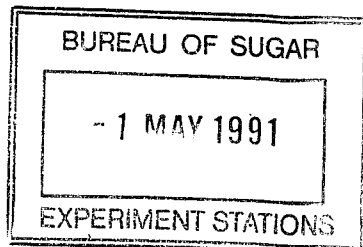


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**THE STANDARD LABORATORY MANUAL
FOR
AUSTRALIAN SUGAR MILLS**

**VOLUME 2
ANALYTICAL METHODS AND TABLES**

**BUREAU OF SUGAR EXPERIMENT STATIONS
BRISBANE, AUSTRALIA**

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CONTENTS

The various methods of analysis have been grouped under headings for ease of reference.

Definitions

Milling train products

- Method 1 Calibration of polarimeter by quartz plate check
- Method 2 Pol — Determination in juice
- Method 3 Brix — Determination in juice by hydrometer
- Method 4 Fibre — Determination in cane
- Method 5 Sampling and analysis for evaluating milling train performance
- Method 6 Pol — Determination in bagasse by wet disintegration
- Method 7 Moisture — Determination in bagasse and cane by drying
- Method 8 Total and soluble phosphate — Determination in cane juice
- Method 9 Starch — Determination in cane juices

Clarification products

- Method 10 Turbidity — Determination in clarified juice
- Method 11 Pol — Determination in filter cake
- Method 12 Moisture — Determination in filter cake
- Method 13 Fibre — Determination in juice and mud streams
- Method 14 Total insoluble solids and mud solids determination in juice and mud streams

Pan stage products and raw sugar

- Method 15 Pol — Determination in pan products
- Method 16 Brix (total solids) — Determination in molasses and cane invert by hydrometer
- Method 17 Brix (total solids) — Determination in sugar solutions by Abbe refractometer
- Method 18 Sucrose — Determination in mill products by double polarisation
- Method 19 Total solids (dry substance) — Determination in mill products
- Method 20 Reducing sugars — Determination in mill juices, clarified juices and pan products by the constant volume modification of the Lane and Eynon method
- Method 21 Reducing sugars — Determination in mill products by the Lane and Eynon method
- Method 22 Total sugars (after inversion) — Determination in molasses and cane invert by the constant volume modification of the Lane and Eynon method
- Method 23 Supersaturation and saturation conditions — Determination in pan products
- Method 24 Viscosity determination in pan products

- Method 25 Filtrability — Determination in products other than raw sugar
- Method 26 Ash — Determination in sugar products by the single sulphation method
- Method 27 Ash — Determination in sugar products by the double sulphation method
- Method 28 Dextran — Determination in raw sugar and sugar products by the alcohol haze method
- Method 29 Phenolic acids — Determination in raw sugar, syrups and cane juice
- Method 30 Polarisation — Determination in raw sugar using visual or automatic polarimeters
- Method 31 Moisture — Determination in raw sugar by drying at atmospheric pressure
- Method 32 Reducing sugars — Determination in raw sugar by the Lane and Eynon method
- Method 33 Total colour (colour index) — Determination in raw sugar
- Method 34 Affined colour — Determination in raw sugar
- Method 35 Amino nitrogen — Determination in raw sugar and mill products
- Method 36 Filtrability — Determination in raw sugar by the celite method
- Method 37 Starch — Determination in raw sugar
- Method 38 Acid-soluble phosphate — Determination in raw sugar by the amidol method
- Method 39 Particle size and size distribution (grist) — Determination in raw sugar
- Method 40 Mean elongation — Determination in raw sugar

Boiler waters, effluents and cleaning solutions

- Method 41 Alkalinity, total alkalinity and caustic alkalinity — Determination in water
- Method 42 Chloride — Determination in caustic (NaOH) cleaning solutions
- Method 43 Chloride — Determination in water
- Method 44 Soluble phosphate — Determination in water by the amidol method
- Method 45 Total dissolved solids — Estimation in water by conductivity measurement
- Method 46 Sulphite — Determination in water
- Method 47 Total hardness (calcium and magnesium) — Determination in water by EDTA titration
- Method 48 Dissolved oxygen (D.O.) — Determination in water by the azide modification
- Method 49 Sucrose soluble alkali — Determination in lime
- Method 50 Sucrose — Determination in effluents by the phenol-sulphuric acid method
- Method 51 Biochemical oxygen demand (BOD₅) — Determination in liquid effluents



REFERENCE TABLES

Table No.			
I	Temperature corrections to readings of brix hydrometers (calibrated at 20 °C).	XVII	Celite 505 standard filter aid
II	Schmitz's table for sucrose (pol) in juice for use in the dry lead method with undiluted solutions. Normal Weight of 26.000 g.		Weights of pure sugar syrup filtered (between 2 and 7 minutes after application of pressure) at various final temperatures, under the standard conditions of the filtrability test.
III	Table of factors for the calculation of pol per cent juice from pol reading for use in the dry lead method with undiluted solutions.	XVIII	Specific rotation of sugars.
IV	Factors for the calculation of sucrose according to Jackson and Gillis Method 2 of double polarisation.	XIX	Crystal contents of massecoites.
V	Temperature corrections for plain polarisation reading in determination of sucrose by double polarisation.	XX	Degree of supersaturation — all values being prefixed by 1.
VI	% Sucrose corresponding to various values of (P-I) for half normal solutions at various temperatures, for use in Jackson and Gillis Method 2 of double polarisation.	XXI	Stock recovery.
VII	% Pol in bagasse This Table gives the % pol in bagasse (at various bagasse moistures) corresponding to the polarimeter reading of wet disintegrator fluid (1 part bagasse to 10 parts water) when read in a 200 mm tube on a sugar polarimeter for wet disintegrator fluid purity of 75-80.	XXII	Density(g/mL) of water at temperatures from 0 to 102 °C. According to M. Thiesen, Wiss. Abh. der Physikalisch-Technischen Reichsanstalt, 4, No. 1; 1904.
VIII	Refractive indices of sugar solutions at 20 °C in air at 20 °C, 760 mm pressure and 50 per cent relative humidity.	XXIII	Corrections for temperature (in g) to be added to weight of water contained to obtain volume (in mL) of vessel at 20 °C. Nominal capacity 1000 mL (for vessels made of soda glass).
IX	Table of temperature corrections for the Abbe refractometer calibrated at 20 °C.	XXIV	Corrections for atmospheric pressure (in g) to be added to or subtracted from the weight of water contained to obtain volume (in mL) of vessel at standard temperature and pressure.
X	The refractive index of pure water at various temperatures, relative to air (sodium line).	XXV	Requirements for apparatus for use in the analysis of cane for payment purposes.
XI	Temperature corrections to be applied to refractometer readings of sucrose solutions of various concentrations, to obtain the refractive index at 20 °C.	XXVI	Corrections to quartz plate polarisation readings for the temperature of the quartz plate.
XII	Degrees brix, true densities and concentrations of sucrose solutions at 20 °C corresponding to refractive index reading.	XXVII	Corrections to polarisations for temperatures of making to the mark — (C_{tm}).
XIII	Solubility of sucrose in water in g sucrose(S) per 100 g water. According to Charles, Amer. Chem. Soc., 1958 Abst. of Papers p. 100. Reported in Honig "Principles of Sugar Technology", 2, 228.	XXVIII	Corrections to polarisations for temperatures of reading — (C_{tr}).
XIV	Solubility of sucrose in water in g sucrose(S) per 100 g solution (Charles).	XXIX	Corrections to polarisation reading for the effect of polarimeter temperature.
XV	Densities of solutions of cane sugar at 20 °C in g/mL. (This table is the basis for standardising hydrometers indicating per cent of sugar at 20 °C).	XXX	Milligrams of reducing sugars required to reduce 10 mL Fehling's Solution (Lane and Eynon method).
XVI	Brix, apparent density, apparent specific gravity, and grams of sucrose per 100 mL of sugar solutions.	XXXI	Showing grams of product to be weighed out per 100 mL of titrating solution, for the determination of reducing sugars in products of various sucrose and reducing sugar contents.
		XXXII	Grams of reducing sugars (as invert sugar) per 100 mL of titrating solution for various end points and sucrose concentrations of 25 g/100 mL.
		XXXIII	Grams of reducing sugars (as invert sugar) per 100 mL titrating solution for various end points, and sucrose concentrations of 0-3 g/100 mL.
		XXXIV	Properties of saturated steam.
		XXXV	Equivalents. Volume and capacity equivalents. Mass equivalents. Density equivalents. Linear measure equivalents.

Surface and area equivalents.

Pressure equivalents.

Heat, energy and and work equivalents.

Heat flow equivalents.

XXXVI Mensuration of surfaces and solids.

XXXVII Capacity of horizontal cylindrical tanks at varying levels.

XXXVIII Boiling point elevation of sugar solutions and cane juices ($^{\circ}\text{C}$) at 760 mm pressure.

XXXIX International atomic weights, 1971 (Published by Perry and Chilton, "Chemical Engineer's Handbook" Fifth Edition).

PREFACE TO VOLUME 2

THE STANDARD LABORATORY MANUAL FOR AUSTRALIAN SUGAR MILLS

Volume 2 of the Laboratory Manual, Analytical Methods, is a departure from the format and binding of previous manuals.

In this edition an A4 format has been adopted with methods and tables bound in a ring binder which will open flat. This is to facilitate the use of the manual as a laboratory workbook.

Methods are numbered and started on separate sheets with page numbering within methods. This is to facilitate the replacement of methods which became obsolete. In this way method updating should be able to be carried out at more frequent intervals and extra methods added as required.

The production of this volume has been undertaken by a group comprising:

Mr P.G. Atherton	BSES
Mr A.G. Noble	BSES
Mr J.B. Lee	CSR
Mr G.S. Rowe	CSR
Mr P.C. Ivin	SRI
Mr D.H. Foster	SRI

Assistance with writing and checking of various sections has been given by a number of members of staff of BSES, Sugar Research Institute and CSR. The work of all concerned is gratefully acknowledged.

C.C. Ryan
Director of Sugar Experiment Stations

NOTES

NOTE 1: Change to Polarimeter Scale

At the 19th Session of the International Commission on Sugar Analysis (ICUMSA), held in 1986, an important change was made to the definition of the International Sugar Scale. The new scale was set in force internationally on July 1st 1988, and was adopted for cane payment in Queensland for the 1989 season.

The definition of the normal sugar solution remains unchanged, i.e. 26.000 grammes of pure sucrose weighed with brass weights in air under normal conditions (1013 kPa, 20 °C, 50% RH) dissolved in pure water and made up to 100.000 cm₃ at 20 °C.

However, the rotation of this solution, measured in a 200.00 mm tube at 20 °C with light of a wavelength of 546.2271 nm has been changed, following precise recalibration, from 40.765 degrees to 40.777 degrees. A table of rotations at various wavelengths is set out below:

Angular rotation of 100 °Z point

Vacuum wavelength, nm	Angular rotation	
	New value, degrees	Previous value, degrees
546.2271	40.777	(40.765)
587.0000*	34.934	(34.924)
589.4400**	34.626	(34.616)
632.9914	29.751	(29.743)

* Effective wavelength of the standard quartz wedge saccharimeter

** Mean effective wavelength of spectrally filtered sodium yellow light.

Readings taken using the new scale are designated °Z to distinguish them from °S. In practical terms the change means that rotations on the new scale (°Z) are 0.99971 of rotations on the old (°S) scale, and this correction factor can be used to correct old values in °S to new values in °Z.

Polarimeters and quartz plates are now standardised in °Z by BSES.

A full dissertation on this change can be found in an article by Dr M.R. Player in the July 1988 edition of the International Sugar Journal, pages 130-131.

NOTE 2: C.C.S. Formula

In order to maintain equity for cane payment a change was made to the c.c.s. formula.

In effect, a factor of 1.00026 has been added into the formula so that it now reads:

$$\text{C.C.S.} = 1.00026 (\text{Pol in cane} - \frac{1}{2} \text{ Impurities in Cane}).$$

DEFINITIONS

Error

An error is a divergence from the truth. The word is generally used to denote the difference between the measured value and the "true" value, although the "true" value is usually unknown. There are two important categories of error: random and systematic.

Random Error

When a given measurement is repeated there is, in general, disagreement between the resulting individual values. An error which individually is unpredictable is a random error. In the long run the average of random errors tends towards zero.

Systematic Error

A systematic error is an error which persists during a series of the same or similar measurements or analyses, and which is not eliminated by the simple process of averaging.

Determinate and Indeterminate Errors

Determinate errors are those which can be evaluated by some logical procedure, either theoretical or experimental. Errors which cannot be so evaluated are called indeterminate.

Random errors are determinate because they can be evaluated by application of statistical theory. Systematic errors may sometimes be evaluated by calibration with standards or by subsidiary experiments.

Precision

Precision is a general term for the closeness of agreement between the results of replicate measurements. If a procedure has small random errors it is said to have high precision. Depending on circumstances, the precision of a procedure may be specified by one or more of the following terms: standard deviation, repeatability and reproducibility.

Standard Deviation

The standard deviation is the square root of the quantity equal to the sum of squares of the deviations of individual results from the mean value divided by one less than the number of results in the set.

$$s = \sqrt{\frac{1}{n-1} \sum_{i=1}^n (X_i - \bar{X})^2}$$

Note that the term "set" is defined as referring to the number 'n' of independent replicate measurements of some property.

The variance, s^2 , is the square of the standard deviation and is the unbiased estimate of the true, but unknown, variance which is generally given the symbol σ^2 .

Relative Standard Deviation (RSD)

This is the magnitude of the deviation compared with the mean value:

$$\text{RSD} = \frac{s}{\bar{X}}$$

Repeatability

The value at or below which the absolute difference between two single test results obtained with the same method on identical test material under the same conditions (same operator, same apparatus, same laboratory and a short interval of time) may be expected to lie with a specified probability; in the absence of other indications, the probability is 95%.

The 95% repeatability is calculated as $2\sqrt{2} s_r$ where s_r^2 is the unbiased estimate of the within-laboratory variance.

Reproducibility

The value at or below which the absolute difference between two single test results obtained with the same method on identical test material under different conditions (different operators, different apparatus, different laboratories and/or different time) may be expected to lie with a specified probability; in the absence of other indications, the probability is 95%.

The 95% reproducibility is calculated as $2\sqrt{2} s_R$ where s_R^2 is an unbiased estimate of the sum of the between-laboratory variance and the within-laboratory variance.

Accuracy

Accuracy is a measure of how close the result is to the true value. A measurement is said to be accurate if the best value or mean value of a set of observations shows no discernible deviation from the true value, i.e. the systematic error is small.

Precision and Accuracy

Measurements may be precise without being accurate, i.e. observation may show small scatter although their best or mean value departs from the true value.

Measurements may be accurate without being precise, i.e. the best or mean value may not deviate from the true value, although the scatter of observations may be wide.

References

- Beers, Y. (1958). "Introduction to the Theory of Errors" Addison-Wesley Publishing Co. 2nd Edition, 1958. BS 5497 — Part 1: 1979.
- Youden, W.J. and Steiner, E.H. (1979). "Statistical Manual of the Association of Official Analytical Chemists". AS2667 — 1983. Chemical Testing — Sampling — Glossary of Terms.
- AS2850 — 1986. Chemical Analysis — Interlaboratory Test Programs — for determining Precision of Analytical Method(s) — Guide to the Planning and Conduct.

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METHOD 1

CALIBRATION OF POLARIMETER BY QUARTZ PLATE CHECK

1.0 SCOPE AND FIELD OF APPLICATION

This method describes a procedure for calibrating manual and automatic polarimeters using a quartz plate as the standard. The procedure determines the "scale correction" for the polarimeter, which corrects for the calibration error of the polarimeter at 20°C. This correction is added to the polarimeter reading when calculating a polarisation result, using the method, "Polarisation — Determination in Raw Sugar".

Scale Corrections outside the range 95°Z to 100°Z

This method determines the scale correction near 100°Z (95°Z to 105°Z). For readings outside this range a proportional scale correction can be used. For example, if the scale correction at 100°Z (SC) is +0.10°Z, then the scale correction at 30°Z (SC₃₀) would be +0.03°Z.

2.0 PRINCIPLE OF METHOD

Initial Scale Correction

An initial scale correction is determined if:

- the polarimeter scale correction has not been measured for an extended period (more than 15 days).
- maintenance has been carried out on the instrument which is likely to have affected its calibration (for example: removal of the scale or adjustment of the interference filter).

Running Average Scale Correction

The "running average scale correction" is the average of the last ten scale correction determinations, and is the scale correction applied during the normal operation of a laboratory. This procedure helps to "smooth out" the normal variances of individual scale correction determinations. As the calibration of a polarimeter does not normally change suddenly, the average of ten readings will accurately represent the calibration of the polarimeter if all the readings have been taken within the last 15 days.

Polarimeters Used for Cane Payment

Polarimeters which are used for cane payment in sugar mills should have a scale correction between -0.1°Z and +0.1°Z.

3.0 CAUSES OF CALIBRATION CHANGE

The calibration of manual polarimeters is very stable, as the calibration can normally only be affected by changing the alignment of the scale.

While the calibration stability of automatic polarimeters is good, they are more prone to drift than manual polarimeters. The most common cause of drift is the ageing of the interference filter used in most automatic circle polarimeters. This is normally a slow process (less than .01°Z per month) but can become as high as .01°Z per day at the end of the filter's life. There are a number of other electronic and optical problems which can cause calibration change.

NOTE: Changing the "REL number" on the "Polar-tronic" polarimeter will change its calibration and therefore the scale correction.

4.0 DEFINITIONS

Q₂₀ — The certified value of the quartz plate at 20°C.

Q_{tp} — The polarisation reading (°Z) of the quartz plate at temperature t_q.

t_q — The temperature of the quartz plate (°C) at the time of reading.

t_p — The ambient temperature of the polarimeter (°C) (assumed to be equal to t_q in this method).

C_{tp} — The correction to be added to the quartz plate polarisation reading to correct for the temperature of the quartz plate.

C_{tp} — The correction to be added to the polarisation reading to correct for the temperature of the polarimeter.

SC — The scale correction of the polarimeter at 100°Z to correct for the scale calibration error at an ambient temperature of 20°C.

SC_p — The scale correction to be used when the polarisation reading (P) is outside the range 95°Z to 105°Z.

5.0 APPARATUS

5.1 **Thermometer**, length 20 cm, 10-35°C, readable to 0.1°C.

5.2 **Certified quartz control plate** in the range 95°Z to 105°Z.

Quartz control plates shall be standard plates which have been certified by BSES, Brisbane, or the National Measurement Laboratories (CSIRO, Sydney). Quartz plates should be recertified every five years.

6.0 INSTRUMENTS

The polarimeter under test.

6.1 **Care of Polarimeter.** Keep splash glasses clean. If the glasses have to be removed for cleaning, care should be taken when reassembling that they are not screwed too tightly — thus being strained and becoming optically active. Variable readings when a sample is rotated in the trough can indicate stress in the optical components of the sample or the instrument.

Clean the air filters on automatic polarimeters regularly (at least once per month) and keep air outlets clear to allow a free flow of air.

6.2 **Location of Polarimeter.** Locate the polarimeter where it will not be heated by direct radiation (e.g. sunlight) or be subjected to excessive vibration.

6.3 **Thermometer Location.** With manual polarimeters place the thermometer in the polarimeter trough or in a holder near the polarimeter. Do not place the thermometer in the trough of automatic polarimeters as this temperature will be above ambient and will not indicate the temperature of the quartz plate. A position near an air *inlet* of the polarimeter is most suitable.

METHOD 1 P.2.

6.4 **Quartz Plate.** Store the quartz plate in a holder near the polarimeter such that it is not heated by direct radiation or by warm air currents from the instrument. With automatic polarimeters, a position near an air inlet is most suitable. Store the quartz plate in this position for at least 30 minutes before measurements are carried out in order that it reaches thermal equilibrium with the thermometer and the polarimeter.

7.0 PROCEDURE

A. SCALE CORRECTION DETERMINATION WITH AUTOMATIC POLARIMETERS

- 7.1 Record the *temperature of the quartz plate*, t_q to 0.1°C (it is not necessary to record the temperature with quartz wedge instruments — see 8.6). Allow the reading of the empty polarimeter to stabilise and *reset the zero*.
- 7.2 Place the quartz plate in the polarimeter trough with a minimum of handling and close the lid gently.
- 7.3 Allow the polarimeter reading to stabilise and record the reading. Obtain four readings rotating the quartz plate through 45° between each reading. Ensure the lid is closed before reading. Average the four readings and record to the nearest 0.01°Z. This is the *quartz plate reading*, Q_{tq} at temperature t_q .
- 7.4 Remove the quartz plate, close the lid and check that the polarimeter returns to zero $\pm 0.02^\circ\text{Z}$. If not, repeat 7.1 to 7.4.

NOTE: IF AFTER A SECOND SET OF READINGS THE ZERO STILL DOES NOT RETURN TO $\pm 0.02^\circ\text{Z}$, A FAULT IN THE INSTRUMENT IS INDICATED. THE INSTRUMENT SHOULD BE CHECKED BY A QUALIFIED SERVICEMAN.

- 7.5 See Section 8 for the calculation of scale correction.
- #### B. SCALE CORRECTION DETERMINATION WITH MANUAL POLARIMETERS
- 7.6 Place the quartz plate in the polarimeter trough with a minimum of handling and close the lid gently.
- 7.7 Set the scale to read the approximate value of the quartz plate and adjust the focus of both the field and the scale telescopes. Rotate the scale setting knob alternately left and right with decreasing amplitude, until colour and intensity of all sections of the field are equal. Always close lid before balancing the field. Read the scale setting to the nearest 0.05°Z for B&S polarimeters or 0.01°Z for S&H polarimeters. (If necessary interpolate between direct vernier readings).
- 7.8 Take two or more sighter readings, do not record. Record the next four readings, rotating the quartz plate 45° between readings. Check and record all digits for at least the first and last of the four readings. If all four readings are not within a range of 0.20°Z, take another set of four readings.
- 7.9 Average the four readings and record to 0.01°Z. This is the *quartz plate reading*, Q_{tq} at temperature, t_q .
- 7.10 Open the trough and read the temperature to the nearest 0.1°C and record. This is the *temperature of the quartz plate*, t_q at the time of reading. It is not necessary to record this temperature with quartz wedge polarimeters (see 8.6).
- 7.11 Remove the quartz plate and close the lid.
- 7.12 To obtain the zero reading set the scale of the

polarimeter to read approximately $\pm 0.2^\circ\text{Z}$. Check the focus of both telescopes and ensure that the trough lid is closed. Again balance colour and intensity of the fields and record the reading. Slightly offset the scale and repeat the balancing and reading. In this way, record four readings. If all four of the readings are not within a range of 0.20°Z, discard and take another set of four readings. Average the four readings and record to the nearest 0.01°Z. This is the *polarimeter zero*, P_o .

7.13 See Section 8 for the calculation of scale correction.

C. DETERMINATION OF AN INITIAL SCALE CORRECTION

- 7.14 To determine an "Initial Scale Correction" at least 20 in-tolerance scale corrections are required. To be in tolerance, all results must be within $\pm .04^\circ\text{Z}$ of the average of the results.
- 7.15 The scale correction determinations should be carried out over a period of 2 to 15 days. To minimise systematic errors, each of the determinations should be separated by a period of at least ½ hour.
- 7.16 It is desirable that all of the sugar analysts who will be using the polarimeter contribute equally to the total of 20 determinations required. This is particularly important with manual polarimeters.

8.0 CALCULATIONS

A. CALCULATION OF A SINGLE SCALE CORRECTION VALUE

- 8.1 The following readings have been recorded:
 Q_{tq} — the quartz plate reading at temperature t_q
 t_q — the temperature of the quartz plate and the polarimeter at the time of reading (required for circle polarimeters only)
 P_o — the polarimeter zero reading (manual polarimeters only)
 Q_{20} — the certified value of the quartz plate at 20°C.
- 8.2 Calculate the average quartz plate reading, Q_{tq} .
- 8.3 Determine the correction to the quartz plate polarisation for the temperature of the quartz plate, C_{tq} from Table XXVI (required for circle polarimeters only).
- 8.4 Calculate the average zero reading, P_o (for manual polarimeters only).
- 8.5 Determine the correction to the polarisation reading for the polarimeter temperature, C_{tp} from Table XXIX (only required for circle polarimeters with an interference filter). Note that $t_p = t_q$.
- 8.6 Calculate the scale correction at 100°Z (SC), from:
$$\text{SC} = Q_{20} - (Q_{tq} + C_{tq} + C_{tp}) + P_o$$

NOTES:

- This equation is an approximation which has an error of less than 0.01°Z when:
 - The scale correction is between -0.2°Z and $+0.2^\circ\text{Z}$.
 - The certified value of the quartz plate is between 95°Z and 105°Z.If these conditions are not satisfied then the following proportional (exact) equation should be used:
$$\text{SC} = (Q_{20} - (Q_{tq} + C_{tq} + C_{tp}) + P_o) \times \frac{100}{Q_{20}}$$
- With quartz wedge compensation polarimeters the terms C_{tq} and C_{tp} will cancel each other in the equation of 8.6 (provided that the quartz plate temperature is the same as the polarimeter ambient

temperature). Hence, it is not necessary to measure the temperature with quartz wedge instruments.

3. If a scale correction outside the range 95°Z to 105°Z is required, it can be calculated from the equation:

$$SC_p = \frac{P}{100} \times SC$$

where: SC is the scale correction at 100°Z.

P is the polarisation in °Z at which the instrument is being used,

SC_p is the scale correction for a polarisation reading of P°Z.

B. CALCULATION OF AN INITIAL SCALE CORRECTION

- 8.7 At least 20 individual scale correction results must be determined (7.14-7.16 and 8.1-8.6). Calculate the average scale correction from all results.
- 8.8 Determine the result which is furthest from the calculated average scale correction. If this result is more than ± .04°Z from the average scale correction, then eliminate it from the list and re-calculate the average scale correction.
- 8.9 Repeat 8.8 until all results are in tolerance. If there are

fewer than 20 results remaining, then further determinations should be made.

- 8.10 The average of at least 20 in-tolerance scale correction determinations is the initial scale correction for the instrument.

C. CALCULATION OF THE RUNNING AVERAGE SCALE CORRECTION

- 8.11 The "Running Average Scale Correction" is determined by calculating the average of the last ten scale correction determinations.
- 8.12 None of the ten values which are used to calculate the running average scale correction should be more than 15 days old. It is desirable to make at least one determination per day. However, all ten values should not be taken on the one day.
- 8.13 If a scale correction determination is more than ± 0.04°Z from the current running average scale correction then the determination should be repeated. If the repeat determination is also out of tolerance then the polarimeter should be checked, and an initial scale correction should then be established.

SCALE CORRECTION DETERMINATION WORK SHEET (Method 1)

Quartz Plate No. Analyst Date

Polarimeter.....

READINGS		CALCULATIONS	
Q.P. readings	(1) °Z (2) °Z (3) °Z (4) °Z	Q.P. Certified Value, Q ₂₀	°Z
Ave Q.P. reading, Q _{tq}	_____ °Z	Q.P. Reading, Q _{tq}	°Z
Q.P. Temp, t _q	_____ °C	Correction C _{tq} (Table XXVI)	°Z
Polarimeter Temp. t _p = t _q		Correction C _{tp} (Table XXIX)	°Z
Zero readings	(1) °Z (2) °Z (3) °Z (4) °Z		
Ave zero reading, P ₀	_____ °Z	Zero reading, P ₀	°Z

CALCULATE THE SCALE CORRECTION AT 100°Z FROM EQUATION:

$$SC = Q_{20} - (Q_{tq} + C_{tq} + C_{tp}) + P_0$$

SC =

SC =

METHOD 1 P.4.

**WORKED EXAMPLE
SCALE CORRECTION DETERMINATION
WORK SHEET (Method 1)**

Quartz Plate No. 1487 Analyst U.R. Smart Date 29.4.85

Polarimeter Manual B & S

READINGS			CALCULATIONS	
Q.P. readings	(1)	98.95 °Z	Q.P. Certified Value, Q_{20}	98.90 °Z
	(2)	98.90 °Z		
	(3)	99.05 °Z		
	(4)	99.00 °Z		
Ave Q.P. reading, Q_{tq}		98.98 °Z	Q.P. Reading, Q_{tq}	98.98 °Z
Q.P. Temp, t_q		24.8 °C	Correction C_{tq} (Table XXVI)	-0.07 °Z
Polarimeter Temp. $t_p = t_q$			Correction C_{tp} (Table XXIX)	- °Z
Zero readings	(1)	-0.10 °Z		
	(2)	+0.05 °Z		
	(3)	0.00 °Z		
	(4)	-0.05 °Z		
Ave zero reading, P_o		-0.03 °Z	Zero reading, P_o	-0.03 °Z

CALCULATE THE SCALE CORRECTION AT 100°Z FROM EQUATION:

$$SC = Q_{20} - (Q_{tq} + C_{tq} + C_{tp}) + P_o$$

$$SC = 98.90 - (98.98 - 0.07 + 0) - 0.03$$

$$SC = -0.04°Z$$

METHOD 2

POL — DETERMINATION IN JUICE

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian sugar industry. It complies with the requirements of the "Regulation of Sugar Cane Prices Acts 1962-1981" for the analysis of 1st expressed juice for payment for cane delivered to mills.

The method is applicable also to the determination of pol in all other cane and mill juices.

2.0 PRINCIPLE

A sample of the juice is clarified by adding basic lead acetate powder (which coagulates colloidal impurities and removes some colorant) followed by filtration. The pol of the clarified solution is read in a polarimeter tube of standard length in a sugar polarimeter.

If the juice from deteriorated cane cannot be clarified by basic lead acetate powder, a solution of lead nitrate/sodium hydroxide (Herles' Reagent) is used.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

3.1 **Sugar Polarimeter.** All sugar polarimeters for first expressed juice analysis for cane payment purposes must be certified for use in the Australian Sugar Industry by BSES. Automatic sugar polarimeters should conform to the Australian Standard K157.

3.2 **Quartz Control Plates.** Quartz control plates must be certified by BSES or by the National Measurement Laboratories (CSIRO, Sydney).

3.3 **Polarimeter tube,** 200 mm long, and cover glasses, conforming with ICUMSA (1974), Subject 2, specifications. A standard or a continuous pol tube can be used. Pol tubes should be certified by BSES.

3.4 **Juice can,** plastic or metal.

3.5 **Stemless filter funnel,** glass or plastic, 2.5 cm dia. 100 mL capacity.

3.6 **Filter glass** or conical beakers, 300 mL.

3.7 **Boiling flask** (florencia flask), 250 mL.

3.8 **Filter paper,** Whatman No. 91 or equivalent.

3.9 **Cold water bath** (not needed for 1st expressed juice).

4.0 REAGENTS

4.1 Basic lead acetate powder (Horne's Dry Lead), having the following specifications:

Basic Lead (as PbO)	: not less than 33%
Total Lead (as PbO)	: not less than 75%
Basicity	: 41.3 - 50.7%
Loss on Drying (2 hr at 105°C)	: not exceeding 1.5%
Insoluble Matter (1 g in 50 mL water)	: not exceeding 2.0%
Insoluble Matter (5 g in 100 mL of 10% acetic acid)	: not exceeding 0.05%
Fineness — through 425 micron	: 100%
— through 125 micron	: not less than 70%

4.2 Herles' Reagent

Solution A — Dissolve 50.0 ± 0.1 g sodium hydroxide in water and dilute to 1 litre in a volumetric flask.

Solution B — Dissolve 500.0 ± 0.1 g lead nitrate in water and dilute to 1 litre in a volumetric flask.

To check the reagent mix equal volumes of solutions A and B and measure the pH of the mixed solution. If the pH is above 7.0, solution A is diluted until the resultant mixed solution gives a pH below 7.0.

Safety

Lead is a highly toxic, cumulative poison. Persons who use or are exposed to dry lead over long periods should have an annual blood test through local hospitals or occupational health authorities.

Label containers : Label all containers, in which any lead solution or compounds is stored or used, with the name and also "Poison".

Avoid ingesting lead : Always wash hands thoroughly before eating. Always wear a mask and avoid breathing dust when handling powder. Do not drink from laboratory glassware or taps.

Avoid absorbing lead : Dry hands with hand towel, not laboratory glassware towel. Wear gloves and protective clothing where possible. Keep lead reagent and leaded solutions away from cuts and abrasions.

Display precautions : On laboratory notice board.
Antidote : Induce vomiting with 10% aqueous magnesium sulphate

and follow with milk or egg white in cold water. Seek medical attention.

Disposal of lead waste: Place all wastes containing any lead reagents into a plastic bucket which is half filled with dry soda-ash (sodium carbonate) powder. The bucket should have a tightly fitting lid and be clearly marked "Poison-Lead Waste". Accumulated lead waste should be disposed of to an authorised trade waste authority.

5.0 CALIBRATION OF THE POLARIMETER

The polarimeter is calibrated against a certified quartz plate. A scale correction is determined, and applied to

METHOD 2 P.2.

the polarisation reading of the test sample. Refer to the method for "Polarimeter Calibration by Quartz Plate Check". Alternatively the calibration of the polarimeter should be adjusted such that the scale correction is insignificant.

6.0 PROCEDURE

A. JUICE CLARIFICATION

6.1 If necessary, cool the juice to room temperature in a cold water bath as soon as the sample is taken.

6.2 Transfer 150-200 mL to a dry 250 mL boiling flask (3.7).

6.3 Immediately add the minimum quantity of dry lead needed to clarify the juice. For normal juices about 1.0 g/200 mL dry lead is sufficient. For juices that are difficult to clarify up to 2.0 g/200 mL may be added. NOTE: Avoid adding excess dry lead which can precipitate fructose and raise the pol.

6.4 Mix well and allow to stand for 2 minutes.

6.5 Filter the solution through a Whatman 15 cm No. 91 filter paper in a clean, dry stemless funnel into a filter glass (3.6). Place a cover slip over the filter funnel to minimise evaporation. Discard the first 10-15 mL and collect about 100 mL of clear filtrate.

NOTE: Supercel (about 1 teaspoon or approx. 5 g) may be added to aid poorly filtering juices.

B. READING THE POLARIMETER

6.6 Record the scale correction, SC, of the polarimeter *OR* alternatively, with a certified quartz plate adjust the polarimeter reading to give a negligible scale correction (between 0 and +0.05°Z). Refer to the Method No. 1 "Calibration of a Polarimeter by Quartz Plate Check".

NOTE: The scale correction should be checked regularly preferably at the beginning of each shift. A running record of the scale correction should be kept.

6.7 Before reading a batch of samples, and at least once each shift, rinse the continuous pol tube (3.3) several times with water, then fill the tube with water.

6.7.1 **Automatic polarimeters.** Allow the reading of the water-filled tube to stabilise and then reset to zero.

6.7.2 **Visual polarimeters.** Place the water filled tube in the polarimeter trough. Adjust the focus of the field and scale telescopes. Rotate the scale setting knob alternatively left and right until the colour and intensity of both portions of the field are equal. Read the scale and record the zero pol reading, P_o , to the nearest 0.05°Z.

NOTE: As an alternative, adjust the polarimeter to make the zero reading negligible (between 0 and 0.05°Z).

6.8 Pour 20-30 mL of the clear filtrate (6.5) through the continuous pol tube.

NOTE: If the continuous pol tube was previously filled with water, then rinse the tube with 30-50 mL of the clear filtrate.

6.9 Allow the pol reading to stabilise. Read the pol, P, of the clarified juice and record to the nearest 0.05°Z.

C. REFRACTORY JUICE

6.10 Juice from badly deteriorated cane may not give a clear filtrate and in such cases the Cane Tester can approve a modified method of clarification using Herles' reagent (4.2).

6.11 To 50.0 ± 0.5 mL of the refractory juice in a 100 mL flask add 5 ± 0.1 mL of Reagent B followed by 5 ± 0.1 mL of Reagent A (4.2). Add water to the neck of the flask and mix by swirling. Make to the mark and mix well.

6.12 Filter and read the pol of the clear juice as in steps 6.4 to 6.9. Multiply the reading by 2 and calculate % pol in juice as in 7.1-7.2.

7.0 CALCULATIONS

7.1 Calculate the % pol of juice from Table II, using the pol reading, P, and the Brix of the juice, at the temperature of polarisation.

Table II (Schmidt's Table) gives the % of Cane Sugar in Mill Juices corresponding to the pol readings of the undiluted juice and the Brix readings at the temperature of polarisation. Table II derives the normality of the juice sample from the Brix of the juice and hence the % pol of the juice.

7.2 Record the results as % pol in juice to the nearest 0.1°Z.

NOTE: (i) If the scale correction (6.6) is greater than +0.05°Z calculate the proportional scale correction, SC_p , and apply the correction to the pol reading P.

$$SC_p = \frac{P}{Q_p} \times SC$$

where: P = Pol reading of the clarified juice (6.9)

Q_p = Certified value of the quartz plate

SC = Scale correction for the quartz plate reading

(ii) If P_o , the pol reading for the water filled tube is greater than 0.05°Z then apply a correction to the pol reading P.
i.e. corrected pol reading

$$= P - P_o + SC_p$$

where: P_o = pol reading of the water filled tube

SC_p = proportional scale correction

8.0 PRECISION

The expected range of results is 0-20°Z. The precision has not been determined.

9.0 REFERENCES

9.1 Queensland Government; Regulation of Sugar Cane Prices Act, 1962-1972; Regulation 57 (1967).

9.2 Laboratory Manual for Queensland Sugar Mills; Ed. 5; Watson and Ferguson (1970).

METHOD 3

BRIX — DETERMINATION IN JUICE BY HYDROMETER

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian sugar industry. Two procedures are described. The first procedure is for the analysis of 1st expressed juice and complies with the requirements of the "Regulation of Sugar Cane Prices Act, 1962-1981" for the payment for cane delivered to mills. The second procedure describes a method for other mill juices.

2.0 DEFINITIONS

Brix: The brix of a solution is the concentration (in grams solute per 100 g solution) of a solution of pure sucrose in water, having the same density as the solution at the same temperature.

3.0 PRINCIPLE OF METHOD

The sample of juice is cooled if necessary and then allowed to stand so that suspended solids can settle out and air bubbles can escape. A brix spindle (hydrometer) is floated in the juice and the brix read from the spindle scale. The temperature of the juice is read and, for temperatures other than 20°C, a temperature correction is applied.

4.0 APPARATUS

4.1 **Brix spindle** (brix hydrometer), for the standard temperature, 20°C, calibrated to 0.1°b_x. Brix spindles should be constructed to comply with the ICUMSA (1924) specifications.

Brix spindles to be used for 1st expressed juice analysis must be certified by BSES.

4.2 **Thermometer**, 10-35°C graduated to 0.5°C. Thermometers for 1st expressed juice analysis must be certified by BSES.

4.3 **Juice Can**, metal or plastic, about 1.5 L capacity.

4.4 **Brix Cylinder**, metal or plastic.

4.5 **Cold Water Bath**.

5.0 PROCEDURE

A. 1ST EXPRESSED JUICE

5.1 Thoroughly stir the bulk juice sample and fill a juice can, taking care to avoid aeration. The juice must not be stirred after filling the juice can from the main sample container.

5.2 Fill a clean, well drained brix cylinder with juice. Pour the juice through a strainer if there is an appreciable amount of suspended matter present.

5.3 After allowing the juice to stand undisturbed for 20 ± 0.5 minutes, remove any traces of froth.

5.4 Select a brix spindle with the appropriate range for the sample. Hold the spindle in the midstem, between thumb and index finger, and lower it carefully into the juice until the flotation level is reached. Do not wet the stem any further than is necessary to form a natural meniscus.

5.5 Withdraw the spindle and gently refloat it to the same

level as in 5.4. The stem should not be wet more than two graduations above the juice level.

5.6 When the spindle has settled, read the brix of the juice. Take the reading (with the eye level with the level of the juice surface), by estimating the point at which the surface would cut the scale. Record the brix to 0.1°.

5.7 With a 10-35°C thermometer measure the temperature of the juice in the brix cylinder. Record the temperature to 0.5°C.

B. MILL JUICES

(Except 1st expressed juice)

5.8 If necessary, cool the juice sample to room temperature by placing the juice can in a water bath held at room temperature.

5.9 Stir the sample vigorously, but with minimum aeration. Rinse the brix cylinder with a portion of the sample, then pour the juice through a strainer into the brix cylinder, filling it completely.

Allow the juice to stand for 15 minutes, then remove any traces of froth from the top of the liquid.

5.10 Select a brix spindle with the appropriate range for the juice sample.

5.11 Proceed as in steps 5.4 to 5.7 to read the brix and temperature of the juice. Record the brix to 0.1° and the temperature to 0.5°C.

6.0 CALCULATIONS

6.1 For routine 1st expressed juice analysis record the brix of the juice at the temperature of reading to 0.1°b_x.

6.2 When more precise results are required determine the temperature correction for the temperature of reading from Table I.

6.3 **Calculate** the brix reading, converted to 20°C.

$$\text{brix at } 20^{\circ}\text{C} = \text{brix reading} + \text{temperature correction}$$

7.0 REFRACTOMETER BRIX

If, in the opinion of the Cane Tester acting for the Central Sugar Cane Prices Board, the reading of a brix hydrometer immersed in a juice is inflated by the presence of soil suspended in the juice, then the Cane Tester may require that the brix be determined by refractometer. If the brix so determined is lower than that indicated by the brix hydrometer and the difference exceeds one unit of brix, the refractometer brix increased by one unit shall be adopted as the brix of the juice.

8.0 PRECISION

Precision data has not been determined.
 The expected range of results is 12-30°b_x.

9.0 REFERENCES

- 9.1 Queensland Government; Regulation of Sugar Cane Prices Acts, 1962-1981; Regulation 57 (1973).
- 9.2 Total Solids (Brix) — Determination in Sugar Solutions by Refractometer.



METHOD 4

FIBRE — DETERMINATION IN CANE

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian sugar industry. It complies with the requirements of Regulation 62 of the "Regulation of Sugar Cane Prices Acts, 1962-1981" for the payment for cane delivered to mills.

This method is applicable to the determination of fibre in all classes of sugar cane. Analytical results derived from this method are used in the calculation of both brix and pol in cane.

2.0 PRINCIPLE

Fibre in cane is directly determined by breaking open all the fibrous cells containing the juice, washing the juice out and drying and weighing the remaining fibre. A representative sample of the prepared, shredded cane is collected and reduced by coning and quartering. The sub-sample is comminuted ("fibrated") in a cutter-grinder, which is presumed to give almost total cell breakage. The "fibrated" cane is mixed and a sample placed in a cotton cambric bag and weighed. The sample in the bag is washed in cold water for 1 hour, with periodic spin drying and then washed in a boiling water bath for a second hour, also with periodic spin drying. The fibre and bags are dried at 100-105 °C to constant weight and the percent of fibre, by mass, in the cane sample calculated.

3.0 APPARATUS

3.1 Jeffco cutter-grinder

Safety

The Jeffco cutter-grinder must be used with care at all times. Display instructions beside the machine.

Guard the motor.

Ensure the machine has stopped and controls isolated before opening the collection box.

Use the plunger, not hands, to feed cane.

