IS 15279 (2003): Sugar and Sugar Products - Methods of Test
[FAD 2: Sugar Industry]

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भारतीय मानक

चीनी एंव चीनी उत्पाद — परीक्षण विधि

Indian Standard

SUGAR AND SUGAR PRODUCTS — METHODS OF TEST

ICS 67.180.10

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

July 2003

Price Group 10

AMENDMENT NO. 1 JUNE 2011
TO
IS 15279 : 2003 SUGAR AND SUGAR PRODUCTS —METHODS OF TEST

(*Page* 11, *clause* **9.7.1**) — Substitute the following for the existing formula:

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

(FAD 2)

Reprography Unit, BIS, New Delhi, India

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Sugar Industry Sectional Committee had been approved by the Food and Agriculture Divisional Council.

This standard is a necessary adjunct to the series of Indian Standards for individual sugar products as it prescribes the methods of test for determining whether the material under test conforms to the requirement of the respective standard.

The methods of test for various parameters are based on the Codex approved ICUMSA (International Commission for Uniform Methods of Sugar Analysis) methods.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

SUGAR AND SUGAR PRODUCTS — METHODS OF TEST

1 SCOPE

This standard prescribes the methods of test for sugar and sugar products.

2 REFERENCES

The following standards 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 indicated below:

IS No.	*Title*
915 : 1975	One-mark volumetric flasks (*first revision*)
1070 : 1992	Reagent grade water (*third revision*)
1117 : 1975	Specification for one-mark pipettes (*first revision*)
4162 (Part 1) : 1985	Specification for graduated pipettes: Part 1 General requirements (*first revision*)

3 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS 1070) shall be used where use of water as reagent is intended.

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

4 DETERMINATION OF LOSS ON DRYING

4.1 Field of Application

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

4.2 Definition

4.2.1 *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 re-entrant 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.

4.3 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.

4.4 Apparatus

4.4.1 *Forced Draught Atmospheric Pressure Oven*, maintained at a temperature of 105 ± 1°C as measured at a distance of 2.5 ± 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.

4.4.2 *Desiccator,* containing self-indicating silica gel.

4.4.3 *Dishes with Tight Fitting Lids* — These should have a diameter of 6 to 10 cm and a depth of 2 to 3 cm. Although they may be made of glass, platinum or nickel, aluminium 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.

4.4.4 *Clean Dry Duster*

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

4.4.6 *Analytical Balance* — capable of weighing to the nearest 0.1 mg.

4.5 Procedure

4.5.1 *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, 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 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 **4.4.1**. 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.

4.5.2 *Weighing to Determine Loss on Drying*

Replace the lids, remove the dishes from the oven and replace 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.

4.6 Expression of Results

4.6.1 *Calculation of Loss on Drying*

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

$$\text{Percent loss on drying} = \frac{100\,(m_2 - m_3)}{m_2 - m_1}$$

where

m_1 = mass of dish, in g;

m_2 = mass of dish + sugar before drying, in g; and

m_3 = mass of dish + 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.

5 DETERMINATION OF POLARIZATION

5.1 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**).

5.2 Apparatus

5.2.1 *Analytical Balance*

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

5.2.3 *Basin*, made of nickel or German silver, large enough to hold the normal weight of sugar.

5.2.4 *Volumetric Flask*, of capacity 100 ml, calibrated at 20°C.

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

5.2.6 *Stemless Funnel*, capable of holding 100 ml.

5.2.7 *Glass Cylinder*, capable of holding 100 ml.

5.2.8 *Watch-Glass*, large enough to cover the stemless funnel.

5.2.9 *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.

5.2.9.1 *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 (°S). To convert values in °S to values in °Z, multiply the °S value by the factor 0.999 71.

Basis of calibration of 100° point on international sugar scale is polarization of normal solution of pure sucrose (26.000 g/100 ml) at 20°C in 200 mm tubes. This solution, polarized at 20°, must give saccharimeter reading of exactly 100°S.

5.3 Reagent

5.3.1 *Alumina Cream* — Dilute 500 ml of 30 percent (*w/v*) aluminium sulphate solution to 2 litres Add gradualiy 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.

5.4 Test Temperature

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

5.5 Procedure

5.5.1 *Preparation of the Solution*

Weigh accurately on the analytical balance the normal weight of the sugar (*see* **5.2.2**) 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 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* **5.4**) 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).

5.5.2 *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 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* **4.2.1**), using a 200-mm tube. The reading gives the polarization percent of the sugar.

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

$$P^{20} = P^{t}[1+ 0.000\,3\,(t-20)]$$

where

P^{20} = saccharimeter reading, at 20°C,

P^{t} = saccharimeter reading, at t°C, and

t = temperature in °C at which the polarization is carried out.

NOTE — The formula is applicable only up to 30°C.

6 DETERMINATION OF POLARIZATION OF RAW SUGAR BY POLARIMETRY

6.1 Field of Application

This method is applicable to all raw sugars and speciality sugars requiring clarification. This method, which may form the basis of sugar purchasing contracts, measures the optical rotation of the normal solution of raw sugar. The polarization is expressed in °Z of the International Sugar Scale.

6.2 Definitions

6.2.1 *Normal Sugar Solution* — It is defined as 26.016 0 g of pure sucrose weighed in vacuum and dissolved in water at 20.00°C to a final volume of 100.000 ml. This corresponds to 26.000 g weighed in air and dissolved in water at 20.00°C to a final volume of 100.000 ml.

6.2.2 *Basis of the 100°Z Point of the International Sugar Scale* — The optical rotation of the normal solution of pure sucrose (*see* **6.2.1**) at the wavelength of the green line of the mercury isotope ^{198}Hg = 546.227 1 nm in vacuum at 20.00°C in a 200.000 mm tube is 40.777 ± 0.001° angular. For spectrally filtered yellow sodium light, 589.440 0 nm in vacuum, this results in a 100°Z point of 34.626 ± 0.001° angular. For quartz wedge instruments the effective wavelength has been fixed at 587.000 0 nm leading to a 100°Z point of 34.934 ± 0.001° angular. This rotation value is of no practical significance but it serves for calculating the sugar value of a quartz control plate for quartz wedge instruments.

6.3 Principle

The optical rotation is the algebraic summation of the predominant effects of the sucrose content of the sample, modified by the presence of other optically active constituents and by the clarification procedure.

This is a physical analysis involving three basic steps:

a) Preparation of a normal solution of raw sugar in water, including its defecation by the addition of basic lead acetate solution;

b) Clarification of the solution by filtration; and

c) Determination of the polarization by measurement of the optical rotation of the clarified solution.

6.4 Reagents

6.4.1 *Basic Lead Acetate* — Conforming to the specification as given under and ground to pass a 0.42 mm sieve.

Basic lead (PbO)	: not less than 33 percent
Loss on drying at 105°	: not more than 1.5 percent
Insoluble in dilute acetic acid	: not more than 0.02 percent
insoluble in water	: not more than 1.0 percent
Chloride (Cl)	: not more than 0.003 percent
Nitrate and nitrite (as NO_3)	: not more than 0.003 percent
Copper (Cu)	: not more than 0.002 percent

Substances not precipitated by
H_2S (as sulphates) : not more than 0.30 percent
Iron (Fe) : not more than 0.002 percent

6.4.2 *Basic Lead Acetate Solution* — Dissolve 560 g of basic lead acetate in about 1 litre of distilled water. Boil for about 30 min and allow to settle. Decant the supernatant liquid and dilute to a density of 1.24 g/ml or 24.4 g PbO/100 ml total lead content with recently boiled distilled water. Check the total lead content either by measuring the density accurately or by determining total lead titrimetrically as described in Annex A. The total lead specification calls for a density, ρ_{20}, of 1.24 ± 0.01 g/ml or a total lead content of 24.4 ± 1.0 g PbO/100 ml. The basic lead content must be between 9.5 and 10.5 g PbO/100 ml. If the quantity of basic lead lies above this range, adjust the reagent by adding glacial acetic acid. After adjustment, re-determine both the total lead and basic lead contents.

Keep the solution in a vessel fitted with an airlock mechanism to avoid any contact with carbon dioxide in the air. Flush the vessel with nitrogen gas before closing.

NOTE — Users of this method are advised to consult the health and safety legislation before handling basic lead acetate and the reagents listed in Annex B.

6.5 Apparatus

6.5.1 *Analytical Balance*, readable to 1 mg.

6.5.2 *Special 100 ml Flasks*, nominal capacity of 100 ml, with a tolerance of ± 0.02 ml.

Where such flasks are not available, use an ICUMSA flask and apply a 'flask correction'. The quantity 'actual volume of flask –100.00', known as the 'flask correction', should be engraved clearly on the bulb of the flask and recorded separately against the code number of the flask. When 'corrected' flasks are used, correct the polarization reading by adding algebraically the 'flask correction'. The 'flask correction' may be positive or negative, and the correct sign must be used.

