PS:1822/SARS 0007-2021
(ICS No. 67.180.10)

PAKISTAN STANDARD SPECIFICATION

FOR

REFINED SUGAR

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PAKISTAN STANDARDS AND QUALITY CONTROL AUTHORITY
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0. FOREWORD

0.1 This Pakistan Standard Specification was adopted by the Pakistan Standards & Quality Control Authority on **03-03-2021** after the finalized by the Sugar Industries Technical Committee had been approved by the National Standards Committee for Agriculture & Food Products.

0.2 In order to keep abreast of the Progress in Industry the Pakistan Standards are subject to periodical review suggestions for improvement shall always be welcomed and put to the relevant committee for its consideration.

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SAARC STANDARD

SARS 0007:2017

First edition
01-03-2017

REFINED SUGAR — SPECIFICATION

ICS 67.180.10

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FOREWORD

The South Asian Regional Standards Organization (SARSO) is a Specialized Body of South Asian Association for Regional Cooperation (SAARC) aimed to achieve and enhance coordination and cooperation among SAARC Member States in the fields of standardization and conformity assessment and to develop harmonized Standards for the South Asian region to facilitate intra-regional trade and to have access in the global market. The Member States of SAARC are Afghanistan, Bangladesh, Bhutan, India, Maldives, Nepal, Pakistan and Sri Lanka. The Agreement on the establishment of SARSO entered into force with effect from 25 August 2011 after ratification by all Member States of SAARC.

The National Standards Bodies of the SAARC Member States participate in the development of SAARC Standards (SARS) through the Sectoral Technical Committees (STCs). The SARS are developed through consensus and are drafted in accordance with the editorial rules of the SARSO Directives, Part 2.

The SARSO Secretariat is the guardian of the authoritative versions of the SAARC Standards and is responsible for keeping master texts of SAARC Standards, both in hard and soft form. The Member States are responsible for making SAARC Standards available for sale, distribution, etc, at the national level. In accordance with the 'SAARC Agreement on Implementation of Regional Standards', the approval of a SAARC Standard implies that Member States have an obligation to give it the status of a National Standard.

This SAARC Standard was considered by the Technical Management Board and approved by the Governing Board of SARSO on the recommendation of the STC-01: Food and Agricultural Products.

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INTRODUCTION

Refined sugar is manufactured from any type of sugar or sugar cane or sugar beet, in general by a process of purification, consisting broadly of affination, melting, chemical treatment, filtration, decolourization and subsequent recrystallization in vacuum pan, the treatment depending upon the nature of the initial material. It may be of any grain size (large, medium or small).

This Standard is aligned with the Codex Standard for Sugars, CODEX STAN 212 - 1999 and the methods of test have been aligned with the International Commission for Uniform Methods of Sugar Analysis (ICUMSA).

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, as per the relevant national Standards of the respective Member State. The number of significant places retained in the rounded off value should be the same as that of the specified value in the relevant referred Standard

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SAARC Standard

REFINED SUGAR — SPECIFICATION

1 SCOPE

This Standard specifies the requirements and the methods of sampling and test for refined sugar.

2 NORMATIVE REFERENCES

The Standards referred in the text, if any, contain provisions which through reference in this text constitute provisions of this Standard. 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 most recent editions of the Standards referred in the text.

3 TERMS AND DEFINITIONS

For the purposes of this Standard, the following term and definition apply.

3.1 Refined Sugar

Purified and crystallized sucrose (saccharose)

4 REQUIREMENTS

4.1 Description

Refined sugar shall be crystalline, white, odourless and free from dirt, iron fillings and other extraneous matter.

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

4.3 Refined sugar shall be manufactured, packed, stored and distributed under hygienic conditions as detailed in the respective National Standards. In case such National Standards are not available, the Codex Alimentarius Commission Standards on the hygienic conditions shall be followed.

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Table 1 - Requirements for Refined Sugar
(*Clause* 4.2)

Sl. No.	Characteristics	Requirement	Method of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Loss on drying, percent by mass, *Max*	0.10	B
ii)	Polarization, *Min*	99.7°Z	C
iii)	Reducing sugar, percent by mass, *Max*	0.04	D
iv)	Colour in ICUMSA units, *Max*	60	E
v)	Conductivity ash, percent by mass. Max	0.04	F
vi)	Sulphur dioxide, mg/kg, *Max*	15	G
vii)	Lead, mg/kg, *Max*	0.5	AOAC 999.10 H (alternative method)
viii)	Chromium, mg/kg, *Max*	20	I
ix)	Copper, mg/kg, *Max*	2	AOAC 960.40 or AOAC 999.10

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x)	Arsenic mg/kg, *Max*	1	AOAC 986.15 or ICUMSA , GS2/3-23 (2005)

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5 PACKAGING

Refined sugar shall be packaged in clean, sound and new jute bags or bags made of polypropylene or bags made of high density polypropylene made of food grade material. The jute bags may be lined with polyethylene film. The mouth of each bag shall be either machine-stitched or rolled over and hand-stitched. If hand-stitched, the stitches shall be in two rows with at least 14 stitches in each row. In the case of smaller consumer packs, the product shall be packed in food grade plastics or any other suitable non-toxic material.

6 MARKING/LABELLING

6.1 Each bag/pack shall bear legibly and indelibly the following particulars:

a) name of the product;

b) brand name or Trade name, if any;

c) name and address of the manufacturer including the country of origin;

d) in case of imports, the name and address of importer;

e) net mass of sugar;

f) month and year of manufacture;

g) batch or Code number;

h) the words ‘Best before . . . .’ (month and year to be indicated); and

i) any other markings as specified as per respective national regulations.

6.2 Certification Marking

The product may also be marked with the Standard Mark of conformity.

7 SAMPLING

Representative samples of refined sugar shall be drawn and the criteria for conformity to this Standard shall be established, according to the method prescribed in Annex A. In case of consumer packs, samples of refined sugar shall be drawn and the criteria for conformity to this Standard shall be established.

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Annex A
(Normative)
(*Clause* 7)

SAMPLING OF REFINED SUGAR

A-1 General Requirements

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

A-1.1 The sample shall be taken in a protected place not exposed to damp air, dust or soot.

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

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

A-1.4 The samples shall be placed in clean, dry and moisture-proof containers.

A-1.5 The sample containers shall be sealed air-tight after filling and marked with full details of sampling, that is, name of the material, the date of sampling, month and year of manufacture, name of the producer, name of the person carrying out the sampling and such other particulars as considered necessary.

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A-2 Scale of Sampling

A-2.1 Lot

All the bags in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the group of bags in each batch shall constitute separate lots.

A-2.1.1 Each lot shall be tested for ascertaining the conformity of refined sugar to the requirement of this Standard.

A-2.2 The number of bags to be selected for sampling shall be in accordance with column 2 and 3 of Table 2.

Table 2 - Number of Bags to be selected for Sampling
***(Clause* A-2.2)**

Sl. No.	No. of Bags in each Lot	No. of Bags to be Selected
(1)	(2)	(3)
i)	2-25	2
ii)	26-100	3
iii)	101-500	5
iv)	501-1000	7
v)	1001 and above	8

A-2.3 These bags shall be selected at random from the lot.

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A-3 Test Samples and Referee Sample

From the top, middle and bottom portions of each of the selected bags (*see* A-2) approximately equal quantity of the sugar shall be taken with the help of a suitable sampling instrument. The sample collected from each of the bags shall be mixed thoroughly to constitute a composite sample of 600 g. The composite sample thus prepared shall be divided approximately into three equal parts; one for the purchaser, one for the supplier and the third for the referee; and sealed air-tight with the particulars as given in A-1.5.

A-4 Number of Tests

The composite sample prepared as under A-3 shall be tested for the characteristics as prescribed in Table 1.

A-5 Criteria for Conformity

The lot shall be declared as conforming to this Standard when the test results on various characteristics obtained on the composite sample satisfy the corresponding requirements as specified in clause 4 and Table 1. When examined each pack shall conform packaging and marking/labeling requirements.

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Annex B
(Normative)
[*Clause* 4.2, Table 1, Sl. No. (i)]
DETERMINATION OF LOSS ON DRYING

B-1 Field of Application

The method is applicable to various types of sugar and sugar products.

