6	336.8	123.4	333.5						
38	124.7	328.1	123.4	324.7						
39	124.7	319.7	123.4	316.4						
40	124.8	311.9	123.4	308.6						
41	124.8	304.4	123.5	301.2						
42	124.9	297.3	123.5	294.1						
43	124.9	290.5	123.5	287.3						
44	125.0	284.1	123.6	280.9						
45	125.0	277.9	123.6	274.7						
46	125.1	272.0	123.6	268.7						
47	125.1	266.3	123.7	263.1						
48	125.2	260.8	123.7	257.7						
49	125.2	255.5	123.7	252.5						
50	125.3	250.6	123.8	247.6						

# TABLE 8 – Invert sugar table for 25 ml of Fehling's solution

\*mg of invert sugar corresponding to 25 ml of Fehling's solution.



FIGURE 1 - Amino acids chromatogram of Kithul jaggery