6.5.3 *Automatic Dispenser for Lead Acetate,* to avoid contact with CO_2 in the air, fit a cartridge containing a mixture of calcium and sodium hydroxides, or equivalent, to the air inlet of the dispenser.

6.5.4 *Filtration Equipment,* use stemless funnels of corrosion-resistant material to filter the solutions prepared for polarization. Mount funnels on filtrate-receiving glasses or beakers which do not lead to excessive splashing, and place cover slips on the funnels to minimize evaporation. Use filter papers of a type suitable for raw sugar polarization work, for example Whatman No. 91 in 15 cm circles having a moisture content in the range of 6 to 8 percent water, determined by drying for 3h at 100°C.

6.5.5 *Polarimeter,* calibrated in sugar degrees (°Z) at 20.00°C.

6.5.6 *Polarimeter Tubes and Cover Glasses,* conforming to the specification as given under Annex B. The tolerance on length of a tube shall conform either with Class A or, where it conforms with Class B, the actual length, correct within the tolerance specified for Class A, shall be engraved on the tube. A tube length correction equal to nominal length divided by actual length is then applied as a multiplier to all polarization readings.

6.5.7 *Quartz Control Plate* — Officially certified in °Z at 20°C. The controlling laboratories inspect the plate to determine whether it is in accordance with the specifications of ICUMSA as given under Annex C. The certified value of the plate should be as close as possible to 100°Z.

6.5.8 *Water Bath,* maintained at 20 ± 0.5°C.

6.5.9 *Thermometer,* readable to 0.1°C over the range 0-50°C.

6.6 Samples

On arrival in the laboratory and before opening, inspect the packed sample to see whether:

a) the sugar has been affected by changes in temperature between the time of packing and the time of arrival, and
b) the package has been damaged or tampered with.

If, as a result of this inspection, there is reason to believe that a sugar received in the laboratory could differ from the sugar as packed, report this to the person or organization for whom the analysis is to be done, whether or not the analysis is carried out.

As far as practicable, keep the humidity of the laboratory in which samples are unpacked within the range 65 to 70 percent relative humidity.

Avoid re-mixing of the sample if possible. If any re-mixing is done, a loss on drying determination should be reported as well as the polarization.

If the sugar has not been freshly mixed and there is reason to suspect that the sample container is not airtight, discard the top 1 to 2 cm of sample immediately before weighing.

6.7 Procedure

6.7.1 *Preparation and Defecation of the Sample Solution*

Weigh out accurately 26.000 ± 0.002 g of the sugar as rapidly as possible. Transfer the sugar to a 100 ml volumetric flask by washing with distilled water to a

volume not exceeding 70 ml and completely dissolve either by hand swirling or using a mechanical agitator. Add distilled water if necessary to bring the volume to 60 or 70 ml. Add basic lead acetate solution according to the expected polarization of the raw sugar: below 99.3°Z use 1.00 ± 0.05 ml and above 99.3°Z use 0.50 ± 0.05 ml. Add the lead solution from the automatic dispenser.

Mix the solution by gentle swirling and using the same motion, add distilled water until the bulb of the flask is full. Allow to stand for at least 10 min to attain room temperature, preferably temperature-controlled at 20 ± 1°C.

Add distilled water to about 2 mm below the mark, ensuring that all the neck is washed. Take care that no air bubbles are entrapped and if necessary, de-froth the meniscus by means of alcohol or ether vapour. Dry the inside of the neck of the flask with a clean roll of filter paper to within a few millimetre of the mark. With the meniscus suitably shaded, hold the flask vertically by the top of the neck, calibration mark at eye-level and viewed against a well-lit background. Add distilled water drop-wise, preferably from a hypodermic needle, until the bottom of the meniscus and the top of the calibration mark just coincide. If fine bagasse or fibre particles are present, flick the side of the neck momentarily so that the true position of the meniscus is sighted.

Dry the inside of the neck of the flask, seal the flask with a clean, dry stopper and shake thoroughly. Insert a clean dry thermometer in the flask and record the temperature, t_m, to 0.1°C. Hold the flask by the top of the neck during this measurement.

6.7.2 *Filtration of the Solution*

Allow the solution to stand for at least 5 min to permit settling of the precipitate. Then filter the solution through a single filter paper using equipment described in **6.5.4**. Discard the first 10 ml of filtrate and collect a volume just sufficient for polarization measurement, usually 50 to 60 ml. Place a cover slip on the filter funnel during filtration to minimize evaporation and also keep the filter out of draughts and direct sunlight. Do not replenish the solution in the filter funnel and do not return filtrate to the filter.

6.7.3 *Determination of the Polarization*

Rinse the polarization tube thoroughly at least twice with the solution to be used and fill in such a way that no air bubbles are entrapped. Screw on the caps of end-filling tubes only as tightly as is necessary to prevent leakage. Tighter screwing may strain the material of the cover glasses and distort the plane of polarized light. Handle the tube as little as possible to avoid warming.

Place the tube in the polarimeter. For a visual polarimeter take four readings to 0.05°Z or better. Rotate the tube through about 45° between each reading. Flow-through or side-filling tubes, of course, cannot be rotated. It should be ensured that the tube compartment lid is closed when balancing and reading. The readings are averaged to 0.01°Z.

Measure the temperature of the solution in the tube, t_r, as soon as practicable after taking the polarimeter reading, and record to 0.1°C.

6.7.4 *Standardization of Polarimeter and Application of Temperature Corrections*

Standardize polarimeter readings by means of certified quartz plates with a nominal value between 95 and 101°Z. According to which method of standardization and application of temperature corrections is adopted certain air, water and quartz plate readings are required together with temperatures of the quartz plate and polarimeter. Make these readings in a manner similar to those for the polarimeter tube to a precision of 0.01°Z.

There are two recommended methods for carrying out the standardization of the polarimeter and calculating the polarizations of test solutions corrected to 20°C. Method 1 requires that, with each batch of test solutions, a standardization with a quartz plate is carried out at the same time. This procedure assumes that the polarimeter is operating at a constant but unmeasured instrument temperature. Method 2 requires that quartz plate readings be carried out periodically but not necessarily at the same time as test solutions are read. Standardization of the polarimeter is achieved by application of a scale correction derived by this procedure. Since Method 1 is more generally applicable it is given here while Method 2 is given in Annex D.

Method 1

Make and record the following readings:

a) Polarimeter zero, that is air reading P_a;
b) Quartz plate reading, Q_{tq}, at temperature t_q, where t_q is the temperature of the quartz plate at time of reading;
c) Reading of a polarimeter tube filled with water, P_w;
d) Reading of test solution, P_{tr}, at temperature t_r; and
e) The temperature of making to the mark, t_m, will already have been recorded.

6.8 Expression of Results

6.8.1 *Calculation*

Taking the results from **6.7.3** and **6.7.4**, calculate the

polarization corrected to 20°C, P_{20}, by applying the corrections given below to the observed polarization, P_{tr}:

a) Subtract zero correction using water, P_w

b) Add temperature of reading, t_r correction

t_r correction = $c \times P_{tr} \times (t_r - 20)$ $0.004 \times R \times (t_r - 20)$

where

c = coefficient in Table 1, and

R = reducing sugars, percent on sample.

c) Subtract temperature of making to mark, t_m correction

t_m correction = $f \times P_{tr} \times (t_m - 2Q)$

where f = coefficient in Table 1.

Table 1 Table of Coefficients for Polarization Temperature Corrections

(*Clause* 6.8.1)

Sl No.	Material of Construction		Coefficients	
	Tube	Flask	c	f
(1)	(2)	(3)	(4)	(5)
i)	BS	BS	0.000 467	0.000 270
ii)	N	BS	0.000 462	0.000 270
iii)	St	BS	0.000 455	0.000 270
iv)	BS	N	0.000 467	0.000 255
v)	N	N	0.000 462	0.000 255
vi)	St	N	0.000 455	0.000 255

BS = borosilicate glass, for example, duran, pyrex.
N = normal glass, for example, window glass.
St = steel, for example, stainless, V2A

d) Subtract combined scale and instrument temperature correction,

scale and instr t corr scale and instr t corr = $Q_{tq} - Q_{20} - P_a - 0.000\ 144 \times Q_{20} \times (t_q - 20)$

where Q_{20} = certified quartz plate value at 20°C.

Thus,

$P_{20} = P_{tr} - P_w + t_r$ corr. $- t_m$ corr. – scale and instr t corr

Express the results to 2 decimal places as °Z. Indicate in the result the corrections that have been made to the result observed without corrections (for example corrected for instrument standardization and to a temperature of 20°C).