B-2 Definition

B-2.1 Loss on Drying — Because water represents the primary heat-volatile liquid in both cane and beet processing it is certainly the main volatile component lost on drying sugar. The matter lost on drying in this method is referred to as 'moisture' or water. Moisture in sugar is considered to be present in three forms:

a) Free moisture, being that contained on the surface of the crystal which is easily and quickly removed on drying.

b) Bound moisture, being that contained in the glassy layer on the surface and in the reentrant angles which is only released slowly as the glass crystallizes.

c) Inherent moisture, this being moisture included within the crystal structure and only released in general by grinding.

B-3 Principle

The principle of the method is oven drying using the atmospheric pressure oven technique (105°C) followed by standardized conditions for cooling after oven drying. It is mainly free moisture which is estimated by this method.

B-4 Apparatus

B-4.1 Forced Draught Atmospheric Pressure Oven, maintained at a temperature of 105°C ± 2°C as measured at a distance of 2.5 cm ± 0.5 cm above the dishes in the test. The oven is to be ventilated and the circulation fan faced with an inter-lock switch which opens when the oven door is opened.

B-4.2 Desiccator, containing self-indicating silica gel.

B-4.3 Dishes with Tight Fitting Lids — These should have a diameter of 6cm to 10cm and a depth of 2 cm to 3 cm. Although they may be made of glass, platinum or nickel, aluminum is recommended. The thickness of the dishes is optional except that due regard should be paid to the weight of the dish in relation to the weight of the sample and to the loss to be determined.

B-4.4 Clean Dry Duster

B-4.5 Surface Pattern Dial Thermometer — An electronic thermometer may be used provided it is fitted with a surface probe.

B-4.6 Analytical Balance — Capable of weighing to the nearest 0.1 mg.

B-5 Procedure

B-5.1 Drying

Carry out the determination in duplicate and preheat the oven to 105°C. Place the empty dishes with lids open in the oven for not less than 30 min. using the duster for handling them, remove the dishes from the oven,

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replace the lids and place in the desiccator. Place the contact thermometer on top of one of the dishes. When the temperature of the dishes has fallen to ambient ± 2°C, weigh them as rapidly as possible to an accuracy of ± 1 mg. As rapidly as possible, place 20 g to 30 g of the sample into each dish, replace the lids and weigh the dish and contents to an accuracy of 0.1 mg. Return the dishes with the lids open to the oven. Their position in the oven will be governed by the requirements of B-4.1. Dry the sample for 3 h exactly. Ensure that there are no other materials in the oven during the drying period.

NOTE: The depth of the sugar in the dish must not exceed 1 cm.

B-5.2 Weighing to Determine Loss on Drying

Replace the lids, remove the dishes from the oven and place in the desiccator with the contact thermometer on one of them. Cool the dishes, until the thermometer indicates a temperature of ambient ± 2°C. Weigh the dishes to an accuracy of 0.1 mg.

NOTE: No attempt should be made to dry to constant weight and care must be taken to ensure that there is no physical loss of sugar at any stage. Dishes should always be held with a clean, dry duster.

B-6 Expression of Results

B-6.1 Calculation of Loss on Drying

Loss in mass is expressed as a percentage or the original oss in mass is expressed as a percentage of the original mass of the sample, that is:

$$\text{Loss on drying, percent by mass} = \frac{100(m_2-m_3)}{m_2-m_1}$$

where

m_1 is the mass of dish, in g;
m_2 is the mass of dish with sugar before drying, in g; and
m_3 is the mass of dish with sugar after drying, in g.

Duplicate results are acceptable if neither is outside the limits of ± 10 percent of the mean value for the test. Tests in which either duplicate exceeds this limit should be repeated.

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Annex C
(Normative)
[*Clause* 4.2, Table 1, Sl. No. (ii)]

DETERMINATION OF POLARIZATION

C-1 Field of Application

The method is applicable to plantation white sugar and refined sugar. The method is used in statutory analysis and is applicable to white sugars and other white refined products of low colour and turbidity, not needing clarification and having a loss on drying of not more than 0.1 percent. White sugars, the polarization of which cannot be measured without clarification, are to be measured according to method prescribed for raw sugars (*see 6*).

C-2 Apparatus and Glassware

C-2.1 Analytical Balance

C-2.2 Normal Weight, 26.000 g (brass weight) as weighed in air at 20°C.

C-2.3 Basin, made of nickel or German silver, large enough to hold the normal weight of sugar.

C-2.4 Volumetric Flask, of capacity 100 ml, calibrated at 20°C.

C-2.5 Long-Stemmed Funnel, with a stem long enough to extend below the neck of the volumetric flask.

C-2.6 Stemless Funnel, capable of holding 100 ml.

C-2.7 Glass Cylinder, capable of holding 100 ml.

C-2.8 Watch-Glass, large enough to cover the stemless funnel.

C-2.9 Saccharimeter, graduated in International Sugar Scale and provided with a 200 mm tube. The saccharimeter should be sheltered in a cabinet, the inside of which is maintained at 20°C. Where this is not possible, necessary corrections shall be applied, based on the characteristics of the particular instrument.

C-2.9.1 Standardization of saccharimeter scale — Saccharimeter scale must be graduated in conformity with International Sugar Scale adopted by ICUMSA. Rotations of this scale are designated as degrees sugar (^{o}S).To convert values in ^{o}S to values in ^{o}Z, multiply the ^{o}S value by the factor 0.999 71. Basis of calibration of 100^{o} point on international sugar scale is polarization of normal solution of pure sucrose (26.000 g/100 ml) at 20^{o}C in 200 mm tubes. This solution, polarized at 20^{o}, must give saccharimeter reading of exactly 100^{o}S.

C-2.9.2 Standardization of polarimeter scale – Polarimeter will be calibrated with traceable quartz plates for 100^{o}Z.

C-3 Reagent

C-3.1 Alumina Cream — Dilute 500 ml of 30 percent (w/v) aluminium sulphate solution to 2 litres. Add gradually 10 percent (w/v) ammonia solution to the above with constant stirring until no precipitate is formed. Allow the precipitate to settle and decant off the clear liquid. Shake the precipitate with 1 litre of distilled water and allow it to settle again for decantation of the clear liquid. Repeat the process of washing and decantation till the supernatant liquid is neutral to litmus and does not precipitate with barium chloride solution. Make the precipitate of the aluminium hydroxide into a thick paste with water and store it in a stoppered bottle.

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C-3.2 Lead Acetate

Dissolve 560 g of basic lead acetate powder in 1 litre of freshly boiled distilled water, which has been previously cooled in a sealed container. Boil for 30 minutes and allow to settle overnight in a sealed container. Decant the supernatant liquid and dilute with freshly boiled distilled water to 1.25 specific gravity (56° Brix).

C-4 Test Temperature

The polarization of the normal solution of sugar shall be carried out at 20°C, as far as possible.

C-5 Procedure

C-5.1 Preparation of the Solution

Weigh accurately on the analytical balance the normal weight of the sugar (*see* C-2.2) in the basin. Transfer this quantity to the volumetric flask with the aid of the long-stemmed funnel. Rinse the basin and the funnel with water, and transfer the washings to the volumetric flask taking care that the volume of the contents of the flask does not exceed 80 ml. Bring the sugar into solution by gently swirling the flask and clarify the solution by adding 1 ml to 2 ml of alumina cream until nearly all the impurities have been precipitated. Make up the volume to 100 ml at the test temperature (*see* C-4) with water. If difficulty in clarification is experienced, repeat the experiment using anhydrous lead sub-acetate in small quantities in place of alumina cream (about 0.6 g of lead sub-acetate is usually required; any excess of lead sub-acetate should be avoided).

C-5.2 Polarization of the Solution

Place the stemless funnel over the glass cylinder and fix a cone of dry filter paper, large enough to hold 100 ml, in the funnel. Pour the whole of the defecated solution on to the cone of dry filter paper and cover it immediately with a watch-glass large enough to cover the funnel. Reject the first 10 ml to 15 ml of the filtrate. Rinse the glass cylinder with a little quantity of the filtrate; discard the solution used for rinsing. Collect the remainder of the filtrate into the rinsed cylinder. Polarize the filtrate in the saccharimeter at 20°C, as far as possible (*see also* C-2.1)**,** using a 200 mm tube. The reading gives the polarization percent of the sugar.

C-5.2.1 If the polarization is done at a temperature other than 20°C, the saccharimeter reading shall be corrected using the following formula (*see* Note):

$$P^{20} = P^{t}[1+0.0003\ (t - 20)]$$

Where

P^{20} is saccharimeter reading, at 20°C,
P^{t} is saccharimeter reading, at t°C, and
t is temperature in °C at which the polarization is carried out.