3.2 Mixing trays, metal.

NOTE: The cutter-grinder and mixing trays must be located in an area sheltered from sun, wind, rain and steam. The cutter blades should be sharpened regularly and maintained in good condition.

3.3 Cold Water Bath, replenished continuously with clean cold water. Minimum holding capacity 40 litres. The bath is fitted with a hinged wire mesh screen to keep the bags submerged during washing.

3.4 Hot Water Bath, with a heating element capable of promoting continuous boiling, replenished continuously with hot water. Minimum holding capacity 40 litres. The bath is fitted with a hinged wire mesh screen to keep the bags submerged during washing.

3.5 Spin Drier. The preferred machine is the Lightburn Model 215 which has the following characteristics:

- 1440 rpm
- 250 mm bowl top dia.
- 300 mm bowl bottom dia.

Any other machine with a gravity factor of approximately 325 G's would be acceptable.

3.6 Metal Can with Lid. 1-2 litres capacity

3.7 Stainless Steel Tongs.

3.8 Fibre Bags as specified by the Senior Inspecting Cane Tester from time to time (8.2):

Material. Loomstate Cotton Cambric with the following characteristics:

- 100% cotton (added matter not more than 8.0%), 150 ± 9 g/m²
- Extractable matter (solvent dichloromethane) 2% (max)
- Total starch and filling in cotton fabric 13% (max)
- Thread count per centimetre 47 ± 1 warp, 39 ± 1 weft
- Thread yarn count 40
- Cloth breaking load min 500 N warp, min 430 N weft per 5 cm strip.

Shape and Size. Bag shape is a modified rectangle with square corners at the top and rounded corners at the bottom. Inside dimensions, depth 240 mm, width 200 mm with the bottom corners rounded on 75 mm radius.

Construction. Single walled, hemmed at the top. The bag is sewn and trimmed, turned inside out and resealed clear of the cut edges. (External French Seam 5 mm wide). In the final stitching, a 450 mm length of 20 mm tape is to be attached with its centre folded round one edge of the bag, 25 mm below the top of the bag. Number of stitches to be not less than 3.5 stitches per cm. Bags should be individually and indelibly numbered with digits being not less than 10 mm high. New bags should be boiled for 30 minutes with an adequate amount of laundry detergent, rinsed, dried and stored in ambient, atmospheric conditions.

Bags used for payment purposes must be purchased from the Central Sugar Cane Prices Board which ensures that the bags are of a uniform quality made from the specified fabric (tested by a N.A.T.A. accredited laboratory).

Fibre bags can be used up to a maximum of six times.

3.9 Sample Containers, e.g. plastic buckets with lids.

3.10 Balance to weigh 600 g to 0.1 g.

3.11 Forced ventilation oven, capable of being controlled at 100-105 °C.

4.0 SAMPLING

The determination of fibre is generally based on the class of cane. Classes of cane may be made up of single varieties or groups of varieties of generally similar fibre content.

Each class of cane should be sampled, under supervision of a cane tester, 4 times per shift, and a sub-sample of 2 kg taken and composited over not less than 2 shifts and not more than thirty six hours.

If an "other" class does not reach a sampling rate of four per shift the cane tester can authorize that two separate samples be taken from the same delivery in order to obtain a sufficient composite sample.

METHOD 4 P.2.

- 4.1 Sample approximately 20 kg of prepared cane each hour, or more frequently if required. The sample may be raked from a conveyor taking prepared cane from the shredder to the first mill feed chute or from some other suitable place. It is required that the method of sampling prepared cane at each mill has the written approval of the Senior Inspecting Cane Tester.
- 4.2 Level the sample immediately and transfer about 2 kg (e.g. 3 large random handfuls through the depth of prepared cane) to a plastic bucket with a well fitted lid and labelled according to the fibre class stored therein. The bucket is used to accumulate samples and should be kept in a cool place, but not refrigerated. Where circumstances require it, a small plastic bucket (with lid) or plastic bag may be used to transfer the sample from the sampling point to the sample storage location.
- 4.3 At the end of a 24 hour sampling period, transfer the composite sample to a galvanised mixing tray. Quickly but carefully break up conglomerates of prepared cane to achieve representative mixing. Reduce the sample size to 2-3 kg by coning and quartering.
- 4.4 Close up the clean, dry cutter-grinder and switch it on. "Wet condition" the machine by passing some of the unwanted portion of the sample through it. Switch the machine off and isolate the motor. When the cutter has stopped turning — and not before — open the collection box and remove the fibrated cane. Do not open the machine.
- 4.5 Close the box, restart the motor and pass the mixed sample (4.3) through it. Use a suitable device (i.e. plunger) to push all the cane through the blades. On no account should the hand be pushed past the outer confines of the machine guard. Switch the machine off and isolate the motor. When the cutter has stopped turning, open the machine and collection box and remove the fibrated material by hand, transferring it to a closed plastic bucket, or other suitable container. Any fibrated material adhering to the collection box or discharge chute is included in the sample. Any cane lying above the cutting disc is not to be included in the sample, but must be discarded before the machine is wet conditioned with the next sample.

5.0 PROCEDURE

A. MIXING

- 5.1 Weigh a fibre bag (3.8) to 0.1 g and record the mass (m_1). The bags should have been stored in ambient atmospheric conditions so that their moisture content is at equilibrium with the atmosphere. Bags may be used up to a maximum of six times.
- 5.2 Transfer the freshly-fibrated cane sample to the mixing tray located in a convenient dry place sheltered from sun, wind and rain and away from steam.
- 5.3 Rapidly mix the fibrated cane. Break up large conglomerates of fibre. Mix again and spread the fibrated cane evenly over the bottom of the tray to a depth of about 5 cm.

NOTE: It is important that the sample is thoroughly but quickly mixed, since it is subject to loss of weight by evaporation while it is exposed to the atmosphere. Take no longer than five minutes for this operation.

- 5.4 Transfer a catch weight of 150 to 200 g from the mixed

sample to a pre-weighed fibre bag by "pinching out" sections of the layer at random. It is important that the full depth of the layer is sampled, and a portion of the floor of the mixing tray is left bare at each point of sampling. The neck of the fibre bag is securely closed and fastened using the attached tape.

- 5.5 Weigh fibre bag and sample to 0.1 g and record (m_2).

B. WASHING

- 5.6 Transfer the bags to the cold water bath. Immerse the bags under fresh cold water and gently squeeze them twice to exclude air and then tease them out to promote intimate contact between the water and the cane. Place the wire screen over them to keep them submerged. Ensure that clean cold water is flowing through the bath.
- 5.7 After 15 minutes, remove the bags from the bath and spin dry the diluted juice from them.
- 5.8 Repeat operations 5.6 and 5.7 a further three times.
- 5.9 Transfer the bags to the boiling water bath with continuous replacement of water. Place the wire screen over the bags to keep them submerged during the hot washing process.
- 5.10 After 15 minutes, remove the bags and spin dry the diluted juice from them.
- 5.11 Repeat operations 5.9 and 5.10 a further three times.
- 5.12 Take a sample of the final spin water and measure the pol in a 200 mm tube (after clarifying and filtering). If the pol reading is greater than 0.2%Z the bags should be subjected to steps 5.9 and 5.10 until this requirement is met. The pol check is to be performed on every batch of fibres.

C. DRYING

- 5.13 Place the bags in an oven at 100-105 °C (3.11). It is best to hang the bags from small hooks within the oven — placing bags flat on the oven shelves may lead to local overheating or even scorching. Occasional ruffling of the bags during drying will hasten complete drying.
- 5.14 After 12-18 hours in the oven, remove the bags one by one and weigh them immediately. When transferring the bags to the balance, store them in a covered metal can. Once weighed, return them to the oven.
- 5.15 Repeat operation 5.14 at hourly intervals until the bag weight is constant between consecutive weighings of the same bags. Record the final mass to 0.1 g (m_3).
- 5.16 Empty the bags of all fibre by turning them inside out and brushing and beating. Return the empty bags to the oven for an hour.
- 5.17 Weigh the dried empty bags to 0.1 g and record (m_4). (Transfer empty bags to the balance in a covered metal can).

6.0 CALCULATIONS

- 6.1 Calculate the % fibre in cane (mass/mass)

$$\begin{aligned} \text{Percent fibre in cane} &= \frac{100}{1} \times \frac{\text{mass dry fibre}}{\text{mass fibrated cane}} \\ &= \frac{100}{1} \times \frac{[m_3 - m_4]}{[m_2 - m_1]} \end{aligned}$$

where: m_1 = mass of bag (5.1)

m_2 = mass of unwashed sample plus bag (5.5)

m_3 = mass of washed and dried sample plus bag (5.15)

m_4 = mass of washed, dried and emptied bag (5.17)

Record the result to the nearest 0.05% (m/m).

Round the result to the first decimal. When the second decimal is 5, round the result to the nearest even first decimal.

7.0 PRECISION

Sample	Concentration Range percent (m/m)	95% Repeatability r	95% Reproducibility R
Prepared Cane	8-20	0.25	Not determined

8.0 REFERENCES

- 8.1 Queensland Government; Regulation of Sugar Cane Prices Acts, 1962-1981; Regulation 62.
- 8.2 Central Sugar Cane Prices Board; Specification for Fibre Bags revised April 1985.



METHOD 5

SAMPLING AND ANALYSIS FOR EVALUATING MILLING TRAIN PERFORMANCE

1.0 INTRODUCTION

To evaluate milling train performance, there is a need to summarise the sampling and analysis procedures required. This method gives details of these procedures and discusses the derivation of milling performance parameters from the bagasse and cane data.

No attempt is made to cover specific requirements for evaluating diffuser performance. However, a great deal of information given here is directly applicable. Milling train sample analysis is carried out for two different purposes: factory control and extraction performance analysis. Accurate factory control requires that prepared cane and final bagasse be composited and analysed over the full shift production regardless of whether milling performance was satisfactory. In this way, the estimate of pol lost in bagasse represents average shift operating conditions. The same shift final bagasse composite may not represent actual milling performance under steady conditions.

Milling train performance implies a more detailed examination of the performance of each element in the extraction train. This performance can be assessed over a short time period taken for one single rake of cane or over a longer period such as a shift. In most sugar factories milling train performance is assessed over a shift. This produces an averaged estimate of the extraction station performance and gives a good indication of milling train efficiency over a week. This is a reasonable approach providing all calculations are based on the results of analyses carried out on carefully preserved samples of prepared cane and mill bagasses. These samples must all be taken with an appropriate time interval between milling units so that relativity is maintained with the one particular rake or parcel of shredded cane chosen. Deviation from these procedures reduces the value of the evaluation. For example, if the fibre per cent cane value is taken from the previous shift then this estimate of milling performance may be inaccurate. Full disintegrator analysis provides all relevant analytical results for each sample.

Since preservation of samples may present problems and sampling of prepared cane and bagasses could be mismatched, the short period test may be preferable. With short period tests there are no preservation worries and analyses are related to the same unique parcel of cane. However, in this case, at least two, and preferably more, people are required to handle adequately the matching and collection of samples along the milling train.

2.0 COLLECTION AND PRESERVATION OF SAMPLES

2.1 Introduction

The procedure for collecting and preserving prepared cane or bagasse samples is basically similar for both short and long period tests although, with short period

tests, care is required to ensure that samples are taken from the same parcel of cane as it proceeds through the train. With long period tests, preservation is important. A short period test should be of at least 15 and preferably 30 minutes duration. There should be no mill stops in a short period test.

Aspects of the testing procedure for both short term and shift-based analyses are covered in the following.

2.2 Elapsed Time Between Tip and Mills

For the short period test, it is important to know the time taken for a parcel of cane to travel from the tip to each of the mills to be sampled. A coloured dye (e.g. ICI Edicol Poinceau 4R) can be used as a marker so that the travel time can be measured. Handful-size plastic bags of the dye are easy to throw into the cane. Generally the dye will need 'boosting' with another bag after two mills. The dye is a strong colorant and can be very messy if handled carelessly.

2.3 Prepared Cane Sample

This is easily taken from the prepared cane elevator using the sampling equipment already installed. Care should be taken to ensure that the full depth of bed of prepared cane is sampled over the full width of the sample hatch opening. The sample is then spread evenly over the collection tray and grab samples taken representatively from the tray through the complete thickness and over the full area of the layer to obtain the sample size required.

Depending upon the frequency of sampling, the subsample size is gauged so that a composite sample weight of 15-20 kg has accumulated at the end of the test period.

2.4 Bagasse Sampling

For the short period test, the elapsed times obtained from the dye test are used to determine when the first sample of bagasse should be taken from each mill after the test has started. Up to ten samples should be taken from each point spread evenly over the duration of the test. Samples should be taken from each mill in sequence, as closely as possible from the same parcel of cane as it moves down the train.

In the shift period test, samples should be taken at intervals of no longer than one hour. Generally, no effort is made to ensure that the prepared cane and bagasse samples come from the same parcel of cane although it is preferable that they do.

Every effort should be made to obtain bagasse samples which are representative of the total bagasse production. Thus bagasse should be collected in sequence from the left side, centre and right side of the bagasse blanket and the tongs used for sampling should penetrate the blanket of bagasse to the plate below.

Subsample sizes should be of the order of 1.5 kg for cane graduating down to about 1.0 kg for final bagasse. Subsampling on site may be necessary to achieve these

METHOD 5 P.2.

weights. A total composite should weigh about 10 to 15 kg depending upon its origin.

2.5 *Back Roller Juices*

If brix per cent bagasse is required and a precision refractometer is not available for the analysis of the disintegrated bagasse extracts, an estimate of bagasse extract purity can be obtained from back roller juice. This analysis is carried out by some mills. Although not strictly correct, this purity value can then be taken as an approximate estimate of the residual juice purity of the bagasse in later calculations (Section 3.4).

2.6 *Bagasse and Cane Sample Preservation*

The samples should be stored in a sealed container to prevent loss of moisture. Unless the samples are to be analysed within an hour or so of collection, steps should be taken to preserve the sample to prevent degradation of the pol by microbiological action.

As chloroform is no longer a permissible preservative, two other procedures are recommended. If storage up to a limit of 24 hours is required, toluene can be added at a rate of 1 mL per 1 000 g sample and the sample should be stored in a sealed plastic bag at a maximum temperature of 4 °C. Alternatively, the sample should be quickly frozen below -20 °C within a sealed plastic bag, sized so that the thickness of the bagasse is not greater than about 80 mm. Samples in thick overfilled bags take too long to freeze and defrost and may then suffer localised deterioration. Sample bags should not be placed on top of one another until frozen.

If sample composite bins are left along the milling train under hot moist conditions for over four hours with no preservative added, sample deterioration will occur. Satisfactory analytical results can be obtained only by immediately preserving each subsample.

The composite sample will not be obtained until all the subsamples have been collected over the required period. Special care should be taken to bring the sealed subsamples quickly back to ambient temperature prior to the mixing together to obtain the composite sample, otherwise deterioration and condensation can affect the analysis. Manipulation of the sample within the sealed bag will be necessary at regular intervals during the defrost period to break up lumps and help heat transfer. Great care must be taken with frozen samples to prevent extra moisture condensing on the bagasse and thereby affecting the analysis.

For a shift composite sample, the only method that has been rigorously established as giving an accurate result involves taking every sample back to a refrigerator (if toluene is added to each sample) or to a freezer. Placing each sample in a bin at ambient temperature for the duration of the shift is suspect due to the probability of microbial degradation especially under warm, humid conditions.

3.0 ANALYSIS OF CANE AND BAGASSE

3.1 *Preparation of Sample*

The possibility of obtaining some separation of large or small particles during collection or mixing must be recognised and suitable techniques (Section 3.2) must be employed to avoid any bias.

Shredded cane is often sufficiently well broken and

divided to require little further attention but any long fibrous pieces (or whole stick portions if they should occur) may be cut up with a cane knife. Some cane varieties which are long fibred will not disintegrate satisfactorily if long pieces are presented to the machine. These tend to wrap around the blades or shaft in the first few seconds after start-up. Long thick pieces also cause errors in moisture measurement. Sections of whole stick which have come through the shredder unbroken may be rejected if they comprise not more than five per cent of the total sample weight.

Bagasse samples should not require further preparation before disintegration.

3.2 *Mixing of Sample*

The following method ensures good mixing and keeps evaporation to a minimum. Two people are required to carry out the operation efficiently. It must be carried out quickly on a clean non-absorbing surface in a sheltered, draught-free area.

The sample is heaped in a cone on the mixing surface and then halved by the two people leaving a free space for mixing between the halves. Each person then spreads their sample half in portions evenly over the cone of a new heap being formed between them. This heap is then rotated by approximately 90 degrees and then halved again.

The heaping and heap rotation is repeated followed by a final halving and heaping. This third mixed heap is then transferred 'en masse' into a suitable container and sealed with a lid. It is important to avoid overhandling the material as this may cause unnecessary evaporation or separation of fines. Subsamples for analysis may then be taken at random from the mixed sample with particular care that there is no bias towards selection of fine or coarse particles.

3.3 *Disintegration*

The sample of bagasse (or cane) is introduced into the disintegrator can, followed by a weighed quantity of water. The mixture is then disintegrated for 30 minutes for bagasse and for 40 minutes for prepared cane. Typical quantities are shown in Table A within this method for SRI type disintegrators. Some disintegrators are designed to handle larger volumes (Refer method for Pol in Bagasse where 9 kg water is used with 0.9 kg of bagasse).

In Table A within this method the amount of water used is a constant 6 kg. The amount of cane or bagasse used is intended to supply approximately 300 g of fibre to each disintegrator sample. This results in a thinner slurry for cane (with its higher natural moisture) and a heavier slurry for the final bagasse (lower moisture). The higher fibre/total moisture ratio for final bagasse is desirable so that a reasonable pol reading can be obtained. It is inadvisable to go above this loading because of excessive heating and lack of mobility of the slurry.

Certain models of the Jeffco brand of disintegrator require a lower total mass but the same ratio of sample to water.

On completion of the disintegration, a quantity of the slurry is removed and strained through a fine wire gauze with an opening of approximately 0.5 mm.

Approximately 250 mL of this strained extract is collected in a stoppered flask and cooled quickly to room temperature. Pol and brix are determined on this solution.

The following points should be noted about disintegrator analysis:

- (i) In the absence of a large capacity single pan balance for weighing the water, the use of a specially constructed fixed volume container with an overflow side-arm is acceptable. This is adjusted to deliver the required mass of water and should be check-calibrated at regular intervals.
- (ii) To minimise evaporation losses and possible thermal degradation of the sample, water cooling should be provided for the disintegrator can.
- (iii) Disintegrator blades should be kept sharp, and 'touched up' with a file after every third or fourth disintegration.
- (iv) The original Sugar Research Institute design (Foster, 1954) is used by various mills. Good disintegration is obtained with three 150 mm blades rotating at approximately 5800 rev/min. The blades should be spaced at 12.5 mm intervals from the shaft end. The shaft end should be about 3 mm from the bottom of the can.
- (v) The Jeffco cane disintegrator fitted with 14 litre capacity bowl with internal baffles is also satisfactory. This should have a top spinner blade in addition to the three cutter blades.
- (vi) A check should be made at intervals to ensure that the disintegration time is adequate. A minimum volume of slurry sample should be taken at say 30, 35, 40 and 45 minutes after start of disintegration for analysis of pol and refractometer brix. These results should indicate whether the disintegration is complete in the standard time period and whether the machine is in adequate condition.

3.4 Brix Determination

Approximately 100 mL of the strained disintegrator extract is shaken in a stoppered flask with two heaped teaspoons (six to seven grams) of dry Supercel. Filtration is then made through an 11 cm paper in a 65 mm diameter funnel into a 100 mL Erlenmeyer flask. After running the first 10 mL of filtrate to waste it may be necessary to return cloudy filtrate to the paper until the runnings are clear. When 15-20 mL of the clear filtrate has been collected, the refractometer brix can be determined. Either the Bellingham and Stanley or Zeiss dipping type precision refractometer, each with jacketed prism, is satisfactory for this purpose. Regular checks on the standardisation should be carried out, either with distilled water or the calibration plates provided. A determination of the refractometer brix of the water used in disintegration must also be made and the brix of the disintegrator extract is corrected accordingly:

$$\text{Brix of extract (corrected)} = \text{Measured brix of extract} - \text{brix of water.}$$

If a suitable precision refractometer is not available, then brix can be inferred from pol using the purity of the back roller juice. This assumes that the bagasse purity is approximately equal to the back roller juice purity.

3.5 Pol Determination

Approximately 150 mL of the strained disintegrator extract is clarified with a minimum quantity of dry lead and filtered. The usual precautions of covering the filter funnel and rejecting the first 15-20 mL of filtrate are observed. A polarimeter is then used to obtain the pol reading for the extract. The polarimeter tubes used are normally 200 mm long in automatic polarimeters and 400 mm long in manual polarimeters. This difference must be taken into account in calculations.

3.6 Moisture Determinations

Spencer type drying ovens are used to determine the moisture content, with sample cans initially containing between 100 g (standard Spencer oven) and 1000 g (SRI type oven) of moist bagasse or prepared cane. Reduction of sample size reduces the available accuracy of the determination. Drying is carried out with air at 105 to 115 °C to constant weight. Usually, the initial weight is measured for SRI type ovens after one hour of drying, with check weights carried out at 15 minute intervals thereafter until two successive weighings agree within 1 g. For other oven types, cans are weighed after three hours and then at hourly intervals. Underdrying not only results in incorrect moisture level determination but also increases the calculated fibre level if fibre content is determined by an indirect (i.e. difference) method (see Section 3.7).

3.7 Calculation of Pol, Brix and Fibre Level

Definitions —

- B = Brix % cane (or bagasse)
 P = Pol of cane (or bagasse)
 M = Moisture % cane (or bagasse)
 F = Fibre % cane (or bagasse)
 b = Brix of disintegrator extract (°bx)
 Y = Mass cane (or bagasse) for analysis (g)
 Z = Mass added water (g)
 d = Apparent density of disintegrator extract (g/mL)
 Q = Purity of back roller juice (assumed equal to purity of bagasse extract)
 R = Pol reading of extract in 200 mm tube (°Z)
 If a 400 mm tube is used, $R = \frac{\text{reading}}{2}$

An assumption that fibre contains 25 per cent hygroscopic moisture is made.

Then

$$B = \frac{b(Z - 0.25Y + 0.0125 YM)}{Y(1 - 0.0125b)}$$

$$F = 100 - B - M$$

$$P = \frac{R \times 0.26 (Z + Y - 0.0125 FY)}{d Y}$$

If accurate brixes of extract for final bagasse cannot be measured, an alternative formula can be used which relies on the assumption that the specific gravity of the extract is 1.000 and that the purity of the residual juice in bagasse is equivalent to that of the back roller juice.

$$P = \frac{R \cdot 0.26(Z - 0.25Y + 0.0125 MY)}{Y(1 - (0.16R)/Q)}$$

A relationship for all bagasses, based on a 10:1 ratio of water to bagasse in the disintegration, assuming disintegrator fluid purities of 80 for first bagasse and

METHOD 5 P.4.

75 for all other bagasses and a hygroscopic water of 25% on dry fibre, is given in Table VII. This Table is a convenient aid to the calculation of results for control laboratory purposes.

3.8 Pol in Open Cells

Although the determination of pol in open cells is normally done on prepared cane, it is sometimes carried out on bagasse. The following method is applicable to prepared cane. Table A within this method describes the variations needed for bagasses. A 15 litre bottleneck can, used for the cold leaching step, is shown in Figure 1, together with details of supply and construction. Cane (1 000 g) and water (10 000 g) are placed in the container and, after sealing, the contents are rotated on a jar roller at 70 r/min for 10 minutes \pm 5 seconds. The extract is immediately strained through a fine wire gauze funnel to avoid further extraction. The pol reading, x , is then determined on the extract.

For the wet disintegrator, cane (2 000 g) and water (6 000 g) are weighed out, placed in the disintegrator and the soluble solids extracted for 40 \pm 1 minutes. The pol reading, p , of the extract is then determined. The ratio, r , is given by:

$$r = \frac{\text{pol reading for tumbler}}{\text{pol reading for disintegrator}} = \frac{x}{p}$$

and the value of % pol in open cells, K , is given by the formula:

$$K = \frac{1000r}{3.838 - .838r}$$

As mentioned in Section 3.3, it may be necessary to deviate from the recommended quantities in Table A. In this case, the following general formula can be used for the determination of per cent pol in open cells (K):

$$K = \frac{100rW_T}{C_T(1 - r)(1 - 1.25F/100) + W_D C_T / C_D}$$

where W_T is weight of water added in tumbler (g),
 W_D is weight of water added in disintegrator (g),

C_T is weight of cane or bagasse added in tumbler (g),

C_D is weight of cane or bagasse added in

disintegrator (g), and

F is fibre per cent cane or bagasse.

3.9 Precision of Analysis

Table B within this method gives information on the precision of methods of cane analysis, obtained from statistical analyses of variance of test data gained from duplicate measurements on separate samples of cane. As only a limited number of samples were measured, the precision estimates should only be used as a guide. Sample size may have a marked effect on the precision of an analytical determination. The following test illustrates the need to select the largest size possible for mill samples.

Five separate samples of prepared cane were analysed for moisture content with three different sizes of subsamples taken in replicate.

A Spencer type bagasse oven was used to dry 1 000 g and 500 g subsamples, each in quadruplicate. A like number of 50 g subsamples were dried in sealable cans in a laboratory oven. The results for each sample weight were then treated with a statistical one-way analysis of variance to determine the individual precision of the moisture determination. The estimated precision obtained for the 1 000, 500 and 50 g sample weights were 0.24, 0.40 and 0.46 per cent relative standard deviation respectively.

4.0 SUMMARY

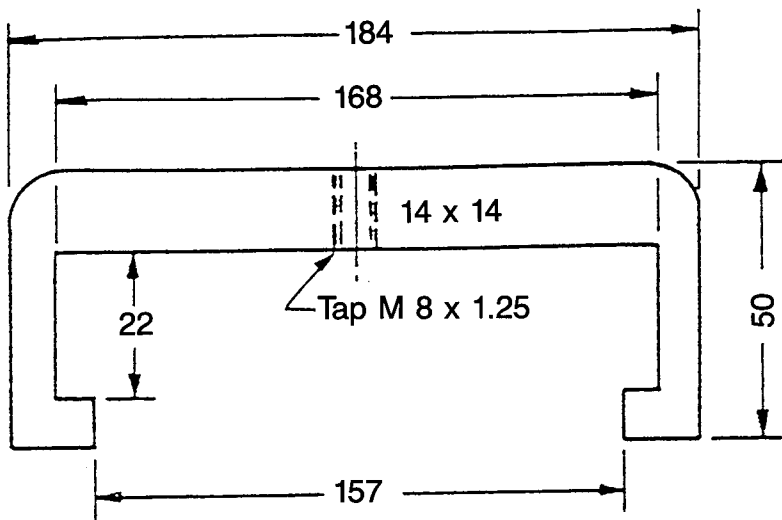
Measurements conducted over a short period of time with samples from the one rake of cane require more intensive analyst involvement but avoid the problems of preservation and ensure that all samples are matched with each other.

The alternative evaluation carried out over the period of a shift may produce a better indication of milling performance but only if the samples are taken when milling conditions are settled.

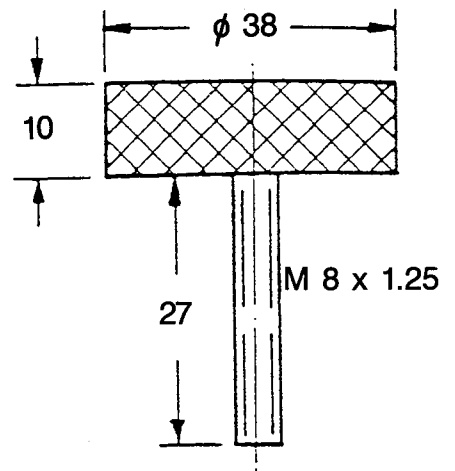
In this case, the bagasse sampling has been selective and the final mill bagasse sample is unlikely to be representative of the bagasse sent to the boiler station and thus may give a result which is misleading for factory control purposes.

5.0 REFERENCES

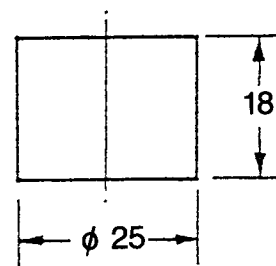
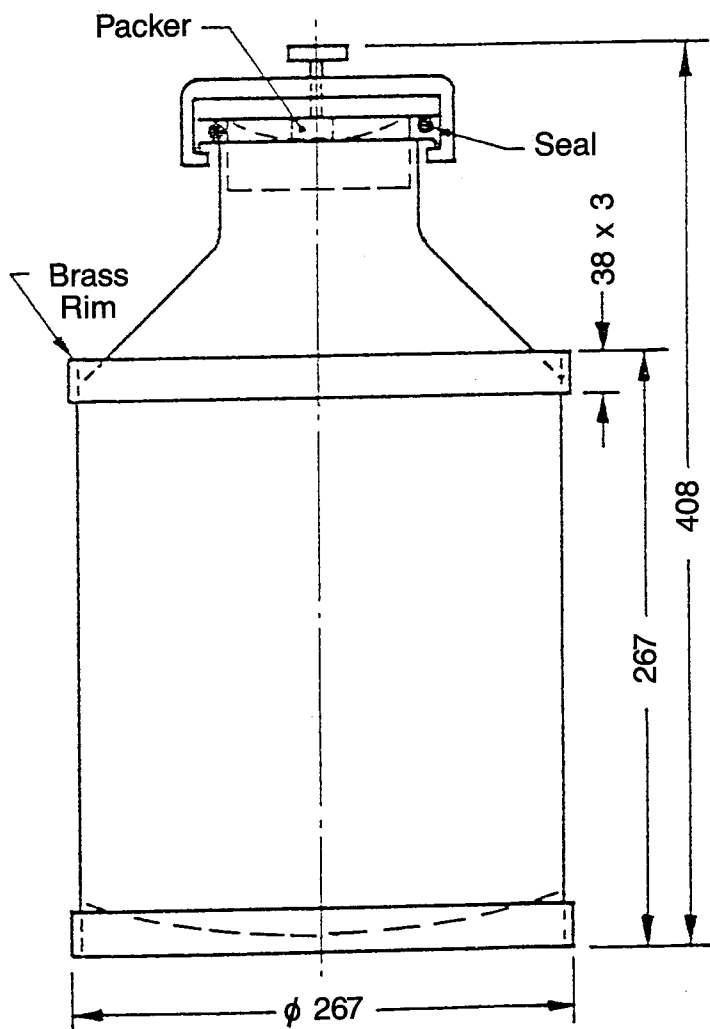
Foster, D.H. (1955). The determination of pol in bagasse. Proc. Qd Soc. Sugar Cane Technol. 279-283.



Holding Down Bracket Material:
14 x 14 Mild Steel



Tightening Screw Material:
Mild Steel



Packer Material: Brass

These measurements may need to be modified to suit the latest design of can.

Materials required:

- 1 only 15 litre Bottleneck Can
(Milking Machine Supplies,
6 Light Street,
Fortitude Valley, Qld.
Phone: (07) 252 2095
- 1 only O-ring, BS436
O.D. 161.9 I.D. 149.2
(Bearing Service Centre)

Figure 1. Modification of 15 litre bottleneck can for pol in open cell determination

METHOD 5 P.6.

TABLE A. ANALYSIS AND CALCULATION OF POL IN OPEN CELLS

Sample	Disintegrator	Tumbler	Formula
Shredded cane	2 000 g + 6 kg water	1 000 g + 10 kg water	$K = \frac{1\ 000\ r}{3.838 - .838r} *$
No. 1 Mill Bagasse	1 000 g + 6 kg water	500 g + 10 kg water	$K = \frac{2\ 000r}{7 - r - \frac{1.25F}{100} (1 - r)}$
Nos. 2 and 3 Mills Bagasse	800 g + 6 kg water	400 g + 10 kg water	$K = \frac{2\ 500\ r}{8.5 - r - \frac{1.25F}{100} (1 - r)}$
Nos. 4, 5 and 6 Mills Bagasse	600 g + 6 kg water	300 g + 10 kg water	$K = \frac{3\ 333r}{11 - r - \frac{1.25F}{100} (1 - r)}$

*Formula for shredded cane generally used in this form. Fibre content of 13.0% is assumed.
 Calculated value with ± 0.5 units over range 11 to 15% fibre.

r = Ratio of pol readings
 K = Per cent pol in open cells
 F = Fibre per cent cane or bagasse

TABLE B. ESTIMATED PRECISION OF METHODS OF CANE ANALYSIS

Analysis	No. of samples	Mean value	Standard deviation	Relative standard deviation (%) ¹	Repeatability ²
Pol % cane	15	18.30	0.062	0.34	0.18
Brix % cane	15	19.85	0.14	0.71	0.40
Moisture % cane	6	71.6	0.27	0.38	0.76
P.O.C. (%)	10	82.1	1.1	1.4	3.2
Fibre % cane, by bag	14	14.1	0.17	1.2	0.49
Fibre % cane, by disintegrator ³	—	—	0.30	—	0.86

¹ The standard deviation (s), expressed as a percentage of the mean. ² Repeatability = $2\sqrt{2}\ s$

³ Inferred from variances of brix % cane and moisture % cane with relationship $F = 100 - B - M$.

METHOD 6

POL — DETERMINATION IN BAGASSE BY WET DISINTEGRATION

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian sugar industry. It applies to bagasse from all mills in a crushing train.

Pol in bagasse is used to determine the extraction of sucrose from cane by successive mills and is an indication of the performance of the crushing station. Adjustments to mill settings are based on pol losses calculated from the results.

2.0 PRINCIPLE OF METHOD

A weighed sample of bagasse is mixed with a measured volume of water and disintegrated in a high speed disintegrator. The fibre cells are broken, releasing the sucrose content. The disintegrated slurry is screened and the liquor obtained cooled and clarified by adding basic lead acetate. The pol of the clarified solution is read in a 200 mm polarimeter tube in a sugar polarimeter. Per cent pol in bagasse is read from a Table.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

3.1 Wet disintegrator.

This is essentially a high speed (5,800 r/min) wet blender. Either a commercially available unit such as the Jeffco wet disintegrator or a specially fabricated unit is suitable.

The disintegrator is fitted with 3 blades, each 150 mm long × 32 mm wide × 1.5 mm thick. The blades are fitted at the end of the shaft with spacing nuts (13 mm) between each blade. Each blade should be at an angle of 60° to the one below it, to balance the shaft and avoid vibrations. The shaft should end about 3 mm above the bottom of the drum. The blades should be sharpened regularly with a file.

NOTE: For more accurate analysis, e.g. investigational work, thinner, 0.8 mm blades should be used to give better extraction.

3.2 Wet disintegrator drum,

20 L capacity, with lid and rubber seal, and fitted with a water-cooled baffled jacket.

Safety

The machine must never be operated without the drum in position. Nuts and blades should be inspected regularly, tightened if necessary, and replaced if worn. When removing or placing the drum into position, make sure that the power isolator switch, as well as the ON/OFF switch for the motor, is turned OFF. No work should be done on the disintegrator unless these switches are OFF and a DO NOT START tag placed on the isolator switch.

A hinged guard to fit around the 20 L drum should be provided in case of blade failure.

3.3 Water dispenser

9 kg ± 50 g capacity. The accuracy should be checked weekly by weighing the water discharged.

3.4 Cooling bath,

with running cold water.

3.5 Sugar Polarimeter.

3.6 Polarimeter tube,

200 mm long for automatic polarimeters

3.7 Balance

to weigh 900 ± 10 g, plus container.

4.0 REAGENTS

4.1 Basic lead acetate powder (dry lead) or basic lead acetate solution (wet lead).

5.0 TREATMENT OF SAMPLE

Intermediate mill bagasse samples are placed in an airtight plastic bag and taken to the laboratory for immediate analysis. Frequency of sampling of intermediate mill bagasse may vary from mill to mill.

Final mill bagasse is sampled hourly. The sample is placed in an airtight plastic bag and taken to the laboratory where 2-3 drops of toluene are added (as a preservative). The bag is sealed and the sample stored in the laboratory freezer until required for analysis. Near the end of each shift the hourly samples are removed from the freezer, completely thawed and thoroughly mixed together to form a composite from which the sample to be analysed is taken.

6.0 PROCEDURE

6.1 Weigh out 900 ± 10 g of bagasse and place into the 20 litre wet disintegrator drum. Place the drum and its contents in position on the wet disintegrator.

NOTE: A larger sample may be needed with some varieties (e.g. varieties with long fibres and those that tend to break into small sections) to ensure a reasonable fibre to water ratio.

6.2 Add 9 kg ± 50 g of water. This water is added volumetrically using the 9 litre water dispenser (3.3).

6.3 Fit the lid on the drum carefully and ensure the rubber seal is correctly positioned and firmly clamped. Turn on the cold water and ensure an adequate water flow. Run wet disintegrator for 30 minutes ± 30 seconds.

6.4 Turn off machine, making sure to turn OFF isolator switch, unclamp and remove lid. Turn off the cooling water and remove hoses from the couplings. Remove drum and pour 500 mL of slurry through a coarse sieve to remove bagasse fibre into a clean, dry jar. Cover with lid and cool to room temperature in a cooling bath.

6.5 Transfer about 150 mL of the cool liquor to a dry 250 mL boiling flask. Immediately add the minimum quantity of dry lead (or wet lead) needed to clarify the juice. Mix well and allow to stand for 2 minutes.

6.6 Filter the solution through a Whatman 15 cm No. 91 filter paper in a covered filter funnel. Discard the first 10-15 mL and collect about 100 mL of clear filtrate.

6.7 Rinse twice then fill the polarimeter tube with water. Read the water reading of the polarimeter and record to 0.01°Z (average of 4 readings).

NOTE: With automatic polarimeters allow the reading of the water filled tube to stabilise and then re-set to zero.

METHOD 6 P.2.

6.8 Rinse twice and fill the polarimeter tube with the clear filtrate. Read the pol in a sugar polarimeter and record the results to 0.01°Z (average of 4 readings).

7.0 CALCULATIONS

7.1 Record the per cent moisture in bagasse obtained by the method, "Moisture — Determination in bagasse and cane by drying".

7.2 Correct the pol reading of the liquor —
Corrected pol reading = pol reading (6.8) — water reading (6.7)

7.3 Per cent pol in bagasse is obtained from Table VII. Table VII gives the pol in bagasse according to the corrected polarimeter reading, (in a 200 mm pol tube) and the per cent water in the bagasse sample.

NOTE: (i) If the pol was read in a 400 mm tube, then divide the corrected pol reading (7.4) by 2,

before obtaining % pol in bagasse from Table VII.

(ii) If the sample to water ratio was not 1 in 10 (see Note 6.1), use the relationship given in Table VII to calculate % pol in bagasse.

7.4 Record the result as % pol in bagasse to 0.01.

8.0 PRECISION

The precision of the method has not been determined. The expected range of results is 1.0% - 15.0% pol in bagasse.

9.0 REFERENCE

Foster, D.H., (1955). The Determination of Pol in Bagasse. Proc. Qd Soc. Sugar Cane Technol., 22nd Conf. 279-283.

METHOD 7

MOISTURE — DETERMINATION IN BAGASSE AND CANE BY DRYING

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry. The moisture content in bagasse is used in calculations of crushing station extraction efficiencies and pol losses. The result is needed also for the determination of pol in bagasse. The method can be used also to directly determine moisture in cane (which has been fibrated as in the method for fibre analyses).

2.0 PRINCIPLE OF METHOD

Per cent moisture in bagasse is the loss of mass after drying a test portion of the bagasse to constant weight in a stream of hot air in a special oven at $110\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$. The determination of water and of pol in bagasse are usually done together on sub-samples of the same sample.

3.0 APPARATUS

- 3.1 **Drying oven**, either a modified Spencer oven or an SRI type oven capable of being controlled at $110\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$
- 3.2 **Drying Cans**, aluminium.
- 3.2.1 For Spencer type ovens, 100 mm dia x 200 mm high, fitted with a 150 micron gauze at the bottom.
- 3.2.2 For SRI type ovens, drying can to hold 500-1000 g bagasse, fitted with 120 micron gauze at top and 36 micron gauze at bottom.
- 3.3 **Stiff Brush**, for cleaning the gauze in the drying can.

4.0 SAMPLE PREPARATION

- 4.1 The bagasse must be sampled through the full depth of the bagasse mat and care must be taken to ensure the sample is representative.
- 4.2 For moisture in cane, a representative sample of prepared cane is collected from the cane elevator and stored in a sealed container. The sample is prepared for analysis by reduction in a cutter grinder or hammer mill as in Method 4, "Fibre Determination in Sugar Cane".
- 4.3 The sample is thoroughly mixed and subsampled. Large samples can be sub-sampled by coning and quartering. Sub-sampling must be done quickly to avoid evaporation losses. A sub-sample of about 3 kg is taken and placed in a sealed plastic bag.
- 4.4 If the sub-sample is to be stored for any time add 2-3 drops of toluene, seal the bag and store in a freezer. The sample should be removed from the freezer, completely thawed and re-mixed before analysis.

5.0 PROCEDURE

- 5.1 Select a clean, dry and empty drying can.
- 5.2 Discard the top layer (3 cm) of the sample and weigh

a sample of bagasse into the can, $100 \pm 0.1\text{ g}$ sample for the Spencer oven, $1,000 \pm 1\text{ g}$ for the SRI oven. Record the mass of can + sample, m_1 .

NOTE: With some Spencer type ovens a larger $400 \pm 0.1\text{ g}$ sample can be used and is preferred.

- 5.3 Place the can plus sample in the drying oven and switch the oven on.
- 5.4 Check that the temperature is controlled at the recommended temperature (3.1), particularly over the last stages of drying.
- 5.5 After three hours drying, switch off the oven, remove the can and sample and weigh while still hot. Record the mass of the can plus dried sample.
- 5.6 Replace the can in the oven and dry for a further 1 hour. Re-weigh the can plus dry sample, while still hot and record the weight.
- 5.7 Repeat step 5.6, until the can plus dried sample reaches constant mass. This is when the difference between two weighings is $<0.1\%$ of the total weight. Record the final mass of the can plus dried sample, m_2 .

Safety

Heat resistant gloves should be worn while handling the hot drying can.

- 5.8 Immediately empty the can, brush out residual dried bagasse from the can and gauze. Weigh the empty can while still hot and record the mass, m_3 .

NOTE: For convenience a standard drying time can be measured for each type of bagasse. With all the cans in the oven filled with one type of bagasse, determine the time taken for the samples to reach constant mass. The standard drying time will be this time plus 1 hour.

6.0 CALCULATIONS

Calculate the per cent (m/m) moisture in the sample

$$\% \text{ moisture} = \frac{[m_1 - m_2]}{[m_1 - m_3]} \times \frac{100}{1}$$

where: m_1 = mass drying can + wet bagasse (5.2)

m_2 = mass drying can
+ dried bagasse (5.7)

m_3 = mass of empty drying can (5.8)

Record the result as % (m/m) moisture in the sample to 0.1%

7.0 PRECISION

The expected range of results 35-65% (m/m) moisture in bagasse.

The precision of the method has not been determined.