7 DETERMINATION OF REDUCING SUGARS BY THE MODIFIED OFNER TITRIMETRIC METHOD

7.1 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.

7.2 Definition

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

7.2.2 *Invert Sugar* — Equi-molar solution of glucose and fructose.

7.2.3 *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.

7.3 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 1 mg of reducing sugars, once the correction for the reducing effect of sucrose has been taken into account.

7.4 Reagents and Materials

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

7.4.1 *Activated Carbon*, powdered.

7.4.2 *Small Pumice Pieces*

7.4.3 *Disodium Hydrogen Phosphate*, $Na_2HPO_4.12H_2O$.

7.4.4 *Glacial Acetic Acid*, M_{20} = 1.05 g/ml.

7.4.5 *Acetic Acid Solution*, approximately 5 mol/l.

7.4.6 *Potassium Sodium Tartrate (Rochelle or Seignette Salt)*

7.4.7 *Copper Sulphate Pentahydrate*, $CuSO_4.5H_2O$.

7.4.8 *Sodium Carbonate*, anhydrous.

7.4.9 *Soluble Starch*

7.4.10 *Hydrochloric Acid*, approximately 1 mol/l.

7.4.11 *Hydrochloric Acid*, approximately 2 mol/l.

7.4.12 *Ofner Solution, Modified* — Weigh out 7.0 g copper sulphate pentahydrate (*see* **7.4.7**), 10.0 g sodium carbonate (*see* **7.4.8**), 300 g potassium sodium tartrate (*see* **7.4.6**) and 50 g disodium hydrogen phosphate (*see* **7.4.3**) in a 1 000 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* **7.4.1**) and stir for 5-10 min. Filter the solution (*see* **7.5.11**).

7.4.13 *Potassium Iodate Solution*, 0.016 67 mol/l — Weigh out 3.566 7 g potassium iodate, KIO_3. Transfer to a 1 000 ml volumetric flask, dissolve in water and fill to the mark.

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

7.4.14 *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 a few minutes.

7.4.15 *Potassium Iodide, KI*

7.4.16 *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* **7.4.11**) and 10.0 ml of 0.016 67 mol/l potassium iodate solution (*see* **7.4.13**). 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 decolorization, adding 1 ml of starch indicator (*see* **7.4.14**) immediately before the endpoint. Calculate the factor f_{Th} of the thiosulphate solution:

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

where

V_{Th} = 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.

7.4.17 *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* **7.4.16**). Pipette 25.0 ml of the iodine solution into a 300 ml Erlenmeyer flask. Add 5 ml of 5 mol/l acetic acid (*see* **7.4.5**) and, after gently shaking the mixture, titrate back with the 0.033 3 mol/l sodium thiosulphate solution (*see* **7.4.16**). Add 1 ml of starch indicator (*see* **7.4.14**) 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} = ml of sodium thiosulphate solution titrated, and

f_{Th} = Correction factor for the sodium thiosulphate solution.

7.5 Apparatus

7.5.1 *Analytical Balance*, capable of weighing to the nearest 0.1 mg.

7.5.2 *Precision Balance*, capable of weighing to the nearest 0.1 g.

7.5.3 *Burettes*, capacity 50 ml.

7.5.4 *Erlenmeyer Flasks*, capacity 300 ml.

7.5.5 *Volumetric Flasks*, 1 000 ml and 200 ml.

7.5.6 *Pipettes*, capacities 1 ml, 15 ml and 50 ml.

7.5.7 *Watch Glasses*, to cover Erlenmeyer flasks.

7.5.8 *Bunsen Burner, Tripod and Wire Gauze*

7.5.9 *Boiling Water Bath*

7.5.10 *Water Bath with Cold Running Water*

7.5.11 *Filter Paper*

7.6 Procedure

7.6.1 *Preparation of the Sample*

The solution prepared for the determination should contain not 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.

7.6.2 *Hot Value*

Mix 50.0 ml of the prepared solution (*see* **7.6.1**) with 50.0 ml of the Ofner solution (*see* **7.4.12**). Add some pumice pieces (*see* **7.4.2**) 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* **7.4.4**). Add iodine solution (*see* **7.4.17**) 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* **7.4.16**) are consumed on back titration. Add 15 ml of the 1 mol/l hydrochloric acid (*see* **7.4.10**) 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* **7.4.16**). 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.

7.6.3 *Cold Value*

Mix 50.0 ml of the prepared sample (*see* **7.6.1**) with 50.0 ml of the Ofner solution (*see* **7.4.12**).

Leave the mixture at room temperature for 10 min. Repeat the procedure outlined in **7.6.2**. Record values V_3 and V_4.

7.6.4 *Blank Value*

Mix 50 ml of water with 50 ml of the Ofner solution (*see* **7.4.12**). Repeat the procedure outlined in **7.6.2**. 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.

7.7 Expression of Results

7.7.1 *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 **7.4.17**, and

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

$$\text{Invert sugar, mg/kg} = \frac{(A - B - C - D) \times 1\,000}{s}$$

where

s = the amount of sample in 50 ml of prepared solution (*see* **7.6.1**)

7.7.2 *Example Calculation*

100 g white sugar is weighed out and diluted to 200 ml. 50.0 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.

$B = (25.00 \times 1.031) - (24.72 \times 1.029) = 0.34$

$A = (25.00 \times 1.031) - (21.71 \times 1.029) = 3.43$

$C = (25.00 \times 1.031) - (25.00 \times 1.029) = 0.05$

$D = 25.0 \times 0.1 = 2.50$

Reducing sugar mg/kg

$$= \frac{(3.43 - 0.34 - 0.05 - 2.50) \times 1\,000}{25} = 21.9 \text{ mg/kg}$$

8 DETERMINATION OF SUGAR SOLUTION COLOUR

8.1 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.

8.2 Definitions

8.2.1 *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:

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

where 100 T = percentage transmittance

8.2.2 *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:

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

8.2.3 *Absorbancy (Extinction),* A_s

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

8.2.4 *Absorbancy 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:

Absorbancy index of the solution, $a_s = \frac{A_s}{bc}$

8.2.5 *ICUMSA Colour*

The value of the absorbancy index multiplied by 1 000 is reported as ICUMSA Colour. The resulting values are designated as ICUMSA Units (IU).

8.3 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 absorbancy of the filtered solution is measured at a wavelength of 420 nm and the solution colour is calculated.

8.4 Reagents

Use only distilled water or water of equivalent purity.

8.5 Apparatus

8.5.1 *Instrument* — Spectrophotometer or colorimeter 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.

8.5.2 *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.

8.5.3 *Membrane Filters* — Pore size 0.45 mm. Diameter 50 mm of cellulose based material.

8.5.4 *Membrane Filter Holder* — Preferably fitted with a stainless steel support.

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

8.5.6 *Refractometer*

8.5.7 *Laboratory Balance* — capable of weighing to the nearest 0.1 g.

8.6 Procedure

8.6.1 *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* **8.5.3**) into a clean dry conical flask. De-aerate the filtered solution for 1 h 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 E.

8.6.2 *Colour Measurement*

Set up the colour measuring instrument (*see* **8.5.1**) 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 (A_s or $-\log_{10} T_s$) of the solution using filtered de-aerated distilled water as the reference standard for zero colour.

8.7 Expression of Results

8.7.1 *Calculation*

Calculate the concentration of sample solids in from the RDS measured in **8.6.1** using Table 2.

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

where

A_S = absorbancy,
b = cell length in cm, and
c = concentration

Express results to the nearest whole number.

Table 2 Concentration of Sample Solids from the RDS Percentage

(*Clause* 8.7.1)

RDS percent	cg/cm³	RDS percent	cg/cm³
(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
49.1	0.601 337	54.0	0.676 065
49.2	0.602 831	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

9 DETERMINATION OF CONDUCTIVITY ASH

9.1 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.

9.2 Definition

9.2.1 *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.

9.3 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.

9.4 Reagents

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

9.4.2 *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.

9.4.3 *Potassium Chloride*, 0.000 2 mol/l — Dilute 10 ml of potassium chloride solution, 0.01 mol/l (*see* **9.4.2**) 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).

9.5 Apparatus

9.5.1 *Sugar Ash Bridge, Null Balance Bridge or Conductivity Meter*

9.5.2 *Volumetric Flasks*, 100, 500 and 1 000 ml.

9.5.3 *Pipettes*, 10 ml, conforming to Class A of IS 1117.

9.5.4 *Analytical Balance*, capable of weighing to the nearest 0.1 mg.