NOTE: The formula is applicable only up to 30°C

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Annex D
(Normative)
[*Clause* 4.2, Table 1, Sl. No. (iii)]

DETERMINATION OF REDUCING SUGARS BY THE MODIFIED OFNER TITRIMETRIC METHOD

D-1 Field of Application

The method measures the reducing power of solutions of white sugar containing reducing substances, for example, invert sugar in a weak alkaline solution of a Cu^{++} complex with tartrate. The method is applicable to various types of sugar and sugar products.

D-2 Definition

D-2.1 Reducing Sugars — Mono and oligosaccharides containing a free aldehydic or ketonic group which show a reducing effect on certain oxidizing agents.

D-2.2 Invert Sugar — Equi-molar solution of glucose and fructose.

D-2.3 Reducing Substances — The sum of reducing sugars and other substances in sugar products defined by their reducing power on reagents which are used for the determination of reducing sugars. Like reducing sugars their amount is in most cases expressed as the equivalent amount of invert sugar, that is the amount of invert sugar which shows the same reducing power under the conditions of the reaction.

D-3 Principle

The complex formed between Cu^{++} ions and potassium sodium tartrate is reduced by reducing sugars to univalent Cu^{+} which is precipitated as Cu_2O. The precipitated Cu_2O is then determined by iodometric titration.

The Cu_2O is oxidized by an excess is back-titrated with sodium thiosulphate. The reaction between the reducing sugars and the Cu^{++} complex is not stoichiometric. The amount of Cu_2O formed depends upon the prescribed reaction conditions which therefore must be strictly followed. It has been determined that 1 ml of 0.016 15 mol/l iodine solution is equivalent to 1mg of reducing sugars, once the correction for the reducing effect of sucrose has been taken into account.

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D-4 Reagents and Materials

Use only distilled water or water of similar quality. All reagents should be of analytical grade of better unless stated.

D-4.1 Activated Carbon, powdered.

D-4.2 Small Pumice Pieces

D-4.3 Disodium Hydrogen Phosphate, $Na_2HPO_4.12H_2O$

D-4.4 Glacial Acetic Acid, M_{20} = 1.05 g/ml.

D-4.5 Acetic Acid Solution, approximately 5 mol/l.

D-4.6 Potassium Sodium Tartrate (Rochelle or Seignette Salt)

D-4.7 Copper Sulphate Pentahydrate, $CuSO_4.5H_2O$.

D-4.8 Sodium Carbonate, anhydrous.

D-4.9 Soluble Starch

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D-4.10 Hydrochloric Acid, approximately 1 mol/l.

D-4.11 Hydrochloric Acid, approximately 2 mol/l.

D-4.12 Ofner Solution, modified — Weigh out 7.0 g copper sulphate pentahydrate (*see* D-4.7*)* 10.0 g sodium carbonate (*see* D-4.8), 300 g potassium sodium tartrate (*see* D-4.6) and 50 g disodium hydrogen phosphate (*see* D-4.3) in a 1000 ml flask. Dissolve in approximately 900 ml water (heating slightly to dissolve if necessary). Heat the solution for 2 h in a boiling water bath. Cool down to room temperature and fill up to the mark. Add approximately 10 g activated carbon (*see* D-4.1) and stir for 5-10 min. Filter the solution (*see* D-5.11).

D-4.13 Potassium Iodate Solution, 0.016 67 mol/l — Weigh out 3.566 7 g potassium iodate, KIO_3. Transfer to a 1000 ml volumetric flask, dissolve in water and fill to the mark.

NOTE: Dry the potassium iodate for 3 h at 100°C before use.

D-4.14 Starch Solution Indicator for Iodine — Dissolve 1 g of soluble starch in 100 ml saturated sodium chloride solution. Bring the solution to the boil for few minutes.

D-4.15 Potassium Iodide, KI

D-4.16 Sodium Thiosulphate Solution, 0.033 3 mol/l — Dilute a 0.1 mol/l sodium thiosulphate solution three fold with water and standardize with potassium iodate. Dissolve 2 g of potassium iodide in 10 ml water. Add 5 ml of approximately 2 mol/l hydrochloric acid (*see D*-4.11) and 10 ml of 0.016 67 mol/l potassium iodate solution (*see D*-4.13). Cover the flask with a watch glass, shake gently and leave the solution in the dark for approximately 30 min. Titrate the iodine formed with the sodium thiosulphate solution to complete decolonization, adding 1 ml of starch indicator (*see D*-4.14) immediately before the end point. Calculate the factor f_{Th} of the thiosulphate solution:

$$f_{Th} = \frac{30.96}{V_{Th}}$$

where

V_{Th} is ml of sodium thiosulphate solution titrated.

NOTE: f_{Th} corrects the used iodine solution to the experimentally determined value of 0.016 15 mol/l, for which 1 ml corresponds to 1 mg reducing sugars.

D-4.17 Iodine Solution, 0.016 67 mol/l - Dilute a 0.05 mol/l iodine solution three-fold with water and standardize with the 0.033 3 mol/l sodium thiosulphate solution (*see* D-4.16). Pipette 25.0 ml of the iodine solution into a 300 ml Erlenmeyer flask. Add 5 ml of 5 mol/l acetic acid (*see* D-4.5) and, after gently shaking the mixture, titrate back with the 0.033 3 mol/l sodium thiosulphate solution (*see* D-4.16). Add 1 ml of starch indicator (*see* D-4.14) just before the endpoints is reached. Calculate the factor, f_1 of the iodine solution:

$$f_1 = \frac{V_{Th} \times f_{Th}}{25}$$

where

V_{Th} is ml of sodium thiosulphate solution titrated, and
f_{Th} is Correction factor for the sodium thiosulphate solution.

D-5 Apparatus and Glassware

D-5.1 Analytical Balance, capable of weighing to the nearest 0.1 mg.

D-5.2 Precision Balance, capable of weighing to the nearest 0.1 g.

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D-5.3 Burettes, capacity 50 ml,

D.5.4 Erlenmeyer Flasks, capacity 300 ml.

D-5.5 Volumetric Flasks, 1 000 ml and 200 ml.

D-5.6 Pipettes, capacities 1 ml, 15 ml and 50 ml.

D-5.7 Watch Glasses, to cover Erlenrneyer flasks.

D-5.8 Bunsen Burner, Tripod and Wire Gauze

D-5.9 Boiling Water Bath

D-5.10 Water Bath with Cold Running Water

D-5.11 Filter Paper

D-6 Procedure

D-6.1 Preparation of the Sample

The solution prepared for the determination should contain no more than 25 mg invert sugar in 50 ml. This requires that 40 g of white sugar be made up with water to 200 ml.

D-6.2 Hot Value

Mix 50.0 ml of the prepared solution (*see* D-6.1) with 50.0 ml of the Ofner solution (*see* D-4.12). Add some pumice pieces (*see* D-4.2) to the mixture. Bring the mixture to the boil within 4 to 5 min using the Bunsen burner, the tripod and the wire gauze. Boil for exactly 5 min. Note the start of boiling is once numerous steam bubbles break over the whole surface. Cool the mixture down in a water bath with cold running water. After approximately 10 min. the mixture should have reached room temperature. Add 1 ml concentrated acetic acid (*see* D-4.4). Add iodine solution (*see* D-4.17) until the colour of the mixture turns a typical iodine colour. This procedure dissolves the formed Cu_2O with an excess of iodine. The surplus iodine should be so high that between 10 ml and 15 ml of sodium thiosulphate (*see* D-4.16) are consumed on back titration. Add 15 ml of the 1 mol/l hydrochloric acid (*see* D-4.10) by pouring it down the inner side of the flask so that the residual droplets are washed down into the solution. Cover the flask with a watch glass and move it gently for 2 min until the precipitate of Cu_2O is completely dissolved. Titrate the sample with 0.033 3 mol/l sodium thiosulphate (*see* D-4.16). Add 1 ml of starch solution immediately before the endpoint is reached. Repeat the above procedure with another prepared solution mixed with Ofner solution and record the average of the two replicated V_1, and V_2 for iodine and thiosulphate respectively.

D-6.3 Cold Value

Mix 50.0 ml of the prepared sample (*see* D-6.1) with 50.0 ml of the Ofner solution (*see* D-4.12). Leave the mixture at room temperature for 10 min. Repeat the procedure outlined in D-6.2. Record values V_3 and V_4.

D-6.4 Blank Value

Mix 50 ml of water with 50 ml of the Ofner solution (*see* D-4.12). Repeat the procedure outlined in D-6.2. Record the values V_5 and V_6.