METHOD 8

TOTAL AND SOLUBLE PHOSPHATE — DETERMINATION IN CANE JUICE

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian sugar industry. The method applies to the determination of total, that is, acid-soluble inorganic phosphate and soluble, that is, water soluble inorganic phosphate.

The concentrations of phosphate in the juice, before and after clarification, give an indication of clarifier performance.

2.0 PRINCIPLE OF METHOD

If total phosphate is being determined, the pH of the juice is adjusted to 4.0 by adding sulphuric acid. Soluble phosphate is determined at the natural pH of the juice.

An aliquot of the juice is filtered under vacuum with acid-washed Supercel. Acid-molybdate, amidol and sodium metabisulphite solutions are added to an aliquot of the filtrate and phosphate is measured as the absorbance, at a wavelength of 660 nm, of the blue colouration which develops. Interference from low concentration of organic phosphates and from anions and cations found in sugar is not significant.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

- 3.1 **Spectrophotometer** suitable for the measurement of absorbance in the region of 660 nm with matched 2 cm cells.
- 3.2 **Bulb pipettes** — Class A (AS 2166) — 2 mL, 3 mL, 5 mL, 8 mL, 10 mL
- 3.3 **Graduated straight pipette** Class A (AS 2167) — 10 mL
- 3.4 **Automatic dispenser**, 10 mL

4.0 REAGENTS

During the analysis, unless otherwise stated, use only analytical grade reagents and only distilled water.

- 4.1 **Acid-molybdate reagent.** Dissolve 16.6 ± 0.1 g ammonium molybdate tetrahydrate in about 600 mL of distilled water in a 1 litre beaker. Add 96 ± 1 mL of concentrated sulphuric acid. Add the acid slowly with stirring to avoid the generation of excessive heat. Cool, transfer to a 1 litre volumetric flask, make to the mark, and mix well.

Safety

Sulphuric acid is very corrosive. Safety glasses and gloves should be used when handling the acid. It must not be pipetted by mouth.

If in contact with skin, remove all contaminated clothing and wash affected area with copious quantities of water. If ingested administer large quantities of aluminium hydroxide or milk of magnesia (magnesium hydroxide). Do not induce vomiting.

- 4.2 **Acid reagent.** Add 96 ± 1 mL of concentrated sulphuric acid ($\rho_{20} = 1.84$ g/mL) to about 600 mL of distilled water in a 1 litre beaker, slowly as in 4.1. Cool, transfer to a 1 litre volumetric flask, make to the mark, and mix well.

- 4.3 **Amidol reagent.** Dissolve 1.00 ± 0.01 g of amidol (2, 4-diaminophenol hydrochloride) and 20 ± 1 g of sodium metabisulphite in distilled water in a 100 mL volumetric flask, make to the mark and mix well. Filter under vacuum through a Whatman No. 3 filter paper with a level teaspoon of acid-washed Supercel (4.4). Store in a dark bottle in a refrigerator, and discard after one week.

Safety

Amidol/sodium metabisulphite reagent and its intermediate oxidation products are toxic and have a pungent odour. They may produce bronchial asthma and hence should not be inhaled. Contact with the skin may give rise to an irritation leading to dermatitis; the reagent should be washed off the skin immediately. Use gloves when handling. Do not pipette by mouth.

- 4.4 **Acid-washed Supercel.** Add 50 ± 5 g of Supercel to 1 litre of distilled water. Add 50 ± 5 mL of concentrated hydrochloric acid and stir for 5 minutes. Filter and wash Supercel cake free of acid with distilled water. Test washings with litmus paper. Dry Supercel for 6 hours at 90-100°C and store in a closed container.

Safety

Concentrated hydrochloric acid is very corrosive and gives off pungent, toxic fumes. It must be handled using safety glasses and gloves in a well ventilated atmosphere. It should not be pipetted by mouth.

First aid is the same as for sulphuric acid (4.1).

- 4.5 **Standard phosphate stock solution**, 1.0 mg P/mL. Dry about 1.5 g of potassium dihydrogen phosphate for 1 hour at 110°C. Weigh out 1.0984 ± 0.0001 g of the dried salt, dissolve in freshly distilled water and make up to 250 mL in a volumetric flask. Do not keep for longer than one month.
- 4.6 **Working standard phosphate solution**, 0.01 mg P/mL. Dilute 10.00 ± 0.05 mL of the standard phosphate stock solution to 1 litre in a volumetric flask, and mix thoroughly.

5.0 STANDARDISATION OF METHOD

- 5.1 Using Class A pipettes, pipette aliquots of 0, 2.00, 4.00, 6.00, 8.00, 10.00, 12.00 mL of working solution (4.6) into separate 50 mL volumetric flasks. These solutions are equivalent to 0, 0.02, 0.04, 0.06, 0.08, 0.10, 0.12 mg phosphate.
- 5.2 To each flask add 2 drops of acid reagent (4.2). Adjust the volume to about 30 mL with distilled water. Add 10.0 ± 0.2 mL of acid-molybdate reagent (4.1) and 4.0 ± 0.2 mL of amidol reagent (4.3). Make each flask to 50 mL with distilled water, stopper and mix well.
- 5.3 Allow the solutions to stand for at least 10 minutes but not more than 30 minutes.

NOTE: If the temperature of the solutions is above 25°C, do not allow to stand for more than 15 minutes.

- 5.4 Read the absorbance of each standard solution in a spectrophotometer at 660 nm, using a 2 cm cell with

METHOD 8 P.2.

water as the reference solution. Record the absorbance to 0.001 units.

As a guide to performance of the method, the 0.06 mg P standard measured at 660 nm in a 2 cm cell should read 0.280 absorbance. The reading for the zero P standard in a 2 cm cell against distilled water should not exceed 0.001 absorbance.

- 5.5 Plot the absorbance of each standard solution against the concentration of phosphate.

If two or more points lie off the line of best fit by more than 5% of the line's absorbance value at that particular phosphate concentration, the standardisation must be repeated.

The calibration graph should be constructed using the mean of independent standardisations by at least two analysts. Each set of results and the mean must comply with the limits above. In addition the slopes of the individual lines must not differ by more than 2%.

Each time a new batch of reagents is prepared, check the calibration at the zero point (reagent blank) and at one other point by repeating steps 5.2 to 5.4. If the results do not lie within the limits set out above repeat the calibration.

6.0 PROCEDURE

A. PREPARATION OF TEST SOLUTION

- 6.1 If the final result is to be expressed on solids (mg/kg) then determine the brix of the sample at 20°C and record to 0.1°brix.

- 6.2 For total phosphate only, transfer about 100 mL of the juice to a 150 mL beaker. While stirring on a magnetic stirrer, add sufficient acid reagent (4.2) to adjust the pH to 4.0 ± 0.5.

NOTE: The analysis must be completed within 1 hour of adding acid.

- 6.3 For both total and soluble phosphate, filter under vacuum through a Whatman No. 3 filter paper. Precoat the filter with a slurry consisting of a level teaspoon of acid washed Supercel (4.4) with about 10 mL of the solution. Use the filtrate from this precoat to rinse the filter flask.

- 6.4 Pipette an aliquot (V) of the sample into each of two 50 mL volumetric flasks.

For mixed juice, take 2.00 ± 0.02 mL.

For clarified juice, take 20.00 ± 0.06 mL

NOTE: The aliquot (V) of the test solution should be selected to give a reading of between 0.10 and 0.40 absorbance.

- 6.5 To one flask add 10.0 ± 0.2 mL of acid-molybdate reagent (4.1) and 4.0 ± 0.2 mL of amidol reagent (4.3). Make up to 50 mL with distilled water, stopper and mix well. Stand for at least 10 minutes, but not more than 30 minutes. This is the test solution.

NOTE: If the temperature of the solution is above 25°C, read between 10 and 15 minutes.

B. PREPARATION OF BLANK SOLUTION

- 6.6 To the second 50 mL flask (6.5) add 10.0 ± 0.2 mL of acid reagent (4.2). Make up to 50 mL with distilled water, stopper and shake. Stand for at least 10 minutes, but not more than 30 minutes.

C. MEASUREMENT OF ABSORBANCE

- 6.7 Read the absorbance of the test solution (6.5) in a spectrophotometer at 660 nm, in a 2 cm cell using the corresponding test blank solution (6.6) as the reference solution. Record the absorbance to 0.001 units.

7.0 CALCULATIONS

- 7.1 Read from the standard graph "mg phosphate" in the test solution.

- 7.2 Calculate the phosphate (mg/L) on juice —

$$P \text{ (mg/L)} = \frac{\text{mg P from graph}}{1} \times \frac{1000}{V}$$

where V is the sample aliquot (6.4)
or calculate P (mg/kg) on solids —

$$P \text{ (mg/kg)} = \frac{\text{mg P from graph}}{1} \times \frac{10^5}{V} \times \frac{1}{\text{density} \times \text{brix}}$$

The density at 20°C corresponding to the measured brix of the juice (6.1) is found from Table XII or Table XV.

Table XII gives "Degrees Brix, Density and Concentration of Sucrose Solutions at 20°C corresponding to Refractive Index Reading".

Table XV gives "Densities of Solutions of Cane Sugar at 20°C in g/mL".

8.0 PRECISION

Sample	Concentration Range	95%	95%
		Repeatability r	Reproducibility R
Mixed Juice or Clarified Juice	5-150 (mg/L) on juice 25-850 (mg/kg) on solids	3 (mg/L)	6 (mg/L)

9.0 REFERENCE

Allen, R. J. L., Biochem, J.; Vol. 34 (1940).

METHOD 9

STARCH — DETERMINATION IN CANE JUICES

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian sugar industry. The method measures a substance (or group of substances) which is basically starch or derived from starch. The method gives a relative measure of the starch content in cane juices. Cane starch can be occluded in the raw sugar crystal and affects the filtration rate of liquor from the refinery carbonatation process.

2.0 PRINCIPLE OF METHOD

The juice is sampled and a preservative added immediately to inhibit naturally occurring starch-degrading enzymes. The brix is measured and the pH adjusted to 6.0. An aliquot of the juice is added to a boiling solution of uranyl acetate and calcium chloride to precipitate protein and other organic impurities. The solution is boiled and then digested at 95-100°C to solubilise starch, and then filtered under vacuum with Celite filter aid.

The filtrate is diluted and potassium iodide/iodate solution is added to an aliquot to form the blue starch iodide complex. The absorbance of this complex is read in a spectrophotometer at 700 nm. At this wavelength the effect on absorbance by the impurities in the cane juice is minimal.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

- 3.1 **Spectrophotometer** suitable for the measurement of absorbance at 700 nm with a set of matched 2 cm cells.
- 3.2 **Water bath** capable of being controlled at 95-100°C.
- 3.3 **Graduated straight pipette** with a wide delivery tip opening (3 mm internal dia).
- 3.4 **Volumetric flask** — Class A (AS 2164), 100 mL
- 3.5 **Bulb pipettes** — Class A (AS 2166), 2 mL, 5 mL, 10 mL, 15 mL, 20 mL
- 3.6 **Automatic pipette**, 5 mL and 25 mL capacity.

4.0 REAGENTS

During the analysis, unless otherwise stated, use only reagents of analytical grade and only distilled water.

- 4.1 **Calcium Chloride Solution 40% (m/m)**. Dissolve 53.0 ± 0.1 g of calcium chloride dihydrate in distilled water and dilute to 100.0 ± 0.1 g with distilled water. As calcium chloride is highly deliquescent, stocks should not be exposed to air. The strength of the calcium chloride solution must be checked and, if necessary, adjusted to $40.0 \pm 0.3\%$ (m/m) with distilled water or with calcium chloride dihydrate (approximately 75.5% CaCl_2).

The solution strength can be checked by measuring its specific gravity. The specific gravity of 40.0% m/m calcium chloride solution is 1.3942 at 24°C. Within the temperature range 20-30°C it decreases on an average by 0.0007 per degree above 24°C and increases by the same amount per degree below 24°C. In the region of 40% (m/m), a change of 0.1% in calcium chloride con-

centration produces a change of 0.001 in specific gravity.

Specific gravity may be measured by pycnometer, hydrometer or other instrument capable of the accuracy required. A convenient method is as follows:

- (i) Allow stoppered containers of calcium chloride solution and distilled water to stand for a number of hours (e.g. overnight) so that both reagents come to ambient temperature.
- (ii) Weigh a clean, dry 100 mL volumetric flask to 0.02 g. Fill to the mark with calcium chloride solution and weigh again to 0.02 g. Note the temperature of the solution to 0.1°C.
- (iii) Rinse out the flask and fill to the mark with distilled water at the same temperature as the calcium chloride solution, and weigh to 0.02 g. From the weights of the solution and water, and the temperature, calculate the specific gravity to 0.001.

- 4.2 **Acetic Acid (1 mol/L approx.)**. Dilute 57 ± 1 mL of glacial acetic ($\rho_{20} = 1.049$ g/mL) acid to one litre with distilled water.
- 4.3 **Acetic Acid (0.1 mol/L approx.)**. Dilute 10.0 ± 0.2 mL 1 mol/L acetic acid (4.2) to 100 mL with distilled water.
- 4.4 **Calcium Chloride/Acetic Acid Reagent**. Using a pH meter and stirrer, adjust the pH of a 100 mL aliquot of calcium chloride solution (4.1) to $\text{pH } 3.0 \pm 0.1$ with 0.1 mol/L acetic acid (4.3). Because of the large salt concentration, allow sufficient time for the pH meter to equilibrate. Adjust the pH of the bulk of the calcium chloride solution in the same proportion. Although the pH of a calcium chloride/acetic acid solution tends to alter on standing, do not readjust to pH 3.0 before using.
- 4.5 **Uranyl Acetate Solution 2.5 g/100 mL**. Dissolve 2.50 ± 0.01 g of uranyl acetate in distilled water and make to 100 mL. If necessary add not more than ten drops of glacial acetic acid per 100 mL of solution to aid solution. This reagent should be discarded after 3 months.

Safety

Uranyl acetate is highly toxic. If ingested, induce vomiting with 10% aqueous magnesium sulphate, followed by milk or egg white in cold water.

- 4.6 **Potassium Iodate Solution, 0.0017 mol/L**. Dry about 0.5 g potassium iodate at 105-110°C for 1 hour. Dissolve 0.3566 ± 0.0002 g of the dried reagent in distilled water and make to 1 litre. Store in the dark in a brown, glass-stoppered bottle.
- 4.7 **Potassium Iodide, 10 g/100 mL**. Dissolve 10.0 ± 0.1 g of potassium iodide in distilled water and dilute to 100 mL. Store in the dark in a brown, glass-stoppered bottle. Discard the solution if it becomes yellow.
- 4.8 **Potassium Iodide/Potassium Iodate Reagent**. This reagent must be prepared on the day it is to be used. Mix 10.0 ± 0.5 mL of potassium iodide solution with 90.0 ± 0.5 mL of distilled water. To this solution add

METHOD 9 P.2.

100.0 ± 0.5 mL of potassium iodate reagent. Mix the reagents and keep in a brown, glass-stoppered bottle. This reagent must be discarded after 1 day.

- 4.9 **Standard Starch Solution, 1000 mg/L.** Use an approved batch of B.D.H. Laboratory Reagent Potato Starch, supplied by CSR Central Laboratory. Determine its moisture content, correct to 3 decimal places, by drying about 2 g (weighed to 0.0001 g) at 105-110 °C for 2 hours.

4.9.1 Weigh into a 25 mL beaker $\frac{(1.0000 \times 100)}{(100 - \%H_2O)}$ ± 0.0002 g of fresh starch, i.e. equivalent to 1.0000 ± 0.0002 g of anhydrous starch.

4.9.2 To the weighed quantity of starch, add 5 mL of cold distilled water and mix with a glass rod. Before the starch settles, transfer the mixture quantitatively to 500 mL of boiling water in a 1 litre conical flask so that no slurry touches the wall of the flask.

4.9.3 With at least three additional 5 mL portions of distilled water, transfer all the starch to the flask. This operation should be completed within 1 minute.

4.9.4 Boil the starch for 3 minutes ± 10 seconds, timed from the moment the first 5 mL of starch slurry enters the boiling water.

4.9.5 Rinse a 1 litre volumetric flask with hot distilled water. Quantitatively transfer the hot solution through a glass funnel to the 1 litre volumetric flask. Wash the conical flask at least twice with hot distilled water, by adding the water to the 25 mL beaker (4.9.1.) and then transferring it to the conical flask. Continue washing the conical flask with hot distilled water and transferring to the volumetric flask until the latter is filled to approximately 900 mL.

4.9.6 Swirl to mix the flask contents and cool under running water to room temperature. Make the solution to 1 litre, stopper and mix well. Store in a refrigerator.

NOTE: The solution will keep for one week.

- 4.10 **Working Standard Starch Solution, 200 mg/L.** Using a Class A bulb pipette, pipette 50.00 mL of the 1000 mg/L standard starch solution (4.9) into a 250 mL volumetric flask. Dilute to 250 mL with distilled water and mix well.

This reagent will not keep and must be prepared on the day the standardisation is carried out.

- 4.11 **Starch Free Sucrose.** Use only sugar, tested to be free of starch. Suitable starch free sugar is available from CSR Central Laboratory.

- 4.12 **Mercury (II) Chloride Solution, 0.5 g/100 mL.**

Safety

Mercury (II) chloride is highly toxic. Gloves should be worn when handling the reagent. It should not be pipetted by mouth.

If in contact with the skin, remove all contaminated clothing, etc. and wash affected area with copious quantities of water.

If ingested, quickly administer a large quantity of egg white in water or medical charcoal in suspension and induce vomiting with 10% magnesium sulphate. Follow with milk or egg white in water. Obtain medical

attention immediately. Speed is essential since this poison is fast acting and extremely dangerous.

- 4.13 **Celite 560 filter aid.**
4.14 **Hydrochloric Acid** — 1 mol/L approximately.
4.15 **Sodium Hydroxide** — 1 mol/L approximately.
4.16 **Blank Sugar Reagent.** Mix 10.0 ± 0.1 mL of an 8 g/50 mL solution of sucrose (4.11) in water with 20.0 ± 0.2 mL of calcium chloride-acetic acid (4.4) and 2.0 ± 0.2 mL uranyl acetate reagent (4.5) in a 50 mL volumetric flask. Heat in a water bath at 95-100 °C for 15 ± 1 minutes, cool, make to the mark, stopper and mix well.

- 4.17 **Sucrose Solution, 30 g/100 mL.**

5.0 STANDARDISATION OF METHOD

A. PREPARATION OF STANDARDS

- 5.1 Pipette, using class A bulb pipettes 0, 2.00, 5.00, 10.00, 15.00 and 20.00 mL aliquots of the 200 mg/L working standard starch solution (4.10) respectively into six 100 mL volumetric flasks.

The final concentration in the flasks will be 0, 4, 10, 20, 30 and 40 mg/L of starch.

- 5.2 To each flask add 10 ± 0.1 mL of 30% sucrose solution (4.17), 40.0 ± 0.2 mL of calcium chloride/acetic acid reagent (4.4) and 4.0 ± 0.2 mL of uranyl acetate solution (4.5). Dilute each flask to about 80 mL with water.

- 5.3 Mix by swirling and place the flasks in a boiling water bath at 95-100 °C for 15 ± 1 minutes. Swirl each flask at 5 and 10 minutes after placing in the bath.

- 5.4 After 15 minutes remove the flasks and cool in a water bath to room temperature. Dilute each flask to the mark with distilled water, stopper and mix well.

B. MEASUREMENT OF ABSORBANCE

- 5.5 Pipette 15.0 ± 0.02 mL of each of the standard solutions into a 25 mL volumetric flask.

- 5.6 Add to each flask 2.50 ± 0.02 mL of 1 mol/L acetic acid (4.2), and 5.00 ± 0.02 mL of the potassium iodide/iodate reagent (4.8). Mix by swirling and make to the mark with water. Mix well.

- 5.7 Immediately read the absorbance of the each standard solution in a 2 cm cell, in a spectrophotometer at 700 nm against distilled water as the reference. Record the absorbance to 0.001.

NOTE: (i) The absorbance of the test solution must be read between two and five minutes after adding the iodide-iodate reagent to the test solution.

- (ii) The reading of the 0 mg/kg starch standard should not exceed 0.010 absorbance for a 2 cm cell. As a guide the reading for the 20 mg/L starch standard should be about 0.250 absorbance in a 2 cm cell at 700 nm.

C. PREPARATION OF GRAPH

- 5.8 Plot mg/L starch (5.1) against absorbance (5.7). A straight line must be obtained for the graph. If three or more points lie off the line of best fit by more than 5% of the line's absorbance value at that particular starch concentration, the standardisation must be repeated.

- 5.9 The graph used by any laboratory for routine analysis should be constructed using the mean of independent standardisations by at least two analysts. Each set of

results and the mean must comply with the 5% limits above (5.8). In addition, the slopes of the individual lines must not differ by more than 5%.

Each time a new batch of reagents is prepared, check the calibration at the zero point and at one other point by repeating steps 5.1 to 5.7.

6.0 TREATMENT OF SAMPLE

A. PREPARATION OF TEST SOLUTION

When the juice is sampled, immediately add two drops (0.1 mL) of the 0.5% mercury (II) chloride solution (4.12) per 100 mL of juice. Cool the juice as quickly as possible to 1-6 °C, in a refrigerator.

NOTE: Mercury (II) chloride is added to the juice to inhibit natural enzymes and to minimise loss of starch. With this treatment and storage at 1-6 °C, starch levels should remain constant for at least 24 hours. On no account should juice be frozen for storage.

7.0 PROCEDURE

- 7.1 Warm the sample to room temperature by placing the container in a water bath or under running tap water. Measure and record the brix to 0.1°bx.
- 7.2 Adjust the pH to 6.0 against suitable pH indicator paper, with 1 mol/L sodium hydroxide or 1 mol/L hydrochloric acid. (First expressed juice generally takes 2-6 drops 1 mol/L sodium hydroxide).
- 7.3 Add to a 100 mL conical flask, 20.0 ± 0.2 mL of calcium chloride/acetic acid reagent (4.4) and 2.0 ± 0.2 mL of uranyl acetate solution (4.5). Mix well.
- 7.4 Bring the solution to boiling and using a 10 mL calibrated pipette with a wide delivery tip (3.3), pipette 10 mL of the juice sample into the boiling solution. Add the first 1-2 mL of juice dropwise to minimise frothing, then quickly add the remainder of the solution.
- 7.5 Continue boiling the solution for 1 minute \pm 10 seconds after adding the juice sample.
- 7.6 Swirl to mix the solution and transfer the flask to a water bath at 95-100 °C for 15 ± 1 minutes. Mix by swirling after 5 and 10 minutes.
- 7.7 After 15 minutes, remove the flask from the bath and immediately add about 0.5 g Celite 560 (4.13). Mix well.
- 7.8 Without delay, filter under vacuum through a 5.5 cm Whatman No. 541 filter paper into a 250 mL Buchner flask. Filter to dryness.
- 7.9 Wash down the sides of the conical flask with 10 mL of hot water and again filter to dryness through the No. 541 paper.
- 7.10 Transfer the filtrate into a 50 mL volumetric flask. Rinse the Buchner flask with 1×5 mL of hot water and add the washings to the 50 mL volumetric flask.
- 7.11 Cool the flask to room temperature and dilute to volume with water. Mix well.

B. MEASUREMENT OF ABSORBANCE

- 7.12 Pipette 15.00 ± 0.02 mL of the filtrate into each of two 25 mL volumetric flasks.
 - (a) sample blank solution
 - (b) sample test solution

- 7.13 Add to each flask, 2.50 ± 0.02 mL of 1 mol/L acetic acid (4.2). Mix by swirling.
- 7.14 Make the sample blank solution (7.12a) to the mark with water. Stopper and mix well.
- 7.15 To the sample test solution (7.12b) add 5.00 ± 0.02 mL of potassium iodide/iodate reagent (4.8). Mix by swirling and make to the mark with water. Stopper and mix well.

- 7.16 Read the absorbance of the sample test solution (7.15) in 2 cm cells in a spectrophotometer at 700 nm, against the sample blank (7.14) as the reference solution. Record the absorbance to 0.001 units.

NOTE: (i) The readings must be made between two and five minutes after the addition of the potassium iodide-iodate reagent to the sample test solution.

- (ii) If the absorbance reading of the test solution exceeds 0.700 repeat step 7.12, taking a 5 mL aliquot of the filtrate. Add 10.0 ± 0.2 mL of the blank sugar reagent (4.16) and proceed as in steps 7.13-7.16.

8.0 CALCULATIONS

- 8.1 Read the concentration in g/mL of the sample solution at 20 °C using Table XII or XV. Table XII gives the Brix, density, and concentration in g/mL of sucrose solutions corresponding to refractive index at 20 °C. Table XV gives the density in g/mL of sucrose solutions corresponding to Brix at 20 °C.
- 8.2 Read the mg/L starch in the test solution from the calibration graph.
- 8.3 Calculate mg/kg of starch (on solids).

$$\text{mg/kg starch} = \frac{\text{mg/L from graph}}{1000} \times \frac{50}{10} \times \frac{1000}{\text{conc. (g/mL at 20°C)}} \times \frac{15}{V}$$

where V is the aliquot taken (7.12), normally 15.0 mL

$$\text{For a 15 mL aliquot, mg/kg starch} = \frac{\text{mg/L from graph}}{\text{conc. (g/mL)}} \times \frac{5}{1}$$

9.0 PRECISION

Sample	Concentration Range mg/kg Starch	95% Repeatability r	95% Reproducibility R
Cane Juice	100-1500	10 mg/kg	25 mg/kg

10.0 REFERENCES

- 10.1 Whistler, R. L. (ed); Methods in Carbohydrate Chemistry; Vol. 4 (Starch); Academic Press (1964).
- 10.2 Balch, R. T.; The Sugar Journal; Vol. 15, No. 8 (1953), p. 11.
- 10.3 Alexander, J. B.; South African Sugar Journal; Sept. 1954, p. 617.
- 10.4 Wei Chen & Mei-Wei Chen; Taiwan Sugar Journal; Jan-Mar 1965, p. 27.



METHOD 10

TURBIDITY — DETERMINATION IN CLARIFIED JUICE

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry. The method is applicable to all clarified cane sugar juices and is indicative of the efficiency of the clarification operation.

2.0 DEFINITION

Turbidity index, s, is a measure of "turbidity" or degree of light scattering —

$$s = \frac{A}{b}$$

where: A is the absorbance measured at a wavelength (900 nm) where the effect of light absorption can be assumed to be zero.

b is the cell length

Because the Turbidity Index is generally a small number, turbidity (S) is expressed as $S = 100s$. Therefore turbidity, $S = 100A$ where A is measured at 900 nm in a 1 cm cell.

3.0 PRINCIPLE OF METHOD

The test sample is cooled to room temperature and the absorbance in a 1 cm cell read in a spectrophotometer at 900 nm against water as the reference.

4.0 APPARATUS

4.1 **Spectrophotometer** suitable for the measurement of absorbance at 900 nm with matched 1 cm cells. The spectrophotometer should comply with the following specification:

- spectral band pass 10 nm or less
- wavelength reproducibility ± 0.5 nm
- absorbance reproducibility ± 0.003 at 1.0 abs

5.0 TREATMENT OF SAMPLE

Flush the sample pipe and rinse the sample container 2-3 times with the hot juice immediately before taking a sample. The sample will be hot, and should be cooled under running cold water to room temperature, as soon as possible. The sample should be analysed as soon as possible but, if necessary, may be stored in a sealed container in a refrigerator for up to 12 hours.

6.0 PROCEDURE

- 6.1 Select a pair of matched 1 cm cells. Rinse twice and fill one cell with the cooled test sample. Fill the other cell with distilled water.
- 6.2 Read the absorbance of the test solution in the spectrophotometer at 900 nm, against the waterfilled cell as the reference. Record the reading to the nearest 0.001 absorbance.

7.0 CALCULATION

Calculate clarified juice Turbidity

$$\text{Turbidity, } S = 100 \times \text{Absorbance}$$

8.0 PRECISION

The expected range of results is 0-100
The 95% repeatability, r is 0.3 units



METHOD 11

POL — DETERMINATION IN FILTER CAKE

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry. The method gives an estimate of pol (% cane sugar) in the wet filter cake from rotary vacuum filters. The result is used to assess filter performance and the weight of pol lost is used in compiling the mill chemical balance.

2.0 PRINCIPLE OF METHOD

The method has three steps:

- Dilution of a 50 g test portion to 200 mL with water.
- Clarification with basic lead acetate followed by filtration.
- Reading the pol of the clarified solution in a polarimeter.

An arbitrary adjustment is made to the weight of the test portion to correct for the presence of insoluble solids and therefore only 50 g of sample per 200 mL is used instead of 52 g (or 1 normal weight).

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

- 3.1 **Polarimeter tube**, 200 mm long for automatic polarimeters,
400 mm long for visual polarimeters.
- 3.2 **Volumetric flask**, Kohlrausch, 200 mL.
- 3.3 **Sugar Polarimeter** (26 g normal weight).

4.0 REAGENTS

Unless otherwise stated use only analytical grade reagents and only distilled water.

- 4.1 **Basic lead acetate** (dry lead), or basic lead acetate solution (wet lead).

5.0 TREATMENT OF SAMPLE

Vacuum filter cake is best sampled from the mud conveyor just prior to the mud hopper. Samples should be taken hourly and placed in a suitable sealed container. Samples from separate filters may be obtained using a metal tray to either sample the entire length or only a section of the filter. These samples should be taken hourly and placed in a sealed container. Each

shift, the composite samples are taken to the laboratory, mixed thoroughly and analysed.

6.0 PROCEDURE

- 6.1 Weigh 50 ± 0.1 g of sample into a basin. Make a slurry with water and wash quantitatively into a 200 mL Kohlrausch flask (3.2) with water. Dilute to about 150 mL with water and mix well.
- 6.2 Add sufficient wet lead (or dry lead) to the flask to clarify the solution. Mix well.
- 6.3 Make exactly to the mark with water using a fine dropper. Stopper the flask, mix well and allow to stand for 5 minutes.
- 6.4 Filter through a Whatman 15 cm No. 91 filter paper in a covered filter funnel. Discard the first 10-15 mL and collect about 100 mL of clear filtrate.
- 6.5 Rinse twice and fill a 200 mm pol tube with water. Read the water tube in the polarimeter. Take 4 readings and record the mean water reading to 0.01°Z.
- NOTE: With automatic polarimeters allow the reading of the water-filled tube to stabilise and then re-set to zero.
- 6.6 Rinse the 200 mm pol tube with the filtrate (6.4) three times then fill. Read the pol of the sample in a polarimeter. Take 4 readings and record the mean reading to 0.01°Z.

7.0 CALCULATIONS

- 7.1 **Correct** the pol reading of the sample (6.6) for the water reading (6.5).

$$\text{Corrected Pol reading} = \text{pol reading (6.6)} - \text{water reading (6.5)}$$

$$\% \text{ Pol in filter cake} = \text{corrected pol reading (for a 200 mm pol tube)}$$

$$\% \text{ Pol in filter cake} = \frac{\text{corrected pol reading}}{2}$$

(for a 400 mm pol tube)

- 7.2 Record results as % pol in wet filter cake to 0.01.

8.0 PRECISION

The expected range of results is 1.0 - 5.0% pol on wet filter cake. The precision of the method has not been determined.



METHOD 12

MOISTURE — DETERMINATION IN FILTER CAKE

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry. The determination of the % moisture in filter cake, in conjunction with a % pol and % fibre in filter cake, is used to determine filter efficiency.

2.0 PRINCIPLE OF METHOD

The per cent water in filter cake is defined as the percentage loss in mass of a test portion dried in an oven overnight at $100\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

3.0 APPARATUS

3.1 **Evaporating basin**, 150 mm dia.

3.2 **Drying oven**, forced draft capable of being controlled at $100\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

4.0 PROCEDURE

4.1 Collect the sample of filter cake and place in a suitable air-tight container. Mix the sample well and take a sub-sample of about 25 g for the analysis.

4.2 Weigh a clean, dry evaporating basin (3.1) and record the mass of the basin (m_1), to 0.1 g.

4.3 Weigh $10.0\text{ g} \pm 0.5\text{ g}$ of sample into the evaporating basin. Record the mass of the basin plus wet sample (m_2), to 0.1 g.

4.4 Spread the sample in the basin to expose the maximum surface area of sample. Do not compress the sample.

4.5 Dry the sample plus basin in the air oven overnight at $100\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ until constant weight is achieved.

4.6 After drying, remove the sample from the oven and place in a desiccator with fresh silica gel, to cool for 45-60 minutes.

4.7 Weigh the basin plus dried sample. Record the mass of the basin plus dried sample (m_3), to 0.1 g.

5.0 CALCULATION

Calculate moisture % (m/m) in mill mud.

$$\% \text{ moisture} = \frac{[m_2 - m_3]}{[m_2 - m_1]} \times \frac{100}{1}$$

where: m_1 = mass dry basin (4.2)

m_2 = mass basin + wet filter cake (4.3)

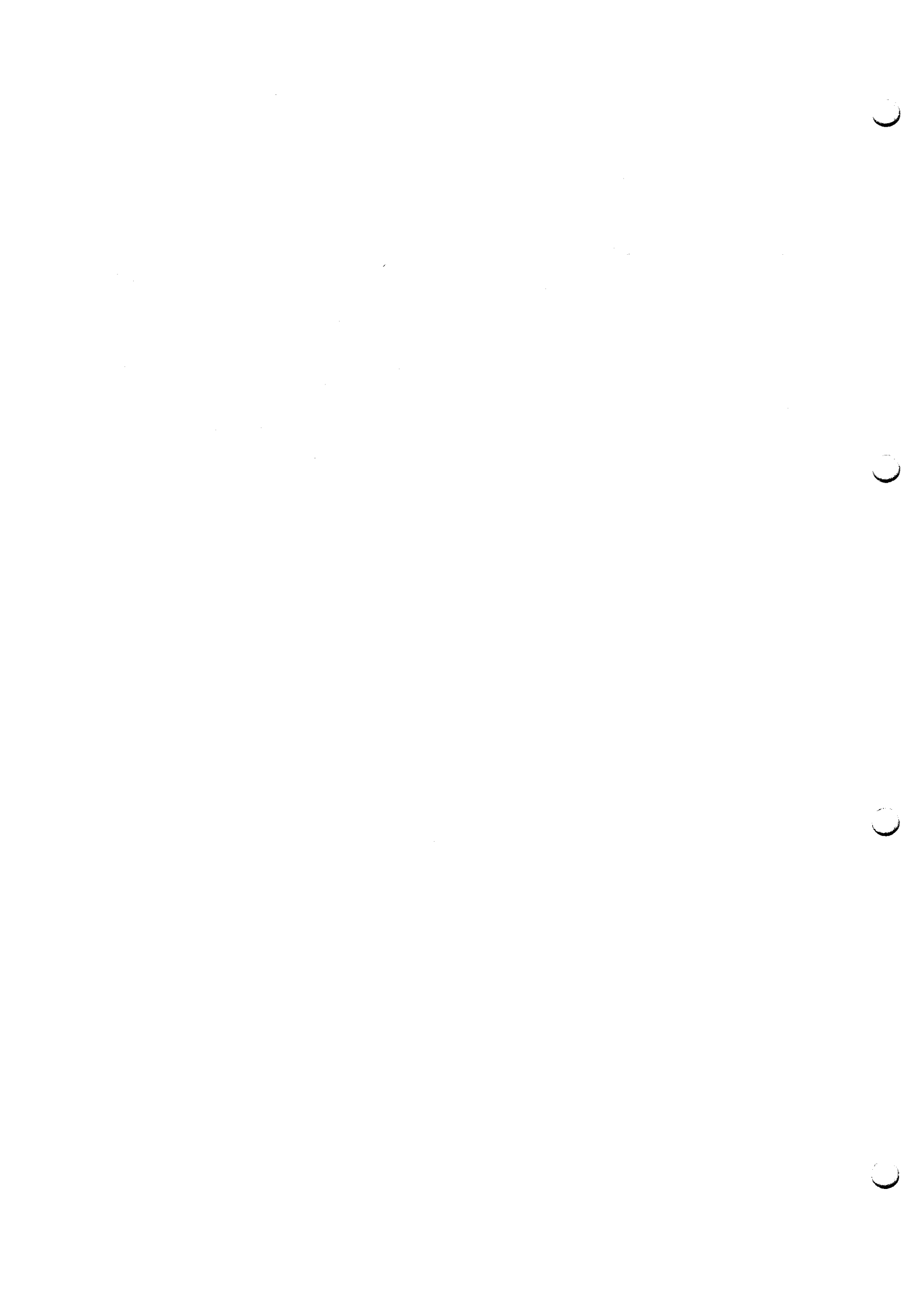
m_3 = mass basin + dry filter cake (4.7)

Record the result as moisture % (m/m) on filter cake to 0.1%.

6.0 PRECISION

The expected range of results is 70-80% moisture on filter cake.

The precision of the method has not been determined.



METHOD 13

FIBRE — DETERMINATION IN JUICE AND MUD STREAMS

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry.

The results from these analyses are used to assess juice screen, clarifier and filter station performance.

2.0 PRINCIPLE OF METHOD

Fibre is determined after filtering the sample through a 75 micron screen and washing with water to remove mud solids and soluble solids. A decantation step removes any sand present. The filtered sample is dried.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

3.1 **Fibre can**, either rolled galvanised iron or stainless steel, approximately 100 mm diameter and 150 mm deep with 75 micron stainless steel screen in the bottom.

3.2 **High pressure fish-tail water jet**. This can be achieved by clamping the end of a rubber hose attached to the tap.

3.3 **Drying oven**, forced draft, capable of being controlled at $100\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

4.0 PROCEDURE

4.1 Weigh out the appropriate quantity of sample as shown in the following Table. Record the mass of sample (m_1) to 0.1 g.

Product	Sample quantity	Sample beaker
Mixed juice	2000 g	4000 mL
Primary mud	200 g	500 mL
Filter feed	200 g	500 mL
Filter cake	50 g	500 mL

4.2 Transfer portion of the sample to the can and wash the mud solids and soluble solids through the 75 micron gauze with a fish-tail jet of high pressure water. Hold the jet fairly close to the bottom of the can to achieve the maximum washing effect and also to avoid loss of fibre by splashing. If the water level builds up

in the can after initial washing, gently swirl the can to assist liquid drainage.

4.3 Repeat the above steps until the sample beaker is empty. Rinse the beaker with water to wash all remaining sample into the can. Continue washing until the clarity of the filtrate is comparable with that of the water source.

4.4 A decantation step is used to remove any sand from the fibre mat. Upend the can and wash all the contents into a 1000 mL beaker, making the final volume to about 500 mL. Swirl the contents of the beaker. The sand settles quickly to the bottom. Return the supernatant containing suspended fibre to the can. Rinse the beaker with water and return any remaining fibre to the can. Allow the can to drain.

4.5 Dry the fibre in the can in an air oven overnight at $100\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ until constant weight is achieved.

4.6 While hot, weigh the sample and can. Record the mass of the can and dried fibre (m_2) to 0.1 g.

4.7 Empty the fibre out of the can, using a brush to remove all traces of fibre. Weigh the empty can while hot, and record the mass (m_3) to 0.1 g.

5.0 CALCULATION

% Fibre in Mixed Juice, Filter Cake, Primary Mud and Filter Feed.

$$5.1 \text{ Fibre \% (m/m)} = \frac{(m_2 - m_3)}{m_1} \times \frac{100}{1}$$

where: m_1 = mass of sample (4.1)

m_2 = mass of can + dried fibre (4.6)

m_3 = mass of fibre can (4.7)

6.0 PRECISION

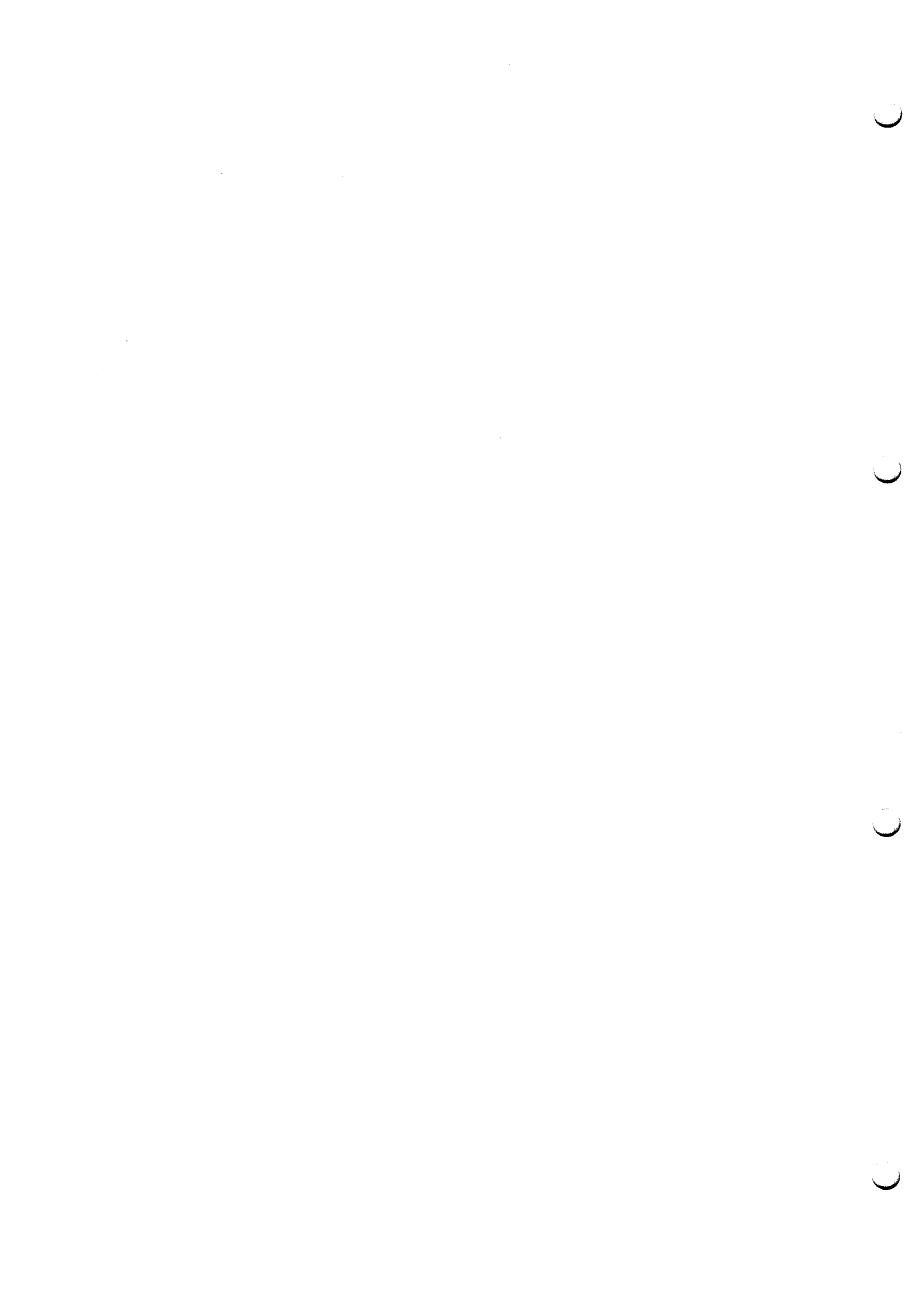
The precision of these methods has not been determined. The expected range of results is:

Mixed Juice: Unscreened 0-2%
Screened 0-0.5%

Primary Mud: 1-4%

Filter Feed: 1-6%

Filter Cake: 4-8%



METHOD 14

TOTAL INSOLUBLE SOLIDS AND MUD SOLIDS DETERMINATION IN JUICE AND MUD STREAMS

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry.