9.6 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 ± 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 ± 0.2°C. Check the measurement using the reference solution (*see* **9.4.3**)

9.7 Expression of Results

9.7.1 *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

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

9.7.2 *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:

$$C_{20^\circ} = \frac{C_T}{1 + 0.026\,(T - 20)}$$

where

C_T = conductivity at temperature T°C

NOTE — The conductivity of the potassium chloride standard solution (*see* **9.4.3**) 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 KCl (*see* **9.4.3**) at T°C = 26.6 [1+0.021 (T – 20)] in the range 20 ± 5°C.

10 DETERMINATION OF SULPHATED ASH

10.1 Field of Application

This standard specifies a method for the determination of sulphated ash in sugar products.

10.2 Definition

10.2.1 *Sulphated Ash* — The residue obtained after incineration of the product, according to the method specified in this standard. It is expressed as a percentage by mass either of the product as received or on the dry basis.

10.3 Principle

Incineration of a test portion, in the presence of sulphuric acid, at a temperature of 525 ± 25°C. The sulphuric acid facilitates the destruction of the organic matter and avoids losses by converting the volatile chlorides into non-volatile sulphates.

10.4 Reagents

During the analysis, use only reagents of recognized analytical quality and only distilled water or water of at least equivalent purity.

10.4.1 *Sulphuric Acid Solution*

Add carefully 100 ml of concentrated sulphuric acid, ρ_{20} 1.83 g/ml, to 500 ml of water and mix.

10.5 Apparatus

Ordinary laboratory apparatus, and in particular the following:

10.5.1 *Incineration Dish*, of platinum or any other material which does not deteriorate under the test conditions (for example, a silica incineration dish), of capacity 100 to 200 ml and with a minimum useful surface of 15 cm^2.

10.5.2 *Electric Furnace with Air Circulation*, capable of being controlled at 60 to 70°C.

10.5.3 *Electric Hot Plate or Gas Burner or Heating Lamp*

10.5.4 *Desiccator*, provided with an efficient desiccant.

10.5.5 *Water Bath*, capable of being controlled at 60 to 70°C.

10.5.6 *Analytical Balance*

10.6 Procedure

10.6.1 *Preparation of the Incineration Dish*

Clean the incineration dish (*see* **10.5.1**), whether it is new or used, with boiling hydrochloric acid solution (*see* **10.4.2**), then rinse generously with water.

Calcinate the incineration dish for 30 min in the furnace (*see* **10.5.2**), controlled at 525 ± 25°C. Allow to cool to ambient temperature in the desiccator (*see* **10.5.4**) and weigh to the nearest 0.000 2 g (the incineration dish should be calcinated to constant mass).

10.6.2 *Preparation of the Test Sample*

Mix the sample carefully and quickly by stirring (for a powder) or by mixing with a spatula (for a liquid) in a sample container.

If the volume of the container is insufficient for this, quickly transfer the whole sample to another, previously dried container of a suitable size.

Take care to avoid any change in the moisture content of the sample. The taking of representative sample of approximately 5 g can be difficult (for example, glucose in lumps). In this case, use one of the procedures described below:

a) Weigh carefully, to the nearest 0.01 g, approximately 100 g of the sample into a dry container, provided with a lid, previously tared with the lid. Add approximately 100 ml of water at 90°C, place the lid on the container and stir until the sample is completely

dissolved. Allow to cool to ambient temperature and weigh to the nearest 0.01 g.

b) Melt the sample in solid from by immersing it, in a container, provided with lid, in the water bath (*see* **10.5.5**), controlled at 60 to 70°C, and placing the lid on the container. Remove the container from the water bath and allow it to cool to ambient temperature, agitating frequently but without removing the lid, and then mix the condensed water with the sample.

10.6.3 *Test Portion*

If a dilution has been carried out, take an aliquot portion of the solution obtained (*see* **10.6.2**), so as to obtain a mass of sample corresponding to a mass of test portion as given below.

In all other cases, weigh, to the nearest 0.001 g, in the incineration dish (*see* **10.5.1**), previously weighed to the nearest 0.000 2 g, a mass of test sample (*see* **10.6.2**) in accordance with the following:

Sulphated Ash Percent	*Mass of Test Portion*
m/m	*G*
< 5	10
>5 <10	5
>10	2

10.6.4 *Preincineration*

Add 5 ml of the sulphuric acid solution (*see* **10.4.1**) to the test portion or the aliquot portion (*see* **10.6.3**). Mix with a glass stirring rod and rinse with a little water, collecting the rinsings in the incineration dish. Heat the incineration dish slowly and carefully, over the electric hot-plate or gas burner or using the heating lamp (*see* **10.5.3**), until completely carbonized (it is recommended that this be carried out under an extraction hood).

10.6.5 *Incineration*

Place the incineration dish in the oven (*see* **10.5.2**), controlled at 525 ± 25°C, and maintain this temperature until the carbon residue has disappeared. A period of 2 h is usually sufficient.

Allow to cool. Take up the residue with several drops of the sulphuric acid solution (*see* **10.4.1**), evaporate on the edge of the oven (*see* **10.5.2**) and incinerate again for 0.5 h. Place the incineration dish in the desiccator (*see* **10.5.4**) and allow it to cool to ambient temperature. Weigh the dish and contents to the nearest 0.000 2 g. The incineration should be continued until constant mass is attained.

Do not put more than four incineration dishes in the desiccator at any one time.

10.7 Expression of Results

10.7.1 *Method of Calculation*

The sulphated ash, expressed as a percentage by mass of product as received, is given by the following formula:

$$\frac{(m_2 - m_1) \times 100}{m_0}$$

The sulphated ash, expressed as a percentage in mass on the dry basis, is given by the following formula:

$$\frac{(m_2 - m_1) \times 100}{m_0} \times \frac{100}{100 - H}$$

where

m_0 = mass, in g, of the test portion, taking into account any dilution (*see* **10.6.3**),

m_1 = mass, in g, of the incineration dish before incineration (*see* **10.6.1**),

m_2 = mass, in g, of the incineration residue and incineration dish after incineration (*see* **10.6.5**), and

H = moisture content of the product.

11 DETERMINATION OF STARCH IN ICING SUGAR

11.1 Reagents

11.1.1 *Fehling's Solution (Soxhlet Modification)* — Prepared by mixing immediately before use, equal volumes of Solution A and Solution B.

11.1.1.1 *Solution A* — Dissolve 34.679 g of copper sulphate ($CuSO_4 . 5H_2O$) in water, add 0.5 ml of concentrated sulphuric acid of sp gr 1.84 and dilute to 500 ml in a volumetric flask. Filter the solution through prepared asbestos.

11.1.1.2 *Solution B* — Dissolve 173 g of Rochelle salt (potassium sodium tartrate, $KNaC_4H_4O_6$, $4H_2O$) and 50 g of sodium hydroxide, in water, dilute to 500 ml in a volumetric flask and allow the solution to stand for two days. Filter this solution through prepared asbestos.

11.1.2 *Methylene Blue Indicator* — Dissolve 0.2 of methylene blue in water and dilute to 100 ml.

11.1.3 *Concentrated Hydrochloric Acid* — sp gr 1.16.

11.2 Procedure

11.2.1 *Preparation of the Solution*

Weigh accurately about 2.5 g of the material in a watch glass. Transfer the material to a conical flask with about 80 ml of water. Add 10 ml of concentrated hydrochloric acid. Heat the flask for 1 h in a briskly boiling water-

bath. Cool and neutralize with sodium hydroxide solution and transfer the solution to a 500-ml volumetric flask and make up the volume to the mark in the flask.

11.2.2 *Estimation of Reducing Sugars*

Pour the prepared solution (*see* **11.2.1**) into a 50-ml burette. Pipette 25 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 s, 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 1 ml of methylene blue indicator solution. Continue boiling the contents of the flask for 1 to 2 min from the commencement of ebullition and then add the prepared solution in small quantities (1 ml or less at a time), allowing the liquid to boil for about 10 s between successive additions, till the blue colour of the indicator just disappears. Note the reading of the burette and calculate the volume of the prepared solution used up in titration.

11.3 Calculation

11.3.1 Record from col 3 of Table 3 the amount of reducing sugar percent in 100 ml of the solution corresponding to the titre value.

$$\text{Starch (moisture-free), percent} \left[\frac{5M}{1\,000} - \frac{WR}{100} - \frac{WS}{95}\right]\frac{90}{W}$$

where

M = amount of reducing sugars present in 100 ml of prepared solution corresponding to the titre,

W = mass in g of the material taken for the test (*see* **11.2.1**),

R = percent reducing sugars by mass (*see* **7.7.1**), and

S = percent of sucrose by mass (*see* **12.4.1**)

12 DETERMINATION OF SUCROSE

12.1 Field of Application

The method measures the sucrose content of sugar products.