NOTE: It is essential that the time between addition of iodine solution and beginning of the back titration is equal for the hot value and the cold value.

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D-7 Expression of Results

D-7.1 Calculation of the Results

Added amount of iodine for hot value = V_1

Added amount of thiosulphate for hot value = V_2

Added amount of iodine for cold value = V_3

Added amount of thiosulphate for cold value = V_4

Added amount of iodine for blank value = V_5

Added amount of thiosulphate for blank value = V_6

Corrected consumption of 0.016 67 mol/l iodine solution:

Calculated hot value, $A = (V_1 \times f_1) – (V_2 \times f_{Th})$

Calculated cold value, $B = (V_3 \times f_1) - (V_4 \times f_{Th})$

Calculated blank value, $C = (V_5 \times f_1) - (V_6 \times f_{Th})$

where

f_1 is the factor of the iodine solution. Calculated in D-4.17 and

Sucrose correction, D, is 0.1 ml iodine solution/g of sucrose in the reaction mixture.

$$\text{Invert sugar, mg/kg} = \frac{(A - B - C - D) \times 1000}{s}$$

where

s is the amount of sample in 50 ml of prepared solution (*see* D-6.1)

D-7.2 Example Calculation

100 g white sugar is weighed out and diluted to 200 ml. 50 ml of this solution contains 25 g sucrose.

Amount of iodine solution added to hot value is 25.00 ml.

Amount of sodium thiosulphate consumed is 21.71 ml.

Amount of iodine solution added to cold value is 25.00 ml.

Amount of sodium thiosulphate consumed is 24.72 ml.

Amount of iodine added to the blank value is 25.00 ml.

Amount of sodium thiosulphate consumed is 25.00 ml.

f_{Th} is calculated to be 1.029

f_1 is calculated to be 1.031

$B = (25.0 \times 1.031) – (24.72 \times 1.029) = 0.34$

$A = (25.0 \times 1.031) – (21.71 \times 1.029) = 3.43$

$C = (25.0 \times 1.031) – (25.0 \times 1.029) = 0.05$

$D = 25.0 \times 0.1 = 2.50$

$$\text{Reducing sugar} = \frac{(3.43-0.34-0.05-2.50) \times 1000}{25} = 21.9 \text{ mg/kg}$$

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Annex E
(Normative)
[*Clause* 4.2, Table 1, Sl. No. (iv)] DETERMINATION OF SUGAR SOLUTION COLOUR

E-1 Field of Application

This method is used for the determination of sugar solution colour. The method can be applied to all crystalline or powdered white sugars provided that a filtered test solution can be prepared by the procedure specified in the method. The method is not suitable for those sugars which contain colouring matter, turbidity or additives to an extent that filtration is not practical.

E-2 Definitions

E-2.1 Transmittance of a Solution

If I_1 represents the radiant energy incident upon the first surface of the solution and I_2 represents the radiant energy leaving the second surface of the solution.
Then

$$\text{Transmittance of the solution, } T = \frac{I_2}{I_1}$$

where

100 T is percentage transmittance

E-2.2 Transmittancy

Let T_{soln}, represent the transmittance of a cell containing the solution and let T_{solv} represent the transmittance of the same or duplicate cell containing the pure solvent.
Then

$$\text{Transmittancy of the solution, } T_S = \frac{T_{soln}}{T_{solv}}$$

E-2.3 Absorbency (Extinction), *As*

$$\text{Absorbancy of the solution, } \blacksquare = -\log_{10} T_s$$

E-2.4 Absorbance Index (Extinction Index)

Let b represent the length, cm, of the absorbing path between the boundary layers of the solution and let *c* represent the concentration, g/ml, of the sugar solution.
Then,

$$\text{Absorbance index of the solution, } \blacksquare = \frac{\blacksquare}{\blacksquare\blacksquare}$$

E-2.5 International Commission for Uniform Methods of Sugar Analysis (ICUMSA) Colour

The value of the absorbance index multiplied by 1000 is reported as ICUMSA Colour. The resulting values are designated as ICUMSA Units (IU).

E-3 Principle

White sugar is dissolved in a distilled water to give a 50 percent sugar solution. The solution is filtered through a membrane filter to remove turbidity. The absorbance of the filtered solution is measured at a wavelength of 420 nm and the solution colour is calculated.

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E-4 Reagents

Use only distilled water or water of equivalent purity.

E-5 Apparatus

E-5.1 Instrument — Spectrophotometer or calorimeter capable of light transmission measurements at a wavelength of 420 nm with the narrowest practical bandwidth, for example ± 10 nm. The instrument should be fitted with a grating, prism or interference filter monochromator. Coloured glass of gelatin filters are not satisfactory.

E-5.2 Associated Optical Cells — Use a cell of at least 4 cm in length. A cell length of 10 cm or more is to be preferred for low colour white sugars. A second or reference cell may be used provided that a test with distilled water has shown that the two cells are within 0.2 percent of being identical.

E-5.3 Membrane Filters — Pore size 0.45 mm of cellulose based material.

E-5.4 Membrane Filter Holder — Preferably fitted with a stainless steel support.

E-5.5 Vacuum Oven, Vacuum Desiccator or Ultrasonic Bath, for de-aeration of the filtered sugar solution.

E-5.6 Refractometer

E-5.7 Laboratory Balance — Capable of weighing to the nearest 0.1 g.

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E-6 Procedure

E-6.1 Sample Preparation

Mix the sample of sugar thoroughly. Weigh 50.0 ± 0.1 g of the sample into a 250 ml conical flask and add 50.0 ± 0.1 g of distilled water and dissolve by swirling at room temperature. Filter the sample solution under vacuum through a membrane filter (*see* E-5.3) into a clean dry conical flask. De-aerate the filtered solution for 1 hour at room temperature in a vacuum oven or an evacuated desiccator. Alternatively de-aerate by immersing the conical flask, containing the sugar solution, in an ultrasonic bath for 3 min. Measure the refractometric dry substance (RDS) of the solution, to an accuracy of ± 0.1 g/100 g by the method given in Annex J.

E-6.2 Colour Measurement

Set up the colour measuring instrument (*see* E-5.1) according to the manufacturer's instructions and adjust the wavelength to 420 nm. Rinse the measuring cell with sugar solution and then fill. Determine the absorbancy (*As* or $-\log_{10}Ts$) of the solution using filtered de-aerated distilled water as the reference Standard for zero colour.

E-7 Expression of Results

E-7.1 Calculation

Calculate the concentration of sample solids from the RDS measured in E-6.1 using Table 3.

$$\text{ICUMSA Colour} = \frac{1000 \times As \quad \text{IU}}{bc}$$

where

As is absorbance,
b is cell length in cm, and
c is concentration

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Table 3- Concentration of Sample Solids from the RDS Percentage

RDS percent	cg/cm^3	RDS percent	cg/cm^3
(1)	(2)	(1)	(2)
45.0	0.541 178	50.1	0.616 333
45.1	0.542 621	50.3	0.619 348
45.2	0.544 064	50.4	0.920 857
45.3	0.545 509	50.5	0.622 368
45.4	0.546 955	50.6	0.623 880
45.5	0.548 402	50.7	0.625 393
45.6	0.549 851	50.8	0.626 907
45.8	0.552 752	50.9	0.628 423
45.9	0.554 204	51.0	0.629 940
46.0	0.555 657	51.1	0.631 459
46.1	0.557 112	51.3	0.634 500
46.2	0.558 568	51.4	0.636 022
46.3	0.560 025	51.5	0.637 546
46.4	0.561 483	51.6	0.639 070
46.5	0.562 943	51.7	0.640 597
46.6	0.564 404	51.8	0.642 124
46.8	0.567 330	51.9	0.643 653
46.9	0.568 794	52.0	0.645 183
47.0	0.570 260	52.1	0.646 715
47.1	0.571 728	52.2	0.648 248
47.2	0.573 196	52.3	0.649 782
47.3	0.574 666	52.4	0.651 317
47.4	0.576 137	52.5	0.652 854
47.5	0.577 609	52.6	0.654 392
47.6	0.579 082	52.7	0.655 932
47.8	0.582 033	52.8	0.657 472
47.9	0.583 510	52.9	0.659 015
48.0	0.584 989	53.0	0.660 558
48.1	0.586 469	53.1	0.662 103
48.2	0.587 950	53.2	0.663 649
48.3	0.569 432	53.3	0.665 196
48.4	0.590 916	53.4	0.666 745
48.5	0.592 401	53.5	0.668 295
48.6	0.593 887	53.6	0.669 846
48.7	0.595 374	53.7	0.671 399
48.9	0.598 353	53.8	0.672 953
49.0	0.599 844	53.9	0.674 509
49.I	0.601 337	54.0	0.676 065
49.2	0.602 83 I	54.1	0.677 624
49.3	0.604 326	54.2	0.679 183
49.4	0.605 822	54.3	0.680 744
49.5	0.607 320	54.4	0.682 306
49.6	0.608 819	54.5	0.685 434
49.7	0.610 319	54.6	0.687 000
49.8	0.611 821	54.7	0.688 568
49.9	0.613 324	54.8	0.688 568
50.0	0.614 828	54.9	0.690 137

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Annex F
(Normative)
[*Clause* 4.2, Table 1, Sl. No. (v)]
DETERMINATION OF CONDUCTIVITY ASH

F-1 Field of Application

The conductivity ash in solutions gives measure of the concentration of ionized soluble salt present in solutions of low conductivity. This method is applicable to plantation white sugar, refined sugar and other types of sugars.