The results from these analyses are used to assess clarifier and rotary vacuum filter performance.

2.0 PRINCIPLE OF METHOD

The analyses consist of physical separations. For all streams except filter cake, total insoluble solids are determined by vacuum filtration of a flocculated test portion of the sample through a Whatman No. 54 filter paper. The soluble solids are removed completely by two series of dilution, settling and decantation steps, prior to vacuum filtration and washing of the formed cake. The filtered sample is dried.

For filter cake, soluble solids are calculated from per cent pol and an assumed juice purity in the cake. Insoluble solids are the difference between total solids (100 - moisture) and soluble solids.

For all streams, mud solids is the difference between per cent total insoluble solids and per cent fibre. The fibre level in filtrate is negligible and only insoluble solids are reported.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

3.1 Drying oven, forced draft, capable of being controlled at 100 °C ± 2 °C.

4.0 PROCEDURE

4.1 Dry a 27 cm Whatman No. 54 filter paper in the oven at 100 °C ± 2 °C for 3 hours. Weigh the dried filter paper and record the mass (m_1) to 0.1 g.

4.2 Weigh out the appropriate quantity of sample as shown in the following Table. Record the mass of sample (m_2) to 0.1 g.

Product	Sample quantity	Sample beaker
Mixed juice	500 g	1000 mL
Primary mud	200 g	500 mL
Filter feed	200 g	500 mL
Filtrate	500 g	1000 mL

4.3 Transfer the sample to a 5 litre beaker, using hot water to wash any residual sample into this beaker. Make the final volume up to about 4.5 litres. Avoid aerating the insoluble solids by pouring the water slowly down the wall of the beaker.

4.4 For mixed juice and filtrate, heat the suspension until just boiling, with stirring to prevent bumping. For mixed juice, add sufficient factory saccharate with stirring to raise the pH of the liquid to within the range 7.5-8.0.

4.5 Add about 5 mL of 0.1% factory flocculant solution to the beaker and stir in gently until flocculation occurs. This allows the flocculated material to settle.

Any floating particles will sink if gently disturbed with a spatula. The solids should compact to about 30% of the original volume. Carefully decant off as much clear liquid as possible without losing solids.

4.6 Repeat steps 4.3-4.5

4.7 Place the pre-dried filter paper from step 4.1 in a 15 cm Buchner funnel so as to form a cup. Connect to the flask and apply vacuum. Rinse the edges of the filter paper using a wash bottle and pour the settled solids into the funnel. Wash all remaining solids into the funnel.

4.8 When the surplus liquid has filtered, flood the cake with hot water to a depth of about 1 cm and vacuum filter until the surplus liquid is removed. Dry the filter cake as much as possible by suction.

4.9 Dry the filter paper plus contents in the air oven overnight at 100 °C ± 2 °C until constant weight is achieved. Weigh the dried cake and paper and record the mass (m_3) to 0.1 g.

5.0 CALCULATION

% Total Insoluble Solids in Mixed Juice, Primary Mud, Filter Feed and Filtrate.

$$\text{Total insoluble solids \% (m/m)} = \frac{(m_3 - m_1)}{m_2} \times \frac{100}{1}$$

$$m_1 = \text{Mass of dried filter paper} \quad (4.1)$$

$$m_2 = \text{Mass of sample} \quad (4.2)$$

$$m_3 = \text{Mass of dried cake + filter paper} \quad (4.9)$$

Report the result as total insoluble solids % (m/m) to 0.1.

6.0 CALCULATION

% Total Insolubles in Filter Cake

6.1 Determine % pol in filter cake (see Method 11).

6.2 Determine % moisture in filter cake (see Method 12).

6.3 Calculate total insoluble solids % (m/m) in filter cake. % total insoluble solids = % total solids — % soluble solids.

$$\text{where: \% total solids} = 100 - \% \text{ moisture}$$

$$\% \text{ soluble solids} = \% \text{ pol} / 0.8$$

where: purity of the juice in the mud is assumed to be 80%. Report the result as total insoluble solids % (m/m) to 0.1.

7.0 CALCULATION

% Mud Solids

7.1 Determine % fibre (see Method 13).

$$\text{Mud solids \% (m/m)} = \% \text{ total insoluble solids} - \% \text{ fibre.}$$

Report the result as mud solids % (m/m) to 0.1

METHOD 14 P.2.

8.0 PRECISION

The precision of these methods has not been determined.

The expected range of results is:

Mixed Juice:	Total insoluble solids	0-2%
	Mud solids	0-2%
Primary Mud:	Total insoluble solids	4-12%
	Mud solids	3-12%
Filter Feed:	Total insoluble solids	4-12%
	Mud solids	3-12%
Filtrate:	Total insoluble solids	0-3%
Filter Cake:	Total insoluble solids	12-30%
	Mud solids	8-20%

METHOD 15

POL — DETERMINATION IN PAN PRODUCTS

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry.

The method is used mainly for factory control purposes. However it should be noted that pol, as a measure of sucrose, becomes less accurate as the purity of the product decreases.

2.0 PRINCIPLE OF METHOD

A test portion of the product is weighed, diluted with water and clarified by adding basic lead acetate powder, followed by filtration. The pol of the clarified solution is read in a sugar polarimeter.

Low purity products need relatively large quantities of basic lead acetate for clarification. This can cause overleading and inflate the pol reading. Acetic acid is therefore added to release fructose from any lead fructose compounds formed.

If a product cannot be clarified by basic lead acetate powder, a solution of lead nitrate/sodium hydroxide (Herles' Reagent) can be used.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

- 3.1 Sugar polarimeter (26 g normal weight).
- 3.2 Polarimeter tube, 200 mm.
- 3.3 Volumetric flasks, Class A (AS 2164) 100 mL and 250 mL.
- 3.4 Pipette, Class A (AS 2166) 50 mL.
- 3.5 Volumetric flask, 50-55 mL.

4.0 REAGENTS

- 4.1 Basic lead acetate powder (dry lead).
- 4.2 Acetic acid, $\rho_{20} = 1.050$ g/mL) diluted 2 + 8 with water.

5.0 PROCEDURE

- 5.1 For syrups, weigh 26.0 ± 0.1 g into a 100 mL volumetric flask. Dissolve in about 50 mL water, then dilute to volume. Proceed as in steps 5.6 - 5.8.
- 5.2 For other products, weigh 50 ± 1 g of product into a tared 250 mL beaker. Record the mass of the samples to 0.1 g. Add approx. 30 mL of hot (60°C) water and dissolve the sample carefully by stirring.
- 5.3 Add water until the total mass is double the mass of the original sample to the nearest 0.1 g. Mix well by stirring and cool in a water bath to room temperature.
- 5.4 Weigh the required amount of the sample into a 100 mL or 250 mL volumetric flask (as specified in the Table below).

Product	Mass of Sample g	Final Volume mL	Mass of Dry Lead g
Syrup	26 straight syrup	100	0.5
A and B Masecuite	26 of 1:1 dilution	100	2.0
A and B Molasses	26 of 1:1 dilution	100	3.0
Magma	26 of 1:1 dilution	100	2.0
C Masecuite	52 of 1:1 dilution	250	5.0
Final Molasses	52 of 1:1 dilution	250	8.0

5.5 Dilute to volume with water. Defroth if necessary with an alcohol blower before the final adjustment to the mark. Stopper and mix well.

5.6 Transfer to a clean dry 500 mL beaker and add the prescribed amount of dry lead (refer to 5.4). Mix well by stirring.

5.7 Filter through a Whatman 15 cm No. 91 filter paper in a covered stemless filter funnel, into a filter glass. Discard the first 10-15 mL and collect the clear filtrate.

5.8 Rinse twice and fill a 200 mm pol tube with water. Read the water tube in the polarimeter. Take 4 readings and record the mean water reading to 0.01°Z.

NOTE: With automatic polarimeters allow the reading of the water-filled tube to stabilise and then re-set to zero.

5.9 Rinse twice and fill a 200 mm polarimeter tube with the clear filtrate. Determine the pol reading of the filled tube in a sugar polarimeter. Take 4 readings and record the average pol reading to 0.01°Z.

5.10 For C Masecuite and Final Molasses only.

Pipette with a Class A pipette 50.0 mL of filtrate (5.7) into a 50-55 mL volumetric flask. Add 2.00 ± 0.02 mL of diluted acetic acid (4.2). Dilute to 55 mL with water. Fill a 200 mm polarimeter tube and read the pol of the solution as in 5.8 and 5.9. Record the average pol reading to 0.01°Z.

NOTE: If the sample cannot be clarified by dry lead, a solution of lead nitrate/sodium hydroxide can be used (Herles' Reagent). Refer to the Method No. 2 "Pol — Determination in Juice".

6.0 CALCULATIONS

6.1 Syrup — 1 normal weight of straight syrup/100 mL.
pol % sample = pol reading (5.9)

6.2 A & B Masecuite] 1 normal weight of 1:1 dilution
A & B Molasses] of sample/100 mL
Magma

6.3 C Masecuite] 2 normal weights of 1:1 dilu-
Final Molasses] tion of sample/250 mL
pol % sample = pol reading (5.9) \times 2
pol % sample = pol reading (5.10) \times 2.75

Record the result to the nearest 0.1% pol

6.4 Calculation where a more precise result is required:

- 6.4.1 Record — the scale correction SC for the polarimeter. [Refer to Method No. 1 "Calibration of Polarimeter by Quartz Plate Check"]
 - the temperature of making to the mark, t_m (5.5)
 - the temperature of reading the polarimeter, t_r (5.9 or 5.10)

- 6.4.2 Calculate — proportional scale correction, SC_p
 - correction for temperature of making to the mark, C_{tm}
 - correction for temperature of reading, C_{tr}

METHOD 15 P.2.

[Refer to Method No. 30 "Polarisation — Determination in Raw Sugar using Visual or Automatic Polarimeters" for the procedure of calculating these corrections]

6.4.3 Calculate the corrected pol reading

Corrected pol reading —

$$= P - P_o + SC_p + C_{tr} + C_{tm}$$

where:

P = pol reading of the filtrate (5.9 or 5.10)

P_o = pol reading of the water filled tube (5.8)

SC_p = proportional scale correction

C_{tr} = correction for temperature of reading

C_{tm} = correction for temperature of making to the mark

6.4.4 Use the corrected pol reading to calculate pol % sample in 6.1, 6.2 or 6.3.

7.0 PRECISION

The expected range of results is 30-90% pol in sample. The precision of the method has not been determined.

METHOD 16

BRIX (TOTAL SOLIDS) —

DETERMINATION IN MOLASSES AND CANE INVERT BY HYDROMETER

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian Sugar Industry. It is used for process control in the factory and for payment analyses for molasses and cane invert sold overseas.

This method is often used as a measure of total dissolved solids in low grade products but becomes less accurate as the purity decreases from syrup to final molasses.

2.0 PRINCIPLE OF METHOD

The test sample is diluted 1:1 by weight, with water, cooled to room temperature and the brix measured by hydrometer (brix spindle).

The contraction in volume associated with the dilution prevents a true measurement of the brix of the original material.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

- 3.1 **Brix Spindles** (Hydrometers), range 30-40 and 40-50 °bx, at 20°C, calibrated to 0.1 °bx.
- 3.2 **Brix cylinder**, metal or glass, at least 75 mm longer than the brix spindle.
- 3.3 **Cold water bath.**
- 3.4 **Balance** to weigh 1000 ± 0.1 g.

4.0 PROCEDURE

NOTE: Store the sample in a sealed container, below 30°C, to minimise evaporation.

- 4.1 Weigh into a tared 1L conical flask 450 ± 10 g of molasses or cane invert. Record the weight to 0.1 g.
- 4.2 Add to — Molasses: approx. 300 mL hot (60°C) water
Cane Invert: approx 300 mL cold water
Dissolve the sample completely by stirring carefully with a brass rod fitted with a rubber policeman. Rinse the rod with water into the dissolved sample.
- 4.3 Add water until the weight of the solution is double the weight of the original sample, to the nearest 0.1 g. Stopper the flask and mix well.
- 4.4 Stand the diluted solution in a running water bath until the solution cools to room temperature.
- 4.5 Mix the test solution well by swirling then rinse the brix cylinder (3.2) with a portion of the solution. Immediately fill the cylinder completely with the solution.
- 4.6 Allow the solution in the brix cylinder to stand for 5 minutes to allow air bubbles and froth to rise to the

surface, then pour in more solution to displace all the froth.

- 4.7 Stand the solution for a further 15 minutes and then remove any traces of froth.
- 4.8 Select a clean dry brix spindle. Hold the spindle in the midstem, between thumb and index finger and lower it carefully into the solution until the flotation level is reached. Do not wet the stem any further than is necessary to form a natural meniscus.
- 4.9 Withdraw the spindle, and then gently refloat it to the same level as in 4.8. The stem should not be wet more than two graduations above the liquid level.
- 4.10 When the spindle has settled, read the brix of the solution. Take the reading (with the eye level with the juice surface), by estimating the point at which the surface would cut the scale. Record the brix to 0.1°.

NOTE: Insoluble solids from molasses samples may settle out in the brix cylinder. A serious error will result if the bottom of the spindle enters this layer. A glass cylinder can be used to ensure the spindle is floating above the insoluble layer.

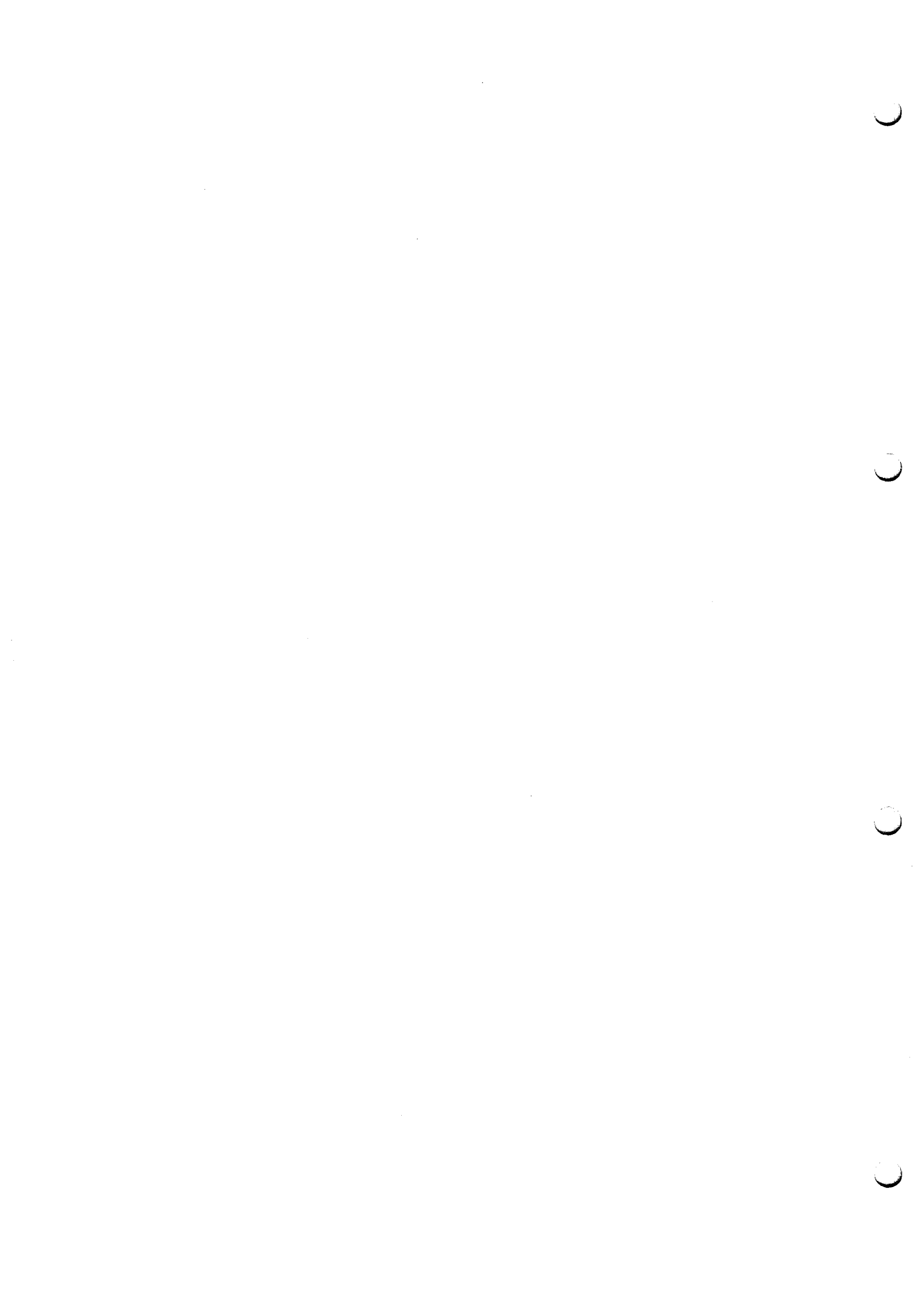
- 4.11 Measure the temperature of the test solution with a 10-35°C thermometer. Record the temperature to 0.1°C.

5.0 CALCULATIONS

- 5.1 **Determine** the temperature correction (to 20°C) from Table I.
Table I gives Temperature Corrections to readings of brix hydrometers (calibrated at 20°C).
- 5.2 **Calculate** the brix reading of the diluted sample, corrected to 20°C:
brix at 20°C = brix reading + temperature correction
- 5.3 **Calculate** the brix of the original sample:
brix of sample = 2 × corrected brix of diluted sample (5.2)
- 5.4 **Report** the result as degrees brix (or % total solids) at 20°C to the nearest 0.1.

6.0 PRECISION

Sample	Concentration Range °bx	95% Repeatability r	95% Reproducibility R
Molasses	65 - 95	0.5	not determined
Cane Invert	80 - 90	0.5	not determined



METHOD 17

BRIX (TOTAL SOLIDS) —

DETERMINATION IN SUGAR SOLUTIONS BY ABBÉ REFRACTOMETER

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry.

The brix, determined by refractometer, gives an approximate measure of the total solids content of an impure sugar solution. It applies to solutions up to a maximum 85% total solids. The approximation becomes increasingly inaccurate as the purity of the solution decreases and care must be taken when interpreting the brix of low purity products, determined by this method.

2.0 PRINCIPLE OF METHOD

The refractive index of sugar solutions is determined using a sugar refractometer. In the Abbé type refractometer the refractive index is measured by observing the critical angle of a thin film of the sample held between two glass prisms. The reading is temperature dependent and is corrected to 20°C by referring to temperature correction tables.

3.0 APPARATUS

3.1 **Abbé type refractometer**, with suitable light source and thermometer.

4.0 STANDARDISATION

4.1 Calibration of Refractometer Scale

The refractometer scale calibration is checked regularly against distilled water, and if necessary adjusted to this standard. The refractive index of distilled water at various temperatures is given in Table X. Abbé type refractometers may also be calibrated at the high end of the scale by means of a glass test piece of known refractive index. The refractive index of the test pieces normally supplied (R.I. = 1.51) represents a brix value in excess of 85°, and test plates are only recommended when accurate measurements are required on high brix materials.

4.2 For the calibration and adjustment of the refractometer refer to maker's instruction manual.

5.0 PROCEDURE

5.1 Connect the prism water jackets to a water supply at ambient temperature and ensure that the water is running steadily. Position the illuminating source and turn it on.

5.2 Clean the prisms with a damp tissue using a dabbing action. Do not rub the prisms, as they scratch very easily. Dry the prisms thoroughly with a dry tissue.

5.3 Place one or two drops of distilled water on the lower prism with a plastic rod and then close the prisms to finger tightness only. The prisms become optically active if strained. Allow sufficient time for the temperature to equilibrate if the sample is not at the same temperature as the refractometer.

5.4 View the field through the telescope and adjust the dispersion compensator at the lower end of the telescope until the light and dark portions of the field are

divided by a sharply focussed horizontal line. Adjust the scale until this line is set accurately at the intersection of the cross hairs of the eyepiece. Take three readings, approaching the cross hairs alternately from above and below. Record the readings to 0.0001 and average, or if the instrument is calibrated with a brix scale, read brix directly to 0.05 units or 0.01 units depending on the instrument scale. Take and record the temperature to 0.1°C.

5.5 Open the prisms and dry by dabbing with a fresh tissue. Place a drop of the sample to be analysed on the lower prism with a plastic rod and close the prisms quickly to finger tightness to avoid evaporation of the sample. Obtain the refractive index and temperature of reading as set out in 5.3 and 5.4.

5.6 Open the prisms and wash away all traces of sugar solution using distilled water from a wash bottle. Clean and dry the prisms by dabbing gently with a fresh tissue. Leave a folded tissue between the prisms when the refractometer is not in use.

6.0 CALCULATION

6.1 **Calculate** the refractometer scale correction.

Scale Correction = R.I. of water (Table X) — observed R.I. of water (5.4).

Record the scale correction to 0.0001.

Table X gives the refractive index of distilled water at various temperatures relative to air.

6.2 **Obtain** temperature correction for sugar solution from Table XI.

Table XI gives temperature corrections to be applied to refractometer readings of sucrose solutions of various concentrations to obtain the Refractive Index at 20°C.

6.3 **Calculate the R.I.** at 20°C of the test sample by subtracting the zero correction and adding or subtracting the temperature correction as required.

R.I. (20°C) = R.I. (t°C) — scale correction ± temperature correction.

Record the refractive index of the test sample to 0.0001.

6.4 **Determine** the brix (or % total solids) of the test sample from Table XII.

Table XII gives brix, true densities and concentration of sucrose solutions at 20°C, corresponding to refractive index reading.

OR if the instrument is calibrated with a brix scale proceed as follows:

6.5 Calculate the scale correction (see Note)

Scale correction = brix of water at temperature of reading corrected for temperature from Table X — Zero

6.6 **Obtain** the temperature correction for the refractometer reading from Table IX.

Table IX gives "Temperature Corrections for the Abbé Refractometer Calibrated at 20°C".

METHOD 17 P.2.

- 6.7 Calculate the brix at 20°C of the test sample by subtracting the scale correction or adding or subtracting the temperature correction as required

Brix at 20°C

= Brix reading (t °C) – scale correction

± temperature correction

- 6.8 Record the result to one fifth of a scale division.

NOTE: Unless a refractometer has suffered physical damage or been tampered with it is rare for the setting to change, and an instrument which is calibrated by BSES once very five years should have negligible scale correction. If the brix scale does not have a calibration for negative values, and extreme accuracy is required, the alternative method (using refractive index) must be employed.

7.0 PRECISION

Sample	Concentration °brix	95%	95%
		Repeatability r	Reproducibility R
Sugar Solutions	0 - 85	0.2°bx	not determined

8.0 REFERENCES

- 8.1 Sugar Analysis — ICUMSA Methods, Ed. F. Schneider; ICUMSA (1979); p.177.
- 8.2 ICUMSA, Proceedings of the Seventeenth Session (1978), Subject 12.
- 8.3 Brown, C.A. and Zerban, F.W., Physical and Chemical Methods of Sugar Analysis, Ed. 3; (1941).

METHOD 18

SUCROSE — DETERMINATION IN MILL PRODUCTS BY DOUBLE POLARISATION

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry. It is based on the Jackson and Gillis Method 2.

The double polarisation gives a more accurate estimate of sucrose than does single (plain) polarisation for products with reducing sugar content above 1%. However it is not an absolute method.

2.0 PRINCIPLE OF METHOD

A solution of the sample is clarified with basic lead acetate, and the pol of the clarified solution determined before and after the sucrose in the solution is inverted by hydrochloric acid, to glucose and fructose. The percentage sucrose in the sample is calculated from the change in polarisation. This procedure eliminates interference from other substances in the sample, that are optically active but are not inverted by hydrochloric acid. In low grade products, oligosaccharides may be hydrolysed to reducing sugars causing significant errors.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

3.1 **Volumetric flasks**, Class A (AS 2164), 100 mL and 250 mL.

3.2 **Bulb pipette**, Class A (AS 2166), 5 mL.

3.3 **Polarimeter tube**, 200 mm, centre filling (glass or glass lined).

3.4 **Cooling bath**.

3.5 **Hot water bath**, capable of being controlled at 68.0 ± 0.5 °C.

3.6 **Sugar polarimeter** (26 g normal weight).

4.0 REAGENTS

Unless otherwise specified, use only analytical grade reagents and only distilled water.

4.1 **Basic lead acetate powder** (dry lead).

4.2 **Hydrochloric Acid** (6.34 mol/L), $\rho_{20} = 1.103$ g/mL. Dilute 630 \pm 5 mL of hydrochloric acid ($\rho_{20} = 1.17$ g/mL), to 1L with water and adjust to exactly 6.34 mol/L, after titrating 5 mL with 1 mol/L NaOH using methyl orange indicator.

4.3 **Ammonia solution**. Dilute 300 mL of NH_4OH , ($\rho_{20} = 0.90$ g/mL), to 1L with water.

4.4 **Ammonium chloride** (NH_4Cl) 226 g/L solution.

4.5 **Supercel**.

4.6 **Norit decolourising carbon**, (SX11).

4.7 **Whatman No. 91** filter paper, 15.0 cm. dia.

5.0 PROCEDURE

A. CLARIFICATION

- 5.1 Weigh the product to 0.01 g into a tared basin.
Juices: 65.00 g (one normal weight/250 mL)
Mill Products: 32.50 g (half normal weight/250 mL)
- 5.2 Transfer the sample to a 250 mL Class A volumetric flask with hot water. Dissolve the sample completely by swirling and cool quickly to room temperature in

a cold water bath. Fill the flask to within a few mm of the mark, defroth with an alcohol blower and adjust exactly to the mark. Stopper and mix well.

- 5.3 Add approximately 8-12 g of dry lead for molasses and less for products of higher purity. Stopper the flask, mix well and allow to stand for 5-10 minutes.

For molasses it is more convenient to place the dry lead in a 400 mL beaker and, using a rubber tipped glass rod, make a thick, smooth slurry with a few mL of the solution. Then, add the remainder of the solution, with continuous stirring and allow to stand for 5-10 minutes. Cover the beaker to minimise evaporation.

- 5.4 If after standing, the clarified solution appears too dark to read in a polarimeter after a 50-50 dilution, add more lead acetate. However the total amount of dry lead must be kept to a minimum.

- 5.5 Filter the solution through a 15 cm Whatman 91 filter paper in a stemless funnel, into a filter glass. Place a cover slip over the filter funnel to minimise evaporation. Discard the first 10-15 mL then collect a total of 120 mL of the clear filtrate.

NOTE: To speed filtration, filter the solution through two filter funnels and combine the filtrate.

B. PREPARATION OF PLAIN AND INVERT SOLUTIONS

- 5.6 Determine the volume, to 0.1 mL, of NH_4OH solution (4.3) required to neutralise 10.0 ± 0.1 mL of 6.34 mol/L HCl (4.2) by titration, using methyl orange as indicator.

- 5.7 **Plain Solution**. Pipette 50.00 ± 0.05 mL of filtrate (5.5) into a 100 mL Class A flask and add 15.0 ± 0.1 mL NH_4Cl solution (4.4). Mix the contents by swirling and make to the mark with water. Filter using Supercel if necessary, through a Whatman 91 filter paper in a covered filter funnel. Discard the first 10 mL filtrate. If the filtrate is too dark to read on the polarimeter, discard and repeat the analysis using additional dry lead for clarification.

- 5.8 **Invert Solution**.

5.8.1 Pipette 50.00 ± 0.05 mL of filtrate (5.5) into a 100 mL Class A flask and run in 10.0 ± 0.1 mL 6.34 mol/L HCl from a burette.

NOTE: If a heavy precipitate of lead chloride forms, the solution has been over-leaded. For the analysis of other samples of this product add a smaller amount of dry lead.

5.8.2 Add 20 ± 1 mL of water from a measuring cylinder and mix. Place the flask in a water bath at 68.0 ± 0.5 °C for 10 min. \pm 10 sec. agitating the flask for the first 3 minutes.

5.8.3 Cool the solution to room temperature in a cold water bath and add the determined volume of NH_4OH solution (5.6) slowly, and with constant swirling of flask. Again cool to room

METHOD 18 P.2.

temperature in a water bath. Make to mark with water and mix well.

- 5.8.4 If the solution appears too dark, add a small quantity (enough to cover a 5 cent coin) of Norit (4.6) and mix. Allow to stand for 5 minutes.
- 5.8.5 If a precipitate forms and/or if Norit has been added, filter using Supercel if necessary, through a Whatman 91 filter paper in a covered filter funnel. Discard the first 10 mL of filtrate.

C. POLARISATION

- 5.9 Record the scale correction, SC, of the polarimeter. Refer to Method No. 1 "Calibration of Polarimeter by Quartz Plate Check".
- 5.10 Rinse twice and fill a clean 200 mm pol tube (3.3) with the plain solution (5.7). Check to ensure that there are no bubbles.
- 5.11 Determine the pol reading of the filled tube in a sugar polarimeter. Take 4 readings. Average all readings and record the mean *plain solution polarisation*, p , to 0.01 °Z.
- 5.12 Record the *temperature of reading of the plain solution*, t_p , in the polarimeter tube to 0.1 °C.
- 5.13 Empty and drain the tube. Rinse twice and fill the same pol tube with invert solution (5.8.4).
- 5.14 Determine the pol reading of the invert solution following the same procedures in 5.11. Place the polarimeter tube in the polarimeter in exactly the same manner and position as in 5.11. Average all the readings and record the *mean invert solution polarisation*, i , to 0.01 °Z.
- NOTE: The readings of the invert solution will be on the negative scale of the polarimeter.
- 5.15 Record the *temperature of reading of the invert solution*, t_i in the polarimeter tube to 0.1 °C.
- NOTE: If the same polarisation tube is used for both solutions the "zero" correction cancels out and need not be determined.

6.0 CALCULATIONS

- 6.1 **Subtract** the *invert solution polarisation*, i , (5.14), from the *plain solution polarisation*, p , (5.11) i.e. $(p - i)$
- 6.2 **Calculate** the proportional scale correction, SC_p
- $$SC_p = \frac{(p - i)}{Q_p} \times SC$$
- where: Q_p is the certified value of the quartz plate
SC is the scale correction for the quartz plate reading
- 6.3 **Add** the proportional scale correction, SC_p to $(p - i)$
 $(p - i)^* = (p - i) + SC_p$
- 6.4 **Correct** $(p - i)$ to normal strength $\left[\frac{1}{\text{normality}} \right]$

and apply dilution factor (multiply by 2).

If 1/2 normal solution (i.e. 32.5 g/250 mL),

$$P - I = 4 (p - i)^*$$

If 1 normal solution (i.e. 65 g/250 mL),

$$P - I = 2 (p - i)^*$$

- 6.5 **Multiply** $P - I$ by the original normality of the solution and determine the factor corresponding to this value from Table IV.

Table IV gives factors for the calculation of sucrose according to Jackson and Gillis, Method 2.

- 6.6 **Calculate** the % Sucrose in the sample

$$\% \text{ Sucrose} = \frac{100 \times [P - I]}{\text{Factor} - 0.53 [t_i - 20]}$$

where t_i = temperature of reading of the invert solution (5.15)

NOTE: The temperature coefficient of Jackson and Gillis is 0.53, comprising 0.03 for sucrose and 0.50 for invert sugar.

Record the result as % sucrose to 0.01%

- 6.7 The calculation assumes that both the plain and invert solutions have the same temperature at reading. Where this difference is greater than 1 °C the plain pol reading p , (5.11) should be corrected to the temperature of the invert solution from Table V.

Table V gives temperature corrections for plain polarisation reading in the determination of sucrose by double polarisation.

- 6.8 % **Sucrose** may be determined directly from $(P - I)$ values listed in Table VI.

Table VI gives % Sucrose corresponding to various values of $(P - I)$ for half normal solutions at various temperatures.

This table should only be used when expressing the result to 0.1% gives sufficient accuracy.

7.0 PRECISION

Sample	Concentration	95%	95%
	Range % Sucrose	Repeatability r	Reproducibility R
Pan			
Products	10 — 90	0.8%	1.4%

8.0 REFERENCES

- (1) Clerget, T., Ann. Chim. Phys., Vol. 26 (1849), p. 175.
- (2) Jackson, R.F. and Gillis, C.L., U.S. Bureau of Standards, Scientific Paper No. 375 (1920).
- (3) Brown, C.A. and Zerban, F.W., Physical and Chemical Methods of Sugar Analysis, Ed. 3, Wiley (1955), pp. 321-5.
- (4) Wilson, R.A.M., International Sugar Journal, Vol. 67 (1965), p. 235.

METHOD 19

TOTAL SOLIDS (DRY SUBSTANCE) — DETERMINATION IN MILL PRODUCTS

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry. The method is used to determine water and/or total solids in factory process products of more than 30° brix.

This is an empirical method which assumes that all the water in the sample is removed by the drying process and that the loss of mass is due only to the removal of this moisture.

2.0 PRINCIPLE

A test portion of the sample is diluted to approximately 30° brix in a weighing tube. The solution is soaked into a coiled strip of filter paper to increase its surface area. This assists water evaporation and avoids formation of a protective skin. The sample is then dried at atmospheric pressure for 6 hours in an oven at 98 °C or in a vacuum oven at 65 °C for 18 hours at a pressure not exceeding 50 mm mercury. The loss of mass after drying represents the water in the sample. It should be pointed out that the reference method is that based on the vacuum oven.

Some sugars, especially fructose, begin to decompose in the temperature range 70-100 °C, producing water and other volatile products which contribute to the loss of mass and can cause high moisture (low total solids) values.

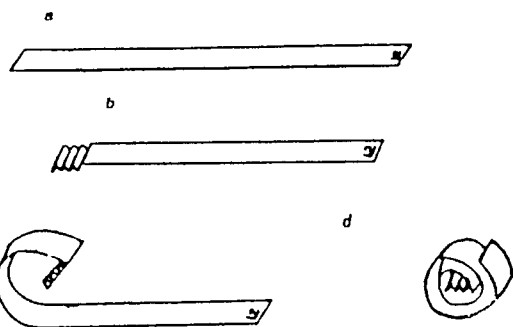
3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

- 3.1 **Desiccator**, with self indicating silica gel.
- 3.2 **Drying oven**, forced draft, capable of being controlled at $98 \pm 2^\circ\text{C}$ or **vacuum oven** capable of being controlled at $60 \pm 2^\circ\text{C}$.
- 3.3 **Weighing bottle**, glass or metal, with lid, 70 mm \times 35 mm.
- 3.4 **Filter paper strip**, 600 mm long \times 50 mm wide. For example, suitable strips may be cut from Whatman No. 4 chromatography paper sheets.

4.0 PROCEDURE

- 4.1 Roll a filter paper strip (3.4) into a coil, mark the number of the weighing bottle (3.3) on the paper with a pencil and place the paper in the weighing bottle. The paper coil is best made by making several pleats (about 5 mm) in the end of the strip and then, using these as the axis, coil the rest of the strip.



- 4.2 Place the weighing bottle containing the paper, in the drying oven with the lid alongside, and dry for 3-6 hours at $98 \pm 2^\circ\text{C}$ or in a vacuum oven and dry for 6 hours at $60 \pm 2^\circ\text{C}$. Cool the tube and paper, with the lid fitted, in a desiccator for 45-60 minutes.
- 4.3 Weigh the bottle and dried paper with the lid on to 0.0001 g (m_1). Store the bottle with lid in place in a desiccator.
- 4.4 Remove the paper from the tube and weigh empty tube and lid to 0.0001 g (m_2).
- 4.5 Stir the sample to be analysed thoroughly with a brass rod, removing surplus material from the rod by wiping it on the side of the sample container.
- 4.6 Transfer the required amount of sample to within 0.1 g to the weighing bottle (see 4.7). Weigh the bottle and lid and sample to 0.0001 g (m_3).
When transferring the sample to the bottle, move the rod carefully down the centre of the bottle, to avoid getting any sample on the walls of the bottle where it is difficult to dissolve. If this occurs and it cannot readily be removed with a tissue or glass towel the bottle should be discarded and a fresh start made with a clean bottle.
- 4.7 Dilute the sample to approximately 30° bx by adding the required volume of distilled water (to within 0.1 mL) with a barrel pipette.

Approximate % solids in product	Mass of product to be taken (g)	Volume of water to be added (mL)
100	2.0	4.5
90	2.2	4.3
80	2.5	4.0
70	2.9	3.6
60	3.3	3.2
50	4.0	2.5
40	5.0	1.5
30	6.7	0.0
Less than 30	7.0	0.0

- 4.8 Boil water in a 1000 mL boiling flask with 7.5 cm diameter glass funnel placed in the neck of the flask.
- 4.9 Dissolve the sample by steam heating the bottle in the glass funnel (4.8). Solution is completed by gently swirling the heated bottle. Do not use a bunsen flame or hot plate to heat the bottle since these may cause decomposition of the sample. Care must be taken to ensure that all the sample has dissolved, particularly in the case of massécuites and molasses. Do not heat any longer than is necessary to obtain dissolution.
- 4.10 Place the loosely rolled filter paper into the bottle and incline bottle about 60° from the vertical. Slowly rotate the bottle to assist absorption of all the solution. Allow the bottle to stand for 10 minutes and check that all the solution has been absorbed.

NOTE: Check that the number on the paper and that on the bottle coincide.

METHOD 19 P.2.

4.11 Place the bottle and contents with lid alongside, in the drying oven and dry for 6 hours \pm 10 minutes at $98 \pm 2^\circ\text{C}$ or dry in a vacuum oven at a pressure not exceeding 50 mm mercury for 18 hours at $65 \pm 2^\circ\text{C}$. After drying, fit the lid on the bottle and cool in desiccator for 45-60 minutes.

4.12 Weigh dry bottle, paper and solids, with lid on, to 0.0001 g (m_4).

5.0 CALCULATIONS

5.1 Calculate % total solids.

$$\begin{aligned}\% \text{ total solids} &= \frac{\text{mass of dry solids}}{\text{mass of sample}} \times \frac{100}{1} \\ &= \frac{(m_4 - m_1)}{(m_3 - m_2)} \times \frac{100}{1}\end{aligned}$$

where: m_1 = mass of weighing bottle + lid + paper (4.3)

m_2 = mass of weighing bottle + lid (4.4)

m_3 = mass of weighing bottle + lid + sample (4.6)

m_4 = mass of weighing bottle + lid + paper + dried sample (4.12)

Record the result as % (m/m) total solids to the nearest 0.1%.

5.2 Calculate % moisture

$$\% \text{ moisture} = 100 - \% \text{ total solids}$$

Record the result as % (m/m) moisture to the nearest 0.1%.

6.0 PRECISION

Sample	Concentration Range	95%	95%
	% (m/m) moisture	Repeatability r	Reproducibility R
Factory products ($> 30^\circ \text{bx}$)	5 - 90	0.4%	1.0%

7.0 REFERENCE

Jossé, A.; Bull. Assoc. Chem. Suc.; Vol. 10 (1893), p. 656.

METHOD 20

REDUCING SUGARS — DETERMINATION IN MILL JUICES, CLARIFIED JUICES AND PAN PRODUCTS BY THE CONSTANT VOLUME MODIFICATION OF THE LANE AND EYNON METHOD

1.0 SCOPE AND FIELD OF APPLICATION

The method has been officially adopted by the ICUMSA and will supersede the previous classical procedure of the Lane and Eynon Method.

The reducing sugar concentration in the test sample should lie within the range of 250 to 400 mg/100 mL. Samples with levels outside this range are either diluted, or standard invert solution is added, prior to analysis. In the latter instance, the concentration in the original test solution is determined by subtraction of the added invert from the total reducing sugars found.

2.0 PRINCIPLE OF METHOD

The principle of this constant volume modification is the same as for the original method of Lane and Eynon, in which the main part of the test solution is added to a definite volume of a strongly alkaline cupric-complex salt solution, Fehling's solution, which is then boiled.

The remainder of the test solution is then added, until, at the end-point, the cupric ions are completely reduced to cuprous oxide and the blue colour of the solution disappears. The sharpness of the end-point is improved by the use of an indicator, methylene blue, which is decolourised in the presence of a minute excess of reducing sugars.

The constant volume modification differs only in the fact that at the end-point, the final volume, and consequently the concentration of the Fehling's solution in the reaction flask, has been kept constant by the prior addition of a predetermined amount of water. For this reason the titration always corresponds to the same amount of invert sugars and allows the use of a simple formula instead of tables. As sucrose will be present, and be partially converted to reducing sugars due to the high pH and temperature of the reaction mixture, the result must be multiplied by a sucrose correction factor. The experimental conditions, including volume and concentration of Fehling's solution, time of boiling and final volume of reaction mixture, are strictly defined.

Therefore, for accurate work, three titrations are necessary, one preliminary test run to determine the volume of added water required and two titrations which fulfil the requirement that all but the last 1 mL of test solution is added before heating.

3.0 APPARATUS

Ordinary laboratory apparatus and

- 3.1 **Burette**, 50 mL, AS 2165, graduated in 0.1 mL for the sugar solution. The burette should have a pinch-cock instead of a glass tap and a bent outlet tube in order to keep the graduated section of the burette out of the steam while additions are made to the boiling mixture.
- 3.2 **Bulb pipettes**, 15, 20 and 25 mL, Class A (AS2166).
- 3.3 **Heat resistant plate** (about 15 cm x 15 cm) with a 5 cm diameter hole.

- 3.4 **Anti-bumping granules** (pumice).
- 3.5 **Boiling flasks** (florencia flask) 200/250 mL, flat bottom.
- 3.6 **Volumetric flask**, 100 mL A class (AS 2164).
- 3.7 **Stainless steel gauze strainer** about 5 cm dia., 100 mesh.
- 3.8 **Stopwatch** with 60 second sweep.
- 3.9 **Bunsen burner**.