12.2 Reagents — Same as those given under **11.1**.

12.3 Procedure

12.3.1 *Preparation of the Solution*

Weigh accurately about 5 g of the sample in a watch glass. Transfer this quantity into a beaker, add about 50 ml of water and warm the mixture in a water bath at 50 to 60°C for about 5 min to dissolve the sucrose content of the sample. Cool and filter through a Whatman filter paper No. 40 or equivalent. Collect the filtrate carefully in a 100-ml volumetric flask. Wash the beaker and the insoluble residue of starch in the filter paper carefully with water. Make up the volume of the filtrate to 100 ml.

Table 3 Invert Sugar Factors for 25 ml of Fehling's Solution

(*Clause* 11.3.1)

Titre	Invert Sugar Factor	Reducing Sugar (as Content Anhydrous Dextrose per 100 ml of Solution)
(1)	(2)	(3)
15	120.2	801
16	120.2	751
17	120.2	707
18	120.2	668
19	120.3	638
20	120.3	601.5
21	120.3	572.9
22	120.4	547.3
23	120.4	523.6
24	120.5	501.9
25	120.5	482.0
26	120.6	463.7
27	120.6	446.8
28	120.7	431.1
29	120.7	316.4
30	120.8	402.7
31	120.8	389.7
32	120.8	377.6
33	120.9	366.3
34	120.9	355.6
35	121.0	345.6
36	121.0	336.3
37	121.1	327.4
38	121.2	318.8
39	121.2	310.7
40	121.2	303.1
41	121.3	295.9
42	121.4	289.0
43	121.4	282.4
44	121.5	276.1
45	121.6	270.1
46	121.6	264.3
47	121.6	258.8
48	121.7	253.5
49	121.7	248.4
50	121.8	243.6

12.3.2 *Inversion*

Take 10 ml of this solution in a conical flask and add 1.5 ml of concentrated hydrochloric acid and about 10 ml of water. Heat the flask at 60 to 70°C for 10 min in a water bath. Cool immediately and transfer quantitatively the inverted solution to a volumetric flask and make up the volume to 100 ml.

12.3.3 Estimate reducing sugars in the inverted solution as in **7.2.2**.

12.4 Calculation

12.4.1 Refer col 3 of Table 3 for value corresponding to the titre and calculate the sucrose content as follows:

$$\text{Sucrose, percent by mass in the material} = \frac{0.95}{W}(Q - W\,R)$$

where

Q = value in col 3 of Table 2 corresponding to the titre,

W = mass, in g, of the material taken for the test (*see* **12.3.1**), and

R = percent reducing sugars by mass (*see* **7.7.1**).

13 DETERMINATION OF SULPHUR DIOXIDE BY THE ROSANILINE COLORIMETRIC METHOD

13.1 Field of Application

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

13.2 Principle

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

13.3 Reagents

13.3.1 *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.

13.3.2 *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 allow the solution to stand for at least 1 h before use.

13.3.3 *Formaldehyde Solution (approximately* 0.2 g/100 ml) — Dilute 5 ml of analytical reagent grade formaldehyde solution, ρ_{20} = 1.070 – 1.080 to 1 000 ml.

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

13.3.5 *Sodium Hydroxide Solution*, 0.1 mol/l.

13.3.6 *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 1 000 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.

13.3.7 *Concentrated Hydrochloric Acid,* ρ_{20} = 1.18 g/ml.

13.3.8 *Hydrochloric Acid Solution*, approximately 1 mol/l.

13.3.9 *Iodine (Starch) Indicator*, ready-made, or a starch solution.

13.3.10 *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 make up to the mark.

13.3.11 *Standard Sulphite Solution* — Dissolve approximately 2.5 g of general purpose reagent grade sodium sulphite heptahydrate in sucrose solution (*see* **13.3.4**) and make up to 500 ml with this pure sucrose solution (*see* **13.3.4**). 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* **13.3.8**) followed by approximately 100 ml of distilled water. 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* **13.3.10**) until the contents of the flask are a pale straw colour. Then add the iodine (starch) indicator (*see* **13.3.9**) (0.2 to 0.5 g) to the flask and continue the titration until the blue colour disappears. Record the titre, t.

13.3.12 *Dilute Standard Sulphite Solution* — Dilute 5 ml of standard sulphite solution (**13.3.11**) to exactly 100 ml with pure sucrose solution (*see* **13.3.4**). The exact value of the sulphite content, c, is calculated as follows from the titre, t, found in **13.3.11**:

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

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.

13.4 Apparatus

13.4.1 *Spectrophotometer or Colorimeter*, for use at approximately 560 nm.

13.4.2 *Volumetric Flasks*, 100, 500 and 1 000 ml, conforming to Class A of IS 915.

13.4.3 *Graduated Pipette*, 10 ml, conforming to Class A of IS 4162 (Part 1).

13.4.4 *Pipettes*, 2, 10 and 25 ml.

13.4.5 *Burette*,10 ml, graduated by 0.05 ml.

13.4.6 *Test Tubes*

13.4.7 *Analytical Balance*, capable of weighing to the nearest 0.1 mg.

13.5 Procedure

13.5.1 *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* **13.4.1**) at about 560 nm using distilled water as a reference.

13.5.2 *Standard Curve*

Pipette aliquots of the dilute standard sulphite solution (*see* **13.3.12**) (1, 2, 3, 4, 5 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* **13.3.4**) 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 min. Measure the absorbances as in **13.5.1** and plot the results on a graph.

The amount of SO_2 in each test tube is:

$$\frac{c \times n}{10} \ \mu gSO_2$$

where

n = the number of ml of dilute sulphite added to each 100 ml flask and c is from **13.3.12**.

13.6 Expression of Results

13.6.1 *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:

$$\frac{(\mu gSO_2 \text{ from graph}) \times 10}{\text{Mass of sugar used in } \mathbf{13.5.1}} \ \text{mg } SO_2/\text{kg sugar}$$

14 DETERMINATION OF INSOLUBLE MATTER BY MEMBRANE FILTRATION

14.1 Field of Application

The membrane filter method of Hibbert and Phillipson is used to determine the amount of water-insoluble matter in sugars.

14.2 Principle

The sugar to be tested is dissolved in hot water and filtered through a membrane filter of pore size 8.0 mm. The membrane and the retained insoluble matter are thoroughly washed, dried and weighed.

The insoluble matter content is calculated from the increase in mass of the membrane filter.

14.3 Reagents

14.3.1 *Chromatographic Spray Reagent* — 1-naphthol/ phosphoric acid solution. Dissolve 1.0 g of 1-naphthol 100 ml of ethanol and add 10 ml of ortho-phosphoric and (ρ_{20} = 1.69 g/ml).

NOTE — The ethanol used may be industrial methylated spirits 98.99 percent *m/m* total alcohols.

14.4 Apparatus

14.4.1 *Membrane Filters,* diameter about 50 mm, pore size 8.0 mm.

NOTE — Pore size is determined by bubble point testing.

14.4.2 *Glass Fibre Pre-Filters,* with an acrylic binder for the modified procedure only.

14.4.3 *Filtration Apparatus,* comprising a holder for the membrane filter (*see* **14.4.1**) fitted into a conical filtration flask, of capacity 4 litre, connected with a vacuum system.

14.4.4 *Stainless Steel Jug,* capacity 2 litre with a stainless steel stirring rod.

14.4.5 *Tweezers*

14.4.6 *Plastic Petri Dishes*

14.4.7 *Drying Oven,* maintained between 60 and 65°C.

14.4.8 *Square Mesh Sieve,* diameter 20 cm, mesh size about 0.4 mm. Place the sieve in a level base pan containing hot distilled water, in such a way that the water is just in contact with the mesh of the sieve. Cover the sieve with a lid.

14.4.9 *Analytical Balance,* readable to 0.1 mg.

14.4.10 *Balance,* capacity 5 kg. Capable of weighing to the nearest 1 g.

14.5 Procedure

Different procedures, **14.5.1** and **14.5.2**, are used according to whether the sugar filters with a good rate or poorly.

14.5.1 *Preparation of Membrane Filter*

For white sugars with a good filtration rate. Wash the membranes (*see* **14.4.1**) by immersion in boiling distilled water for 6 min. Drain the excess water from

the members and transfer individually to clean, dry petri dishes (*see* **14.4.6**) by using tweezers. Dry the membranes in their dishes with the lids removed for 1 h at 60 to 65°C in the drying oven. After drying replace the lids and cool for 30 min in a desiccator. Record the mass of the cooled membranes to the nearest 0.1 mg.