F-2 Definition

F-2.1 Conductivity Ash — The ash determined conductimetrically, known as conductivity ash cannot be directly compared with the gravimetric ash determined by incineration and weighing of the ash. Conductivity ash has its own individual significance. The factors for converting conductivity to ash are chosen in such a way that the conductivity ash value corresponds approximately to values for sulphated ash. This coefficient is conventional and cannot be experimentally verified.

F-3 Principle

The specific conductivity of a white sugar solution at a concentration of 28 g/100 g is determined. The equivalent ash is calculated by the application of a conventional factor.

F-4 Reagents

F-4.1 Purified Water — For preparation of all solutions (sugar and potassium chloride) use twice-distilled or deionized water with a conductivity of less than 2 µS/cm.

F-4.2 Potassium Chloride, 0.01 mol/l — Weigh out 745.5 mg after first dehydrating by heating to 500°C (dull red heat). Dissolve in water in a 1 litre volumetric flask and make up to the mark.

F-4.3 Potassium Chloride, 0.000 2 mol/l — Dilute 10 ml of potassium chloride solution, 0.01 mol/l (*see* F-4.2) and make up to the mark in a 500 ml volumetric flask. This solution has a conductivity of (26.6 ± 0.3) µS/cm at 20°C (after deduction of the specific conductivity of the water used).

F-5 Apparatus and Glassware

F-5.1 Sugar Ash Bridge, Null Balance Bridge or Conductivity Meter

F-5.2 Volumetric Flasks, 100, 500 and 1 000 ml.

F-5.3 Pipettes, 10 ml

F-5.4 Analytical Balance - Capable of weighing to the nearest 0.1 mg.

F-6 Procedure

Dissolve 31.3 g ± 0.1 g of sugar in water in a 100 ml volumetric flask and make up to volume at 20°C (or dissolve 28.0 g ± 0.1 g of sugar in water to give a solution of mass 100.0 g). In the case of liquids, the amount taken must be such that the test solution contains 31.3 g of solids/100 ml, or 28.0 g solids/100 g of solution. After thorough mixing, transfer the solution into the measuring cell and measure the conductivity at 20°C ± 0.2°C. Check the measurement using the reference solution (*see* F-4.3).

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F-7 Expression of Results

F-7.1 Calculation of Results

If C_1 is the measured conductivity in µS/cm at 20°C and if C_2 is the specific conductivity of the water at 20°C, then the corrected conductivity (C_{28}) of the 28 g/100 g solution is:

$$C_{28} = C_1 - 0.35\, C_2$$

and

$$\text{Conductivity ash, percent} = 6 \times 10^{-4}{}_{28} \times C$$

F-7.2 Temperature Correction

If the determination cannot be made at the standard temperature of 20°C make a temperature correction to the final result provided that the range of ± 5°C is not exceeded.

The correction is: $\dfrac{C_T}{1 + 0.026\,(T - 20)}$

where

$C_{20°} =$

C_T is conductivity at temperature T°C

NOTE: The conductivity of the potassium chloride standard solution (see F-4.3) is given for a temperature of 20°C. If the measurement cannot be made at the standard temperature of 20°C then the conductivity of the potassium chloride standard solution has to be determined by the formula;

Conductivity of KCI (see F-4.3) at T°C = 26.6 [1+0.021 (T- 20)] in the range 20°C ± 5°C.

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Annex G
(Normative)
[*Clause* 4.2, Table 1, Sl. No. (vi)]

DETERMINATION OF SULPHUR DIOXIDE BY THE ROSANILINE CALORIMETRIC METHOD

G-1 Field of Application

This method is based on the calorimetric determination of sulphur dioxide and is applicable to plantation white sugar, refined sugar and sugar products.

G-2 Principle

The colour of a sulphite/rosaniline complex is measured photometrically, at a wavelength near to 560 nm, after reaction with formaldehyde.

G-3 Reagents

G-3.1 Rosaniline Hydrochloric Solution (Saturated)

Suspend 1 g of rosaniline hydrochloride in 100 ml of distilled water, heat to 50°C and cool with shaking. After standing for 48 h, filter the solution.

G-3.2 Decolourized Rosaniline Solution — Transfer 4 ml of saturated rosaniline hydrochloride solution to a 100 ml volumetric flask. After addition of concentrated hydrochloric acid (6 ml) make the mixture up to the mark. Decolourization takes places in short time but allows the solution to stand for at least 1 h before use.

G-3.3 Formaldehyde Solution (approximately 0.2 g/ 100 ml) — Dilute 5 ml of analytical reagent grade formaldehyde solution, p^{20} = 1.070 – 1.080 to 1 000 ml.

G-3.4 Pure Sucrose Solution — Dissolve 100 g of analytical reagent grade sulphite-free sucrose in water and make up to 1 000 ml.

G-3.5 Sodium Hydroxide Solution, 0.1 mol/l.

G-3.6 Iodine Solution, 0.05 mol/l - Dissolve 20 g of analytical reagent grade iodate-free potassium iodide in 40 ml of distilled water in a 100 0 ml volumetric flask. After the addition of 12.69 g of analytical reagent grade iodine shake the flask until all the iodine is dissolved and then make up to the mark with distilled water.

G-3.7 Concentrated Hydrochloric Acid, p^{20} = 1.18 g/ml.

G-3.8 Hydrochloric Acid Solution, approximately 1 mol/l.

G-3.9 Iodine (Starch) Indicator, ready-made, or a starch solution.

G-3.10 Sodium Thiosulphate Solution, 0.1 mol/l — Dissolve 24.817 g of analytical reagent grade sodium thiosulphate pentahydrate in 200 ml of distilled water in a 1 000 ml volumetric flask and then makeup to the mark.

G-3.11 Standard Sulphite Solution — Dissolve approximately 2.5 g of general purpose reagent grade sodium sulphite heptahydrate in sucrose solution (*see* G-3.4) and make up to 500 ml with this pure sucrose solution (*see* G-3.4). Determine the titre of this solution as follows. Place 25 ml of the 0.05 mol/l iodine solution in a 300 ml conical flask and add 10 ml of the 1 mol/l hydrochloric acid solution (*see* G-3.8) followed by approximately 100 ml of distilled water.

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Pipette 25 ml of standard sulphite solution into this flask while swirling the flask. Then titrate the excess iodine with the 0.1 mol/l sodium thiosulphate solution (*see* G-3.10) until the contents of the flask area pale straw colour. Then add the iodine (starch) indicator (*see* G-3.9) (0.2 g to 0.5 g) to the flask and continue the titration until the blue colour disappears. Record the titre, *t*.

G-3.12 Dilute Standard Sulphite Solution — Dilute 5 ml of standard sulphite solution (*see* G-3.11) to exactly 100 ml with pure sucrose solution (*see* G-3.4). The exact value of the sulphite content, *c* is calculated as follows from the titre, *t*, found in G-3.11:

$$c = (25 - t) \times 3.203 \times 2 \text{ mg } SO_2/\text{ml}$$

NOTE: Users of this method are advised to consult their national health and safety legislation and chemical suppliers before handling rosaniline hydrochloride, formaldehyde and the other reagents here mentioned.

G-4 Apparatus and Glassware

G-4.1 Spectrophotometer or Colorimeter, for use at approximately 560 nm.

G-4.2 Volumetric Flasks, 10 ml, 500 ml and 1 000 ml.

G-4.3 Graduated Pipette, 10 ml.

G-4.4 Pipettes, 2 ml,10 ml and 25 ml.