4.0 REAGENTS

- 4.1 **Fehling's Solutions** (Soxhlet's Modification)
Mixed Fehling's solution does not keep indefinitely and the ingredients are therefore dissolved in two separate solutions, A and B, which are mixed together immediately before use. This mixing is done by adding a volume of solution A to an exactly equal volume of solution B. It is essential that the mixture be carried out in this order otherwise the precipitate of cupric hydroxide initially formed may not redissolve completely.
Solution A: cupric sulphate pentahydrate, AR (69.28 ± 0.01 g) is dissolved and diluted to 1 L in distilled water.
Solution B: sodium potassium tartrate tetrahydrate, AR (346 ± 1 g) and sodium hydroxide AR (100 ± 0.5 g) are dissolved in distilled water and diluted to 1 L.
- 4.2 **Standard Invert Sugar Solution, 1 g Invert Sugar/100mL:**
Stock Solution
4.2.1 Weigh 9.500 ± 0.001 g of sucrose and transfer quantitatively to 1 L volumetric flask with 100 ± 5 mL of water. Swirl to dissolve.
4.2.2 While swirling gently, add 5 mL of hydrochloric acid, ($\rho_{20} = 1.17$ g/mL). Cover the flask mouth with a small beaker and stand for 3-4 days at 20-25 °C for complete inversion of the sucrose.
4.2.3 Dilute the invert solution to about 800 mL with water. Dissolve 2.0 ± 0.1 g benzoic acid in hot water, cool and add to the invert solution. Dilute to exactly 1L and mix well. Stored in a stoppered bottle, this solution will keep for several weeks.
- 4.3 **Neutral Standard Invert, 0.25 g/100 mL Solution**
Pipette 50mL of 1% invert solution (4.2) into a 200 mL volumetric flask. Add 2 drops phenolphthalein solution and while swirling gently add 1 mol/L NaOH solution from a burette until the solution is just pink. Approximately 2.5 mL of 1 mol/L NaOH will be needed.
Add 1-2 drops of 0.5 mol/L HCl to just discharge the pink colour. Dilute the solution to exactly 200 mL with water and mix well.
- 4.4 **Methylene Blue Indicator, 1 g/100 mL**
Pure methylene blue (1.0 ± 0.05 g) is dissolved and diluted to 100 mL using distilled water, and then filtered if necessary.

METHOD 20 P.2.

5.0 PROCEDURE

5.1 Standardisation of the Fehling's Solution

The copper content of the Fehling's solution, prepared as described above (4.1), varies slightly from one solution to another and must therefore be adjusted. To 20 ± 0.06 mL of Fehling's solution are added 15 ± 0.05 mL distilled water and 39.0 ± 0.1 mL (by burette) of invert sugar solution (0.25 g/100 mL). Using the procedure of section 5.4 from the second paragraph onwards, the titration is completed with the invert sugar solution (0.25 g/100 mL). The total volume of invert sugar solution required should be 40 ± 0.1 mL if the Fehling's solution contains the correct amount of copper (equivalent to 100 mg invert sugar). The Fehling's solution should be adjusted as necessary by the addition of a calculated amount of cupric sulphate or water, followed by thorough mixing. Another standardisation should be carried out after any such adjustment.

5.2 Preparation of the Test Solution

(a) Mill Juice and Clarified Juice

The concentration of the test solution should, if necessary, be adjusted to fall between 250 and 400 mg invert sugar/100 mL.

(b) Pan Products

The dilution required for sugar mill products must be ascertained by a trial and error basis to produce test solutions with a reducing sugar concentration with the range of 250 to 400 mg/100 mL.

Calcium present in molasses forms a complex with glucose and fructose, resulting in slower reaction rates and apparently low reducing sugar content. The removal of calcium is therefore achieved by adding a complexing agent such as EDTA which forms a stronger complex with calcium than do the hexoses. An effective decrease in colour and an improved end-point are obtained. 4 mL of EDTA solution (4 ± 0.01 g/100 mL ethylene diamine tetra acetic acid, disodium salt) per g of cane molasses is recommended.

The required volume of EDTA solution is added to the subsample of pan product once it is dissolved but prior to making to the mark with distilled water.

5.2.1 For juices, where a volume is taken, determine the brix to 0.1 °bx by spindle or refractometer.

5.2.2 Where the invert sugar concentration of the test solution falls below 250 mg/100 mL, additional invert in the form of a known volume of standard invert (e.g., 1.0 g/100 mL) should be added to bring the resultant diluted sample up to a final invert concentration within the desired range.

5.3 Preliminary Test

A preliminary test should be carried out to ascertain the volume of water to be added to the 20 mL of Fehling's solution in order to obtain a final total volume of 75 mL when the end-point of the titration is reached.

The following mixture is prepared:
 20 ± 0.06 mL of Fehling's solution,

25 ± 0.06 mL of the test solution, and
 15 ± 0.5 mL of distilled water.

The last two additions correspond to the 40 mL of dilute invert sugar solution used in the calibration procedure. The mixture is then titrated with additional test solution using the procedure of the following section (5.4) from the second paragraph. The bulk of the test solution should be added as early as possible during the initial warm up before the boiling period. If the reddish colour of the boiling solution persists after the addition of the methylene blue indicator, this indicates that the test solution must be discarded and a less concentrated solution used.

If more than 50 mL of test solution, added to 20 mL of the Fehling's solution, are required to obtain the reddish colour, this indicates that a more concentrated solution should be used.

The volume of water to be added is calculated as 75 mL (total) — 20 mL (Fehling's solution) — volume (mL) of test solution = volume (mL) of water to be added.

5.4 Titration

Mixed Fehling's solution (4.1) (20 ± 0.06 mL) is pipetted into a glass flask (3.5); the volume of distilled water indicated by the preliminary test is then added. The burette (3.1) is rinsed and filled with the test solution. The whole volume of test solution required in the preliminary test less 1 mL is run into the flask. A few fragments of pumice (3.4) are added and the contents of the flask are well mixed by gentle swirling. The flask is placed on a wire gauze (3.7) over a bunsen flame and heated to boiling (within approximately 2 minutes 30 seconds). (See 5.5).

The liquid is kept boiling moderately fast for precisely 2 minutes and then 3 or 4 drops of methylene blue indicator (4.4) are added directly into the boiling mixture. The mixture should assume a distinctly blue colour.

The titration is completed in 1 minute \pm 5 seconds by the further addition of small increments, initially of 0.2 mL, then 0.1 mL and finally of single drops until the end-point is reached. It is indicated by the complete disappearance of the blue colour of the indicator and the appearance of the reddish colour due to the precipitated cuprous oxide.

5.5 Precautions

The titration should be completed in 3 minutes \pm 5 seconds from the commencement of boiling.

The heating device* used for boiling the reaction mixture during the titration is of prime importance when accurate results are to be guaranteed. During the whole time, the flask should remain on the wire gauze and boil at a moderate rate. The continuous emission of steam from the neck prevents atmospheric oxidation of the Fehling's solution or of the indicator. During the additions of sugar solution to the boiling liquid, the main burette tube (3.1) must be kept out of the steam outlet while the jet is brought over the mouth of the flask.

* An electric heater called the "United Molasses Combined Heater / Illuminator" is recommended for this purpose and is available from R&L Slaughter Ltd., 162 Balgore Lane, Gidea Park, Romford, Essex, U.K.

6.0 CALCULATIONS

- 6.1 Where a measured volume of sample is taken, calculate the weight of sample per 100 mL based upon the density (g/mL) obtained from Table XV, using the brix value measured in 5.2.
- 6.2 If necessary, calculate the amount of sucrose per 100 mL titrating solution from the weight of sample and its approximate percentage sucrose. If a measured volume of sample is taken, estimate the sucrose content by using the density obtained from Table XV using the brix value (from 5.2) and assumed purity, e.g. if assumed purity = 80% and brix = 20%. Then density = 1.081 g/mL (Table XV). And —

$$\text{Sucrose (g per 100 mL)} = \frac{8}{100} \times 20 \times \frac{1}{1.081}$$

- 6.3 The reducing sugar content is given by the formula:

$$\text{Reducing sugar, (\%)} = \frac{1000f}{V_t C_t} - \frac{C_s V_s}{C_t}$$

Where C_t is concentration (g/100 mL) of sample in the test solution, V_t is the volume (mL) of the test solution used in the titration, C_s is the concentration (g/100 mL) of the standard invert added, if necessary, V_s is the volume (mL) of any invert added in the test solution and f is the correction factor deduced from the Emmerich Table (Table 1) based on the amount of sucrose present.

The amount of sucrose present in the mixture (g) is equal to sucrose (g per 100 mL) in the titrating mixture $\times 0.01 V_t$.

When no invert is added, the second term is zero and

$$\text{Reducing sugar, (\%)} = \frac{1000f}{V_t C_t}$$

For amounts of sucrose intermediate between two consecutive figures in Table 1, the correction factor is obtained by interpolation or from the formula:

$$f = 1 - 0.0292 S^{2/3}$$

where: S = approximate sucrose content in test solution.

7.0 RANGE AND PRECISION

The expected range of results is —

Direct method 0.6 to 100%

Added invert 0.05 to 0.6%

The precision that should normally be obtained by following the direct procedure is:

Standard deviation for one analyst in one laboratory: 0.1%

Standard deviation for different analysts in different laboratories: 0.5%

95% repeatability*: 0.3%

95% reproducibility*: 1.4%

8.0 REFERENCES

Pieck, R. Subject 14: Reducing Sugars. Proc. 17th Session ICUMSA, 1978, 190-211.

Laursen, J. Subject 14: Reducing Sugars. Proc. 18th Session ICUMSA, 1986 232-267.

- * These figures are to be used when there are two and only two, determinations.

TABLE 1
SUCROSE CORRECTION TO BE APPLIED IN LANE
AND EYNON CONSTANT VOLUME METHOD
(ACCORDING TO PIECK, ICUMSA, 17TH SESSION
(1978) SUBJECT 14, TABLE 4, 194)

Sucrose in boiling mixture (g)	Correction factor (f)	Sucrose in boiling mixture (g)	Correction factor (f)
0.0	1.000		
0.5	0.982		
1.0	0.971	6.0	0.904
2.0	0.954	7.0	0.893
3.0	0.939	8.0	0.883
4.0	0.926	9.0	0.874
5.0	0.915	10.0	0.864

These corrections are being investigated and may undergo slight changes in the near future.



METHOD 21

REDUCING SUGARS — DETERMINATION IN MILL PRODUCTS BY THE LANE AND EYNON METHOD

1.0 SCOPE AND FIELD OF APPLICATION

This method is used in the Australian sugar industry for the determination of reducing sugars in factory process materials. The results obtained are used for process control.

2.0 PRINCIPLE OF METHOD

An appropriate amount of the product, based on the expected reducing sugars and sucrose contents is weighed and dissolved in water. The reducing sugars are determined by the Lane and Eynon titration.

3.0 APPARATUS

Ordinary laboratory apparatus and

- 3.1 **Bulb pipette**, 5 mL Class A. (AS 2166)
- 3.2 **Bulb pipette**, 10 mL Class A. (AS 2166)
- 3.3 **Burette**, 50 mL Class A, (AS 2165) graduated in 0.1 mL for the sugar solution. The burette tap is replaced by a rubber tube and pinch-cock, connected to a bent outlet tube in order to keep the graduated section of the burette out of the steam while additions are made to the boiling mixture.
- 3.4 **Heat resistant plate** (about 15 cm x 15 cm) with a 5 cm diameter hole.
- 3.5 **Anti-bumping granules**.
- 3.6 **Boiling flasks** (florencia flask) 200/250 mL, flat bottom.
- 3.7 **Volumetric flask**, 100 mL A-class. (AS 2164).
- 3.8 **Stainless steel gauze strainer** about 5 cm dia., 100 mesh.
- 3.9 **Stop-watch** with 60 second sweep.
- 3.10 **Bunsen burner**.

4.0 REAGENTS

During the analysis unless otherwise stated, use only reagents of analytical grade and only distilled water.

- 4.1 **Fehling's Solution A**.
 - 4.2 **Fehling's Solution B**.
 - 4.3 **Methylene Blue**, 1 g/100 mL solution in water.
 - 4.4 **Standard Invert Solution**.
 - 4.5 **Differential Invert Solution**.
- NOTE: Directions for preparing the above reagents are given in Method 32 "Reducing Sugars-Determination in raw sugar by the Lane and Eynon Method".
- 4.6 **Preserving solution for mill juices**. Dissolve 50 g of anhydrous sodium carbonate in water, add 50 mL of formalin (37 g/100 g formaldehyde), dilute to 1 litre and mix.
 - 4.7 **EDTA**, 4.00 ± 0.01 g/100 mL solution in water of ethylenediaminetetra-acetic acid, di-sodium salt.

5.0 TREATMENT OF SAMPLE

- 5.1 When sampling mill juices which are to be composited and stored before analysis, immediately add 1.0 mL preserving solution (4.6) for each 10.0 mL juice. Composite the samples, mix well and store in a sealed bottle at room temperature.

6.0 PROCEDURE

A. STRAIGHT METHOD (for products with >0.6% RS)

- 6.1 Weigh out the appropriate quantity of product indicated in Table XXXI. For juices, using a measuring cylinder take 20, 30, 40 or 50 mL of the preserved sample as required.
Determine the brix of the juice sample to 0.1° by spindle or refractometer.
- 6.2 Wash the sample into a 100 mL volumetric flask. Hot water may be needed for some products.
NOTE: For invert syrup, prepare 1L of solution. For molasses, prepare 200 mL.
- 6.3 Dissolve the sample by shaking and cool to room temperature. Add water to fill the bulb of the flask and mix well. Make to the mark, stopper and mix well.
- 6.4 Using a 5 mL class 'A' pipette, add 5.00 mL of Fehling's A solution to a 200 mL boiling flask. Using a safety pipette, add 5.0 ± 0.1 mL of Fehling's B solution and swirl to complete the preparation of the alkaline copper solution. Fehling's B should be added just before the titration is to be done.
- 6.5 Add a few pieces of anti-bumping granules to the boiling flask to prevent bumping.
- 6.6 Rinse (about 10 mL) and fill a clean 50 mL Class A burette with titrating solution (6.3). Squeeze the rubber tubing above the pinch-cock to remove air-bubbles; open the pinch-cock and incline the jet to remove the remaining air-bubbles. Adjust the level of solution to the zero mark.
- 6.7 Add 15 mL of the solution from the burette to the alkaline copper solution (6.4), swirl, and place the flask on a heat resistant plate (3.4) over a bunsen burner.
- 6.8 Bring the mixture to the boil, making sure that the flame does not impinge on the flask above the level of the solution.
- 6.9 Start the stopwatch when the mixture commences to boil vigorously and adjust the flame to give moderate even boiling.
- 6.10 After boiling for 15 secs, if the solution is still blue, add rapidly successive small portions of solution from the burette (but without interrupting boiling) until the reddish colour shows that the end-point is near. With experience this can be judged to within 2-3 mL. This estimated end-point should be reached within the first minute of boiling.
- 6.11 After 2 minutes of boiling add 3 drops of methylene blue indicator (4.3).
- 6.12 Continue titrating adding a few drops at a time, allowing 5-10 secs between each portion, until the blue colour is completely discharged, and note the final burette reading. The total boiling time should be 3.0 ± 0.5 min. and boiling should not be interrupted during the titration (to prevent air entering the flask and oxidising the contents).
- 6.13 For routine work, the above incremental method gives satisfactory results, provided that no more than 3 mL

METHOD 21 P.2.

of solution is needed to complete the titration after step 6.10. For accuracy a second titration is required.

- 6.14 Prepare another alkaline copper solution (6.4), add all but 1-2 mL of the required amount of titrating solution from the burette, and repeat steps 6.8 and 6.9.
- 6.15 Continue boiling for 2 minutes before adding the methylene blue indicator and complete the titration as before. The total boiling time should be kept as close as practicable to 3 minutes (but no more than 3½ minutes). Record the end-point to nearest 0.1 mL. The end point must lie between 17 and 35 mL.
- B. 1ST DIFFERENTIAL METHOD** (for products with 0.05-0.6% RS)
- 6.16 Weigh to ± 0.01 g a quantity of the product that will contain 25 g of sucrose.
- 6.17 Wash the sample into a 100 mL Class 'A' volumetric flask. Hot water may be needed for some products. Add water to 50-60 mL and dissolve by shaking.
- 6.18 Using a 10 mL Class 'A' bulb pipette, add 10.00 mL neutralised differential invert solution (4.5), add water to fill the bulb of the flask, swirl, make to the mark, stopper and shake well. This is the test solution.
- 6.19 Prepare a "blank" solution by weighing out 25.00 ± 0.01 g standard sugar and repeating steps 6.17 and 6.18.
- 6.20 Titrate the test solution (6.18) as described in steps 6.4 to 6.15 and record the end point to 0.1 mL.
- 6.21 Titrate the blank (6.19) as described in steps 6.4 to 6.15 but run in 31 mL solution for the first titration. (The end-point should lie between 32 and 36 mL). Record the end-point to 0.1 mL.
- C. 2ND DIFFERENTIAL METHOD** (for sweetwaters, etc.)
- 6.22 Weigh a quantity of the sample containing not more than 0.2 g RS into a 200 mL volumetric flask.
- 6.23 Pipette 25.0 mL of neutralised differential invert solution (4.5) into the flask. Add 4.0 mL of EDTA solution (4.7) for each gram of product if it is necessary to mask calcium salts.
- 6.24 Make to the mark with water and mix well. Filter through a Whatman No. 31 filter paper. This is the test solution.
- 6.25 Prepare a blank solution with 25.0 ± 0.1 mL of neutralised differential invert solution and water in a second 200 mL volumetric flask. Add the same volume of EDTA if this reagent was used in (6.23).
- 6.26 Titrate the test solution as in 6.4 to 6.15. Record the end point to 0.1 mL.

- 6.27 Titrate the blank solution as in 6.4 to 6.15. Record the end point to 0.1 mL.

7.0 CALCULATIONS

- 7.1 **Table XXXI** gives the weight of product to be weighed out per 100 mL of titrating solution for the determination of RS in products of various sucrose and RS content.
- 7.2 If necessary, calculate the amount of sucrose per 100 mL of titrating solution from the weight of sample and the approximate % sucrose. If a measured volume of sample is taken, calculate the amount of sucrose from the density at 20 °C (Table XII or XV) and the assumed purity. Make allowance for any dilution.
- 7.3 Determine the grams RS per 100 mL of titrating solution corresponding to the end point of the titration from Tables XXXII or XXXIII, (e.g. 0 g sucrose for 2nd differential method).
- 7.4 For 1st differential method read grams RS in 100 mL blank solution under the 25 g sucrose column of Table XXXII.
For 2nd differential method read grams of RS in 100 mL blank solution under the 0 g sucrose column of Table XXXIII.
- 7.5 Calculate the % RS in product.

For straight method:

$$\% \text{ RS} = \frac{100 \times \text{g RS per 100 mL titrating solution}}{\text{g sample per 100 mL titrating solution}}$$

For differential methods:

$$\% \text{ RS} = \frac{100 \times [\text{g RS per 100 mL titrating solution} - \text{g RS per 100 mL blank}]}{\text{g sample per 100 mL titrating solution}}$$

- 7.6 **Record** the result as % RS in product to
 Juices 0.01%
 Mill products 0.1%

8.0 PRECISION

Sample	Concentration range (percent reducing sugar)	95% Repeatability r	95% Reproducibility R
Molasses	5-20	0.30	1.40

METHOD 22

TOTAL SUGARS (AFTER INVERSION) — DETERMINATION IN MOLASSES AND CANE INVERT BY THE CONSTANT VOLUME MODIFICATION OF THE LANE AND EYNON METHOD

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian Sugar Industry. It is the method specified under the Australian Molasses Pool contract and is based on the method published by the United Molasses Co. Ltd., London, (U.M.C. Method).

2.0 PRINCIPLE OF METHOD

The test sample is diluted to a fixed concentration and sucrose in the sample hydrolysed (inverted) with hydrochloric acid at 60°C under carefully controlled conditions. The total reducing sugars in the inverted solution are determined by the constant volume modification of the Lane and Eynon titration. EDTA solution is added to the inverted solution to suppress interference from calcium.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

- 3.1 **Bulb pipette**, Class A (AS 2166) — 5 mL, 20 mL, 25 mL, 50 mL
- 3.2 **Burette**, 50 mL Class A, (AS 2165) graduated in 0.1 mL for the sugar solution. The burette tap is replaced by a rubber tube and pinch-cock and connected to a bent outlet tube in order to keep the graduated section of the burette out of the steam while additions are made to the boiling mixture.
- 3.3 **Heat resistant plate** (about 15 cm x 15 cm) with a 5 cm diameter hole.
- 3.4 **Anti-bumping granules**.
- 3.5 **Boiling flasks** (florencia flask) flat bottom, 250 mL.
- 3.6 **Volumetric flask**, A-class (AS 2164) — 100 mL, 200 mL, 250 mL, 1000 mL
- 3.7 **Stop watch** with 60 second sweep.
- 3.8 **Bunsen burner**.
- 3.9 **Water bath**, maintained at $60 \pm 1^\circ\text{C}$.

4.0 REAGENTS

Unless otherwise specified use only analytical grade reagents and only distilled water.

- 4.1 **Fehling's Solution A**. A solution of 69.28 ± 0.05 g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in 1 litre of water.
- 4.2 **Fehling's Solution B**. Dissolve 346 ± 1 g sodium potassium tartrate in distilled water. Add that volume of standardised 5 mol/L sodium hydroxide solution which contains 100.0 ± 0.5 g NaOH. Dilute the solution to 1 litre.

Safety

This reagent is strongly caustic. It should not be pipetted by mouth. If in contact with skin, remove all contaminated clothing and wash the affected area with copious quantities of water. If ingested administer large quantities of 1% acetic acid. Do not induce vomiting.

- 4.3 **Methylene Blue Solution** — 1 g/100 mL solution in water.
- 4.4 **Standard Invert** — 10 g/litre solution.
 - 4.4.1 Weigh $9.500 \pm .001$ g of sucrose and transfer

quantitatively to 1L volumetric flask with 100 ± 5 mL of water. Swirl to dissolve.

- 4.4.2 While swirling gently, add 5 mL of hydrochloric acid, $\rho_{20} = 1.18$ g/mL. Cover the flask mouth with a small beaker and stand for 3-4 days at 20-25°C for complete inversion of the sucrose.
- 4.4.3 Dilute the invert solution to about 800 mL with water. Dissolve 2.0 ± 0.1 g benzoic acid in hot water, cool and add to the invert solution. Dilute to exactly 1L and mix well. Stored in a stoppered bottle, this solution will keep for several weeks.
- 4.5 **Neutral Standard Invert**, 0.25 g/100 mL Solution
 - Pipette 50 mL of 1% standard invert solution (4.4) into a 200 mL volumetric flask. Add 2 drops phenolphthalein solution and while swirling gently add 1 mol/L NaOH solution from a burette until the solution is just pink. Approximately 2.5 mL of 1 mol/L NaOH will be needed.
 - Add 1-2 drops of 0.5 mol/L HCl to just discharge the pink colour. Dilute the solution to exactly 200 mL with water and mix well.
- 4.6 **Sodium Hydroxide**, approx. 2 mol/L solution.
- 4.7 **Hydrochloric Acid**, approx. 0.5 mol/L solution.
- 4.8 **Phenolphthalein**, 1 g/100 mL solution in alcohol.
- 4.9 **Hydrochloric Acid**, 6.34 mol/L solution $\rho_{20} = 1.103$ g/mL. Dilute 630 ± 5 mL of hydrochloric acid, $\rho_{20} = 1.18$ g/mL, to 1L with water and adjust to exactly 6.34 mol/L after titrating 5 mL with 1 mol/L NaOH using methyl orange indicator.
- 4.10 **Di-Sodium Salt of EDTA**, 4 g/100 mL solution.
- 5.0 **STANDARDISATION OF FEHLING'S SOLUTION A**
 - 5.1 Using Class A pipettes with safety bulb, pipette 50 mL of Fehling's A (4.1) followed by 50 mL of Fehling's B (4.2) into a clean, dry beaker. Mix by swirling.
 - 5.2 Pipette 20 mL of the mixed Fehling's solution into a boiling flask. Add 15 mL water from the burette.
 - 5.3 Rinse and then fill the burette (3.2) with standard invert solution (4.4). Add 39 mL of standard invert to the boiling flask, giving a total volume of 74 mL. Add a few pieces of anti-bumping granules.
 - 5.4 Place the flask on a heat resistant plate (3.3) over the bunsen burner. Bring the solution to the boil and adjust the flame to give even moderate boiling.
 - 5.5 Start the stop watch when the solution begins to boil and allow to boil for exactly 2 minutes. Add 4 drops of methylene blue solution and continue to titrate the boiling solution with standard invert from the burette.
 - 5.6 Add a few drops at a time, allowing 5-10 secs between each addition, until the blue colour is completely discharged. The total boiling time should be 3 min. ± 5 secs and boiling should not be interrupted during the titration (to prevent air entering the flask and oxidising the contents).
 - Record the total titre to 0.05 mL.

METHOD 22 P.2.

- 5.7 If the Fehling's solution is the correct strength then 20 mL of this solution should require 40 mL of standard invert solution giving a total solution volume of 75 mL before commencing boiling.
- 5.8 If the titre is less than 40 mL then the Fehling's A solution is deficient in copper. Therefore add $\left[1 - \frac{\text{Titre}}{40.0}\right] \times 69.28$ g $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ to each litre of Fehling's A. Dissolve 105% of the calculated amount in the solution and titrate again. In this way the strength of the Fehling's A will finally be adjusted by dilution, the simpler procedure.
- 5.9 If the titre is more than 40 mL the Fehling's A is too strong and each titre must be diluted by an amount equal to $\left[\frac{\text{Titre}}{40.0} - 1\right] \times 100$ mL water. Add 95% of the calculated volume of water and titrate again.
- 5.10 When the Fehling's A solution is the correct strength 20 mL of mixed Fehling's solution (5.1) is equivalent to 100 mg of invert sugar. If the titre is between 39.90 and 40.10 mL then a correction factor can be calculated and applied —
- $$F = \frac{\text{standard titre}}{40.0}$$
- 6.0 PROCEDURE**
- 6.1 Prepare a 5% solution of molasses or cane invert. Weigh 10.00 ± 0.01 g of the sample into a 250 mL beaker. Dissolve in approximately 50 mL of warm water (cold water for cane invert), cool and transfer quantitatively to a 200 mL volumetric flask. Dilute to exactly 200 mL with water and mix well.
- 6.2 Prepare a 0.5% solution by pipetting with a Class A pipette, 25 mL of the 5% solution into a 250 mL volumetric flask.
- 6.3 Add, while swirling the solution, 5.00 ± 0.05 mL of 6.34 mol/L HCl (4.9) using an automatic dispenser.
- 6.4 Immerse the flask in the water bath at 60°C. Swirl gently for 3-4 minutes to raise the temperature of the solution as quickly as possible. Leave the flask in the bath for a total of 15 minutes.
- 6.5 Remove the flask from the hot water bath and cool rapidly under running cold water.
- 6.6 Add 95-100 mL water and 3 drops of phenolphthalein solution. Add sufficient 2 mol/L NaOH solution till the solution is just pink and then discharge the colour by adding a few drops of 0.5 mol/L HCl.
- 6.7 Add 4.0 mL EDTA solution (4.10) mix well and dilute to volume with water. Mix well.

PRELIMINARY TITRATION

A preliminary titration is made to find the volume of water to be added to 20 mL of mixed Fehling's solution to obtain a final total volume of 75 mL at the end-point of the titration.

- 6.8 Pipette with a Class A pipette, 20 mL of mixed Fehling's solution (5.1) into a boiling flask.
- 6.9 Rinse and then fill the burette (3.2) with the inverted test solution (6.7). Add 25 mL of the solution to the boiling flask. Add a few pieces of anti-bumping granules and 1 drop paraffin oil to prevent frothing.
- 6.10 Place the flask on a heat resistant plate (3.3) over the bunsen burner. Bring the solution to the boil and adjust the flame to give even moderate boiling.
- 6.11 Start the stop watch when the solution begins to boil and allow to boil for exactly 2 minutes. Add 4 drops of methylene blue solution and continue to titrate with the test solution from the burette.
- 6.12 Add a few drops at a time, allowing 5-10 secs. between each addition, until the blue colour is completely discharged. The total boiling time should be 3 mins. ± 5 secs. Record the titre to 0.05 mL.

TITRATION

- 6.13 Carry out a further titration but this time add to the boiling flask —
- 20 mL of mixed Fehling's solution
 - the volume of test solution from the first titration (6.12) less 1 mL
 - sufficient water, from a burette to give a total volume of 74 mL
- Carry out the titration as in steps 6.10-6.12. Record the titre to 0.05 mL.
- 6.14 Carry out a third titration, repeating step 6.13. The two titrations (6.13 and 6.14) must agree to within 0.2 mL or the titrations must be repeated.

7.0 CALCULATIONS

Calculate the per cent total sugars (as invert) in the sample —

$$\% \text{ Total Sugars} = \frac{1000 \times F}{C \times T}$$

where: T = the mean titre (6.14)

C = concentration of the sample (6.2), usually 0.5 g/100 mL

F = standardisation factor (5.10)

7.0 PRECISION

The expected range of results is:

Molasses 45-60% Total Sugars

Cane Invert 78-81% Total Sugars

The precision of the method has not been determined.

METHOD 23

SUPERSATURATION AND SATURATION CONDITIONS — DETERMINATION IN PAN PRODUCTS

1.0 DEFINITIONS

The determination of the degree of supersaturation of molasses is of considerable importance in the study of pan boiling and crystallization. The explanation of the theory associated with the determination of coefficient of supersaturation involves the use of terms which are defined as follows:

- (a) Concentration is the percentage ratio by mass of solute to solvent in the solution under consideration. For molasses this corresponds to the percentage ratio of sucrose to water and must be determined analytically.

$$\text{Concentration} = \frac{\% \text{ sucrose in molasses}}{100 - \% \text{ dry substance}} \times 100$$

- (b) Saturation is the condition in which the quantity of solute (sucrose) is the maximum that can be dissolved in the solvent (water). When less than this quantity is dissolved, the solution is referred to as unsaturated (or undersaturated). If water is evaporated from a saturated solution or a saturated solution is cooled, then more sucrose is in solution than would exist at equilibrium and the solution is referred to as supersaturated. Although the condition of supersaturation is thermodynamically unstable, the tendency to revert to the equilibrium saturated condition by crystallization is sometimes very feeble.

- (c) Solubility is the concentration of solute in the solvent at saturation. The solubility of sucrose in water increases with increasing temperature in a non-linear relationship which has been determined experimentally. There are various ways of expressing the solubility, with grams of sucrose per 100 grams of water (Table XIII) being the most appropriate for the calculation of supersaturation coefficient. The solubility of sucrose in water is also influenced by the presence of soluble impurities, the effect varying with the quantity and type of impurity. Some impurities give sucrose an enhanced solubility while others cause solubility depression. The overall effect of the impurities present in cane molasses is usually to lower the solubility of sucrose.

- (d) Solubility coefficient is the ratio of the solubility of sucrose in the impure liquid phase of a sample to the solubility of sucrose in pure water, at the same temperature. As such, it quantifies the overall effect of the soluble impurities on the sucrose solubility, as discussed above.

$$\begin{aligned} \text{S.C.} &= \frac{\text{S/W ratio in molasses saturated at } T^{\circ}\text{C}}{\text{S/W ratio in pure water saturated at } T^{\circ}\text{C}} \\ &= f(T, I/W, \text{ impurity constituents and concentrations}) \end{aligned}$$

where:

I/W = mass ratio of impurities to water in the molasses

- (e) Supersaturation coefficient, usually abbreviated to supersaturation or S.S., is the ratio of the actual sucrose concentration in a sample to the solubility of sucrose in the liquid phase in the sample, at the same temperature and the same impurity to water ratio (I/W).

$$\begin{aligned} \text{S.S.} &= \frac{\text{actual S/W ratio in the molasses}}{\text{S/W ratio in saturated molasses at the same T and I/W ratio.}} \\ &= \frac{\text{actual S/W ratio in molasses at } T^{\circ}\text{C}}{\text{S/W ratio in saturated pure water at } T^{\circ}\text{C} \times \text{S.C.}} \end{aligned}$$

where S.C. is determined for the molasses at the same T and I/W ratio.

The supersaturation coefficient is the fundamental driving force for crystal growth and accordingly is very important in any crystallization studies. However, the determination of S.S. is difficult and some approaches are inappropriate. For example, stirring a supersaturated crystal sucrose-molasses mixture at constant temperature will not achieve a saturated condition by crystallization, in a finite time. This results from the fact that, although growth rate is linearly related to supersaturation, there exists a null zone at low supersaturation values in which crystal growth rates are extremely low. Thus any attempts to achieve a saturated condition must approach from an unsaturated condition, i.e. by dissolution, for which no such null zone exists.

2.0 MEASUREMENT OF SATURATION CONDITIONS

The measurement of the saturation conditions of a molasses sample is generally undertaken using one of two available methods.

1. The first is the saturation cell method in which the molasses sample under test, pre-mixed with fine seed crystal, is heated until dissolution commences. The temperature at which this occurs, called the saturation temperature, is noted. Originally the onset of dissolution was observed through a microscope (saturascope apparatus). The technique has been automated to some extent by the Sugar Research Institute by incorporating a photocell to monitor the change in light transmission that occurs at the start of dissolution. At present this version of the saturation temperature apparatus is not available commercially.

The saturation cell method provides a measure of the saturation temperature and from this, and

METHOD 23 P.2.

the analysis of the test sample, the solubility coefficient at the saturation temperature can be estimated. This is useful for many crystallization applications but is less suitable for fundamental research into the solubility of molasses where saturation measurements at different temperatures are required.

2. The second is the equilibration method where the test molasses is brought to saturation by tumbling at constant temperature from an initially undersaturated condition in the presence of excess sugar crystals. At the end of the equilibration time, the remaining crystals are observed under a microscope to check that the edges have been rounded, confirming that an initial undersaturated condition existed. A sample of the equilibrated molasses, obtained by pressure filtration, is analysed, allowing the solubility coefficient to be determined. Unfortunately, the application of this technique is limited to an impurity/water ratio of about 3.0 or less because of the tendency of molasses to degrade (and gasify) if held for extended periods above about 65 °C.

3.0 CALCULATION OF SUPERSATURATION

The case of a supersaturated solution of sucrose in pure water is very simple. If the solution has a temperature T_1 and on heating is found to be saturated at T_2 , then the actual concentration of sucrose present is that corresponding to the solubility of sucrose at T_2 which can be obtained from appropriate tables. The concentration of sucrose required to saturate the solution at its initial temperature T_1 is merely its solubility at T_1 from the same table.

$$\text{Thus S.S. at } T_1 = \frac{\text{sucrose solubility at } T_2}{\text{sucrose solubility at } T_1}$$

For impure solutions such as syrups and molasses, the situation is complicated by the effect that the impurities have on the sucrose solubility. Consider a supersaturated molasses sample at temperature T_1 . Then:

$$\text{S/W actual} = \text{sucrose solubility at } T_1 \times \text{S.C. at } T_1 \times \text{S.S.}$$

If this material is heated to its saturation temperature T_2 , then the concentration is unchanged but the supersaturation has dropped to unity. Therefore at T_2 :

$$\text{S/W actual} = \text{sucrose solubility at } T_2 \times \text{S.C. at } T_2 \times 1$$

Equating these and rearranging —

$$\text{S.S. at } T_1 = \frac{\text{sucrose solubility at } T_2 \times \text{S.C. at } T_2}{\text{sucrose solubility at } T_1 \times \text{S.C. at } T_1}$$

where the solubility coefficients at the two temperatures are determined at the same I/W ratio. Generally it is difficult to determine the supersaturation coefficient accurately because the values of the solubility coefficient are unknown and would require determinations by the equilibration method. As a first approximation, depending on the range of temperatures involved, it may be feasible to assume the solubility coefficients at the two temperatures are equal. This simplifying assumption then gives:

$$\text{S.S. at } T_1 = \frac{\text{sucrose solubility at } T_2}{\text{sucrose solubility at } T_1}$$

However, for cane molasses, the solubility coefficient is not independent of temperature, so care is required as the simplifying assumption made above may give rise to serious errors.

METHOD 24

VISCOSITY DETERMINATION IN PAN PRODUCTS

1.0 GENERAL

Molasses and massecuites generally exhibit non-Newtonian flow properties with the viscosity of these materials decreasing as higher rates of shear are applied. This is known as pseudoplastic flow behaviour.

Except for routine comparative analysis where a consistent rate of shear is encountered, it is not sufficient to define a single viscosity value for such materials but the viscosity must be determined over the shear rate range of interest for the process.

2.0 MODEL OF RHEOLOGICAL BEHAVIOUR

The flow behaviour of a material under a shear force is generally presented as a plot of shear stress against shear rate (flow curve). For Newtonian fluids, a constant proportionality exists between the shear stress τ and the shear rate γ in laminar flow viz.

$$\tau = \mu\gamma$$

where μ = viscosity of the fluid (S.I. units, Pa.s)

For non-Newtonian materials the velocity gradient changes shape at the interface of the fluid and the shear inducing element so that the viscosity is no longer constant but depends on the shear stress/shear rate. An absolute measurement of the viscosity of the non-Newtonian solution requires that a correction for the change in shear rate is made.

$$\mu = \tau / \gamma_{\text{corr}}$$

where μ = viscosity (absolute viscosity)
 τ = true shear stress at the interface
 γ_{corr} = corrected shear rate at the interface.

If no correction is made for the change in the shear rate profile due to the non-Newtonian behaviour, an apparent viscosity (μ_{app}) is determined:

$$\mu_{\text{app}} = \frac{\tau}{\gamma_{\text{uncorr}}}$$

where γ_{uncorr} = shear rate induced in a Newtonian fluid for the same relative motion (rotational speed, average velocity).

For measurements in simple commercial viscometers, this is the viscosity value determined using the supplied calibration factors.

In the usual practical range of shear rate for pipeline flow in factories, massecuites and molasses generally show pseudoplastic flow behaviour. In common with other such materials, a "Power Law" model usually adequately describes their non-Newtonian behaviour, i.e.

$$\tau = K\gamma_{\text{corr}}^n$$

$$\text{and } \mu = \tau / \gamma_{\text{corr}} = K\gamma_{\text{corr}}^{n-1}$$

where K is the consistency; the higher the value of K the more viscous the material (S.I. units $\text{Ns}^{-n}\text{m}^{-2}$)

and n is the flow behaviour index; a measure of the

degree of the non-Newtonian behaviour. For pseudoplastic fluids, n lies between zero and unity with values further removed from unity indicating a more pronounced non-Newtonian behaviour.

Thus, for such materials, the consistency and flow behaviour index should be specified rather than defining a single viscosity value. Figure 1 shows a typical viscosity versus shear rate relationship for a pseudoplastic material.

Because the flow behaviour index may vary over a wide range of shear rate then, whenever possible, the viscosity determinations should be made at the shear rate range of interest for the process. If extrapolation of data to a different shear range is necessary for design or process calculations, care should be exercised that significant errors are not introduced.

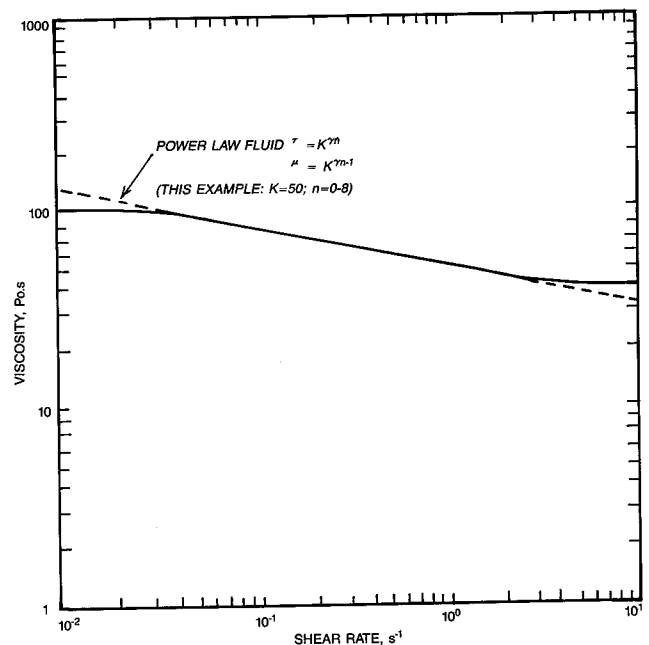


Figure 1. Viscosity as a Function of Shear Rate for a Material showing Pseudoplastic Flow Behaviour.

3.0 VISCOSITY MEASUREMENTS

To adequately measure the flow behaviour of molasses or massecuites, a viscometer is required in which the actual shear stress and shear rate can be calculated and with which operation over a range of shear rates is possible.

Two viscometers that provide a well defined shear field and are therefore suitable for this purpose are the rotational viscometer and the pipeline viscometer. Both of these types of viscometer have proved useful for rheological studies of pan products.

4.0 ROTATIONAL VISCOMETER

With the rotational viscometer, a controlled speed electric motor is used to apply a known rate of rotation

METHOD 24 P.2.

to the measuring element and the torque resulting from viscous drag on the element is measured. By providing the instrument with a range of rotational speeds, measurements may be made over a range of shear rates. Measuring geometries which are suitable for use in non-Newtonian fluids include the coaxial cylinder, the cone-and-plate, and the parallel plate design. The shear stress can be calculated from the measured torque and the dimensions of the rotating element and is independent of whether the sample is a Newtonian or non-Newtonian fluid. The equation for the shear rate is a function of the geometry of the measuring elements and does depend on the nature of the flow behaviour of the fluid. From the measurement of these quantities the consistency and the flow index for a Power Law fluid can be determined. Several commercial designs of rotational viscometers are available from instrument suppliers.

5.0 PIPELINE VISCOMETER

With the efflux type pipeline viscometer, the sample is maintained in a reservoir and gas pressure is applied to drive the material through a horizontal or vertical tube of accurately known dimensions. A schematic arrangement of a pipeline viscometer is shown in Figure 2.

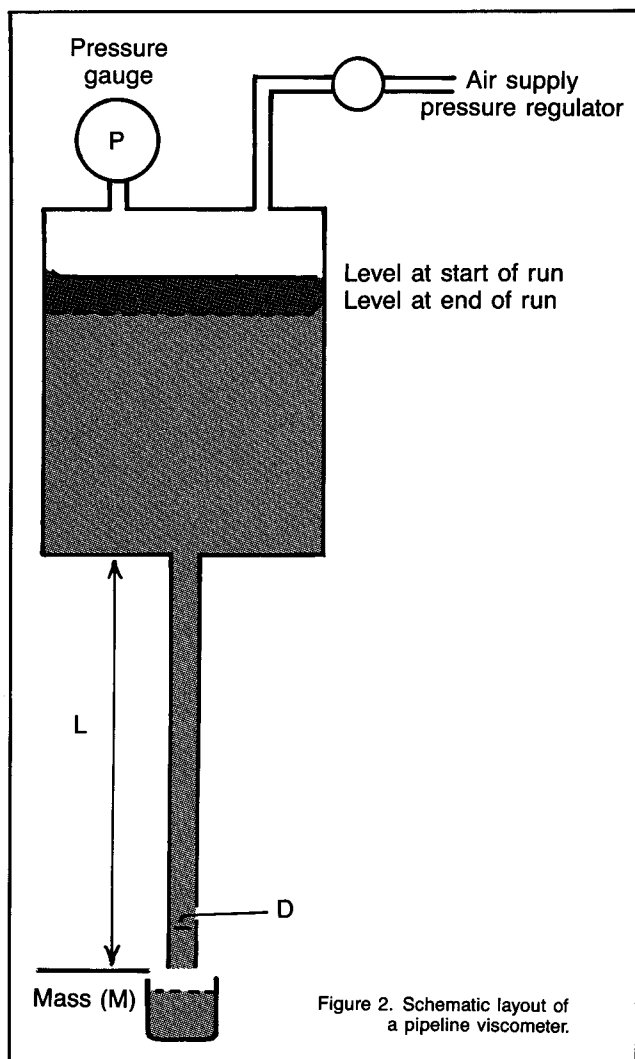


Figure 2. Schematic layout of a pipeline viscometer.