14.5.2 *Preparation of Membrane Filter and Pre-Filter*

For white sugar with poor filtration characteristics. Wash the membranes by immersing them in boiling distilled water for 6 min. Place a washed membrane into the filter holder and position a pre-filter on the top of the membrane. Pour 1 500 ml of hot distilled water at about 95°C through the filter to remove water-soluble material from the pre-filter. Remove the membrane and the pre-filter from the holder and place in a petri dish. Dry the membrane and the pre-filter in the dish with the lid removed for 1.5 h at 60 to 65°C. After drying replace the lid and cool in a desiccator for 30 min. Weigh the membrane with the pre-filter to the nearest 0.1 mg.

14.5.3 *Preparation of Sample Solution*

For refined sugars with an expected insoluble matter content of 20 mg/kg or less, weigh 1 000 ± 1 g of the sample directly into the stainless steel jug (*see* **14.4.4**). For plantation white sugars with an expected insoluble matter content 20 to 50 mg/kg reduce the sample mass to 500 ± 1 g. For plantation white sugars with an expected insoluble matter content greater than 50 mg/kg reduce the sample mass to 200 ± 1 g.

Add hot distilled water at about 95°C to the jug to give a final volume of about 1 800 ml. Stir the mixture with the stainless steel rod and heat to about 95°C; continue stirring until all the sugar has dissolved.

NOTE — Clothes for drying apparatus may be a serious source of contamination. It is therefore important that all apparatus should be rinsed thoroughly with distilled water immediately prior to use, but not dried with a cloth.

14.5.4 *Filtration of the Sugar Solution*

Moisten a weighed membrane filter by floating it on distilled water in the petri dish. Place the moistened filter in the filter holder (*see* **14.4.3**) and pass the hot sugar solution through the membrane filter under reduced pressure. Carefully rinse the jug and stirring rod into the filter holder with hot distilled water. Wash the retained insoluble matter and the membrane in the filter holder using a total volume of hot distilled wash water of about 1 000 ml.

NOTE — Do not allow air to be drawn through the membrane after washing, because there may be a significant amount of particulate matter in the atmosphere. In the case of white sugar with poor filtration characteristics moisten the membrane and pre-filter with distilled water and replace them in the filter holder ensuring that the pre-filter is not clamped by the filter holder. After filtering the hot sugar solution, use 1 500 ml of wash water instead of the 1 000 ml stated above.

14.5.5 *Final Washing of the Membrane Filter*

Carefully remove the membrane or the membrane with its pre-filter from the filter holder and place it/them on the wet mesh of the sieve (*see* **14.4.8**) for 1 h.

14.5.6 *Drying and Weighing of the Membrane*

After the final washing return the membrane or the membrane with its pre-filter to its/their original petri dish. Dry the dish with the lid removed in the oven for 1 h at 60 to 65°C. Replace the lid and cool the dish for 30 min in a desiccator. Re-weigh the membrane to the nearest 0.1 mg. For poor filtering sugars dry the membrane and pre-filter for 1.5 h.

The effectiveness of the final washing is essential to the accuracy of the test. This may be checked by spraying occasional membranes, after use, with the l-naphthol/phosphoric acid chromatographic spray reagent (*see* **14.3.1**), and heating to 105°C. The membrane should be entirely free of any trace of violet colouration.

14.6 Expression of Results

14.6.1 *Calculation*

The insoluble matter content of the sugar expressed in milligrams of insoluble matter per kilogram of sample is given by:

$$\text{Insoluble matter mg/kg} = \frac{m_2 - m_1}{m_0} \times 10^6$$

where

m_1 = mass in g of the membrane filter (*see* **14.5.1**) or mass of membrane + pre-filter (*see* **14.5.2**),

m_2 = mass in g of filter + insoluble matter or mass of filter + pre-filter + insoluble matter (*see* **14.5.6**), and

m_0 = mass in g of sample taken for test (*see* **14.5.3**).

15 DETERMINATION OF LEAD BY COLORIMETRIC METHOD

15.1 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.

15.2 Principle

15.2.1 *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 *p*H 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.

15.2.2 *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.

15.2.3 *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.

15.3 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.

15.3.1 *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.

15.3.2 *Dithizone Solution, Approximately* 20 mg/l — Prepare a fresh solution daily, or as required, by diluting 2 ml of the stock solution (*see* **15.3.1**) to 100 ml with analytical reagent grade chloroform. Store the solution in an amber bottle and in a cupboard away from the light.

15.3.3 *Ammonia Solution*, ρ_{20} ~ 0.88 g/ml.

15.3.4 *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* **15.3.1**) to ensure complete removal of heavy metals, a state indicated by the green colour persisting in the chloroform layer. Remove excess dithizone from the aqueous phase by successive extractions with small amounts of chloroform until the aqueous solution is colourless.

15.3.5 *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.

15.3.6 *Concentrated Nitric Acid*, ρ_{20} = 1.42 g/ml.

15.3.7 *Concentrated Hydrochloric Acid*, ρ_{20} = 1.18 g/ml.

15.3.8 *Concentrated Sulphuric Acid*, ρ_{20} = 1.84 g/ml.

15.3.9 *Concentrated Perchloric Acid*, ρ_{20} ~ 1.54 g/ml.

15.3.10 *Nitric Acid, Approximately 1 Percent (v/v)* — Dilute 10 ml of concentrated nitric acid (*see* **15.3.6**) containing less than 0.005 mg Pb/kg to 1 litre with lead-free water at 20°C.

15.3.11 *Nitric Acid, Approximately* 1 mol/l — Dilute 15.6 ml of concentrated nitric acid (*see* **15.3.6**) to 250 ml with lead-free water.

15.3.12 *Bromothymol Blue Indicator* — Dissolve 0.04 g in 20 percent ethanol and make up to 100 ml with 20 percent ethanol.

15.3.13 *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* **15.3.11**). Solution B is freshly prepared by diluting 10 ml of the stock solution A to 1 litre with distilled water.

15.3.14 *Hydroxylamine Hydrochloride Solution* — Dissolve 20 g of analytical reagent grade hydroxylamine hydrochloride in 100 ml of distilled water.

15.3.15 *Sodium Hexametaphosphate Solution* — Dissolve 10 g in 100 ml of distilled water.

15.3.16 *Magnesium Nitrate Solution* — Dissolve 10 g in 100 ml of distilled water.

15.3.17 *Chloroform*, ρ_{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 reagents 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 armoured glass screen.

15.4 Apparatus

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.

15.4.1 *Pipettes* — 5 and 10 ml, conforming to Class A of IS 1117.

15.4.2 *Graduated Pipettes* — 10 ml, conforming to Class A of IS 4162 (Part 1).

15.4.3 *Volumetric Flasks* — 100 and 1 000 ml, conforming to Class A of IS 915.

15.4.4 *Separating Funnels* — 100 ml.

15.4.5 *Microburettes* — 2 and 5 ml (graduated 0.05 ml) and 25 ml.

15.4.6 *Nessler Tubes*, 25 ml.

15.4.7 *Test Tubes* — Pyrex glass, 200 mm × 24 mm, marked at 10 and 25 ml.

15.4.8 *Platinum Dish*

15.4.9 *Muffle Furnace*

15.4.10 *Water Bath*, boiling

15.4.11 *Erlenmeyer Flasks* — 100 ml.

15.4.12 *Analytical Balance* — capable of weighing to the nearest 0.1 mg.

15.4.13 *Electrical Hot-Plate*

15.5 Procedure

15.5.1 *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* **15.3.3**) 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* **15.3.4**) and 1.0 ml of potassium cyanide solution (*see* **15.3.5**). Where it is known that iron is present in significant concentrations 1.0 ml of hydroxylamine hydrochloride solution (*see* **15.3.14**) should be added before the cyanide.

Stopper the separating funnel and mix the contents well by shaking. Add between 5 and 10 ml of the 20 mg/l dithizone solution (*see* **15.3.2**) 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* **15.3.10**). 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.

15.5.2 *Raw Sugar*

a) *Sample preparation by dry ashing* — Weigh 5 g of the sample in a clean platinum dish or silica crucible and treat with 10 ml magnesium nitrate solution (*see* **15.3.16**).

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 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* **15.3.7**) 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 **15.5.1** (Extraction of lead and Determination of lead).

15.5.3 *Low-Grade Products, Liquids and Solids*

a) *Sample preparation by wet ashing (calcium absent)* — Conduct all digestion operations in a fume cupboard. Refer to the note under **15.3**.

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* **15.3.8**) and 3 ml perchloric acid (*see* **15.3.9**) together with 3 glass beads. If necessary, warm gently to initiate charring.

Treat slowly with 2 to 3 ml concentrated nitric acid (**15.3.6**) 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 **15.3**).

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* **15.3.6**) and 2 ml of perchloric acid (*see* **15.3.9**). 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 30 s to dissolve any solid material present. Add 6 ml of ammonium citrate solution (*see* **15.3.4**) and 10 ml of sodium hexametaphosphate solution (*see* **15.3.15**). 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).