G-4.5 Burette, 10 ml, graduated by 0.05 ml.

G-4.6 Test Tubes

G-4.7 Analytical Balance - Capable of weighing to the nearest 0.1 mg.

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G-5 Procedure

G-5.1 Colour Development

Dissolve 10-40 g of a sample of white sugar in distilled water in a 100 ml volumetric flask. After addition of 0.1 mol/l sodium hydroxide solution (4 ml) make the contents of the flask up to the mark and mix: For levels

0-5 mg SO_2/kg use 40 g of sample

5-15 mg SO_2/kg use 20 g of sample

15-30 mg SO_2/kg use 10 g of sample

Transfer a 10 ml aliquot to a clean, dry test tube. Add 2 ml of decolourized rosaniline solution and 2 ml of formaldehyde solution and allow the tube to stand at room temperature for 30 min. Measure the absorbance in a 1 cm cell in a spectrophotometer (*see* G-4.1) at about 560 nm using distilled water as a reference.

G-5.2 Standard Curve

Pipette aliquots of the dilute standard sulphite solution (*see* G-3.12) (1 ml, 2 ml, 3 ml, 4 ml, 5 ml and 6 ml) into a series of 100 ml volumetric flasks. Take an empty flask as well for the zero sulphite level. To each flask add 4 ml of 0.1 mol/l sodium hydroxide and make the contents up to the mark with pure sucrose solution (*see* G-3.4) and mix. From each flask transfer a 10 ml aliquot to a clean, dry test tube. Add 2 ml of decolourized rosaniline solution and 2 ml of formaldehyde solution and allow the tubes to stand at room temperature for 30 minutes. Measure the absorbance as in G-5.1 and plot the results on a graph.

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$$\text{The amount of } SO_2 \text{ in each test tube} = \frac{c \times n}{10} \mu gSO_2$$

where

c is concentration of sulphite
n is the number of ml of dilute sulphite added to each 100 ml flask and *c* is from G-3.12.

G-6 Expression of Results

G-6.1 Calculation

Calculate the concentration of sulphite by reference to the standard curve and express the result as mg SO_2/kg white sugar as follows:

$$\frac{\mu gSO_2 \text{ from graph} \times 10}{\text{Mass of sugar used in G-5.1}} \text{ mg } SO_2 \text{ /kg sugar}$$

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Annex H
(Normative)
[*Clause* 4.2, Table 1, Sl. No. (vii)]
(Alternate Method)
DETERMINATION OF LEAD BY COLORIMETRIC METHOD

H-1 Field of Application

The method is based upon a colorimetric procedure and is suitable for white and raw sugar, as well as low grade products with lead contents not exceeding 0.5 mg Pb/kg. A dry ashing step for raw sugar and wet ashing for low-grade products is first required to eliminate organic matter. However, this pre-treatment is not necessary for white sugar.

H-2 Principle

H-2.1 For White Sugar

Lead is extracted directly from the prepared solution by shaking with a solution of dithizone in chloroform and discarding the aqueous layer. Dithizone forms a distinctive red, chloroform-soluble complex with lead in solution. Complete extraction from the aqueous phase is possible when the pH is between 9 and 11.5. Interference from other ions is prevented by the addition of ammonium citrate and potassium cyanide. A final colour matching is carried out by adding a known amount of lead in solution to a dithizone blank.

H-2.2 For Raw Sugar

Organic constituents cause emulsification of the dithizone in chloroform, resulting in poor separation of the aqueous and chloroform phases. Removal of the organic constituents is achieved by ashing the sugar, after addition of magnesium nitrate at a temperature not exceeding 500°C.

H-2.3 For Low-Grade Products

A wet-ashing procedure is recommended, as dry ashing methods have not been found to be convenient for routine analysis. This procedure involves the use of a sulphuric/perchloric/nitric acid mixture and is particularly suited to liquid products. When calcium is known to be present a slightly modified two-acid procedure, based upon using nitric and perchloric acid, is preferred.

H-3 Reagents

Although traces of lead in the reagents are allowed for by carrying a blank through the entire procedure, it is desirable that solutions be prepared from analytical grade reagents or those specified as suitable for trace metal analysis.

H-3.1 Dithizone Solution, Approximately 0.1 g/100 ml — Prepare this stock solution by dissolving 0.1 g of diphenylthiocarbazone in 100 ml of analytical reagent grade chloroform.

H-3.2 Dithizone Solution, Approximately 20 mg/l — Prepare a fresh solution daily, or as required, by diluting 2 ml of the stock solution (*see* H-3.1) to 100 ml with analytical reagent grade chloroform. Store the solution in an amber bottle and in a cupboard away from the light.

H-3.3 Ammonia Solution, p_{20} ~ 0.88 g/ml.

H-3.4 Ammonium Citrate Solution — Dissolve 62.5 g of tri-ammonium citrate in 200 ml of lead-free water. Add 5 ml of the ammonia solution and dilute to 250 ml with distilled water at 20°C. Extract the solution with successive volumes of the 0.1 g/100 ml dithizone solution (*see* H-3.1**)** to ensure complete removal of heavy metals, a state indicated by the green colour persisting in the chloroform layer. Remove excess dithizone from

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the aqueous phase by successive extractions with small amounts of chloroform until the aqueous solution is colorless.

H-3.5 Potassium Cyanide — Dissolve 5 g of analytical reagent grade potassium cyanide in distilled water and dilute to 100 ml at 20°C. Allow to stand for 2 days before use, to allow for the oxidation of traces of sulphur.

H-3.6 Concentrated Nitric Acid, p_{20} = 1.42 g/ml.

H-3.7 Concentrated Hydrochloric Acid, p_{20} = 1.18/ml.

H-3.8 Concentrated Sulphuric Acid, p_{20} = 1.84/ ml.

H-3.9 Concentrated Perchloric Acid, p_{20} =1.54/ml.

H-3.10 Nitric Acid, Approximately 1 Percent (v/v) — Dilute 10 ml of concentrated nitric acid (*see* H-3.6) containing less than 0.005 mg Pb/kg to 1 litre with lead-free water at 20°C.

H-3.11 Nitric Acid, Approximately 1 mol/l — Dilute 15.6 ml of concentrated nitric acid (*see* H-3.6) to 250 ml with lead-free water.

H-3.12 Bromothymol Blue Indicator — Dissolve 0.04 g in 20 percent ethanol and make up to 100 ml with 20 percent ethanol.

H-3.13 Standard Lead Solution — Prepare a stock standard solution A by dissolving 0.160 g of previously dried analytical reagent grade lead nitrate in 100 ml of 1 mol/l nitric acid (*see* H-3.11). Solution B is freshly prepared by diluting 10 ml of the stock solution A to 1 litre with distilled water.

H-3.14 Hydroxylamine Hydrochloride Solution — Dissolve 20 g of analytical reagent grade hydroxylamine hydrochloride in 100 ml of distilled water.

H-3.15 Sodium Hexametaphosphate Solution — Dissolve 10 g in 100 ml of distilled water.

H-3.16 Magnesium Nitrate Solution — Dissolve 10 g in 100 ml of distilled water.

H-3.17 Chloroform, p_{20} = 1.49 g/ml.

NOTE: Users of this method are advised to consult their national health and safety legislation before handling potassium cyanide, lead nitrate, perchloric acid and other reagent a here listed. Users of the wet digestion method are also advised that indiscriminate use of perchloric acid can result in an explosion hazard. Carry out the wet digestion in a fume cupboard behind an arrnoured glass screen.

H-4 Apparatus and Glassware

Wash all apparatus, including separating funnels, microburettes, pipettes and new glassware in 10 percent sodium hydroxide, followed by dilute nitric acid and finally rinse in distilled water.

H-4.1 Pipettes — 5 ml and 10 ml, conforming to Class A.

H-4.2 Graduated Pipettes — 10 ml, conforming to Class A.

H-4.3 Volumetric Flasks — 100 ml and 1 000 ml**,** conforming to Class A.

H-4.4 Separating Funnels — 100 ml.

H-4.5 Microburettes — 2 ml and 5 ml (graduated 0.05 ml) and 25 ml.

H-4.6 Nessler Tube — 25 ml.

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H-4.7 Test Tubes — Pyrex glass, 200 mm × 24 mm, marked at 10 ml and 25 ml.

H-4.8 Platinum Dish

H-4.9 Muffle Furnace

H-4.10 Water Bath, boiling

H-4.11 Erlenmeyer Flasks — 100 ml.