A series of measurements of the efflux rate of the material from the tube at different applied pressures allows measurements of the wall shear stress to be made over a range of shear rates. Usually between five and seven measurements in the shear rate range of interest are obtained on a single sample.

The true shear stress and shear rate at the wall of the tube can be calculated from the total pressure differential, the tube dimensions, and the average velocity of the fluid through the tube and from these the consistency and flow index for a "Power Law" fluid can be determined.

Systematic errors can be introduced through end and wall effects unless care is taken with the design of the viscometer. An instrument of a standardized design with specified dimensions to minimize such errors is required. For this, the Sugar Research Institute and BSES have jointly developed a 'standard' design of pipeline viscometer recommended for use in Australian sugar mills.

Notes on the construction, test procedure and data analysis are available from either organization.

6.0 VISCOSITY OF MOLASSES AND MASSECUTE

The rotating cylinder method has been adopted by ICUMSA as an Official Method for determining the viscosity of molasses. Various types of rotating cylinder viscometers are available and among these the coaxial cylinder viscometers are well suited. These are particularly useful for determinations when only small volumes of samples are available, e.g. as obtained from pressure filtration of massecuites. The pipeline viscometer has also been adopted by ICUMSA as an alternative technique for measuring the viscosity of molasses.

When viscosity determinations on molasses are undertaken, pertinent variables such as temperature, concentration, purity and presence of entrapped gases should be recorded.

The measurement of the flow behaviour of massecuite is more complex than that of molasses due to the effects of the solid phase. The pipeline method is being assessed by ICUMSA as a suitable technique for determining the rheological properties of massecuites. At present it is regarded as the most appropriate method for a detailed assessment of massecuite viscosities. Unfortunately, the technique is time consuming (e.g. one to two hours per sample) and for greater convenience and speed of measurement the rotational cylinder viscometer is useful for comparative assessments. This version of the rotational viscometer comprises a cylinder of small diameter rotating in a large volume of sample, e.g. 600 mL low form beaker. Besides the properties of the mother molasses, the massecuite viscosity is influenced by the crystal content and to a lesser extent by the mean size and spread of the crystal population.

METHOD 25

FILTRABILITY —

DETERMINATION IN PRODUCTS OTHER THAN RAW SUGAR

1.0 SCOPE AND FIELD OF APPLICATION

This method can be used to provide comparisons between similar factory products and can be used to assist in factory process control and trouble-shooting.

2.0 DEFINITION

The filtrability of a product is the ratio of the filtration rate of a solution of the product, adjusted by the addition of refined sugar to approximately raw sugar purity, to that of a solution of pure sugar of the same concentration, filtered under identical conditions.

3.0 PRINCIPLE OF THE METHOD

The method is very similar to that used for determination of the filtrability of raw sugars.

A solution of the product is made up to 60.0 ± 0.5 °bx and a purity similar to that of raw sugars by the addition of water and refined sugar. Celite 505 and buffer are added to this solution to bring the pH to between 8.5 and 9.1. The solution is filtered under constant pressure. Filtrate is initially run to waste for two minutes, to allow a uniform Celite filter cake to form. The filtrate is then collected over a precisely timed five minute period, weighed and expressed as a percentage of the weight of pure sucrose solution which would be collected under identical conditions of density, pH, temperature and pressure.

The per cent filtrability is defined at 20 ± 1 °C. At temperatures above 20°C, a lower filtrability is obtained and at temperatures below 20°C the filtrability is higher. If a test is done at a temperature other than 20 ± 1 °C, the result can be expressed only as "filtrability at t°C".

4.0 APPARATUS

Ordinary laboratory glassware and apparatus as for Method No. 36, "Filtrability of Raw Sugar by the Celite Method".

5.0 REAGENTS

5.1 Standard Filter Aid — Celite 505 standardised at CSR Central Laboratory.

5.2 Filtrability Buffer

5.2.1 Prepare 1L of 50% (m/m) solution of glycerol in water.

5.2.2 Dissolve 15.0 ± 0.1 g of calcium acetate in 200-300 mL of 50% glycerol solution in a 500 mL beaker.

5.2.3 Dissolve 400.0 ± 0.5 g of diethanolamine in 200-300 mL of 50% glycerol solution in a 500 mL beaker.

5.2.4 Transfer the calcium acetate solution and the diethanolamine solution to a 1L volumetric flask, rinsing both beakers with 50% glycerol solution.

5.2.5 Make up to 1L with 50% glycerol solution, mix well and allow to stand overnight. Add a teaspoon of Celite 505 and filter in the pressure filter

through a No. 54 filter paper. The filtered solution can be stored for 6 months.

5.2.6 The pH of the test solution should be greater than 10.5.

Safety

Diethanolamine is a mild eye and skin irritant and wearing safety glasses and rubber gloves is recommended.

5.3 Sucrose or high purity refined sugar.

5.4 Filter paper — Whatman Grade 54, 5.5 cm diameter. The filter papers are selected from the one 'making' and should have a mean Gurley time of 7.6 seconds (air flow measured by Gurley Densometer). Suitable selected Whatman 54 filter papers can be obtained from CSR Limited, Central Laboratory.

6.0 STANDARDISATION OF CELITE 505 FILTER AID

The Celite 505 to be used for the test is standardised at Central Laboratory. With each batch of standardised Celite 505, Central Laboratory issues a reference Table (No. XVII). The Table gives the weights of pure syrup, filtered between 2 and 7 minutes after application of pressure at various temperatures, under the standard condition of the test. Each Table is specific to a particular batch of Celite 505.

7.0 PROCEDURE

7.1 For samples of clarified juice, syrup and molasses, measure the brix of the solution (b).

7.2 Weigh out the appropriate quantity of sample as shown in Table I.

7.3 Weigh out and add the amounts of refined sugar and water obtained from Table I.

TABLE I
QUANTITIES REQUIRED FOR CELITE
FILTRABILITY OF FACTORY PRODUCTS

Product	Sample Mass (g)	Refined Sugar (g)	Water (g)	Celite (g)	Buffer (mL)
ESJ	200.0	5(60-b)	0	0.03 (100-b)c	3.2
Syrup	$\frac{2440}{b}$	150.0	$116.3 \frac{24.4(100-b)}{b}$	1.744c	1.5
A & B Masseccutes	50.0	250.0	193.4	2.95 c	3.5
A & B Molasses	$\frac{1300}{b}$	350.0	$242-13 \left(\frac{100-b}{b} \right)$	3.63 c	2.8
Magma	50.0	250.0	186.7	2.92 c	2.5
C Sugar	50.0	250.0	198	3.0 c	2.2

NOTE: b = brix of sample

c = addition rate of Celite (% on solids)

7.4 From Table XVII, obtain the addition rate of the batch of Celite 505 in current use (C% on solids). Weigh out and add the amount of Celite shown in Table I.

7.5 Dissolve the sugar without heating, using an electric stirrer at 900 ± 100 r/min.

METHOD 25 P.2.

7.6 With the stirrer still going, add the appropriate volume of special buffer to the solution down the side of the beaker (see Table I). Mix for 2 minutes \pm 10 seconds after addition of all buffer. Switch the stirrer off and remove from the solution. Cover the beaker and allow to stand for 15 minutes \pm 30 seconds.

NOTE: The buffer used for these products is *not* the same as used for raw sugars (see 5.2).

7.7 Filter the sample in the same manner as for raw sugars.

NOTE: For a guide to the precautions and procedure in the above steps refer to Method No. 36 "Filtrability — Determination in Raw Sugar by the Celite Method".

8.0 CALCULATIONS

8.1 Calculate the per cent filtrability of the sample as for raw sugars.

9.0 PRECISION

No information is available on the precision of the method. The results cannot be used to predict the filtrabilities of raw sugars made from the products or of other factory products. However, the information can give a useful guide to filtrability levels.

METHOD 26

ASH — DETERMINATION IN SUGAR PRODUCTS BY THE SINGLE SULPHATION METHOD

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian Sugar Industry. Ash is a factor in the net titre formula, on which Australian raw sugar payments are based and the determination of ash is used by sugar mills for control purposes.

A single sulphation method gives a rapid measure of ash which is reproducible.

2.0 DEFINITION

Ash is inorganic matter. It is composed mainly of chlorides, sulphates, nitrates, carbonates and phosphates of K, Ca, Mg, Na, Fe and Al, as well as salts of numerous organic acids. Silica is also usually present.

3.0 PRINCIPLE OF METHOD

This is a gravimetric method. The sample is weighed into a platinum crucible, evaporated if necessary, and moistened by dropwise addition of concentrated sulphuric acid. Charring commences at this stage, and is accelerated by slow heating. The charred sample is ignited in a muffle furnace at a temperature of $525 \pm 25^\circ\text{C}$.

The sulphation of the sugar prior to ignition assists in obtaining an ash that is free of carbon at the low ignition temperature used. After sulphation, the cations are recovered as sulphates together with phosphates and silicates.

The sulphates are less volatile and less hygroscopic. Because sulphated ash is heavier than the ash obtained by direct incineration, a 10% deduction is applied to sulphated ash determinations.

4.0 APPARATUS

Ordinary laboratory apparatus and

- 4.1 **Muffle furnace** capable of being controlled at $525 \pm 25^\circ\text{C}$ and having provision for a flow of air through the heated cavity.
- 4.2 **Platinum crucibles** of a suitable size to take the required quantity of sample in a layer about 10 mm thick.
- 4.3 **Automatic pipette** or burette for concentrated sulphuric acid.
- 4.4 **Aluminium or steel block**, approx. 150 mm \times 150 mm \times 25 mm.
- 4.5 **Tongs**, stainless steel, about 400 mm long with insulated handles.
- 4.6 **Wire**, platinum or stainless steel, approx. 100 mm long.
- 4.7 **Tweezers**, rubber tipped.
- 4.8 **Stainless steel tray** (approx. 150 mm \times 150 mm \times 40 mm) containing a ceramic tile and fitted with a lid.
- 4.9 **Soft brush**, approximately 5 mm diameter.

5.0 REAGENTS

- 5.1 **Concentrated Sulphuric Acid** $\rho_{20} = 1.84$ g/ml. This must be free of mineral impurities. Before use,

each bottle of acid should be checked as follows:

With a measuring cylinder, transfer 25.0 ± 0.5 mL of the acid into a clean tared platinum crucible. Evaporate slowly to avoid spitting and then ignite the residue in the muffle furnace at $525 \pm 25^\circ\text{C}$. The acid is suitable if the residue, on ignition, is less than 0.0025 g, i.e. 0.0001 g/mL.

Safety

Concentrated sulphuric acid is very corrosive. Safety glasses and gloves must be used when handling the acid. It must not be pipetted by mouth.

If in contact with skin, remove all contaminated clothing etc. and wash affected area with copious quantities of water.

If ingested, administer large quantities of aluminium hydroxide gel or milk of magnesia (magnesium hydroxide). Do not induce vomiting.

6.0 TREATMENT OF SAMPLE

- 6.1 **Juice.** Allow juice samples to stand for 1 hour to allow suspended matter to settle and then decant.

7.0 PROCEDURE

- 7.1 Switch on the muffle furnace at least an hour before use and adjust the temperature to $525 \pm 25^\circ\text{C}$.
- 7.2 Weigh into a clean dry platinum crucible $5.0 \pm .05$ g raw sugar. Record the mass of the crucible plus sample to 0.0001 g (m_1).
- 7.3 **For juice samples only.** Evaporate the sample in the crucible, to dryness in an oven at about 100°C , and allow to cool before adding sulphuric acid.
- 7.4 **For all samples.** Moisten the sugar in the crucible with 2.0 ± 0.1 mL of concentrated sulphuric acid (5.1), adding slowly from a burette or automatic pipette. Distribute the acid evenly over the sample. This operation, and the subsequent charring and incineration steps, should be carried out in a fume cupboard.
- 7.5 Place the crucible on a pipeclay triangle on a tripod, and heat slowly with a bunsen until charring is complete, i.e. no further swelling occurs. To prevent spillage during charring, tilt the crucible with tongs. Avoid impinging the reducing section of the flame onto the platinum crucible.

NOTE: Steps 7.4 and 7.5 should be done behind a perspex safety shield and the analyst must wear eye protection, e.g. safety glasses. If gas burners are not available, adequate charring may be achieved by burning off at the mouth of the muffle furnace (or on a hot plate). Care should be taken to do this slowly to avoid spillage, as charring is sometimes violent.

- 7.6 Transfer the crucible and its contents to the muffle furnace using long tongs and incinerate for 75-90 minutes, or until all the carbon has been burnt. The temperature of the muffle should be $525 \pm 25^\circ\text{C}$. If the temperature is above 550°C , the ash may fuse, and

METHOD 26 P.2.

if below 500 °C the time required for complete incineration is too long. The final ash should have an even white appearance with no dark areas of unburnt carbon.

- 7.7 Remove the crucible from the muffle and check for unburnt carbon by breaking up the ash residue with a platinum or stainless steel wire. If particles of carbon are present, replace the crucible in the muffle until completely incinerated.

Allow the platinum crucible to cool on a steel or aluminium block to room temperature, then place on a ceramic tile in a covered, stainless steel tray for fifteen minutes. Covering of the crucible is important to prevent loss of ash caused by draughts.

- 7.8 Using rubber tipped tweezers, transfer the crucible to the balance and weigh to 0.0001 g and record (m_2). Brush out the ash residue, weigh to 0.0001 g and record (m_3).

8.0 CALCULATIONS

Calculate the % ash in sugar

$$\begin{aligned} \% \text{ ash} &= \frac{9}{10} \times \frac{\text{mass of ash residue}}{\text{mass of sample taken}} \times \frac{100}{1} \\ &= 90 \times \frac{(m_2 - m_3)}{(m_1 - m_3)} \end{aligned}$$

where: m_1 = wt. of crucible + sample (7.2)

m_2 = wt. of crucible + ash (7.8)

m_3 = wt. of empty crucible (7.8)

Report result as percent ash to 0.01 for sugars and juices and to 0.10 for mill products.

9.0 PRECISION

Sample	Concentration Range % Ash (m/m)	95% Repeatability r	95% Reproducibility R
Refined Sugar	0.00 — 0.02	0.01	0.02
Raw Sugar	0.20 — 0.60	0.03	0.06
1st Expressed Juice	0.3 — 0.6	0.30	0.50
Molasses (wet basis)	9 — 15	0.80	1.60

10.0 REFERENCES

- 10.1 Browne, C.A. and Zerban, F.W.; Physical and Chemical Methods of Sugar Analysis; Ed. 3; Wiley (1948), p. 1021. Refer: Scheibler, C.; Rubenzuckerind; Vol. 14 (1864), p. 188.
- 10.2 Jamison, U.S. and Withrow, J.R.; Industrial and Engineering Chemistry; Vol. 15 (1923), p. 386.
- 10.3 Browne, C.A. and Gamble, C.A.; Facts About Sugar; Vol. 17 (1923), p. 552.
- 10.4 De Whalley, H.C.S.; ICUMSA Methods of Sugar Analysis; Elsevier (1964), p. 37.
- 10.5 Spencer, E.F. and Meade, G.P.; Cane Sugar Handbook; Ed. 9; Wiley (1963), p. 489.
- 10.6 Browne, C.A. and Zerban, F.W.; Physical and Chemical Methods of Sugar Analysis; Ed. 3; Wiley (1948), p. 1189.

METHOD 27

ASH — DETERMINATION IN SUGAR PRODUCTS BY THE DOUBLE SULPHATION METHOD

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry. It is based on the official ICUMSA method, except that the second incineration is done at 550°C instead of 650°. The method is applicable to all sugar products.

The determination of ash is used by sugar mills for process control purposes.

2.0 PRINCIPLE OF METHOD

This is a gravimetric method. Sulphuric acid is added to the test portion in a platinum crucible which is then heated gently to completely char the sample. The charred sample is incinerated in a furnace at 550°C until all organic matter is destroyed, cooled, further sulphuric acid added, followed by a second incineration at 550°. Ash is calculated as the percent loss of mass after the two incinerations, (without any deduction for the sulphation).

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

3.1 **Muffle Furnace** capable of being controlled at 550°C and having provision for a flow of air through the heated cavity.

3.2 **Platinum crucible** of a suitable size to take the required quantity of sample in a layer about 10 mm thick.

3.3 **Automatic pipette** or burette for sulphuric acid (4.1).

4.0 REAGENTS

4.1 **Sulphuric acid** $\rho_{20} = 1.84\text{g/mL}$.

Safety

Concentrated sulphuric acid is very corrosive. Safety glasses and gloves must be used when handling the acid. It must not be pipetted by mouth. If in contact with skin, remove all contaminated clothing etc. and wash affected area with copious quantities of water. If ingested, administer large quantities of aluminium hydroxide gel or milk of magnesia (magnesium hydroxide). Do not induce vomiting.

5.0 TREATMENT OF SAMPLE

5.1 **Juice.** Stand samples for 1 hour to allow suspended matter to settle and then decant.

6.0 PROCEDURE

6.1 Weigh a clean dry platinum crucible to 0.0001 g and record the mass (m_1). Weigh the appropriate amount of sample into the crucible.

Cane Juice : 30.0 ± 0.5 g
Raw Sugar : 5.0 ± 0.5 g
Liquor : 5.0 ± 0.5 g
Syrup and Molasses : 3.0 ± 0.2 g

Record the mass of the crucible plus sample to 0.0001 g (m_2).

6.2 **For juice samples only.** Evaporate the sample in the crucible, to near dryness in an oven at about 95°C, before adding sulphuric acid.

6.3 **For all samples.** Add 1.0 mL of sulphuric acid, dropwise over the total surface of the sample. This operation, and the subsequent charring and incineration steps should be carried out in a fume cupboard. The analyst should wear safety glasses.

6.4 Heat the crucible slowly on a hot plate or over a bunsen burner until the test portion is completely charred.

6.5 Transfer the crucible and its contents to the muffle furnace and incinerate at 550°C until no trace of unburnt carbon is visible. This usually takes 2-3 hours.

6.6 Remove the crucible from the muffle and allow to cool.

6.7 Add 1.0 mL of sulphuric acid to wet the residue, allow this to evaporate at the mouth of the muffle furnace and incinerate at 550°C for 2 hours (or to constant weight).

6.8 Remove the platinum crucible from the muffle and place in a desiccator and allow to cool to room temperature.

6.9 Weigh the crucible plus ash to 0.0001 g and record the mass (m_3).

7.0 CALCULATIONS

Calculate the % ash in sugar

$$\% \text{ ash} = \frac{\text{mass of ash residue}}{\text{mass of sample taken}} \times \frac{100}{1}$$

$$\frac{(m_3 - m_1)}{(m_2 - m_1)} \times \frac{100}{1}$$

where: m_1 = mass of empty crucible (6.1)

m_2 = mass of crucible + sample (6.1)

m_3 = mass of crucible + ash (6.9)

Report result as per cent ash to 0.01% for sugars and juices and to 0.1% for mill syrups and molasses.

8.0 PRECISION

Sample	Concentration	95%	95%
	Range % Ash	Repeatability r	Reproducibility R
Raw Sugar	0.20-0.60	0.03	0.06
1st Expressed Juice	0.3-0.6	0.30	0.50
Molasses (wet basis)	9-15	0.80	1.60

9.0 REFERENCES

ICUMSA, Proceedings of Eighteenth Session, 1982; Subject 16.



METHOD 28

DEXTRAN — DETERMINATION IN RAW SUGAR AND SUGAR PRODUCTS BY THE ALCOHOL HAZE METHOD

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian Sugar Industry. Dextran, for the purpose of this analysis, is a high molecular weight and predominately straight chained glucose polymer with a majority of 1-6 glucosidic linkages formed by the action of certain species of bacteria on sucrose during cane and juice storage.

High dextran concentrations in juice or raw sugar cause severe processing problems in mills and refineries. This method is applicable to cane juice, raw sugar and process materials of 80 purity or greater.

2.0 PRINCIPLE OF METHOD

The test sample is dissolved in water. Soluble starch is destroyed by incubation with a suitable enzyme. Inorganic salts are removed by treatment with mixed ion-exchange resins. Protein is removed by precipitation with trichloroacetic acid followed by filtration with acid washed Supercel.

The dextran haze is produced by diluting an aliquot of the treated, filtered solution, to twice the aliquot volume by the addition of ethanol. The turbidity of the dextran haze is measured by reading in a spectrophotometer at a wavelength of 720 nm.

The method is standardised against Pharmacia Dextran T500.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

3.1 Spectrophotometer

suitable for the measurement of absorbance at 720 nm with matched 2 cm cells. The spectrophotometer should comply with the following specifications:

- spectral band pass 10 nm or less
- wavelength reproducibility ± 0.5 nm
- absorbance reproducibility ± 0.003 at 1.0 abs

3.2 Water bath

capable of operating at 85-95°C.

3.3 Flask shaker.

3.4 Balance,

capable of being read to 0.0001 g.

3.5 Stopwatch.

3.6 Volumetric flasks,

Class A (AS 2164) — 100 mL, 25 mL.

3.7 Bulb pipettes,

Class A (AS 2166) — 1 mL, 2 mL, 5 mL, 10 mL.

3.8 Graduated straight pipettes,

Class A (AS 2167) — 2 mL, 5 mL, 10 mL

3.9 Measuring cylinder,

100 mL with plastic stopper.

3.10 Funnel

fitted with 150 μ m mesh screen to fit into measuring cylinder.

3.11 Automatic dispenser

or safety pipette, 10 mL.

3.12 Buchner funnel,

5.5 cm and 250 mL Buchner flask.

3.13 Burette,

50 mL capacity or suitable automatic burette.

4.0 REAGENTS

During the analysis, unless otherwise stated, use only reagents of analytical grade and only distilled water.

4.1 Standard dextran,

1 mg/mL solution. Use an approved batch of Pharmacia Dextran T500. Determine its moisture content, in duplicate, correct to 3 decimal places by drying approximately 2 g of the solid at 105°C in an oven for three hours. Record the weights to 0.0001 g. Individual determinations must be within 5% of the mean of the measured value.

Weigh a quantity of the undried dextran that contains 0.1000 ± 0.0001 g of anhydrous dextran, i.e.

$$\text{weigh out } \frac{0.1000 \times 100}{(100 - \% \text{ water in dextran})} \text{ g}$$

of undried dextran into a 100 mL beaker.

Dissolve the dextran by adding 1-2 mL water to form a slurry. Allow the particles to become uniformly hydrated by standing and occasionally stirring. Add water gradually to avoid having an excess present while gel masses remain. When about 25 mL has been added and no gel is present, wash the slurry into a 100 mL flask with water to a volume of about 80 mL. Place the flask in a boiling water bath for 30 min. Cool to room temperature and make to the mark (100 mL) with water. Prepare standard dextran solution daily, do not store overnight.

4.2 Trichloroacetic acid (TCA),

100 g/L solution. Dissolve 10.0 ± 0.1 g of trichloroacetic acid in distilled water and dilute to 100 mL. This reagent will keep for two weeks, stored under refrigeration in a dark brown bottle.

Safety

Trichloroacetic acid at this concentration attacks protein and should not be allowed to come in contact with any part of the body, particularly the tongue. Under no circumstances pipette TCA by mouth. Do not store TCA in plastics. If in contact with skin, remove all contaminated clothing etc. and wash affected area with copious quantities of water. Safety glasses and gloves must be worn while transferring the solid chemical, dissolving it or pipetting the solution. Label all containers and solutions of trichloroacetic acid with "CAUSES SEVERE BURNS" and "SERIOUS RISK OF POISONING BY INHALATION, SWALLOWING OR SKIN CONTACT".

If ingested, administer large quantities of aluminium hydroxide gel or milk of magnesia (magnesium hydroxide). Do not induce vomiting.

4.3 Denatured absolute alcohol.

Store in an airtight container.

Safety

Alcohol is highly flammable. The methanol used for denaturing adds substantially to its toxicity. Avoid breathing its vapours. It reacts with varying degrees of violence with a wide range of oxidants. DAA's flash point is in the range 10-12°C.

Alcohol fires may be extinguished with water spray, dry powder, carbon dioxide or vaporising liquids. Label

METHOD 28 P.2.

- all containers of DAA "HIGHLY FLAMMABLE".
- 4.4 **Dextran and starch-free sucrose** (standard sugar). Use only pure refined sugar certified as being suitable for dextran analysis. Check the haze which develops when 12.0 mL of a 200 g/L solution of this standard sugar (4.6), plus 0.5 mL of TCA reagent (4.2), are diluted to 25 mL with denatured absolute alcohol (4.3), according to the method procedure. Absorbance should not exceed 0.003 (in a 2 cm cell) at 720 nm. Suitable certified refined sugar is supplied by CSR Central Laboratory.
- 4.5 **Sucrose, 500 g/L solution.** Dissolve 50.0 ± 0.1 g of standard sugar (4.4) in distilled water and dilute to 100 mL. This solution should be made up freshly as required.
- 4.6 **Sucrose, 200 g/L solution.** Dissolve 20.0 ± 0.5 g of standard sugar (4.4) in distilled water and dilute to 100 mL. This solution should be made up freshly as required.
- 4.7 **Acid-washed Supercel.** Supercel (50 ± 5 g) is added to 1 L distilled water; concentrated hydrochloric acid (50 ± 5 mL) is added and the mixture stirred for 5 mins. After filtration the Supercel is washed free from acid with distilled water, testing the washings with litmus paper. The washed Supercel is dried for 6 hours at 96-100°C and stored in a closed container. Suitable acid washed Supercel can be obtained from CSR Central Laboratory.
- 4.8 **Starch removing enzyme.** Use a heat stable alpha-amylase such as Termamyl 120L (Novo Industri Denmark) or Takalite L340 (Miles Laboratories USA). These enzymes will keep for 12 months, if stored to manufacturer's instructions.
- 4.9 **Ion exchange resin mixture.** The recommended resin is SAC/SBA mixed bed resin supplied by Permutit Australia Ltd., Sydney. This resin is supplied wet and should be washed by shaking with at least twice its weight of distilled water, drained dry, then washed briefly by shaking with an equal weight of acetone for no longer than two minutes, and the solvent immediately removed as before. Air dry after pressing between filter papers or oven dry at low temperature, approximately 30°C.
- 4.10 **Formaldehyde Solution, 37-41% (w/v)**

Safety

The liquid and the vapour irritate the eyes severely. Formaldehyde solution in contact with the skin has a

hardening or tanning effect and causes irritation. Severe abdominal pains with nausea and vomiting and possibly loss of consciousness follow ingestion. The vapour irritates all parts of the respiratory system. High concentration of vapour inhaled for long periods may cause laryngitis, bronchitis or bronchial pneumonia and prolonged exposure may cause conjunctivitis. If in contact with the skin for long periods, it will cause cracking of skin and ulceration, particularly around the fingernails.

The laboratory should be well ventilated to ensure that its concentration in the air is kept below 3 mg m^{-3} , the maximum allowable concentration for safe working over prolonged periods.

A 37% solution has a flash point of 50 °C. Its fire can be extinguished with water spray, dry powder, carbon dioxide or vaporising liquid. Safety glasses and gloves must be worn while handling this solution. Label all containers of formaldehyde solution "FLAMMABLE", "SERIOUS RISK OF POISONING BY INHALATION OR SWALLOWING", "CAUSES BURNS" and "IRRITATING TO SKIN, EYES AND RESPIRATORY SYSTEM".

5.0 STANDARDISATION OF METHOD

A. PREPARATION OF STANDARDS

- 5.1 **Working standards.** In eleven 25 mL, A-class volumetric flasks prepare the standard solutions shown in Table 1.
- 5.2 Into each flask pipette 0.5 ± 0.05 mL (graduated pipette) of the TCA 100 g/L solution (4.2). Do not pipette by mouth — a safety bulb must be used.
- 5.3 Add 5.00 ± 0.05 mL of 500 g/L sucrose solution (4.5) to each flask.
- 5.4 Using A-class bulb or graduated pipettes, add respectively to the first ten flasks, aliquots of the standard dextran solution (4.1) in accordance with Table 1.
- 5.5 Using a 10 mL graduated pipette add respectively to the 10 flasks aliquots of distilled water according to Table 1, to make a total volume of 12.5 mL.
- 5.6 Make flask number 11 to the mark using distilled water and mix by shaking. This is the blank solution.
- 5.7 Add denatured absolute alcohol (4.3) slowly from a burette to the 25 mL mark of the first flask while gently swirling the flask. The time for the alcohol addition should be between 30 and 60 secs. Mix the contents of the flask by inverting gently three times. Start the

TABLE 1
STANDARD DEXTRAN SOLUTIONS

Flask No.	1	2	3	4	5	6	7	8	9	10	11
100 g/L TCA solution (4.2) (mL)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
500 g/L sucrose solution (4.5) (mL)	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Standard Dextran solution (4.1) (mL)	0	0.2	0.4	0.6	1.0	1.5	2.0	3.0	5.0	7.0	0
Distilled Water (mL)	7.0	6.8	6.6	6.4	6.0	5.5	5.0	4.0	2.0	0	19.5
Dextran Content (mg/L)	0	8	16	24	40	60	80	120	200	280	BLANK

stopwatch immediately after the mixing step is complete.

B. MEASUREMENT OF ABSORBANCE

- 5.8 Determine the cell corrections of a pair of matched 2 cm cells using distilled water.
- 5.9 Approximately 15 minutes after the completion of the mixing step (5.7), rinse a 2 cm cell twice with the blank solution, and then fill the cell.
- 5.10 In a similar way, rinse and fill another 2 cm cell with one of the standard solutions (5.5). Clean the optical faces of the cells with a tissue.
- 5.11 At 20 minutes \pm 10 seconds after completion of the mixing step (5.7) read and record to 0.001 the absorbance of the test solution against that of the blank solution at 720 nm.
- 5.12 Repeat steps 5.7, 5.10, and 5.11 for each concentration of dextran involved in the standardisation. It is not necessary to refill the blank solution cell (5.9) for each determination.

C. PREPARATION OF CALIBRATION GRAPH

- 5.13 Apply any cell corrections (5.8) to the absorbance of each standard solution.
- 5.14 Plot 'dextran mg/L' against 'absorbance'. Points representing 60 to 280 mg/L dextran should be approximately on a straight line. The plot of low dextran concentrations (0 to 60 mg/L) is however non-linear and this section of the calibration graph should appear as a gradual curve.
- 5.15 The graph used by any laboratory for routine analysis should be constructed using the mean of independent standardisations by at least two analysts. Individual points must be within 5% of the mean line of best fit.
- NOTE: As a guide to performance of the method, the 80 mg/L dextran standard measured at 720 nm in a 2 cm cell should give an absorbance of about 0.210.

6.0 TREATMENT OF SAMPLE — Cane Juice

Raw cane juices require special treatment. Unless the juice is to be analysed within one half hour of collection, preservation is necessary. If the juice is to be stored for longer than half an hour prior to analysis, the following preservation step should be carried out immediately after collection of the sample.

Add 0.5 mL formaldehyde solution (4.10) to each 100 mL of juice, stir and store in a closed vessel in a refrigerator (do not freeze the juice). Juices preserved by this method will keep for 24 hours. If refrigerated storage is not available, the juice, preserved as above, should not be stored for longer than 8 hours. Use this method of storage only as a last resort.

7.0 PROCEDURE

A. PREPARATION OF TEST SOLUTIONS —

Raw Sugar and Process Materials

- 7.1 (a) Weigh out 40.0 ± 0.1 g of raw sugar, transfer to a 100 mL flask, dissolve and make to the mark with distilled water, stopper and mix well.
- (b) Prepare 100 mL of approximately 40°Bx solution of process material and measure the refractive index.
- 7.2 Using a 50 mL measuring cylinder, transfer 50 mL of the solution into a 100 mL conical flask. Add 0.1 mL

of the enzyme (4.8). Mix the contents well and stopper the flask.

- 7.3 Place the flask in a water bath at $90 \pm 5^\circ\text{C}$ for 15 ± 2 mins. Agitate every 5 minutes.
- 7.4 At the end of 15 minutes cool to room temperature and add mixed ion exchange resins; 1 g for raw sugars and 10 g for process materials.
- 7.5 Shake the flask for 30 ± 2 minutes on a flask shaker.
- 7.6 Pour the mixture through a funnel containing a $150 \mu\text{m}$ gauze into a 100 mL measuring cylinder which can be stoppered.
- 7.7 With distilled water, rinse the flask thoroughly, through the funnel into the measuring cylinder.
- 7.8 Dilute the sample and washings to 100 mL, then add 10 ± 1 mL TCA reagent (4.2). Do not pipette by mouth. A safety bulb must be used.
- 7.9 Add two heaped teaspoonfuls (about 6-8 g) of acid-washed Supercel (4.7). Stopper and mix well. Proceed according to steps 7.15 to 7.23.

B. PREPARATION OF TEST SOLUTION —

Cane Juice

- 7.10 If the juice has been refrigerated, bring it rapidly to room temperature with running cold water. Hot water must not be used for this purpose because some solubilisation of starch may occur.
- 7.11 Measure and record, corrected to 20°C , the brix of the juice to 0.1°.
- 7.12 Shake 60 ± 1 mL juice with 2.0 ± 0.2 g mixed resin (4.9) for 10 ± 1 minutes and filter through $150 \mu\text{m}$ gauze. Transfer 50 ± 1 mL (cylinder) of juice to a 100 mL beaker.
- 7.13 Add 10.0 ± 0.1 mL TCA reagent (4.2). Do not pipette by mouth. A safety bulb must be used.
- 7.14 Add two heaped teaspoons of acid-washed Supercel (4.7), stopper and mix well. Proceed according to steps 7.15-7.23.

C. FILTRATION AND PRODUCTION OF HAZE

- 7.15 Filter mixture (7.9 or 7.14) through a 5.5 cm Buchner funnel (Whatman No. 5 filter paper), using the first 10 to 15 mL of filtrate to rinse the funnel and flask.
- 7.16 Using a 25 mL graduated pipette, add 12.5 mL of the filtrate to each of two clean, *dry* 25 mL volumetric flasks.
- 7.17 Add denatured absolute alcohol (4.3) slowly from a burette to the 25 mL mark of one flask, gently swirling the flask. The time for the alcohol addition should be between 30 and 60 secs. Mix the contents of the flask by inverting gently three times. Start the stopwatch immediately the mixing step is complete.

NOTE: (i) Alcohol must be added within 20 minutes of commencement of step 7.8 (sugars etc.) or 7.14 (juices).

(ii) Vigorous shaking of the flask may cause coagulation of the dextran haze, and must be avoided.

- 7.18 To the other flask add distilled water to the 25 mL mark and mix. This is the test blank.

D. MEASUREMENT OF ABSORBANCE

- 7.19 Determine the cell corrections of a pair of matched 2 cm cells using distilled water.
- 7.20 Approximately 15 minutes after the completion of the mixing step (7.17), rinse a 2 cm cell twice with the blank solution, and then fill the cell.

METHOD 28 P.4.

7.21 In a similar way, rinse and fill another 2 cm cell with the test solution. Clean the optical faces of the cells with a tissue.

7.22 At 20 minutes \pm 10 seconds after the completion of the mixing step for the test solution (7.17), read and record to 0.001 the absorbance of the test solution against that of the blank solution at 720 nm.

NOTE: If the dextran haze reading is beyond the calibration limits of the standard graph, smaller aliquots of the filtrate (7.16) should be taken. Dilute the aliquot in each 25 mL flask to a total volume of 12.5 mL by the addition of a 200 g/L sucrose solution (4.6). Proceed as from step 7.16.

7.23 Immediately after reading, visually inspect the contents of the test solution cell to check for flocculation. If the haze has flocculated, repeat the analysis.

8.0 CALCULATIONS

8.1 Apply any cell correction (7.19) to the observed absorbance.

8.2 Obtain from the standardisation graph "mg/L dextran in the test solution" corresponding to the corrected observed absorbance.

8.3 For process materials use Table XII or XV to determine the concentration in g/mL of the sugar solution from the appropriate brix measurement (7.1).

Table XII gives the degrees brix, density and concentration in g/mL of sucrose solutions at 20°C, cor-

responding to refractive index readings.

Table XV gives the density and concentration in g/mL of sucrose solutions at 20°C, corresponding to degrees brix.

8.4 Calculate dextran in sample mg/kg.

$$\text{Dextran (mg/kg)} = \frac{\text{mg/L dextran} \times y^* \times 0.5}{\text{conc. (g/mL)} \times \text{aliquot}}$$

y^* = 110 for raw sugars and process materials.

y^* = 60 for raw cane juices

aliquot = volume pipetted in step 7.16 (usually 12.5 mL)

8.5 Record the results as mg/kg dextran in the sample to the nearest 1 mg/kg.

Record results below 50 mg dextran in the sample as "< 50 mg/kg".

9.0 PRECISION

Sample	Concentration Range mg/kg Dextran	95% Repeatability r	95% Reproducibility R
Raw Sugar	0-1,000	20	Not determined
Cane Juice	0-12,000	Not determined	Not determined
Process Materials	0-1,000	Not determined	Not determined

12.0 REFERENCES

Nicholson R.I., and Horsley M.; J. Ag. and Food Chem.; Vol. 7, No. 9 (1959), p. 64.

METHOD 29

PHENOLIC ACIDS —

DETERMINATION IN RAW SUGAR, SYRUPS AND CANE JUICE

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry.

Phenolic and amino acids are the two major groups of colour precursors in cane sugar. Phenolic acids form coloured complexes with iron and copper, and some are readily oxidised to form coloured polymers. The concentration of phenolic acids therefore relates to the tendency of the sugar to darken in storage.

The phenolic acids found in sugar cane are hydroxy derivatives of benzoic acid or cinnamic acid.

2.0 PRINCIPLE OF METHOD

A known quantity of the sample in water is reacted with E.D.T.A., sodium nitrite and acidified sulphanilic acid. The intensity of the yellow colour developed after 60 minutes is measured in a spectrophotometer at 420 nm.

Gallic acid is used as the standard. Gallic acid and the phenolic acids in sugar have varying sensitivity to the diazo reaction, and some flavonoids respond to it also. This analysis therefore indicates only relative levels of phenolic acids in different samples.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

- 3.1 **Volumetric flasks**, Class A, (AS2164), 500 mL, 100 mL, 50 mL.
- 3.2 **Bulb pipettes**, Class A, (AS2166), 1 mL, 2 mL, 3 mL, 4 mL, 5 mL, 10 mL.
- 3.3 **Buchner funnel**, 5.5 cm with 250 mL buchner flask.
- 3.4 **Automatic pipette**, or dispenser to deliver 10.0 ± 0.2 mL.
- 3.5 **Spectrophotometer**, with matched 2.0 cm cells.
- 3.6 **Balance** to weight 100.00 ± 0.02 g.

4.0 REAGENTS

During the analysis, unless otherwise stated, use only reagents of analytical grade and only distilled water.

- 4.1 **E.D.T.A., 0.01 mol/L Solution**. Weigh 3.72 g of E.D.T.A. (di-sodium salt) and dissolve in water in a 1L volumetric flask.
- 4.2 **Sulphuric acid, 0.5 mol/L**. Prepare from a commercial concentrated volumetric solution supplied in a sealed ampoule.
- 4.3 **Sulphanilic acid, 0.8% (w/v) solution**. Dissolve 0.800 ± 0.001 g of sulphanilic acid in about 50 mL of hot water in a 150 mL beaker; add 10 mL of 0.5 mol/L sulphuric acid (4.2). Mix to dissolve, transfer to a 100 mL volumetric flask, cool and dilute to volume, mix well by shaking.
This reagent will not keep and a fresh solution must be made up daily or when required.
- 4.4 **Sodium Nitrite, 4% (w/v) solution**. Dissolve 1.00 ± 0.01 g in 25 mL water. Prepare a fresh solution every 2 hours.

- 4.5 Gallic acid standard solution, 200 mg/L. Weigh 0.1000 ± 0.0001 g of gallic acid into a 500 mL Class A volumetric flask. Dissolve in distilled water and make to the mark. Stopper and mix well.

5.0 STANDARDISATION OF METHOD

The absorbance of the coloured complex varies with sugar concentration and it is necessary to prepare a separate calibration curve for juices which omits addition of sucrose. For juices, omit step 5.1.

- 5.1 Weigh 8.00 ± 0.02 g of sucrose into each of seven 100 mL Class A volumetric flasks. Add about 50 mL water to each flask and dissolve the sugar using a shaker.
- 5.2 Using Class A bulb pipettes, pipette 0, 1.0, 2.0, 4.0, 6.0, 8.0 and 10.0 mL of Gallic acid standard solution (4.5) respectively into the 100 mL flasks. Make each flask to the mark with water, stopper and mix well.
The solutions contain 0, 0.2, 0.4, 0.8, 1.2, 1.6 and 2.0 mg of gallic acid per 100 mL.
- 5.3 With a Class A pipette, pipette 10.0 mL of each of the gallic acid solutions (5.2) into each of two 50 mL volumetric flasks [(a) standard blank (b) standard solution].
- 5.4 With an automatic pipette add 10.0 ± 0.2 mL, E.D.T.A. solution (4.1) to each flask.
- 5.5 Dilute the standard blank to the mark with water. Mix well.
- 5.6 Pipette 4.00 ± 0.02 mL sulphanilic acid solution (4.3) and 2.0 ± 0.1 mL of sodium nitrite solution (4.4) into the standard solutions. Mix by swirling after each addition. Dilute each solution to the mark with water and mix well. Allow the solutions to stand for 60 ± 5 minutes after the addition of sodium nitrite solution.
NOTE: For a large number of samples, add the nitrite to successive samples at 3-5 minute intervals to allow time to rinse and refill the blank and standard cells between absorbance readings.
- 5.7 Read the absorbance of the standard solutions and the standard blanks in 2.0 cm cells in a spectrophotometer at 420 nm with water as the reference. Record the absorbance to 0.001 units.
Subtract the absorbance obtained for the blank from the absorbance obtained for the standard solution to give the true absorbance for each standard.
As a guide to performance of the method, the 0.8 mg gallic acid standard measured at 420 nm in 2.0 cm cells should read about 0.290 absorbance.
- 5.8 Plot mg/100 mL phenolic acids (as gallic acid) against absorbance for each standard solution. If 2 or more points lie off the line of best fit by more than 5% of the line's absorbance value at that particular gallic acid concentration, the standardisation must be repeated.
The slopes of independent calibration lines should not differ by more than 2%.

METHOD 29 P.2.

6.0 PROCEDURE

(a) Raw Sugar and Syrups

- 6.1(a) Weigh — for raw sugar 8.00 ± 0.02 g
 — for liquor the equivalent of 8.00 g of solids

Transfer to a 100 mL volumetric flask, dissolve in water and make to the mark. Mix well.

- 6.2(a) Filter the solution through a Whatman No. 5, 5.5 cm paper in a buchner funnel.

- 6.3(a) Pipette 10.00 ± 0.02 mL of the filtrate into each of two 50 mL volumetric flasks. Mark one flask as 'test solution', and the other flask as 'test blank'.

Continue at step 6.4.