15.6 Expression of Results

15.6.1 *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:

$$\frac{10V}{M} \text{ mg P/b kg}$$

where

V = volume, in ml, of the dilute standard lead solution used in matching, and

M = mass, in g, of the original sample.

15.6.2 *Raw Sugar Calculation*

Where the sample mass used is 5 g then 1 ml of solution B is equivalent to 2 mg Pb/kg of raw sugar. Use the formula given in **15.6.1** for other sample masses.

15.6.3 *Low-Grade Products, Liquid and Solids Calculation*

Where the sample mass used is 2 g then 1 ml of solution B is equivalent to 5 mg Pb/kg of product, liquid or solid. Use the formula given in **15.6.1** for other sample masses.

16 DETERMINATION OF CHROMIUM CONTENT

16.1 Principle

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

16.2 Apparatus

16.2.1 *Atomic Absorption Spectrophotometer*

Spectrophotometer capable of operating at conditions as given under:

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

16.3 Reagents

16.3.1 *Deionized 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 manufacturer's instructions.

16.3.2 *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.

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

16.3.4 *Chromium Solution*

16.3.4.1 *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* 16.3.4.1)

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

16.3.4.2 *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 **16.2.1**. Add 1.5 ml nitric acid 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.

16.3.5 *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.

16.3.6 *Ammonium Pyrrolidine Dithiocarbamate Solution* – Dissolve 1 g ammonium pyrrolidine dithiocarbamate in 100 ml water. Prepare fresh daily.

16.4 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.

16.5 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 *p*H of the sample and standard solutions to 2.5 with hydrochloric acid using a *p*H-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 a 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.

16.6 Calculation

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

ANNEX A

(*Clause* 6.4.2)

DETERMINATION OF LEAD IN BASIC LEAD ACETATE SOLUTION

A-1 DETERMINATION OF TOTAL LEAD

A-1.1 Principle

Lead is quantitatively titrated with EDTA in the presence of hexamethylenetetramine added as a buffer in order to keep the *p*H above 5.6. The end-point is detected by the sharp change in colour of a metal indicator, xylenol orange, screened by methylene blue.

A-1.2 Reagents

Distilled water may be replaced by de-ionized water. It is recommended that the reagents are prepared from distilled water which has been boiled and subsequently cooled to eliminate carbon dioxide. Use analytical grade reagents unless stated or unless not available, for example for, indicators.

A-1.2.1 *Standard Lead Solution*, 0.05 mol/l — Weigh 3.312 g of lead nitrate, previously dried for 2 h at 103°C to 105°C and transfer to a 200 ml volumetric flask with distilled water. Dissolve and make up to the mark.

A-1.2.2 *EDTA Solution, Approximately* 0.05 mol/l — Weigh 18.6 g EDTA (disodium salt) and transfer to a 1 litre volumetric flask with distilled water. Dissolve and make up to the mark.

A-1.2.3 *Hexamethylene-tetramine (HMTA) Solution, Approximately* 1 mol/l — Weigh 140 g HMTA and transfer to a 1 litre volumetric flask with distilled water. Dissolve and make up to the mark.

A-1.2.4 *Xylenol Orange Solid Metal Indicator*, 1 percent *m/m* — Weigh 0.10 g xylenol orange and 9.90 g potassium nitrate. Grind both together with a mortar and pestle. Keep in a tightly stoppered bottle.

A-1.2.5 *Methylene Blue Indicator Solution*, 0.2 g/l — Weigh 40 mg methylene blue, dissolve in distilled water and make up to 200 ml.

A-1.2.6 *Acid Solution, Approximately* 1 mol/l — Prepare using acetic or nitric acid and distilled water.

A-1.3 Procedure

A-1.3.1 *Sample Preparation*

Filter the decanted basic lead acetate solution (*see* **6.4.2**) through a slow tiller paper. Pipette 10 ml into a 200 ml volumetric flask which already contains sufficient 1 mol/l acid solution to ensure a clear final solution. Add distilled water and if necessary, add more acid just to clear the solution, then make up to the mark. Record the total volume, V_{gt} of acid used.

A-1.3.2 *Titration*

Pipette 20 ml of the diluted wet lead solution prepared above in **A-1.3.1** into a 250 ml. Erlenmeyer flask. Add 20 ml HMTA solution from a measuring cylinder followed by about 100 mg of solid xylenol orange indicator and 4 drops of methylene blue solution.

Titrate the mixture with the 0.05 mol/l EDTA solution until the purple colour of the indicator changes to a neutral grey and then directly to green. During the titration, a white precipitate appears but this completely dissolves before the end point is reached. The end point corresponds to the first appearance of the green colour. Volume of EDTA solution is V_1.

A-1.3.3 *Standardization of the* 0.05 mol/l *EDTA Solution*

Pipette 20 ml of the standard 0.05 M standard lead solution into a 250 ml Erlenmeyer flask and titrate as described in **A-1.3.2**. Volume of EDTA solution used is V_2.

A-1.4 Calculation of Results

$$\text{Total lead (g PbO/100 ml)} = \frac{22.32\ V_1}{V_2}$$

A-2 DETERMINATION OF BASIC LEAD

A-2.1 Principle

The total lead is complexed by the addition of a stoichiometric quantity of EDTA previously determined by a total lead titration. The liberated acid is then titrated with sodium hydroxide solution in the presence of a *p*H indicator.

A-2.2 Reagents

The note about distilled water, given in **A-1.2** of the preceding method, is equally applicable. Use analytical grade reagents unless stated otherwise or not available, for example, for indicators. In addition to the reagents required for the total lead determination (*see* **A-1.2.1** to **A-1.2.6** in the preceding method), the following are required.

A-2.2.1 *Sodium Hydroxide Solution, Approximately* 0.1 mol/l

A-2.2.2 *Mixed Indicator Solution*, p*H* 7.4 — Dissolve 100 mg of sodium salt of bromothymol blue in distilled water to make 100 ml and call this solution A. Dissolve 100 mg of sodium salt of phenol red in distilled water to make 100 ml and call this solution B. Mix equal volumes of solutions A and B.

A-2.3 Procedure

A-2.3.1 *Preparation of Sample*

Prepare the sample solution as in the total lead determination (*see* **A-1.3.1** above). Note the volume, V_a, of 1 mol/l acid added.

A-2.3.2 *Titration* — Determine the total lead first by titrating 20 ml of the prepared solution with 0.05 mol/l EDTA as described in **A-1.3.2** of previous method. Volume of EDTA solution used is V_1.

Perform a further titration by pipetting 20 ml of the prepared solution into a 250 ml Erlenmeyer flask and adding an amount of 0.05 M EDTA solution equal to that required in the preceding titration (V_1) then adding a few drops of mixed indicator solution, and titrating the mixture with 0.1 mol/l sodium hydroxide solution.

At the end-point the colour changes from green through neutral grey to pale purple (*p*H = 7.4) and then to strong purple (*p*H = 7.6). Take the end-point when the solution becomes pale purple. The volume of sodium hydroxide solution used is V_3.

A-2.3.3 *Standardization of the* 0.1 mol/l *Sodium Hydroxide Solution*

Titrate 20 ml of the 0.05 M lead nitrate solution according to the procedure given in **A-2.3.2** above after adding V_2 ml of 0.05 mol/l EDTA solution. The volume of sodium hydroxide solution used is V_4.

A-2.3.4 *Standardization of* 1 mol/l *Acid Solution*

Pipette 20 ml of the 1 mol/l acid solution used in **A-2.3.1** into a 200 ml volumetric flask and make up to the mark with distilled water. Pipette 20 ml of this diluted solution into a 250 ml, Erlenmeyer flask and add a few drops of mixed indicator solution. Titrate with 0.1 mol/l sodium hydroxide solution. The volume of sodium hydroxide solution used is V_5.

A-2.4 Calculation of Results

$$\text{Basic lead content (g PbO/100 ml)} = 22.32\left[\frac{V_1}{V_2} - \frac{V_3}{V_4} + \frac{V_a}{V_4} \times \frac{V_5}{20}\right]$$

ANNEX B

(*Clauses* 6.4.2 *and* 6.5.6)

SPECIFICATION FOR POLARIMETER TUBES AND COVER GLASSES

B-1 POLARIMETER TUBES

Polarimeter tubes must be suitable for the instrument in which they are to be used and comply with requirements as to materials, shape and construction. The following specification contains the requirements common to all types of polarimeter tubes.

B-1.1 Material and Construction

B-1.1.1 The material of which the tubes are made, as well as their design, must be such as to obviate any change in their length being brought about by chance deformation. On tubes of flow-through type, the inlet and outlet tubes shall be as close to the ends as possible. The tubes shall be amenable to mechanical cleaning both inside and outside, and shall be made of materials that will withstand cleaning fluids of both high and low *p*H, including 50 percent acetic acid. The tubes shall have a high quality finish.