H-4.12 Analytical Balance — Capable of weighing to the nearest 0.1 mg.

H-4.13 Electrical Hot-Plate

H-5 Procedure

H-5.1 White Sugar

a) Sample preparation — Dissolve a 10 g sample in 20 ml of distilled water contained in a beaker and transfer the solution to a 100 ml separating funnel, using only 5 ml for rinsing. Add 2.5 ml of concentrated hydrochloric acid, stopper the funnel and shake for 5 min. Prepare a blank by adding the same volume of hydrochloric acid (2.5 ml) to 25 ml of distilled water contained in another funnel.

b) Extraction of lead — Add 5 to 6 drops of the bromothymol blue indicator to the solution for extraction, and neutralize with ammonia solution (*see* H-3.3) added drop wise from a burette until a blue colour is obtained. Add an additional 1.5 ml of ammonia solution followed by 1.0 ml of ammonium citrate solution (*see* H-3.4) and 1.0 ml of potassium cyanide solution (*see* H-3.5). Where it is known that iron is present in significant concentrations 1.0 ml of hydroxylamine hydrochloride solution (*see* H-3.14) should be added before the cyanide. Stopper the separating funnel and mix the content and well by shaking. Add between 5 and10 ml of the 20 mg/l dithizone solution (*see* H-3.2) incrementally from a 10 ml burette and repeat the mixing. If necessary, add more dithizone solution and continue the extraction until the lower chloroform layer has changed from the brick red colour of lead dithizone to a green, blue or purple colour indicating the extraction of lead from the aqueous phase. Add an equal volume of dithizone solution to the blank and mix in the same manner.

Transfer the chloroform phase to a second clean separating funnel and add about 2 ml of 20 mg/l dithizone solution to the aqueous phase remaining in the original funnel. Shake and transfer the chloroform layer to the original chloroform extract in the second funnel and treat the combined chloroform extract with 10 ml of 1 percent (v/v) nitric acid (*see* H-3.10). Shake vigorously so that the lead is transferred to the aqueous phase as indicated by the restoration of a pure green colour in the chloroform phase. Add more dilute nitric acid if the green colour is not obtained. Discard the exhausted chloroform phase, taking care to avoid any loss of the nitric acid solution. Treat the blank similarly.

c) Determination of lead — Treat the nitric acid solutions from the sample and blank with 0.2 ml ammonium citrate solution, 5 drops of ammonia solution and 0.2 ml potassium cyanide solution. Mix the solutions and add enough 20 mg/l dithizone solution to the sample funnel to change the brick red colour with shaking, to green, blue or purple. Add an equal amount of dithizone to the blank funnel, followed by shaking.

Transfer the complete contents of the separating funnels to Nessler tubes. Slowly add the dilute standard lead solution B from a 2 ml microburette to the blank tube until, on shaking, the colour matches the colour of the solution in the sample tube. Note the volume of solution B needed for matching.

H-5.2 Raw Sugar

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a) Sample Preparation by dry ashing — Weigh 5 g of the sample in a clean platinum dish or silica crucible and treat with 10ml magnesium nitrate solution (*see* H-3.16). Evaporate to dryness and ash the residue in a muffle furnace set at a temperature not greater than 500°C. It is essential that the temperature does not

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exceed 500°C, as it is known that some lead compounds are volatilized above this temperature. When the ash has been completely decarbonized, remove the dish or crucible and allow to cool. Dissolve the ash in 1 ml of concentrated hydrochloric acid (*see* H-3.7) and dilute with lead-free water to 25 ml.

b) Determination of lead — Heat the solution on a water bath for 15 min and determine the lead by following the procedure described in H-5.1 (Extraction of lead and Determination of lead).

H-5.3 Low-grade Products, Liquids and Solids

a) Sample preparation by wet ashing (calcium absent) — Conduct all digestion operation in fume cupboard. Refer to the note under H-3. Transfer the sample, equivalent to not more than 2 g of dry matter, to one of the marked test tubes and add 1 ml of distilled water. Carefully add 1 ml of concentrated sulphuric acid (*see* H-3.8) and 3 ml perchloric acid (*see* H-3.9) together with 3 glass beads. If necessary, warm gently to initiate charring. Treat slowly with 2 to 3 ml concentrated nitric acid (H-3.6) added drop-wise from a burette. Heat gently to boiling and continue the digestion until die solution is clear and almost colourless. Carefully add a few more drops, dropwise until Charring ceases. Continue heating to drive off excess perchloric acid, leaving a final volume of between 1 ml and 2 ml.

b) Sample preparation (when calcium is present) — Carry out all digestion operations in a fume cupboard (*see* Note under H-3). Transfer the sample, equivalent to not more than 2 g of dry matter, to a 100 ml Erlenmeyer flask. Add 1 ml of distilled water, followed by 3 ml of nitric acid (*see* H-3.6) and 2 ml of perchloric acid (*see* H-3.9). Heat from cold on an electrical hot-plate, using a layer of asbestos sheet to moderate the heat. When the liquid turns brown, add more nitric acid drop-wise (not more than 3 ml) and warm until the white fumes of perchloric acid are given off. A colourless solution indicates completion of the digestion phase .

c) Determination of lead — Boil the solutions prepared under the above sections for about 30s to dissolve any solid material present. Add 6ml of ammonium citrate solution (*see* H-3.4) and 10ml of sodium hexa meta - phosphate solution (*see* H-3.15) If not clear, boil again. Dilute the solution to 25 ml with distilled water and quantitatively transfer the solution to a separating funnel for extraction using the procedure described under 15.5.1(a) and (c).

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H-6 Expression of Results

H-6.1 White Sugar Calculation

The lead content is expressed as mg Pb/kg white sugar. Where the sample mass used is 10 g, then 1 ml of solution B is equivalent to 1 mg Pb/kg of white sugar. Where the sample mass differs from 10 g the lead content may be calculated as follows:

$$\frac{10\,V}{M}\ \text{mg Pb/kg}$$

Where

V is volume, in ml, of the dilute standard lead solution used in matching, and
M is mass, in g, of the original sample.

H-6.2 Raw Sugar Calculation

Where the sample mass used is 5 g then 1ml of solution B is equivalent to 2 mg Pb/kg of raw sugar. Use the formula given in H-6.1 for other sample masses.

H-6.3 Low-Grade Products, Liquid and Solids Calculation

Where the sample mass used is 2 g then 1ml of solution B is equivalent to 5 mg Pb/kg of product, liquid or solid. Use the formula given in H-6.1 for other sample masses

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Annex I
(Normative)
[*Clause* 4.2, Table 1, Sl. No. (viii)]
DETERMINATION OF CHROMIUM CONTENT

I-1 Principle

Metals in solution are determined directly by atomic absorption spectrophotometry. Suspended metals are separated by membrane filtration or suspension is dissolved and analyzed.

I-2 Apparatus

I-2.1 Atomic Absorption Spectrophotometer

Spectrophotometer capable of operating at conditions as given under

Wavelength nm	Flame	Optimum Range mg/l
(1)	(2)	(3)
357.9	Reducing air — acetylene	1 to 200

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I-3 Reagents

I-3.1 De-ionized Distilled Water — Distilled, ammonia free. Pass through ion exchange column of mixed strongly acidic cation and strongly basic anion exchange resins. Regenerate resins according to mixed strongly acidic cation and strongly basic anion exchange resins.

I-3.2 Nitric Acid — Dilute 500 ml re-distilled nitric acid to 1 000 ml with water.

NOTE: Perform distillation in hood with protective ash in place.

I-3.3 Hydrochloric Acid — Dilute 500 ml hydrochloric acid to 1 000 ml with water and distill in all-Pyrex or equivalent glass apparatus.

I-3.4 Chromium Solution

I-3.4.1 Chromium stock solution — Accurately weigh amount of metal specified in Table 4 into a beaker and add dissolving medium. When metal is completely dissolved, transfer quantitatively into 1 000 ml volumetric flask and dilute to volume with water.

Table 4 - Preparation of Chromium Standard Solution
***(Clause* I-3.4.1)**

Weight	Compound	Dissolving Medium (1 litre Total)
(1)	(2)	(3)
1.923	Chromium oxide(Cr_2O_3)	Water + 10 ml redistilled nitric acid

I-3.4.2 Chromium Working Solution — Prepare daily. Dilute aliquots of stock solutions with water to make more than or equal to 4 standard solutions within the range of detection as given in I-2.1. Add 1.5 ml nitric acid

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per litre to all working standard solutions before diluting to volume. Add 1 ml lanthanum chloride for every 10 ml making the working standard solution.