(b) Juices

- 6.1(b) Take 30 mL juice, add about 3 g Supercel, mix and filter on a buchner funnel using a No. 1 or other suitable paper.

- 6.2(b) Determine the refractive index of the filtered juice and hence %bx and concentration (g sucrose/mL). With a Class A pipette, pipette 3 mL of filtered juice into a 50 mL Class A volumetric flask and make up to the mark.

- 6.3(b) Pipette $10 \text{ mL} \pm 0.02$ mL of the diluted juice into two 50 mL volumetric flasks. Mark one flask as 'test solution' and the other flask as 'test blank'.

- 6.4 With an automatic pipette add 10.0 ± 0.2 mL E.D.T.A. solution (4.1) to both flasks.

- 6.5 Dilute the 'test blank' to the mark with water. Mix well.

- 6.6 Pipette 4.00 ± 0.02 mL of sulphanilic acid solution (4.3) and 2.00 ± 0.01 mL of sodium nitrite solution (4.4), into the test solution. Mix by swirling after each addition. Dilute to the mark and mix well.

Allow the solution to stand for 60 ± 5 minutes after adding sodium nitrite solution for full colour development.

- 6.7 Read the absorbance of the test solutions and the test

blanks in a spectrophotometer set at 420 nm, in 2 cm cells with water (6.5) as a reference. Record the absorbance to 0.001 units.

Subtract the absorbance obtained for the test blank from the absorbance obtained for the test solution to give the absorbance due to phenolic acids.

7.0 CALCULATIONS

- 7.1 Read from the standard graph the mg phenolic acids in 100 mL of the sample solution.

- 7.2(a) For raw sugar or syrup samples, calculate phenolic acids, mg/kg, in the sample using:

$$\text{phenolic acids, mg/kg} = \frac{\text{mg phenolic acid/100 mL (from graph)}}{m} \times 1000$$

where: m is the sample mass (g)

$$\text{phenolic acids, mg/kg} = \frac{\text{mg phenolic acid (from graph)} \times 125}{\text{sample}} \times 1000$$

- 7.2(b) For juice samples, calculate phenolic acids, mg/kg, on sugar solids, using:

$$\text{phenolic acids, } (\mu\text{g/mL}) \text{ in the juice} = \frac{(\text{mg phenolic acid/100 mL from graph}) \times 1000}{2 \times 3}$$

$$\text{mg/kg phenolics, on sugar solids} =$$

$$\frac{\text{g phenolic acids/mL juice}}{\text{g sucrose/mL juice}}$$

8.0 PRECISION

Sample	Concentration	95%	95%
	Range (mg/kg)	Repeatability r	Reproducibility R
Raw Sugar	0-250	Not determined	Not determined

METHOD 30

POLARISATION — DETERMINATION IN RAW SUGAR USING VISUAL OR AUTOMATIC POLARIMETERS

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian Sugar Industry for the determination of the polarisation of raw sugar. This method is based on the official ICUMSA method.

Sucrose is the major component of raw sugar and the polarisation (pol) of a raw sugar solution gives a commercial estimate of the sucrose content.

This method is applicable to all raw sugars.

2.0 DEFINITIONS

2.1 **The normal sugar solution** is defined by ICUMSA as 26.0160 g of pure sucrose weighed in vacuo and dissolved in pure water at 20.00 °C to 100.00 cm³. This corresponds to a concentration of 26.0000 g of sucrose, weighed with brass weights in air, under normal conditions [101 kPa pressure, 20 °C, 50% relative humidity] in 100.00 cm³ of solution at 20.0 °C.

2.2 ICUMSA — 19th Session 1986, re-defined the “**International Sugar Scale**”. The new definition came into effect in July 1988.

In the new definition the rotation of the normal sugar solution in a 200.000 mm tube at the mercury wavelength, 546.2271 nm (in vacuo), at 20.00 °C is the **100 °Z point** of the International Sugar Scale [replacing the previously defined 100 °S point].

3.0 PRINCIPLE OF METHOD

This is a physical analysis involving three steps. A normal solution of raw sugar is prepared. The solution is clarified by adding a solution of basic lead acetate, (which coagulates colloidal impurities and removes some colourant matter), followed by filtration. The pol of the clarified solution is read in a tube of standard length in a sugar polarimeter.

4.0 APPARATUS

Ordinary laboratory apparatus and glassware and

4.1 **Balance** to weigh rapidly 26 g, plus container, to ± 0.002 g.

4.2 **Volumetric Flasks (S-flask)**. Flasks used for raw sugar polarisation should conform with the specification set out by ICUMSA in Subject 2, 1978 in respect of “ICUMSA Special Flasks”. That is a 100 mL S-flask will be used which has an accuracy of 100.00 ± 0.02 mL. Corrected flasks may be used, the flask correction being applied to the result obtained by this method.

4.3 **Sugar Polarimeters**. All sugar polarimeters must be certified for use in the Australian Sugar Industry by BSES. Sugar polarimeters should satisfy standards laid down by ICUMSA (1970) Subject 5.

Automatic sugar polarimeters should conform to the Australian Standard K157.

Circle polarimeters or quartz wedge polarimeters (saccharimeters) may be used. However, temperature

correction tables may be different for each type of polarimeter (5, 9).

4.4 **Quartz Control Plates** (Nominal value between 95 °Z and 105 °Z). The design, material, workmanship, dimensions and properties of the quartz plates shall be of the standard laid down by ICUMSA Subject 6. Quartz control plates shall be standard plates which have been certified by BSES or by the National Measurement Laboratories (CSIRO, Sydney). Quartz plates should be recertified every five years. Quartz plates should be stored in a holder adjacent to the polarimeter.

4.5 Long stem, corrosion resistant metal funnel, approximately 10 cm diameter, in stand.

4.6 Stemless glass or plastic filter funnel, 7.5 cm diameter, 100 mL capacity.

4.7 **Filter glasses** or conical beakers, 300 mL capacity.

4.8 **Polarimeter tube**, 200 mm long, and cover glasses, conforming to specifications given in ICUMSA (1974) Subject 2. Polarimeter tubes should be certified by BSES, Brisbane.

4.9 **Safety burette**, 50 mL capacity, fitted with CO₂ absorption tubes.

4.10 **Hypodermic syringe**, 5 mL capacity.

4.11 **Black clips** for “S” flasks.

4.12 **Constant temperature bath** (not essential) — capable of being controlled at the ambient room temperature ± 1 °C.

4.13 **Alcohol blower**.

4.14 **Thermometers**, length 20 cm, 10-35 °C, reading to 0.1 °C.

4.15 **Whatman No. 91 filter paper** (15 cm) or equivalent with moisture content in range 6-8% water, determined by drying it for 3 hours at 100 °C. If necessary store the papers in containers, in which the relative humidity is such that the equilibrium moisture content of the papers is in the range 6-8%.

5.0 REAGENTS

During the analysis, unless otherwise stated use only reagents of analytical grade and only distilled water.

5.1 **Basic Lead Acetate Solution (Wet Lead)**.

The reagent should meet the following specifications:

Basic lead content: 10.0 ± 0.2 g PbO/100 mL

Total lead content: 24.4 ± 0.2 g PbO/100 mL

(equivalent to a specific gravity of 1.240 ± 0.002 g/mL)

Basicity: $41.0 \pm 1.2\%$

Each batch of wet lead should be stored in airtight containers, labelled with the batch number, date and specification.

Wet lead working solution is normally dispensed by a safety burette fitted with CO₂ absorption tubes. Cartridges of “Mallcosorb” (Mallinckrodt Chemicals) should be included in all lines open to the air to remove carbon dioxide.

METHOD 30 P.2.

Safety: Lead is a cumulative poison.

Label containers: Label all containers, burettes, etc., in which basic lead acetate is stored or used: "Basic Lead Acetate" or "Wet Lead" and also "Poison".

Avoid ingesting lead: Always wash hands thoroughly before eating. Do not drink from filter glasses or laboratory taps.

Avoid absorbing lead: Dry hands with hand towel, not laboratory towels used for drying glassware. Keep lead reagent and leaded solutions clear of cuts and abrasions.

Display precautions: On laboratory notice board.

Antidote: Induce vomiting with 10% aqueous magnesium sulphate and follow with milk or egg white in cold water. Seek medical attention.

5.2 Distilled Water

Check the distilled water supply daily by a thymol test for the presence of sugar. If the sugar content exceeds 5 mg/kg, then it must not be used for polarisation analysis.

6.0 CALIBRATION OF THE POLARIMETER

There are two recommended procedures for calibrating the polarimeter and calculating the polarisation of test solutions corrected to 20°C. *Procedure 1* requires that with each batch of test solutions a calibration 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. *Procedure 2* requires that quartz plate readings be carried out periodically but not necessarily at the same time as the test solutions are read. Calibration of the polarimeter is achieved by application of a scale correction.

7.0 TREATMENT OF SAMPLE

If the sugar has not been freshly mixed, or there is reason to suspect that the sample container is not airtight, the top 1-2 cm of sample should be discarded immediately before weighing.

8.0 PROCEDURE

A. SOLUTION PREPARATION

8.1 Weigh just more than 26 grams of sample into a tared basin and reseal the sample container immediately. Quickly adjust the weight of the sample to 26.000 ± 0.002 g.

8.2 Place a 100 mL 'S' flask (4.2) under a long stem funnel. The end of the funnel stem should be in the centre of the flask bulb, and there should be a gap of not less than 5 mm between the top of the flask and the funnel apex, to allow air to escape.

Wash the sugar into the flask with distilled water, rinse the tare basin (both sides), funnel mouth and the outside of the funnel stem as it is withdrawn from the flask.

8.3 Fill the flask with distilled water to a volume not exceeding 70 mL.

Dissolve the sugar by swirling or by use of a mechanical shaker. If a mechanical shaker is used it may be necessary to fill the flask to less than 70 mL to prevent splashing or air entrainment. Hold the flask to the light and check that all sugar is dissolved.

8.4 Add distilled water, if necessary, to bring the volume to 70-80 mL.

8.5 Run about 2 mL of wet lead to waste from the burette to remove any solution that may have absorbed CO₂ from the air.

8.6 Add 1.00 ± 0.05 mL of wet lead to the flask. After the lead has been added mix thoroughly by gentle swirling.

8.7 Add distilled water, swirling continuously until the bulb of the flask is full.

8.8 *If laboratory has a cooling bath* (for air conditioned laboratories only), place the flask in the cooling bath for at least 15 minutes. The temperature of the bath should be controlled to within $\pm 1^\circ\text{C}$ of the laboratory temperature.

Remove the flask from the water bath, quickly wipe dry and place the flask on the bench.

If a cooling bath is not used, stand the flask on the bench for at least 15 minutes to allow it to attain room temperature.

NOTE: ALWAYS HANDLE THE FLASK BY THE TOP OF THE NECK TO AVOID WARMING THE SOLUTION.

8.9 Add distilled water, slowly running it down the inside of the neck of the flask (to minimise aeration) until the level is about 1 mm below the calibration mark. Ensure that the whole of the inside of the neck is washed.

8.10 Incline the flask and tap with the finger to dislodge bubbles, then defroth the solution with an alcohol blower and dry with a clean roll of filter paper inside the neck of the flask to within a few millimetres of the mark.

8.11 Place a black clip on the neck of the flask about 1 mm below the mark, to throw a shadow on the meniscus. Hold the flask vertically by the top of the neck with the calibration mark at eye level. Add distilled water dropwise from a hypodermic syringe until the bottom of the meniscus and the top of the calibration mark just coincide (the flask should be viewed against a well-lit background).

If fine bagasse or fibre particles are present, flick on to the side of the neck momentarily so that the true position of the meniscus can be seen. Again dry the inside of the neck of the flask using a clean roll of filter paper.

8.12 Seal the flask with a clean, dry stopper and mix thoroughly by inverting and shaking several times.

8.13 Insert a 10-35°C clean, dry, short stemmed thermometer into the flask with the bulb of the thermometer in the middle of the flask bulb. Record the *temperature of making to the mark* (t_m) to the nearest 0.1°C. Reseal the flask. Clean and dry the thermometer.

8.14 Stand the solution for 5 minutes to allow the precipitate to settle.

B. FILTRATION

8.15 Fold a Whatman 91 filter paper (or equivalent) and

place in a clean, dry, stemless glass or plastic funnel. Do not use a fluted paper or filter aid. Place the funnel containing the filter paper on a waste filter glass.

- 8.16 Fill the filter paper by decanting the solution, ensuring that the paper is fully wetted but not overfilled. Immediately cover the filter funnel with a cover slip. Allow approximately 10 mL of solution to run into the waste filter glass and then transfer the filter funnel to a clean, dry filter glass. The filtrate should be clear and bright. If the filtrate in the filter glass is not clear and bright, allow a further 10 mL to filter, then transfer the funnel to another clean dry filter glass. If the filtrate is still not clear, discard and repeat the preparation.

NOTE: TO MINIMISE EVAPORATION, THE SOLUTION IN THE FILTER PAPER MUST NOT BE REPLENISHED, AND MUST BE KEPT OUT OF DRAUGHTS AND DIRECT SUNLIGHT.

- 8.17 Collect approximately 50 mL of the filtrate (sufficient to rinse and fill a polarimeter tube), remove the filter funnel and place the cover slip on the filter glass.

NOTE: TO MINIMISE EVAPORATION THE SOLUTION MUST BE READ AS SOON AS POSSIBLE AFTER SUFFICIENT FILTRATE HAS BEEN COLLECTED.

C. POLARISATION — FILLING TUBE

NOTE: POLARIMETER TUBES SHOULD BE HELD BY THE METAL FLANGES, NOT THE GLASS, AS HEAT FROM THE HAND CAN CAUSE THE TEMPERATURE OF THE SOLUTION IN THE TUBE TO RISE.

- 8.18 Assemble one end of the tube with a cover glass, rubber washer, and metal cap in that order. Screw the cap on only as tightly as necessary to prevent leakage.
- 8.19 Hold the tube by the metal flange and remove the cover slip from the filter glass. Pour the solution into the tube taking care that no solution spills onto the outside of the tube.

NOTE: IF THE OUTSIDE OF THE TUBE BECOMES WET IT MUST BE DRIED IMMEDIATELY AS EVAPORATION FROM THE OUTSIDE OF THE TUBE CAN COOL THE SOLUTION IN THE TUBE.

- 8.20 About two thirds fill the tube with filtrate, seal the open end with a finger, invert, and then empty to waste. Shake the tube during the draining. Repeat this procedure twice.
- 8.21 Fill the tube and top the solution up to form a liquid bead above the end of the tube. Slide on a cover glass, taking care to avoid trapping an air bubble. Screw on the cap, containing a rubber washer, only as tightly as necessary to prevent leakage.
- NOTE: IF A CAP IS SCREWED ON TOO TIGHTLY, STRAIN CAN MAKE THE COVER GLASS OPTICALLY ACTIVE AND CAUSE ERRORS IN THE POLARIMETER READINGS.

- 8.22 Hold the polarimeter tube by the metal flanges, end-on to the light, and check that the solution is not cloudy, or too dark, and that there are no air bubbles or striations. If there are air bubbles present, unscrew the cap and

top up the solution in the tube. If there are striations present, re-rinse and fill the tube. If the solution is cloudy or too dark, discard the solution and repeat the whole analysis.

- 8.23 Dry the outside of the tube to prevent cooling through evaporation. Place the tube in a tray near the polarimeter.

NOTE: KEEP POL TUBES AWAY FROM SOURCES OF HEAT OR COLD. IN PARTICULAR, DO NOT PLACE POL TUBES NEAR THE AIR OUTLET VENT OF AUTOMATIC POLARIMETERS.

9.0 READING THE POLARIMETER — PROCEDURE 1

Procedure 1 requires that with each batch of test solutions a standardisation 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.

A. PROCEDURE USING VISUAL POLARIMETERS

- 9.1 Turn on the polarimeter at least 30 minutes before readings are to be made.
- 9.2 Store the quartz plate in the polarimeter trough or in a stand adjacent to the polarimeter (but away from any source of heat). Allow the quartz plate to equilibrate for at least 30 minutes.
- 9.3 Place the quartz plate in the polarimeter trough, approximately 5 cm from analyser end with a minimum of handling.
- 9.4 Place a 10-35 °C, short stemmed thermometer (4.14) in the trough with bulb nearest the quartz plate. Close the lid gently without jarring.
- 9.5 Set the scale to read the approximate value of the quartz plate and adjust the focus of both the field and the scale telescopes. Rotate the scale setting knob alternately left and right with decreasing amplitude, until colour and intensity of all sections of the field are equal. Always close lid before balancing field. Read the scale setting to the nearest 0.05 °Z for B&S polarimeters or 0.01 °Z for S&H polarimeters. (If necessary interpolate between direct vernier reading).
- 9.6 Take two or more sighter readings, do not record. Record the next four readings, rotating the quartz plate 45° between readings. Check and record all digits for at least the first and last of the four readings. If all four readings are not within a range of 0.20 °Z, take another set of four readings.
- 9.7 Average the four readings and record to 0.01 °Z. This is the *quartz plate reading*, Q_{tq} , at temperature, t_q .
- 9.8 Open the trough and read the temperature (9.4) to the nearest 0.1 °C and record. This is the *temperature of the quartz plate*, t_q , at the time of reading.
- NOTE: DO NOT HANDLE THE THERMOMETER.
- 9.9 Remove the quartz plate and close the lid.
- 9.10 Place the filled tube (8.23) in the trough of the polarimeter, approximately 5 cm from the analyser end and close the lid without jarring. Set the scale of the polarimeter to read approximately 100 °Z, and check the focus of the telescopes.
- 9.11 Rotate the scale setting knob alternately left and right with decreasing amplitude, until the colour and inten-

METHOD 30 P.4.

sity of the separate portions of the field are equal. Read the scale setting to the nearest 0.05°Z for B&S polarimeters or 0.01°Z for S&H polarimeters. (If necessary interpolate between direct vernier readings).

- 9.12 Take two sighter readings; do not record. Take and record four subsequent readings. Rotate the tube through 45° between each reading and always close the lid before balancing the field. Check and record all digits for at least the first and the last of the four readings. If all the four readings are not within a range of 0.20°Z discard and take another set of four readings. Average the four readings and record to the nearest 0.01°Z. This is the *polarisation of the test solution*, P_{tr} at temperature, t_r . Remove the tube from the trough.
- 9.13 Hold the tube vertically by the bottom metal flange, unscrew the cap and remove the cover glass. Flick the tube such that small portions of the solution are discarded until the level of solution falls just below the top of the metal flange. Insert a 10-35°C clean, dry, short stemmed thermometer into the tube so that the bulb is near the centre of the tube. Rotate the thermometer between finger and thumb for a few seconds until the mercury column is steady. Record the *temperature of reading*, t_r , to the nearest 0.1°C. Do not handle the tube by the glass before reading the temperature. Wash and dry the thermometer after each temperature measurement.
- 9.14 Calculation — see Section 10 below.

B. PROCEDURE USING AUTOMATIC POLARIMETERS

- 9.15 Turn on the polarimeter at least 45 minutes before readings are to be made.
- 9.16 Stand the quartz plate in a stand adjacent to the polarimeter (but away from any source of heat). Fix a thermometer (4.14) adjacent to the quartz plate to measure the quartz plate temperature. Allow the quartz plate to equilibrate to ambient temperature for at least 30 minutes.
- 9.17 Record the *temperature of the quartz plate*, t_q , to 0.1°C. Allow the reading of the empty polarimeter to stabilise and reset the zero.
- 9.18 Place the quartz plate in the trough of the polarimeter with a minimum of handling. Close the lid without jarring.
- 9.19 Allow the polarimeter reading to stabilise and record the reading. Obtain four readings rotating the quartz plate through 45° between each reading. Ensure the lid is closed before reading. Average the four readings and record to the nearest 0.01°Z. This is the *quartz plate reading*, Q_{tq} , at temperature t_q .
- 9.20 Remove the quartz plate and close the lid.
- 9.21 Place the filled tube (8.23) in the trough of the polarimeter and close the lid without jarring.
- 9.22 Allow the polarimeter reading to stabilise and record the reading. Obtain four readings, rotating the tube 45° between each reading. Average the four readings and record to the nearest 0.1°Z. This is the *polarisation reading of the test solution*, P_{tr} , at temperature, t_r .
- 9.23 Remove the tube from the trough, close the lid and check that the polarimeter returns to zero $\pm 0.02^\circ\text{Z}$. If not, repeat section 9.17 to section 9.21.

NOTE: IF AFTER THE SECOND SET OF READINGS THE ZERO STILL DOES NOT

RETURN TO $\pm 0.02^\circ\text{Z}$, A FAULT IN THE INSTRUMENT IS INDICATED. THE INSTRUMENT SHOULD BE CHECKED BY A QUALIFIED SERVICEMAN.

- 9.24 Hold the tube vertically by the bottom metal flange, unscrew the cap and remove the cover glass. Flick the tube such that small portions of the solution are discarded until the level of solution is about 2 cm below the top of the metal flange. Insert a clean, dry 10-35°C calibrated thermometer into the tube so that the bulb is near the centre of the tube. Rotate the thermometer between finger and thumb for a few seconds until the mercury column is steady. Record the *temperature of reading*, t_r , to the nearest 0.1°C. Do not handle the tube by the glass before reading the temperature. Wash and dry the thermometer after each temperature measurement.

10.0 CALCULATION (FOR READING POLARIMETER BY PROCEDURE 1)

- 10.1 The following readings have been recorded:
 Q_{tq} — the quartz plate reading at temperature t_q (9.7 or 9.19).
 t_q — the temperature of the quartz plate at the time of reading (9.8 or 9.17).
 t_m — the temperature of making to the mark (8.13).
 t_r — the temperature of reading (9.13 or 9.24).
 P_{tr} — the polarisation of the test solution at temperature t_r (9.12 or 9.22).
 Q_{20} — the certified value of the quartz plate at 20°C, for the type of polarimeter used.
- 10.2 Determine C_{tq} , the correction to the quartz plate polarisation for the temperature of the quartz plate, from Table XXVI.
- 10.3 Determine C_{tm} , the correction to polarisation for the temperature of making to the mark, from Table XXVII.
- 10.4 Determine C_{tr} , the correction to polarisation for the temperature of reading, from Table XXVIII.
- 10.5 Calculate P_{20} , the polarisation at 20°C, from $P_{20} = P_{tr} + Q_{20} - Q_{tq} - C_{tq} + C_{tm} + C_{tr}$
- 10.6 Report the result to the nearest 0.01°Z.

11.0 READING THE POLARIMETER — PROCEDURE 2

NOTE: Procedure 2 requires that quartz plate readings are carried out periodically but not necessarily at the same time as the test solution is read. The polarimeter is calibrated by applying a scale correction determined by the procedure described in Method 1. A correction for the polarimeter temperature is required for quartz wedge polarimeters or circle polarimeters with a white light source and interference filter.

A. PROCEDURE USING VISUAL POLARIMETERS

- 11.1 Turn the polarimeter on 30 minutes before readings are due to be made.
- 11.2 For quartz wedge polarimeters, e.g. Schmidt & Haensch, place a thermometer in the trough and record the *polarimeter temperature*, t_p , to 0.1°C.
- 11.3 Place the filled tube (8.23) in the trough of the polarimeter, approximately 5 cm from the analyser end and close the lid without jarring.

- 11.4 Set the scale of the polarimeter to read approximately 100°Z and adjust the focus of both the field and scale telescopes. Rotate the scale setting knob alternately left and right with decreasing amplitude, until the colour and intensity of the separate portions of the field are equal. Read the scale setting to the nearest 0.05°Z for B&S polarimeters or 0.01°Z for S&H polarimeters. (If necessary interpolate between direct vernier readings).
- 11.5 Take two sighter readings; do not record. Take and record four subsequent readings. Rotate the tube through 45° between each reading. Always close the lid before balancing the fields. Check and record all digits for at least the first and the last of the four readings. If all four readings are not within a range of 0.20°Z, discard and take another set of four readings. Average the four readings and record to the nearest 0.01°Z. This is the *polarisation reading of the test solution*, P_{tr} at temperature, t_r . Remove the tube from the trough.
- 11.6 Hold the tube vertically by the bottom metal flange, unscrew the cap and remove the cover glass. Flick the tube so that small portions of the solution are discarded until the level of solution falls just below the top of the metal flange. Insert a clean, dry, 10-35°C short stemmed thermometer into the tube so that the bulb is near the centre of the solution. Rotate the thermometer between finger and thumb for a few seconds until the mercury column is steady. Record the *temperature of reading*, t_r , to the nearest 0.1°C. Do not handle the tube by the glass before reading the temperature. Wash and dry the thermometer after each temperature measurement.
- 11.7 To obtain the zero reading set the scale of the polarimeter to read approximately $\pm 0.15^\circ Z$. Check the focus of both telescopes and ensure that the trough lid is closed. Again balance colour and intensity of the fields and record the reading. Slightly offset the scale and repeat the balancing and reading. In this way, record four readings. If all the four readings are not within a range of 0.20°Z, discard and take another set of four readings. Average the four readings and record to the nearest 0.01°Z. This is the *polarimeter zero*, P_o .
- 11.8 To calculate the results see 12.1 to 12.6.
- 11.9 To obtain the *scale correction*, SC, see Method 1.

B. PROCEDURE USING AUTOMATIC POLARIMETERS

- 11.10 Turn the polarimeter on at least 45 minutes before readings are to be made.
- 11.11 Allow the polarimeter reading to stabilise then reset the display to zero. Place the filled tube (8.23) in the trough of the polarimeter and close the lid without jarring.
- 11.12 Record the *polarimeter temperature*, t_p , to 0.1°C each time a tube is placed in the polarimeter. The thermometer (4.14) to measure the polarimeter temperature should be fixed vertically close to the polarimeter, but away from any source of heat.
- 11.13 Allow the polarimeter reading to stabilise and record the reading. Obtain four readings, rotating the tube 45° between each reading. Ensure the lid is closed before each reading. Average the four readings and record to the nearest 0.1°Z. This is the *polarisation reading of the test solution*, P_{tr} , at temperature, t_r .

- 11.14 Remove the tube from the trough, close the lid and check that the polarimeter returns to zero $\pm 0.02^\circ Z$. If not, repeat section 10.2 to section 10.3.

NOTE: IF AFTER THE SECOND SET OF READINGS, THE ZERO STILL DOES NOT RETURN TO $\pm 0.02^\circ Z$, A FAULT IN THE INSTRUMENT IS INDICATED. THE INSTRUMENT SHOULD BE CHECKED BY A QUALIFIED SERVICEMAN.

- 11.15 Hold the tube vertically by the bottom metal flange, unscrew the cap and remove the cover glass. Flick the tube so that small portions of the solution are discarded until the level of solution is about 2 cm below the top of the metal flange. Insert a clean, dry, 10-35°C short stemmed thermometer into the tube so that the bulb is near the centre of the tube. Rotate the thermometer between finger and thumb for a few seconds until the mercury column is steady. Record the *temperature of reading*, t_r , to the nearest 0.1°C. Do not handle the tube by the glass before reading the temperature. Wash and dry the thermometer after each temperature measurement.
- 11.16 To obtain the *scale correction*, SC, see Method 1.

12.0 CALCULATIONS (FOR READING THE POLARIMETER BY PROCEDURE 2)

- 12.1 The following readings have been recorded:
 P_o — the polarimeter zero reading for visual polarimeter (11.7).
 t_m — the temperature of making to the mark (8.13).
 t_r — the temperature of reading (11.6 or 11.15).
 t_p — the polarimeter temperature (11.2 or 11.12)
 SC — the polarimeter scale correction (Method 1).
 P_{tr} — the polarisation of the test solution at temperature t_r (11.5 or 11.13).
- 12.2 Determine C_{tm} , the correction to polarisation for the temperature of making to the mark, from Table XXVII.
- 12.3 Determine C_{tr} , the correction to polarisation for the temperature of reading, from Table XXVIII.
- 12.4 Determine C_{tp} , the correction to polarisation for the polarimeter temperature, from Table XXIX (for quartz wedge polarimeters or circle polarimeters with interference filters).
- 12.5 Calculate P_{20} , the polarisation at 20°C from
 $P_{20} = P_{tr} - P_o + SC + C_{tm} + C_{tr} + C_{tp}$
 NOTE: P_o , THE POLARIMETER ZERO READING APPLIES ONLY TO VISUAL POLARIMETERS.
- 12.6 Report the result to the nearest 0.01°Z.
 NOTE: REFER TO PAGE 6 FOR EXAMPLE WORK SHEET.

13.0 RANGE AND PRECISION

Concentration Range 'Degrees Z'	95% Repeatability r	95% Reproducibility R
96.00-100.00	0.147	0.251

14.0 REFERENCES

- 14.1 ICUMSA; Proceedings of Thirteenth Session (1962), Subject 11.
- 14.2 ICUMSA; Proceedings of Fourteenth Session (1966), Subject 11.

METHOD 30 P.6

- 14.3 ICUMSA; Proceedings of Fifteenth Session (1970), Subject 11.
- 14.4 ICUMSA; Proceedings of Sixteenth Session (1974), Subject 11.
- 14.5 ICUMSA; Proceedings of Seventeenth Session (1978), Subject 11.
- 14.6 ICUMSA; Proceedings of Eighteenth Session (1982), Subject 11.
- 14.7 ICUMSA; Proceedings of Nineteenth Session (1986), Subject 11.
- 14.8 Internat. Sug. J., 59 (1957), p. 156.
- 14.9 Internat. Sug. J., 82 (1978), p. 263.
- 14.10 Internat. Sug. J., 67 (1965), p. 234 and 265.
- 14.11 Sugar Analysis. Official and Tentative Methods Recommended by ICUMSA Ed. F. Schneider (1979), p. 2.

WORK SHEET (For reading Pol by Procedure 2)

Sample No.: Analyst: Date:

Sample Details: Polarimeter:

READINGS	CALCULATIONS
Pol readings	
(1) °Z	
(2) °Z	
(3) °Z	
(4) °Z	
Ave Pol reading	Pol of test solution
$\frac{P_{tr}}{\quad}$ °Z	$\frac{P_{tr}}{\quad}$ °Z
Pol zero readings	
(1) °Z	
(2) °Z	
(3) °Z	
(4) °Z	
Ave Pol zero	Polarimeter zero
$\frac{P_o}{\quad}$ °Z	$\frac{P_o}{\quad}$ °Z
	Scale correct. SC (Method 1) °Z
Temp. of making to mark	Correction C_{tm} (Table XXVII) °Z
$\frac{t_m}{\quad}$ °C	C_{tr} (Table XXVIII) °Z
Temp. of reading	Correction C_{tr} (Table XXVIII) °Z
$\frac{t_r}{\quad}$ °C	C_{tp} (Table XXIX) °Z
Polarimeter temp.	Correction C_{tp} (Table XXIX) °Z
$\frac{t_p}{\quad}$ °C	

CALCULATE POL AT 20°C FROM EQUATION:

$$P_{20} = P_{tr} - P_o + SC + C_{tm} + C_{tr} + C_{tp}$$

$$P_{20} =$$

$$P_{20} =$$

NOTE: POLARIMETER ZERO P_o NOT REQUIRED WITH AUTOMATIC POLARIMETERS.

METHOD 31

MOISTURE — DETERMINATION IN RAW SUGAR BY DRYING AT ATMOSPHERIC PRESSURE

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian Sugar Industry. Moisture is defined as the loss of mass of the sample when it is heated for five hours at 98°C.

2.0 PRINCIPLE OF METHOD

The test portion is dried in a laboratory oven at 96-100°C and the loss of mass is recorded. This analysis *assumes* that all the water in the sample is removed by the drying process and that the loss of mass is due only to the removal of this water. The temperature of 98°C is used to minimise any decomposition of reducing sugars.

3.0 APPARATUS

Ordinary Laboratory apparatus and

3.1 **Laboratory drying oven** capable of being controlled at $98 \pm 2^\circ\text{C}$. A forced draft (fan) oven is preferred.

3.2 **Drying basin**, of metal or glass of a suitable size to take the required (5 g) quantity of sample in a layer about 10 mm thick.

3.3 **Lid** to fit over basins.

3.4 **Desiccator** with active, self-indicating silica gel (silica gel can be reactivated by heating in an oven at 110-150°C until the dark blue colour is restored, or by heating for a few minutes in a microwave oven).

4.0 PROCEDURE

4.1 Weigh a clean, dry, empty drying basin and lid to the nearest 0.0001 g. Record the mass of the basin and lid (m_1).

4.2 Add 5 ± 0.5 g raw sugar to the basin and place lid on top. Weigh basin and lid and sugar to the nearest 0.0001 g and record the mass (m_2).

4.3 Heat basin and sugar with lid removed, in the oven for 5 hours \pm 10 minutes at 96-100°C. Basins must

be at least 3 cm from the sides of the oven, 10 cm from the top, and 10 cm away from air vents. Avoid opening the door of the oven during drying.

4.4 Place the lid on the basin and remove from the oven and cool in a desiccator for 30 ± 5 minutes.

4.5 Reweigh basin, lid and sugar to the nearest 0.0001 g. Record the mass (m_3). This operation should be completed within half a minute of removing the basin from the desiccator.

5.0 CALCULATIONS

Calculate the % moisture in the sugar.

$$\begin{aligned} \% \text{ moisture} &= \frac{100}{1} \times \frac{\text{loss of mass}}{\text{mass of test sample}} \\ &= 100 \times \frac{m_2 - m_3}{m_2 - m_1} \end{aligned}$$

where:

$$m_1 = \text{mass of the empty basin + lid} \quad (4.1)$$

$$m_2 = \text{mass of basin + lid + sample} \quad (4.2)$$

$$m_3 = \text{mass of basin + lid + dry sample} \quad (4.5)$$

Report the result as per cent water to 0.01%.

6.0 PRECISION

Sample	Concentration	95%	95%
	Range % Moisture (m/m)	Repeatability r	Reproducibility R
High pol sugar	0.2-0.5	.03	.04
Low pol sugar	0.3-1.0	.05	.07

7.0 REFERENCES

7.1 Plews, R.W. (Ed.): Analytical Methods Used in Sugar Refining; Elsevier (1970).

7.2 Schneider, F. (Ed.): Sugar Analysis ICUMSA Methods; ICUMSA (1979).



METHOD 32

REDUCING SUGARS — DETERMINATION IN RAW SUGAR BY THE LANE AND EYNON METHOD

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian Sugar Industry. Reducing sugar is a factor in the net titre formula, on which Australian raw sugar payments are based.

It is assumed for this method that the reducing sugars are glucose and fructose.

2.0 PRINCIPLE OF METHOD

Reducing sugars reduce copper (II) salts, in alkaline solution, to a red precipitate of copper (I) oxide. In the Lane and Eynon method, a 10 mL volume of the copper solution (Fehling's solution) is titrated with the sugar solution until all the copper is reduced.

The Fehling's solution is prepared immediately before use by mixing equal volumes of Fehling's A (copper sulphate solution) and Fehling's B (potassium tartrate/sodium hydroxide solution). The main part of the test solution is added to the Fehling's solution which is then boiled, and the final amount of the test solution is added to the boiling solution until the copper (II) ions are completely reduced. Methylene blue is added near the end-point as an internal indicator. Reaction time is standardised at 3 ± 0.5 minutes from the start of boiling.

For accurate work at least two titrations are done: One preliminary titration to find the approximate end-point; then one or more titrations in which all except 1-2 mL of the sugar solution is added before boiling. The amount of reducing sugars which corresponds to the fixed volume of Fehling's solution varies with the total volume of the reaction mixture and also with the concentration of sucrose (some sucrose is converted to reducing sugars at the high pH). A table is therefore used to calculate the amount of reducing sugars in the test solution.

When the amount of reducing sugars in the test solution is less than 0.06 g, a known volume of standard reducing sugar solution is added so that the titration volume remains in the standard range. This procedure is referred to as the first differential method.

3.0 APPARATUS

Ordinary laboratory apparatus and

- 3.1 **Bulb pipette**, 5 mL Class A. (AS 2166).
- 3.2 **Bulb pipette**, 10 mL Class A. (AS 2166).
- 3.3 **Burette**, 50 mL Class A, (AS 2165) graduated in 0.1 mL for the sugar solution. The burette tap is replaced by a rubber tube and pinch-cock, connected to a bent outlet tube in order to keep the graduated section of the burette out of the steam while additions are made to the boiling mixture.
- 3.4 **Heat resistant plate**, (about 15 cm \times 15 cm) with a 5 cm diameter hole.
- 3.5 **Anti-bumping granules**.
- 3.6 **Boiling flasks**, (florencia flask) 200 mL.
- 3.7 **Volumetric flask**, 100 mL A-class. (AS 2164).

3.8 **Stainless steel gauze strainer**, about 5 cm dia., 150 micron.

3.9 **Stop-watch**, with 60 second sweep.

3.10 **Bunsen burner**.

4.0 REAGENTS

During the analysis, unless otherwise stated, use only reagents of analytical grade and only distilled water.

4.1 **Fehling's Solution A**. For each litre of solution, dissolve 69.28 ± 0.05 g of cupric sulphate pentahydrate in distilled water. Add about 10 drops of concentrated sulphuric acid and dilute to 950 mL. (The sulphuric acid retards the formation of moulds which can grow in copper sulphate solutions during long storage).

4.2 **Fehling's Solution B**. Dissolve 346 ± 1 g sodium potassium tartrate in distilled water. Add that volume of standardised 5 mol/L sodium hydroxide solution (4.11) which contains 100.0 ± 0.5 g NaOH. Dilute the solution to 1L.

Safety

This reagent is strongly caustic. It should not be pipetted by mouth. If in contact with skin, remove all contaminated clothing and wash the affected area with copious quantities of water. If ingested, administer large quantities of 1% acetic acid. Do not induce vomiting.

4.3 **Methylene Blue**, 1 g/100 mL solution in water.

4.4 **Standard Invert Solution**. This solution is used for standardising Fehling's solution A and contains 0.2048 g of invert sugar per 100 mL.

4.4.1 Determine the water content of the standard sugar (sucrose) to be used. Take a weight of the undried sugar which will contain 1.946 ± 0.001 g of dry substance and dissolve in about 40 mL distilled water in a 100 mL flask, i.e.

$$\text{weight sugar} = \frac{1.946 \times 100}{(100 - \%H_2O)} \pm 0.001 \text{ g}$$

4.4.2 Add 3.0 mL of 10 mol/L HCl using a 10 mL safety pipette and mix. Heat in a water bath at $68.0 \pm 0.5^\circ\text{C}$ for 10 minutes \pm 10 secs. During the first three minutes agitate the flask continually. Cool and wash the solution into a one litre volumetric flask.

4.4.3 Neutralise the solution with solid sodium carbonate, using a pH 7.0 indicator paper. Dilute to the mark and shake well. This solution must be freshly prepared for each set of titrations of Fehling's Solution A during standardisation.

4.5 **Differential Invert Solution** (for the differential method)

4.5.1 Dissolve 12.0 ± 0.1 g of standard sugar in about 40 mL of distilled water and invert as described above (4.4). Cool, add 0.5 ± 0.1 mL 1% phenolphthalein solution, dilute to 1 litre without neutralising and store. The solution will keep for several weeks if not neutralised as the acid

METHOD 32 P.2.

prevents the growth of bacteria. The actual concentration of invert sugar in this solution is not critical, as a blank test is carried out each time it is used.

4.5.2 When required, neutralise with solid sodium carbonate, sufficient of the differential invert solution for the number of titrations to be performed, (each titration requires 10 mL). When correctly neutralised the solution should be faintly pink. If too much Na_2CO_3 is added, add acid invert solution from the stock until correct colour is obtained. Store the neutralised solution in a stoppered reagent bottle.

4.6 **Sucrose** (or Standard Sugar supplied by CSR Central Laboratory).

4.7 **Sodium Carbonate.**

4.8 **Hydrochloric Acid** $\rho_{20} = 1.17 \text{ g/mL}$.

4.9 **Sulphuric Acid** $\rho_{20} = 1.84 \text{ g/mL}$.

4.10 **Phenolphthalein**, 1 g/100 mL solution in alcohol.

4.11 **Sodium hydroxide** 5 mol/L solution. The concentration of this solution must be determined by standardisation so that it is known within 0.02 mol/L.

5.0 STANDARDISATION OF FEHLING'S SOLUTION A

The strength of Fehling's Solution A is determined by titration against standard invert solution (4.4). The procedure must be identical to that described in the procedure (7.3 to 7.12). The amount of invert required should be $25.00 \pm .05 \text{ mL}$; adjustments to the Fehling's Solution A must be made until this is obtained.

5.1 If the Fehling's Solution A is too strong, i.e. stopping point more than 25 mL, each litre must be diluted by an amount equal to

$1000 \times [(stopping\ point/25.0) - 1.0] \text{ mL}$ of water. Add 95% of the required volume and titrate again.

5.2 If the Fehling's Solution A is too weak, i.e. stopping point less than 25 mL, each litre must be strengthened by the addition of

$69.28 \times [1.0 - (stopping\ point/25.0)] \text{ g}$ of cupric sulphate.

Dissolve 105% of the calculated amount in a portion of the solution, add to the bulk of solution and titrate again. In this way, the strength of the Fehling's Solution A will be finally adjusted by dilution, the simpler procedure. The stock solution should be thoroughly mixed after each adjustment.

6.0 STANDARDISATION OF METHOD

The copper-invert sugar equivalents, using various concentrations of sucrose, were established by Lane and Eynon. Provided the conditions specified in their procedure are carefully followed, no separate standardisation is required. Tables III and VII are based on the original Lane and Eynon standardisation.

7.0 PROCEDURE

A. STRAIGHT METHOD (for raw sugar with $>0.6\%$ RS)

7.1 Weigh out $25.0 \pm 0.1 \text{ g}$ of raw sugar. Wash the sample into a 100 mL, A Class volumetric

flask, add distilled water to 50-60 mL and dissolve by shaking.

7.2 Add distilled water to fill the bulb of the flask and mix well. Make to the mark with distilled water and shake well.

7.3 Using a 5 mL class 'A' pipette, add 5.00 mL of Fehling's Solution A to a 200 mL boiling flask (3.6). Using a safety pipette add $5.0 \pm 0.1 \text{ mL}$ of Fehling's solution B and swirl, to complete the preparation of the alkaline copper solution. Fehling's B should be added just before the titration is to be done.

Add a few pieces of anti-bumping granules to the boiling flask to prevent bumping.

7.4 Rinse (about 10 mL) and fill a clean 50 mL A-class burette (3.3) with titrating solution (7.1). Squeeze the rubber tubing above the pinchcock to remove air-bubbles; open the pinchcock and incline the jet to remove the remaining air-bubbles. Adjust the level of solution to the zero mark.

7.5 Add 15 mL of the solution from the burette to the alkaline copper solution (7.3), swirl, and place the flask on a heat resistant plate (3.4) over a bunsen burner (3.10).

7.6 Bring the mixture to the boil, making sure that the flame does not impinge on the flask above the level of the solution.

7.7 Start the stopwatch when the mixture commences to boil vigorously and adjust the flame to give moderate, even boiling.

7.8 After boiling for 15 secs, if the solution is still blue, add rapidly successive small portions of solution from the burette (but without interrupting boiling) until the reddish colour shows that the end-point is near. With experience this can be judged to within 2-3 mL. This estimated end-point should be reached within the first minute of boiling.