B-1.1.2 The clear aperture of the tubes shall not exceed 10 mm and shall not be less than 7 mm.

B-1.1.3 The ends of the tubes shall be square to their axis, flat and parallel within the limits given in Table 5.

B-1.2 Tolerances on Length

Two classes of accuracy are specified : Class A for the higher grade and Class B for the lower. The measured length of a tube shall not depart at 20°C from its nominal length by more than the amount shown in Table 6.

Table 5 Limits for the Squareness, Flatness and Parallelism of the End of the Polarimeter Tubes

(*Clause* B-1.1.3)

Sl No.	Nominal Length	Flatness, Maximum Errors	Parallelism, Maximum Errors	Squareness, Maximum Errors Minutes of Arc	Maximum Value of Surface Finish
	mm	µm	µm		µm
(1)	(2)	(3)	(4)	(5)	(6)
i)	10	1	1	2	0.2
ii)	20	1	2	3	0.3
iii)	50	2	4	4	0.4
iv)	100	3	6	6	0.6
v)	200	4	9	8	0.8
vi)	400	5	10	10	1.0

Table 6 Tolerances on Length of Polarimeter Tubes

(*Clause* B-1.2)

Sl No.	Nominal Length mm	Class A ± µm	Class B ± µm
(1)	(2)	(3)	(4)
i)	10	1	20
ii)	20	2	40
iii)	50	5	100
iv)	100	10	200
v)	200	20	400
vi)	400	40	800

B-1.3 Supports

The tube and its supports shall be so constructed that the axis of the tube is aligned with the axis of the light path within 0.5° of arc. The axis is usually 15 ± 0.1 mm above the bottom of the trough.

NOTE — If glass tubes with threaded metal collars are used, the collars shall be fitted so that they do not project beyond the glass lends of the tube, and the ends of the tube shall not project more than 1 mm beyond the collars.

B-1.4 Inscriptions

Each tube shall have permanently and legibly marked on it:

a) letter A or B, indicating the class of accuracy,
b) Identification number,
c) Nominal length, and
d) Maker's or Vendor's name or mark.

B-2 COVER GLASSES

Cover glasses shall comply with the following requirements:

B-2.1 They shall be made from clear, colourless optical glass, thoroughly annealed and free from internal strain, and of 1 to 2 mm thickness. The edges shall be slightly rounded.

B-2.2 They shall have faces with a good optical finish, flat within 0.01 percent of the length of the tube with which they are used, and parallel within 5 min of arc.

B-2.3 They shall be sufficiently free from birefringence that, when the polarimeter tube is rotated through 180° about its own axis, the variation in polarimetric reading shall not exceed 0.01°S.

ANNEX C

(*Clause* 6.5.7)

SPECIFICATION FOR QUARTZ CONTROL PLATE

C-1 DEFINITION OF NORMAL QUARTZ CONTROL PLATE

The normal quartz control plate is the quartz plate which shows the rotation value 40.765 +0.001° of arc at 20°C and the wavelength of the green line of the isotope 198 Hg = 546.227 1 nm. The sugar value of this plate is 100.000°S.

C-2 MATERIAL AND CONSTRUCTION

C-2.1 Quartz, including all parts covered by the mounting, should be completely free from all non-homogeneities such as twinning, striae and inclusions. No defects shall be visible on the surface of the plate when examined interferometrically.

C-2.2 A quartz double plate from dextrorotatory and laevorotatory quartz shall be used for values less than 24°S. The thickness of each plate shall be not less than 0.4 mm. The thickness of both plates together shall not exceed 1.6 mm.

C-2.3 The thickness of the plate shall not vary by more than 150 nm over the whole area of the plate.

C-2.4 Each surface of the plate shall be between two imaginary parallel planes 500 nm apart.

C-2.5 Squareness of the plate to the axis of the mounting tube should be within 10 min of arc.

C-2.6 The overall diameter of the plate must not be smaller than 15 mm, nor greater than 17 mm, and at least 4 mm greater than the clear aperture.

C-2.7 The aperture of the plate shall be at least 10 mm in diameter.

C-2.8 The angle between the optic axis of the plate and that normal to the faces of the plate must be less than 10 min of arc.

C-2.9 The axial play of the plate in its cell must be between 5 μm and 30 μm.

C-3 INSCRIPTIONS

Each quartz control plate shall have permanently and legibly marked on it:

a) Nominal value, in °S,
b) Corresponding wavelength,
c) Maker's or Vendor's name or mark, and
d) Identification number of the plate.

ANNEX D

(*Clause* 6.7.4)

METHOD 2 FOR STANDARDIZATION OF SUGAR POLARIMETER AND APPLICATION OF TEMPERATURE CORRECTIONS

D-1 PROCEDURE

D-1.0 This method requires that quartz plate readings be carried out periodically but not necessarily at the same time as test solutions are read. Standardization of the polarimeter is achieved by application of a scale correction derived by this procedure.

D-1.1 Take the following readings and record:

a) Measurements made at the time of periodic quartz plate readings:
   1) Polarimeter zero, that is, air reading, P_a.
   2) Quartz plate reading, Q_{tq}, at temperature t_q of quartz plate at time of reading.
   3) The temperature of the polarimeter, t_p, if it is of a type for which a temperature coefficient is prescribed, for example, quartz wedge instrument.
b) Measurements made at time of reading test solutions:
   1) Reading of a polarimeter tube filled with water, P_w.
   2) Readings of test solution, P_{tr}, at temperature t_r.
   3) The temperature of the polarimeter, t_{pr}, if it is of a type for which a temperature coefficient is prescribed.
   4) The temperature of making up to the mark. t_m will already have been recorded.

D-2 EXPRESSION OF RESULTS

D-2.1 Before the polarization corrected to 20°C can be calculated, determine the scale error derived from the quartz plate standardization data. The scale error so determined is often recorded in a book kept especially for this purpose and the scale correction to be applied may be a value averaged over some selected period.

Determine the scale error ∇, by correcting the observed quartz plate value to 20°C and comparing it with the certified value. In practice, calculate by adding to or subtracting from the observed quartz plate value, Q_{tq}, the quantities given below:

a) Subtract polarimeter zero, P_a
b) Subtract quartz plate temperature correction $= 0.000\ 144 \times Q_{20} \times (t_p - 20)$
c) Add Polarimeter temperature correction
   Quartz wedge: $0.000\ 144 \times Q_{20} \times (t_p - 20)$.
   Circular : No correction is needed unless indicated by manufacturer's specifications.

d) Subtract certified quartz plate value at 20°C, Q_{20}.

D-2.2 Calculate the polarization corrected at 20°C, P_{20}, by applying the corrections given below to the observed polarization, P_{tr}:

a) Subtract water and polarimeter tube correction, P_w

b) Add temperature of reading, t_r correction

t_r correction $= c \times P_{tr} \times (t_r - 20) - 0.004 \times R \times (t_r - 20)$,

where

c = coefficient in Table 1, and

R = reducing sugars, percent on sample

c) Subtract temperature of making to mark, t_m correction

t_m correction $= f \times P_{tr} \times (t_m - 20)$

where f = coefficient in Table 1

d) Add polarimeter temperature, t_p correction

Quartz wedge: t_p correction $= 0.000\ 144 \times P_{tr} \times (t_{pr} - 20)$.

Circular : No correction is needed unless indicated by manufacturer's specifications.

e) Subtract scale correction which is the scale error, ∇ calculated above (*see* **D-2.1**)

Thus,

$P_{20} = P_{tr} - P_w + t_r$ corr. $- t_m$ corr. $- t_{pr}$ corr. (if applic.) $- \nabla$

Express the results to 2 decimal places as °Z. Indicate in the result the corrections that have been made to the result observed without corrections (for example, 'corrected for instrument standardization and to a temperature of 20°C').

ANNEX E

(*Clause* 8.6.1)

DETERMINATION OF REFRACTOMETRIC DRY SUBSTANCE (RDS Percent)

E-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.

E-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.

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

E-3 APPARATUS

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

E-3.2 Light Source, for example, tungsten lamp.

E-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.

E-3.4 Thermometer, 150 mm, range 0 –50°C.

E-3.5 Beaker, capacity 50 ml.

E-3.6 Water Bath and Pump, Thermostatted generally at 20°C.

E-4 PROCEDURE

E-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 7 for corrections to be applied.

Table 7 Temperature Correction

(*Clause* E-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

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 – 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.

E-5 EXPRESSION OF RESULTS

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

E-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 8.

Table 8 International Refractive Index Scale for Pure Sucrose Solution at 20°C

(*Clause* E-5.1.1)

This table give 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	1.414 059	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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