I-3.5 Lanthanum Stock Solution — Slowly add 250 ml hydrochloric acid to 58.65 g lanthanum oxide (La_2O_3), purity 99.9 percent by mass, dissolve and dilute to 1 000 ml.

I-3.6 Ammonium Pyrrolidine Dithiocarbamate Solution — Dissolve 1 g ammonium pyrrolidine dithiocarbamate in 100 ml water. Prepare fresh daily.

I-4 Preparation of Sample

Take 5 g of sample and dissolve in distilled water and make up the volume to 100 ml with water in a 100 ml volumetric flask. Transfer an aliquot of well mixed sample to the beaker and add 3 ml nitric acid. Heat and evaporate to dryness (do not boil). Cool and add 3 ml nitric acid and heat until digestion is complete, generally indicated by light coloured residue. Add 2 ml hydrochloric acid (1:1, v/v) and heat gently to dissolve the residue. Wash the watch-glass and beaker with water and filter. Wash the filter and discard. Dilute the filtrate with water to such a concentration that it is within the range of the instrument.

I-5 Determination

Transfer an aliquot of the sample to a 250 ml beaker and dilute to 100 ml with water. Prepare blank and standard solution in the same manner. Adjust the pH of the sample and standard solutions to 2.5 with hydrochloric acid using a pH-meter. Transfer quantitatively to a 200 ml volumetric flask, add 2.5 ml of ammonium pyrrolidine dithiocarbamate solution and mix. Add 10 ml methyl iso-butyl ketone and shake vigorously for 1 min. Let the layers be separated and then add water until the ketone layer is in the neck of the flask. Centrifuge, if necessary. Aspirate the ketone layer and record readings of standards and samples against blank. The fuel-to-air ratio should be adjusted to as blue flame as possible since organic solvents add to fuel supply. Prepare the calibration curve from the average of each standard and read the sample concentration.

I-6 Calculation

Chromium content, mg/l = Chromium, in mg, in the aliquot/litre.

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Annex J
(Normative)
[*Clause* E-6.1]

DETERMINATION OF REFRACTOMETRIC DRY SUBSTANCE (RDS Percent)

J-1 Scope and Field of Application

The method is used in trading for the determination of the refractometric Brix or the refractometric dry substance (RDS percent) of sugar solution.

J-2 Principle

The refractive index of aqueous sugar solutions depends upon the amount of dissolved material and can therefore serve as a measure of the sugar content. This is valid only for pure sugar solutions; however, the non-sugars present in sugar products influence the refractive index in a similar way to sucrose. For these reasons, the measurement of refractive index can be utilized for an approximate determination of the dry substance content of solutions containing mainly sucrose.

Measurements are generally carried out with sugar refractometer graduated in percent sucrose (g/100 g); alternatively this result may be obtained from refractive index tables for pure sucrose solutions.

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J-3 Apparatus and Glassware

J-3.1 **Refractometer**, for example, Abbe type, calibrated at 20°C and having a water-jacketed prism.

J-3.2 **Light Source**, for example, tungsten lamp.

J-3.3 **Plastic Rod**, approximately 3 mm diameter.

NOTE: A plastic rod for example perspex or polypropylene, is preferred for this duty. When using a glass rod there is a possibility of inadvertently scratching the prism faces. Scratched prisms yield an indefinite boundary line and will eventually call for an expensive repolishing operation. Scratching may also occur during prism cleaning, therefore care should be exercised when removing molasses from the prism faces. When cleaning the prism faces, use cool water and soft tissues; do not use hot water for this purpose.

J-3.4 **Thermometer**, 150 mm, range 0°C - 50°C.

J-3.5 **Beaker**, capacity 50 ml.

J-3 6 **Water Bath and Pump**, Thermostatted generally at 20°C.

J-4 Procedure

J-4.1 Reading the Refractometer

Ensure that the instrument has been set up and checked according to the manufacturer's instructions and that the prism faces are clean and dry. The following apply to the Abbe type.

With the prisms closed, allow temperature controlled water (20°C) to flow through the prisms jacket for a period long enough for equilibrium to be reached; 5 min is usually sufficient.

NOTE: When operating at temperature other than 20°C refer Table 5 for corrections to be applied

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Table 5- Temperature Correction
[*Clause* J-4.1]

Temperature	Measured Sucrose (Mass Fraction)		
	45	50	55
(1)	(2)	(3)	(4)
15	− 0.38	− 0.38	− 0.38
16	− 0.30	− 0.31	− 0.31
17	− 0.23	− 0.23	− 0.23
18	− 0.15	− 0.15	− 0.15
19	− 0.08	− 0.08	− 0.08
20	0.00	0.00	0.00
21	+ 0.08	+ 0.08	+ 0.08
22	+ 0.16	+ 0.16	+ 0.16
23	+ 0.24	+ 0.24	+ 0.24
24	+ 0.32	+ 0.32	+ 0.32
25	+ 0.40	+ 0.40	+ 0.40
26	+ 0.48	+ 0.48	+ 0.48
27	+ 0.56	+ 0.56	+ 0.56
28	+ 0.65	+ 0.65	+ 0.64
29	+ 0.73	+ 0.73	+ 0.72
30	+ 0.82	+ 0.81	+ 0.80
31	+ 0.90	+ 0.90	+ 0.89
32	+ 0.99	+ 0.99	+ 0.98
33	+ 1.08	+ 1.07	+ 1.07
34	+ 1.16	+ 1.16	+ 1.15
35	+ 1.25	+ 1.25	+ 1.24
36	+ 1.34	+ 1.34	+ 1.33
37	+ 1.43	+ 1.43	+ 1.41
38	+ 1.53	+ 1.52	+ 1.50
39	+ 1.62	+ 1.61	+ 1.59
40	+ 1.71	+ 1.70	+ 1.68

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Transfer a drop of water to the refractometer prism to first determine whether a reading of zero is obtained or if a correction needs to be applied.

Transfer a small amount sugar solution from the container to the beaker and adjust the sugar solution temperature to approximately that of the instrument, 18°C - 28°C is suitable.

Open the refractometer prism and apply a drop of sugar solution to the fixed prism face by means of the plastic rod. Extend the sugar solution quickly as a line along the face without touching the prism surface with the rod, taking care to avoid the formation of air bubbles. Close the prisms quickly.

Take the refractometer reading according to the instrument manufacturer's handbook. Apply any scale correction to the reading to obtain a corrected reading.

J-5 Expression of Results

J-5.1 Express results to the nearest 0.1°Brix (0.1 percent RDS).

J-5.1.1 Calculation

Where the Refractometer is calibrated in refractive index, read the nearest 0.000 05 units and determine the °Brix (RDS percent) from Table 6.

Table 6 - International Refractive Index Scale for Pure Sucrose Solution at 20°C [*Clause* J-5.1.1]

This Table gives values of refractive index against air with sucrose mass fraction.

Sucrose G/100 g	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
46	1.411 808	1.412 011	1.412 215	1.412 420	1.412 624	1.412 828	1.413 033	1.413 238	1.413 443	1.413 648
47	1.413 853	1.44 059	1.414 265	1.414 470	1.414 676	1.414 882	1.415 089	1.415 295	1.415 502	1.415 708
48	1,415 915	1.416 122	1.416 330	1.416 537	1.416 744	1.416 952	1.417 060	1.417 368	1.417 576	1.417 785
49	1.417 993	1.418 202	1.418 411	1.418 620	1.418 829	1.419 038	1.419 247	1.419 457	1.419 667	1.419 877
50	1.420 087	1.420 297	1.420 508	1.420 718	1.420 929	1.421 140	1.421 351	1.421 562	1.421 774	1.421 985
51	1.422 197	1.422 409	1.422 621	1.422 833	1.423 046	1.423 258	1.423 471	1.423 684	1.423 897	1.424 110
52	1.424 323	1.424 537	1.424 750	1.424 964	1.425 178	1.425 393	1.425 607	1.425 821	1.426 036	1.426 251
53	1.426 466	1.426 681	1.426 896	1.427 112	1.427 328	1.427 543	1.427 759	1.427 975	1.428 192	1.428 428
54	1.428 625	1.428 842	1.426 059	1.429 276	1.429 493	1.429 711	1.429 928	1.430 146	1.430 364	1.430 582
55	1.403 800	1.431 019	1.431 238	1.431 456	1.434 675	1.431 894	1.432 114	1.432 333	1.432 553	1.432 773

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