7.9 After 2 minutes of boiling, add 3 drops of methylene blue indicator (4.3).

7.10 Continue titrating, adding a few drops at a time, allowing 5-10 secs between each portion, until the blue colour is completely discharged, and note the final burette reading. The total boiling time should be $3.0 \pm 0.5 \text{ min}$. and boiling should not be interrupted during the titration (to prevent air entering the flask and oxidising the contents). For routine work, the above incremental method gives satisfactory results, provided that no more than 3 mL of solution is needed to complete the titration after step 7.8. For accuracy, a second titration is required.

7.11 Prepare another alkaline copper solution (7.3), add all but 1-2 mL of the required amount of titrating solution from the burette, and repeat steps 7.6 and 7.7.

7.12 Continue boiling for 2 minutes before adding the methylene blue indicator and complete the titration as before. The total boiling time should be kept as close as practicable to 3 minutes (but no more than 3.5 minutes). Record the end-point to nearest 0.1 mL. The end point must lie between 17 and 35 mL.

B. 1ST DIFFERENTIAL METHOD (for sugar with 0-0.6 % RS)

7.13 For raw sugar, weigh out $25.00 \pm 0.01 \text{ g}$. For other products, weigh a suitable quantity that will contain 25 g of sucrose.

Wash the sample into a 100 mL Class 'A' volumetric flask, add distilled water to 50-60 mL and dissolve by shaking.

- 7.14 Using a 10 mL Class 'A' bulb pipette, add 10.00 mL neutralised differential invert solution (4.5), add distilled water to fill the bulb of the flask, swirl, make to the mark, and shake well.
- 7.15 Prepare a "blank" solution by weighing out 25.00 ± 0.01 g standard sugar and repeating steps 7.13 and 7.14.
- 7.16 Titrate the unknown (7.14) as described in steps 7.3 to 7.12 and record the end point to 0.1 mL.
- 7.17 Titrate the blank (7.15), as described in steps 7.3 to 7.12, but run in 31 mL solution for the first titration. (The end-point should lie between 32 and 36 mL). Record the end-point to 0.1 mL.

8.0 CALCULATIONS

- 8.1 Table XXXII gives the grams of Reducing Sugars per 100 mL of titrating solution corresponding to the titration (7.12 or 7.17) for a sucrose concentration of 25 g/100 mL.

- 8.2 Calculate the % RS in the sample.

For the straight method:

$$\% \text{ RS} = \frac{100 \times \text{g RS per 100 mL titrating solution}}{\text{mass sample per 100 mL titrating solution}}$$

For the differential method:

$$\% \text{ RS} \times \frac{100 \times [\text{g RS per 100 mL titrating solution} - \text{g RS per 100 mL blank}]}{\text{mass sample per 100 mL titrating solution}}$$

where: mass sample/100 mL titrating solution for raw and refined sugar = 25 g

Report the results as % RS in raw sugar to 0.01%

9.0 PRECISION

Sample	Concentration range per cent reducing sugar	95% Repeatability r	95% Reproducibility R
Raw Sugar	0.60 - 1.0 (straight method)	0.017	0.037
Raw and Refined Sugar	0 - 0.60 (differential method)	0.017	0.037

10.0 REFERENCES

- 10.1 De Whalley, H.C.S., ICUMSA Methods of Sugar Analysis; Elsevier (1964).
- 10.2 Browne, C.A. and Zerban, F.W.; Physical and Chemical Methods of Sugar Analysis; Ed. 3; Wiley (1955).
- 10.3 Honig, P. (Ed.); Principles of Sugar Technology; Vol. 1, Ed. 1., Elsevier (1953).
- 10.4 Lane, J.H. and Eynon, L.; J.Soc.Chem.Ind.; Vol. 42, (1923).
- 10.5 Lane, J.H. and Eynon, L.; J.Soc.Chem.Ind.; Vol. 50, (1931).



METHOD 33

TOTAL COLOUR (COLOUR INDEX) — DETERMINATION IN RAW SUGAR

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian Sugar Industry. A financial incentive scheme is based on this method.

The amount and nature of the colouring matter in raw sugar is one of the more important aspects of raw sugar quality. High colour in a raw sugar adds significantly to refining cost.

2.0 DEFINITIONS

Colour Index, C.I.: The colour index at one particular wavelength is the attenuation index at that wavelength after a correction factor for turbidity has been applied. In this method, twice the attenuation index at 720 nm is subtracted from the attenuation index at 420 nm.

$$\text{C.I.} = a_{420}^* - 2a_{720}^*$$

Attenuation Index, a^* : Attenuation index is the absorbance multiplied by 1000, and divided by path length and concentration.

$$a^* = \frac{A \times 1000}{bc}$$

where: A = absorbance

b = cell length, in cm

c = concentration, in g/mL

3.0 PRINCIPLE OF METHOD

The raw sugar is dissolved in water and the pH of the solution is adjusted to 7.0. (The attenuation index of raw sugar solutions is markedly dependant on pH). The solution concentration is measured by refractometer after adjusting the pH. The absorbance at 420 nm and 720 nm is measured in a spectrophotometer and the colour index calculated. (This method assumes that twice the attenuation index at 720 nm is an accurate measure of the turbidity contribution to the attenuation index at 420 nm.)

4.0 APPARATUS

Ordinary laboratory apparatus and glassware and

4.1 **Spectrophotometer**, suitable for the measurement of absorbance in the region of 420 nm and 720 nm. This spectrophotometer should comply with the following specification:

- spectral band pass 10 nm or less
- wavelength reproducibility ± 0.5 nm
- absorbance reproducibility ± 0.003 at 1.0 abs.

4.2 **Matched glass cells**, 1 cm, for the spectrophotometer.

4.3 **pH meter**, to measure to 0.01 pH.

4.4 **Refractometer**.

4.5 **Magnetic stirrer**.

5.0 REAGENTS

During the analysis, unless otherwise stated, use only reagents of analytical grade and only distilled water.

5.1 **Hydrochloric acid**, approx. 0.1 mol/L.

5.2 **Sodium hydroxide**, approx. 0.1 mol/L.

5.3 **Buffer solution pH 4**. Dissolve 10.21 ± 0.01 g of dried

potassium hydrogen phthalate in distilled water and make to exactly 1 litre to give a 0.05 mol/L solution of pH 4.00 at 20°C and 4.01 at 28°C. The potassium hydrogen phthalate should be dried for 2 hours at 110°C before weighing.

5.4 **Buffer solution pH 6.8**. Dissolve 3.40 ± 0.01 g of potassium dihydrogen orthophosphate and 3.55 ± 0.01 g of anhydrous di-sodium hydrogen orthophosphate in distilled water and make up to exactly 1 litre. The di-sodium hydrogen orthophosphate should be dried at about 110°C for 2 hours before weighing. This will give a solution of 0.025 mol/L with respect to both salts, with pH 6.88 at 20°C and 6.85 at 28°C. NOTE: Buffer solutions can be prepared conveniently from commercially available concentrated volumetric solutions supplied in sealed ampoules. (However these can be outside the stated concentration and should be checked).

6.0 STANDARDISATION OF pH METER

The pH meter must be standardised before use each day and should be checked regularly during the day. For standardisation two buffers (pH 4.0 and pH 6.8) should be used. For checking, one buffer at near pH 6.3 should be used.

7.0 PROCEDURE

A. PREPARATION OF TEST SOLUTION

7.1 Weigh out 12.5 ± 0.1 g of sugar and transfer to a 100 mL volumetric flask. Add approximately 40 mL of distilled water and dissolve the sugar by swirling or shaking on a mechanical shaker.

7.2 Make up to 100 mL with distilled water. Stopper the flask and mix thoroughly. Pour approximately 40 mL of this solution into a clean, dry 50 mL beaker.

7.3 Clean and dry the electrode of the pH meter and immerse it in the sugar solution (7.2).

7.4 Adjust the pH to 7.0 ± 0.1 by adding 0.1 mol/L HCl (5.1) or 0.1 mol/L NaOH (5.2), as required from a fine dropper. Stir the solution continuously with a magnetic stirrer while the pH is being adjusted.

7.5 Remove the electrode from the solution and wash with distilled water. When not in use, the electrode should be kept immersed in distilled water.

7.6 Immediately, determine the refractive index of the sample solution (7.4). Take three readings and record the refractive index to 0.0001 R.I. units. Record the solution temperature to 0.1°C. Refer to Method No. 17 for "Solids (Brix) — Determination in Sugar Solutions by Abbe Refractometer".

B. MEASUREMENT OF ABSORBANCE

7.7 Determine the cell corrections at 420 nm and 720 nm on a pair of matched 1 cm cells using distilled water.

7.8 Read the absorbance of the sample solution (7.5) in a spectrophotometer at 420 nm and 720 nm in a 1 cm cell, using water as the reference solution.

METHOD 33 P.2.

Record the absorbance of sample solution at 420 nm and at 720 nm to 0.001 units.

8.0 CALCULATIONS

8.1 Calculate the refractive index of the sugar solution (7.6) at 20°C.

8.1.1 Table X gives the refractive index of pure water at various temperatures relative to air.

8.1.2 Table XI gives temperature corrections to be applied to refractometer readings of sugar solutions, at 20°C.

8.2 Calculate the concentration (g/mL) of the sugar solution (7.6).

8.2.1 Table XII gives degrees brix, density, and concentration (g/mL) of sucrose solutions corresponding to the refractive index at 20°C.

8.3 Apply any cell corrections (7.7) to the absorbance at 420 nm and at 720 nm (7.8).

8.4 The attenuation indices at 420 and 720 nm are given by:

$$a^* = \frac{1000 \times (\text{corrected absorbance at } \lambda \text{ nm})}{\text{concentration (g/mL)} \times \text{cell size (cm)}}$$

where λ = wavelength of reading.

8.5 The colour index of the sugar sample is given by:

$$\text{Colour Index} = a^*_{420} - 2a^*_{720}$$

Record the colour index to the nearest 10 units.

9.0 RANGE AND PRECISION

Sample	Concentration Range (Colour Units)	95% Repeatability r	95% Reproducibility R
High pol raw sugar	1000-2700	90	Not determined
Low pol raw sugar	3000-5000	190	Not determined

10.0 REFERENCES

- 10.1 Carpenter, F.G. and Deitz, V.R.; J.Am.Soc.Sugar Beet Technologists; Vol. 12, No. 4 (1963), p. 326.
- 10.2 ICUMSA; Report of Proceedings of the 13th Session (1962), pp. 40-41.
- 10.3 Browne, C.A. & Zerban, F.W.; Physical and Chemical Methods of Sugar Analysis; Wiley (1941), pp. 1212-3.

METHOD 34

AFFINED COLOUR — DETERMINATION IN RAW SUGAR

1.0 SCOPE AND FIELD OF APPLICATION

This method is used by CSR Central Laboratory to assess the affining quality of raw sugars. It follows closely the method developed by the Amstar Corporation and included in their general contract provisions. Similarly based procedures are included in nearly all USA raw sugar contracts.

Affined colour represents the colour to be expected after a raw sugar has passed through a refinery affination station. It is the sum of the colour in the residual film of syrup coating which has not been possible to remove under the standard conditions of the test and the colour included in the raw sugar crystal itself. Affined sugar colour represents the colour load to be expected in the main course of a refinery.

The first part of this test can be used to prepare a raw sugar for analysis of any affined sugar parameter.

The method, which is applicable to all raw sugars, consists of two parts:

- (i) Removal of that part of the syrup coating the raw sugar crystal that is likely to be removed during refinery affination.
- (ii) Determining the colour of the affined sugar after dissolving it in water.

2.0 PRINCIPLE OF METHOD

The sugar is mixed with 64.0°bx high purity liquid sugar and the resultant mixture (magma) centrifuged. The affined sugar is spread in a thin layer to dry and the dried, affined sugar is stored for the determination of colour (or any other affined sugar parameter). The colour of the affined sugar is measured (after dissolving in water and adjusting the pH) as the absorbance at 420 nm, corrected for turbidity measured at 720 nm.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

- 3.1 **Centrifuge**, fitted with 200-230 mm dia basket, faced with 500 μ m mesh metal screen, capable of operating at 3000 r/min.
- 3.2 **Mixer**, with flat beaters and approx. 2L bowl (Kitchen-Aid-Model K5, with No. K-5-A-B flat beaters, from Hobart Manuf. Co. Troy, Ohio, USA) or similar mixer.
- 3.3 **Dispensing burette**, or peristaltic pump, to deliver liquid sugar at constant rate of 84-85 mL/minute.
- 3.4 **Balance**, to weigh 1000 \pm 5 g.
- 3.5 **Sheets glazed paper**, about 1 m².

4.0 REAGENTS

- 4.1 **Liquid Sugar**, 64.0°bx. Prepare from high quality refined sugar or from superfine liquid sugar. Adjust to 64.0°bx at 20°C.

5.0 PROCEDURE

A. AFFINATION

- 5.1 Weigh 1000 \pm 5 g of well mixed raw sugar into the mixer bowl (3.2). Turn the mixer on to low speed (No. 1 setting approximately 100 r/min).
- 5.2 With a dispensing pipette or pump (3.3), add 380 mL of 64°bx liquid sugar at a uniform rate over 4.5 minutes (84-85 mL/minute).
- 5.3 Continue to mix the raw sugar and liquid sugar for one more minute. The total mixing time from the commencement of adding liquid sugar is 5.5 minutes.
- 5.4 Immediately transfer the entire magma (raw sugar and liquid sugar mixture) to the centrifuge basket (3.1).
- 5.5 Bring the centrifuge up to 3000 r/min in 15 seconds and spin at 3000 r/min for exactly two minutes.
- 5.6 Remove the sugar from the centrifuge basket and spread on the glazed paper in a layer not to exceed 5 mm thick.
- 5.7 Allow the sugar to dry (in a clean dust free area). Mix the sugar periodically by hand, with a flat spatula, so that the sample dries uniformly and is well mixed.
- 5.8 Store the dry affined sugar in a sealed heavy-duty plastic bag for subsequent determination of colour or other parameters.

B. COLOUR DETERMINATION

Measure the colour of the affined sugar according to the procedure described in the Method No. 33 "Total Colour (Colour Index) — Determination in Raw Sugar".

6.0 CALCULATION

Follow the procedure in Method No. 33 and calculate the colour index of the affined sugar.

Record the colour index of the affined sugar to the nearest 10 colour units.

7.0 PRECISION

Sample	Concentration Range (Colour Units)	95%	95%
		Repeatability r	Reproducibility R
Raw sugar	500-1500	96	Not Determined

8.0 REFERENCE

Method No. 33, "Total Colour (Colour Index) — Determination in Raw Sugar".



METHOD 35

AMINO NITROGEN — DETERMINATION IN RAW SUGAR AND MILL PRODUCTS

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry. It is based on the official ICUMSA Method.

The ninhydrin/hydrindantin method is sensitive to other organic nitrogen compounds, so the analysis must not be regarded as an absolute measure of amino acid content. However, it measures all the major amino acids likely to be present in raw sugar and its liquors with adequate accuracy, and gives results which assist in the prediction of the likelihood of colour formation in raw sugars.

2.0 PRINCIPLE OF METHOD

Ninhydrin reacts with amino-acids and ammonium ions to give a blue-violet colour which can be extracted into an aqueous solution of isopropanol. The absorbance of this solution is measured at 570 nm.

Standard solutions are prepared with L-aspartic acid because asparagine and aspartic acid are the major amino acids occurring in raw sugar.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

3.1 **Volumetric flasks**, 100 mL Class A (AS 2164).

3.2 **Test tubes**, Pyrex, calibrated to 15 mL with stoppers (Quickfit type, B19/26 neck).

3.3 **Metal test-tube rack**.

3.4 **Boiling water bath**.

3.5 **Bulb pipettes**, Class A (AS 2166) — 1.0 mL, 2.0 mL, 3.0 mL, 4.0 mL, 5.0 mL

3.6 **Automatic dispenser**, to deliver 1.0 ± 0.1 mL

3.7 **Balance**, to weigh 100 g to ± 0.02 g.

3.8 **Spectrophotometer**, with matched 1 cm cells.

3.9 **Stop watch**.

4.0 REAGENTS

Use only AR grade reagents and only freshly distilled water.

4.1 **Acetate Buffer Solution** (pH 5.5). Weigh out 54.4 g of sodium acetate trihydrate into a 100 mL volumetric flask and add 40 mL water. Heat in a hot bath and shake until dissolved. Cool, and add 10 mL of glacial acetic acid, mix, make to mark, and shake well.

Check the pH of the solution which should be 5.51 ± 0.03 . If necessary, adjust pH using glacial acetic acid or NaOH (0.1 g of NaOH is approximately equivalent to 0.04 pH units). This solution keeps indefinitely under normal refrigerated conditions (approx. 4°C).

4.2 **2-methoxyethanol**, (Methyl Cellosolve) peroxide free.

Safety

2-methoxyethanol is a suspected carcinogen. Safety gloves must be worn at all times.

4.3 **Ninhydrin/Hydrindantin Solution**. Make up in a 500 mL volumetric flask. For the preparation of 400 mL volume: Weigh out 8.0 g ninhydrin powder and 1.2 g hydrindantin powder and dissolve in 300 mL of 2-methoxyethanol (4.2). Add 100 mL of buffer (4.1).

Transfer immediately to a clean, dry reagent bottle, previously filled with nitrogen or carbon dioxide gas. Insert the automatic dispenser (3.6) and carefully add 25 mL of laboratory grade paraffin oil to form a layer over the liquid surface. Cover the bottle with aluminium foil and store in a refrigerator.

This reagent must be discarded after 3 weeks.

Before using the reagent, run a little to waste to clear the delivery tip.

Safety

Ninhydrin is a poison and irritates the skin. It is a suspected carcinogen. Safety gloves must be worn at all times.

4.4 **Propan-2-ol solution 50%** (v/v). Dilute 50 mL of propan-2-ol to 100 mL with water.

4.5 **Standard solution of L-aspartic Acid** [100 mg/L of nitrogen]. Weigh out 0.0951 ± 0.0001 g of L-aspartic acid. Wash into a 100 mL volumetric flask and make to the mark with distilled water. This solution must be prepared freshly before use.

5.0 STANDARDISATION

5.1 **Working Standards**. With Class A bulb pipettes (3.5), pipette aliquots of the standard L-aspartic acid solution into separate 100 mL volumetric flasks (3.1), as shown in the following table. Make each flask to the mark with distilled water and mix well.

Volume of standard L-aspartic acid solution (4.5)	Amino nitrogen in working standards (expressed as amino nitrogen in aspartic acid)	Concentration of amino nitrogen in 15 mL test solution
(mL)	(mg N/L)	(mg N/15 mL)
1.0	1.0	0.01
2.0	2.0	0.02
3.0	3.0	0.03
4.0	4.0	0.04
5.0	5.0	0.05

5.2 Pipette 1.00 ± 0.01 mL of each of the working standards (5.1) into separate test tubes (3.2).

5.3 Into two separate test tubes, pipette $1.00 \pm .01$ mL of distilled water. These will be the standard blanks.

5.4 Using an automatic pipette (3.6), add 1.0 ± 0.1 mL of ninhydrin/hydrindantin mixture (4.3) to each test tube, mix by swirling. Avoid excess splashing of reagent on the sides of the test tube.

5.5 Plug the necks of the test tubes loosely with cotton wool and place them in the metal test tube rack.

5.6 Place the rack containing the test tubes into a boiling water bath for 15 mins. ± 10 secs. Ensure the water level is higher than the level of solutions in the test tubes.

5.7 Remove from the bath, immediately dilute with 50% (v/v) isopropanol solution (4.4) to about 0.5 cm below the 15 mL mark. Stopper the tubes and shake vigorously for 15 seconds.

METHOD 35 P.2.

- 5.8 Cool in cold water for 15 mins. then on bench out of sunlight for 30 mins. Dilute each solution to the 15 mL mark with 50% (v/v) isopropanol solution (4.4) and mix again, for 15 secs. The solutions must be read within the next 30 mins.
- 5.9 Read the absorbance of each standard blank and each standard solution in a spectrophotometer at 570 nm in matched 1.0 cm cells, using water as the reference solution. Record the absorbance to 0.001 units.
- 5.10 Subtract the average of the absorbance of the 2 standard blanks from each of the standard solutions.
- 5.11 Plot the absorbance of each standard solution against the concentration of nitrogen in mg/L.
As a guide, the absorbance of the 2.0 mg/L nitrogen standard should read about 0.195 absorbance units.
- 5.12 The calibration line should be established from duplicate results by two independent analysts.
The standardisation is repeated if:
- Two or more points lie off the line of best fit by more than 5% of the absorbance value at the particular point;
 - The line of best fit does not pass within 0.003 absorbance units from the origin;
 - The slope of the calibration line obtained by individual analysts differs by more than 2%.

6.0 PROCEDURE

- 6.1 Weigh out 5.00 ± 0.02 g for a high pol sugar
 3.00 ± 0.02 g for a low pol sugar
 0.50 ± 0.02 g for a mill syrup
Measure and record brix of the syrup.
- 6.2 Wash the sample into a 100 mL volumetric flask and completely dissolve the sample in approximately 60 mL distilled water. Make the solution to the mark, stopper the flask and mix well.
- 6.3 Pipette 1.00 ± 0.01 mL of distilled water into two test tubes. These are the test blanks.
- 6.4 Pipette 1.00 ± 0.01 mL of the test solution (6.2) into a test tube.
- 6.5 Using an automatic pipette, add 1.0 ± 0.1 mL of ninhydrin/hydrindantin solution to each test tube. Mix by swirling. Avoid splashing the reagent on the walls of the test tubes.
- 6.6 Plug the necks of the test tubes loosely with cotton wool and place them in the test tube rack.
- 6.7 Place the rack with test tubes into a boiling water bath for 15 mins \pm 10 secs. Ensure that the water level in the bath is higher than the level of the solutions in the test tubes.

- 6.8 Remove from the water bath, immediately dilute each solution with 50% (v/v) isopropanol solution (4.4) to 5 mm below the 15 mL mark, stopper and shake vigorously for 15 secs. in pairs. Cool in cold water bath for 15 mins, then on bench out of sunlight for 30 mins.
- 6.9 Make to 15 mL mark with 50% (v/v) isopropanol solution and mix, again for 15 secs. The solutions must be read within the next 30 mins.
- 6.10 Read the absorbance of the blanks and test solutions in a spectrophotometer at 570 nm in matched 1.0 cm cells, using water as a reference solution.

7.0 CALCULATIONS

- 7.1 Average the two blank absorbance readings and subtract the average blank absorbance from the absorbance of each test solution.
- 7.2 Read the mg N in 15 mL test solution directly from the calibration graph.
- 7.3 The amino nitrogen content in the sample, mg/kg, (expressed as amino nitrogen in L-aspartic acid) is given by:
- $$\text{amino nitrogen (mg/kg)} = \frac{\text{mg/L N, in 15 mL test solution}}{\text{sample mass}} \times 100$$
- 7.4 Record the amino nitrogen, (mg/kg) in sample, expressed as amino nitrogen in L-aspartic acid to the nearest whole number.

NOTE: If needed, amino nitrogen, (mg/kg on solids), can be calculated using the brix measurement in (6.1).

8.0 PRECISION

Sample	Concentration range (mg/kg N) (as amino nitrogen in L-aspartic acid)	95%	95%
		Repeatability r	Reproducibility R
Raw sugar	20-200	9	Not determined

9.0 REFERENCES

- 9.1 ICUMSA, Report of the Proceedings 14th Session (1966): Subj. 18.
- 9.2 ICUMSA, Report of the Proceedings 15th Session (1970): Subj. 18.
- 9.3 Schneider, F.; "Sugar Analysis"; ICUMSA Official and Tentative Methods; 1979.
- 9.4 Greenstein, J.P. and Winitz, M.; "Chemistry of Amino Acids"; Vol. 2 (1961).
- 9.5 Harris, W.A.; J. Amer. Sugar Beet Technol.; Vol. 12 (1962).

METHOD 36

FILTRABILITY — DETERMINATION IN RAW SUGAR BY THE CELITE METHOD

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian Sugar Industry. A financial incentive scheme is based on this method in relation to payment for raw sugar delivered.

The filtrability result of a raw sugar reflects the efficiency of the mill clarification process and is broadly indicative of refinery carbonatation/filtration performance.

2.0 DEFINITION

The filtrability value of a raw sugar is the ratio of the filtration rate of a solution of the raw sugar with that of a solution of pure sugar of the same concentration, filtered under identical conditions.

3.0 PRINCIPLE OF METHOD

A raw sugar solution is made up to $60.0 \pm 0.5^\circ\text{bx}$, and Celite 505 and buffer are added to this solution. The buffer brings the pH of the solution to between 8.5 and 9.1. The solution is filtered under constant pressure of 345 kPa. Filtrate is initially run to waste for two minutes, to allow a uniform Celite filter cake to form. The filtrate is then collected over a precisely timed five minute period, weighed and expressed as a percentage of the weight of pure sucrose solution which would be collected under identical conditions of density, pH, temperature and pressure.

The per cent filtrability is defined at $20 \pm 1^\circ\text{C}$. At temperatures above 20°C , a lower filtrability is obtained, and at temperatures below 20°C the filtrability is higher. If a test is done at a temperature other than $20 \pm 1^\circ\text{C}$, the result can be expressed only as "filtrability at $t^\circ\text{C}$ ".

4.0 APPARATUS

Ordinary laboratory apparatus and glassware and

4.1 **Pressure filter**, of brass or stainless steel including cylinder body, screw on top with air hose connection, screw on base, filter disc, retaining ring and rubber gaskets (Figure 1).

4.2 **Support stand** and locking ring.

4.3 **Variable speed stirrer**, capable of being controlled at 900 ± 100 r/min with either three blade impeller (38 mm diameter with 12 mm square blades, pitch 20°) or 35 mm propeller type blade (11 mm width, pitch 20°).

4.4 **Compressed air** or nitrogen supply, with reducing valve, regulator, safety relief valve, three-way valve and Bourdon type pressure gauge with a range of 0-700 kPa.

4.5 **Balance**, to weigh up to 500.00 ± 0.05 g.

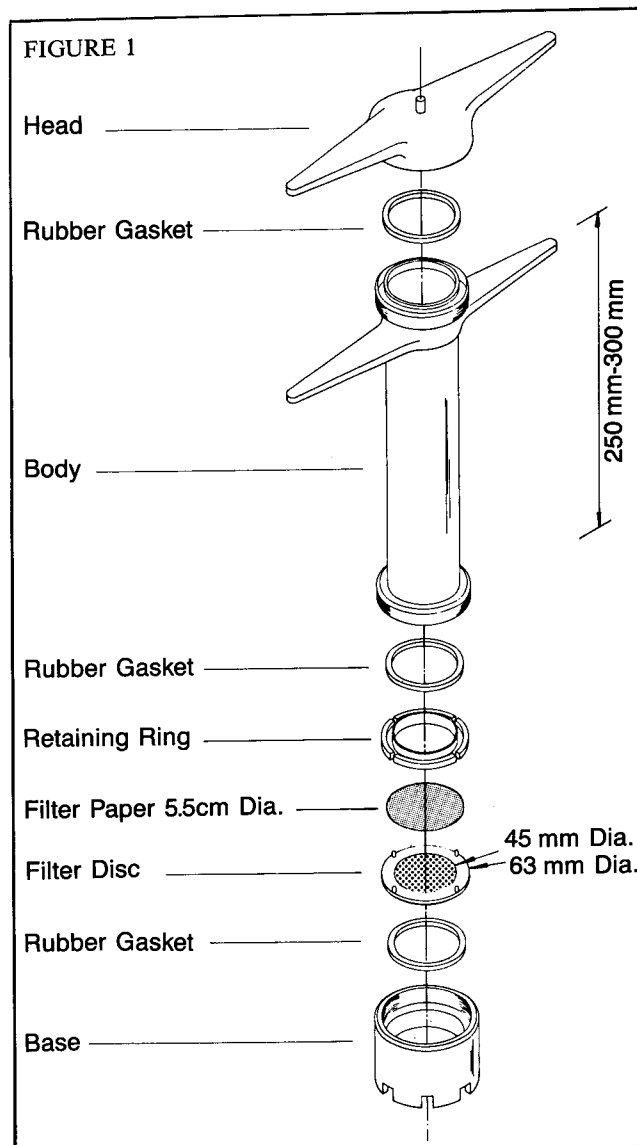
4.6 **Balance**, to weigh up to 100.00 ± 0.01 g.

4.7 **Stopwatch**, with 60 seconds per revolution in units of one second with a range of 30 minutes.

4.8 **Thermometer**, 200-400 mm long, $10\text{-}35^\circ\text{C}$, reading to 0.2°C .

4.9 **Pipette**, 5.0 ± 0.1 mL [graduated straight pipette or automatic pipette].

4.10 **Dispenser**, to deliver 99.4 ± 0.1 mL.



5.0 REAGENTS

5.1 **Standard Filter Aid** — Celite 505 standardised at CSR Central Laboratory.

5.2 Filtrability Buffer

5.2.1 Prepare 1L of a 50% (m/m) solution of glycerol in water.

5.2.2 Dissolve 15.0 ± 0.1 g of calcium acetate in 200-300 mL of 50% glycerol solution in a 500 mL beaker.

5.2.3 Dissolve 400.0 ± 0.5 g of triethanolamine in 200-300 mL of 50% glycerol solution in a 500 mL beaker.

5.2.4 Transfer the calcium acetate solution and the triethanolamine solution to a 1L volumetric flask, rinsing both beakers with 50% glycerol solution.

METHOD 36 P.2.

5.2.5 Make up to 1L with 50% glycerol solution, mix well and allow to stand overnight. Add a teaspoon of Celite 505 and filter in a Buchner filter through a No. 54 filter paper. The filtered solution can be stored for 6 months.

5.2.6 The pH of the test solution should be greater than 10.5 pH.

Safety. Triethanolamine is a mild eye and skin irritant and wearing safety glasses and rubber gloves is recommended.

5.3 **Sucrose**, or high purity refined sugar certified starch free. Suitable refined sugar is available from CSR Limited, Central Laboratory.

5.4 **Filter paper**, Whatman Grade 54, 5.5 cm diameter. The filter papers are selected from the one "making" and should have a mean Gurley time of 7.6 seconds [air flow measured by Gurley Densometer]. Suitable selected Whatman 54 filter papers can be obtained from CSR Limited, Central Laboratory.

6.0 CALIBRATION OF FILTER DISC

The filter disc calibration is checked by measuring the filtration rate of a pure sugar syrup (60.0°bx), filtered with the Celite under the same conditions as the test. The Celite 505 to be used for the test is standardised at CSR Central Laboratory. With each batch of standardised Celite 505, CSR Central Laboratory issues a reference Table (No. XVII). The Table gives the mass of pure syrup, filtered between 2 and 7 minutes after application of pressure at various temperatures, under the standard condition of the test. Each table is specific to a particular batch of Celite 505.

6.1 Prepare a 60.0°bx solution of starch-free refined sugar (5.3) by dissolving 600 ± 5 g of refined sugar in water and diluting to 1000g.

6.2 Add 15 g of Celite 505 (2.5% by weight on solids) and mix well. Filter the syrup through a fresh Whatman 54 filter paper (in a Buchner funnel, under vacuum or in a pressure filter). Discard the first 40-50 mL of filtrate.

6.3 Transfer the filtered syrup to a 1L conical flask and mix well. Check the brix by refractometer. Adjust the syrup to 60.0 ± 0.1 °bx by weight with distilled water or refined sugar. Stopper the flask and mix well.

6.4 Weigh 333 g of the 60.0°bx pure sugar syrup (6.3) into a 500 mL beaker, i.e. 200 ± 0.1 g solids.

6.5 From Table XVII, determine the correct amount of Celite 505 to be added to 200 g solids of pure sugar syrup *for the particular batch of Celite 505*.

6.6 Weigh the correct weight of Celite 505 into the 200 g solids of pure sugar. Stir the mixture with an electric stirrer at 900 ± 100 r/min (see 7.4).

6.7 Add, while stirring, 1.00 ± 0.02 mL filtrability buffer (5.2) with a graduated pipette. Continue stirring for 2 minutes \pm 10 seconds after adding buffer. Cover and allow to stand for 15 minutes \pm 30 seconds.

6.8 Filter the syrup following the procedure outlined in sections 7.6-7.13.

6.9 The mass of filtrate should be within 4% of the mass given in Table XVII for the particular batch of Celite. NOTE: If the solution blows through before the completion of 7 minutes filtration repeat from step 6.4 using an increased quantity of pure sugar syrup (additional Celite will also be required

to maintain the correct percentage on solids for the particular batch of Celite).

7.0 PROCEDURE

A. PREPARATION OF RAW SUGAR SOLUTION

7.1 Weigh out in a 250 mL beaker, the appropriate amount of sugar from the following Table:

% Water in Sugar	Mass of Sample \pm 0.05 g	
	Average filtrability sugars	High filtrability sugars
0.0-0.2	149.00	198.70
0.2-0.5	150.00	200.00
0.5-0.8	151.60	201.50
0.8-1.0	152.60	202.70

7.2 Add 99.4 ± 0.1 mL at 20°C (equivalent to 99.1 ± 0.1 g) distilled water from an automatic pipette or by weighing. For high filtrability sugars, add 132.5 ± 0.1 mL at 20°C (equivalent to 132.1 ± 0.1 g) distilled water.

7.3 From Table XVII, determine the correct mass of Celite 505 to be added. Add in the dry form, the correct mass of Celite 505 to the sugar in the 250 mL beaker.

NOTE (1) Table XVII is applicable to a specific batch of standardised Celite 505 and should only be used with that batch.

(2) Before use, mix the Celite by gently tumbling in the bottle.

7.4 Dissolve the sugar without heating, using an electric stirrer rotating at 900 ± 100 r/min. *Check visually to ensure that all crystals are dissolved.* This usually requires 20-30 minutes stirring although low filtrability sugars may take longer to dissolve.

If the stirring time exceeds 45 minutes, the analysis should be repeated.

NOTE: To avoid aerating the solution, the stirrer shaft must be vertical and centered in the vessel. The base of the shaft should be 1 cm above the bottom of the vessel. To minimise grinding of Celite 505 particles, stirrer speed must be 900 ± 100 r/min, and stirring time (before addition of buffer) should be less than 45 minutes.

7.5 With the stirrer still going, add 2.00 ± 0.02 mL standard buffer (5.2) (down the side of the beaker to prevent aeration), or 3.00 ± 0.3 mL for high filtrability sugars, to the solution. Mix for 2 minutes \pm 10 seconds after addition of all buffer. Switch the stirrer off and remove from the solution. Cover the beaker and allow to stand for 15 minutes \pm 30 seconds.

B. FILTRATION

7.6 Assemble the filter in the order: base, rubber gasket, filter disc, filter paper, retaining ring, second rubber gasket. Screw the assembled end on to the body of the filter. See Figure 1.

7.7 Tare 150 mL beaker to 0.1 g and record mass (m_1).

7.8 At the end of the 15 minutes \pm 30 seconds standing time, stir solution (7.5), using electric stirrer, for 1 minute \pm 10 seconds. Switch off the stirrer and remove from the solution. Transfer all the solution to the filter.

7.9 Screw the top on to the filter body, place in stand and clamp tightly, connect pressure hose, and apply pressure of 345 ± 3.5 kPa (50.0 ± 0.5 psig). Start stopwatch immediately pressure is applied.

- 7.10 Run filtrate to waste for 2 minutes \pm 1 second then collect the filtrate in the previously tared 150 mL beaker (7.7) for the next 5 minutes \pm 1 second.
- 7.11 At 7 minutes \pm 1 second after application of pressure, remove beaker.
- 7.12 Weigh beaker and contents to 0.1 g and record the mass (m_2). Read the temperature of the filtrate to 0.1°C and record (T).
- 7.13 Turn off compressed gas, release pressure from filter, remove air hose, unclamp filter from stand, and open it. Wash and dry the filter, and reassemble it for the next run.

8.0 CALCULATIONS

8.1 Calculate the mass of filtrate delivered during the interval from 2 to 7 minutes after the application of pressure:

$$\text{Weight filtrate} = m_2 - m_1$$

where: m_1 = mass beaker (7.7)

m_2 = mass beaker and filtrate (7.12)

8.2 Determine the weight of pure 60°bx sugar syrup that would have filtered between 2 and 7 minutes after application of the pressure at the temperature T, from Table XVII.

Table XVII gives mass of pure sugar syrup filtered (between 2 and 7 minutes after application of pressure) at various temperatures, under the standard conditions of the filtrability test.

NOTE: The table is applicable to specific batches of Standard Celite 505, and is issued from CSR Central Laboratory when Celite is obtained. It should only be used in conjunction with the appropriate batch.

8.3 Calculate the mass of raw sugar syrup filtered in the 2-7 minute interval as a percentage of the weight of pure sugar syrup that would have been filtered in the same time and at the sample temperature.

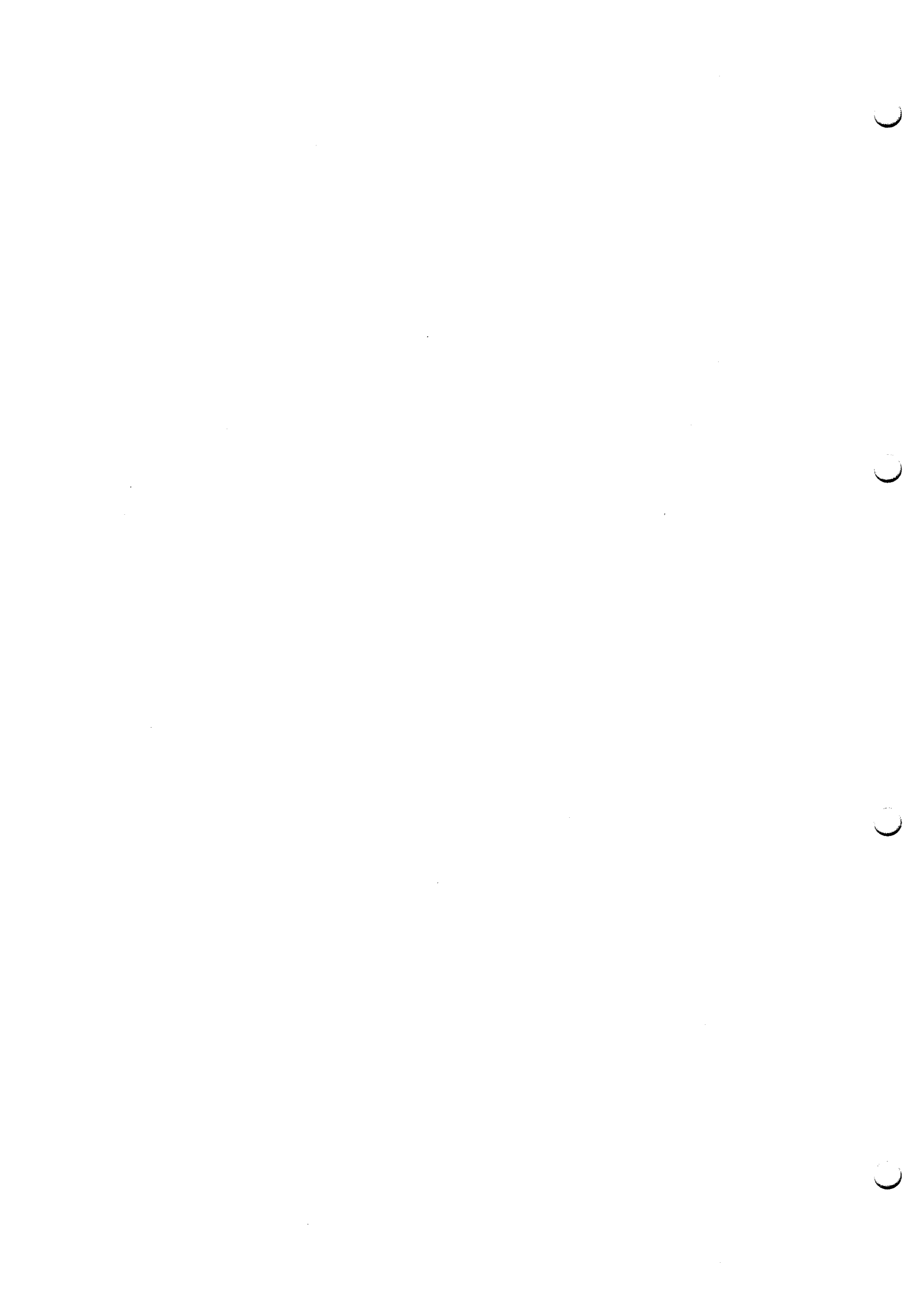
% Filtrability

$$= \frac{\text{mass filtrate test solution at Temp. T}}{\text{mass filtrate pure sucrose at Temp. T}} \times \frac{100}{1}$$

Record the result to 0.1% filtrability.

9.0 PRECISION

Sample	Concentration range (% Filtrability)	95% Repeatability r	95% Reproducibility R
Raw sugar	0-100	3.8	6.0



METHOD 37

STARCH — DETERMINATION IN RAW SUGAR

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian Sugar Industry. The method measures a substance (or group of substances) which is basically starch or derived from starch.

Cane starch is composed of amylose and amylopectin with amylose being the fraction that forms a blue colour with iodine. *Cane starch* can be occluded in the raw sugar crystal and affects the filtration rate of liquor from the refinery carbonation process. The amylose fraction is responsible for this effect.

2.0 PRINCIPLE OF METHOD

The sugar is dissolved in water and the solution is digested with hot calcium chloride/acetic acid to solubilise any starch present. Potassium iodide/iodate solution is added to form the blue starch-iodine complex. The absorbance of this complex is read in a spectrophotometer at 700 nm. At this wavelength, the effect on the absorbance of the impurities in raw sugar is minimal.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

- 3.1 **Spectrophotometer** suitable for the measurement of absorbance at 700 nm with a set of matched 2 cm cells.
- 3.2 **Water bath** capable of being controlled at 95-100°C.
- 3.3 **Flask shaker.**
- 3.4 **Volumetric flask**, with wide neck (Kohlrausch), 50 mL capacity.
- 3.5 **Bulb pipettes**, Class A (AS 2166), 1 mL, 2 mL, 3 mL, 4 mL, 5 mL, 20 mL
- 3.6 **Automatic dispenser**, 15 mL
- 3.7 **Balance**, to weigh up to 100.00 ± 0.01g.

4.0 REAGENTS

During the analysis, unless otherwise stated, use only reagents of analytical grade and only distilled water.

- 4.1 **Calcium Chloride Solution 40% (m/m).** Dissolve 53.0 ± 0.1 g of calcium chloride dihydrate in distilled water and dilute to 100.0 ± 0.1 g with distilled water. As calcium chloride is highly deliquescent, stocks should not be exposed to air. The strength of the calcium chloride solution must be checked and, if necessary, adjusted to 40.0 ± 0.3% (m/m) with distilled water or with calcium chloride dihydrate (approximately 75.5% CaCl₂).

The solution strength can most readily be checked by measuring its specific gravity. The specific gravity of 40% m/m calcium chloride solution is 1.3942 at 24°C. Within the temperature range 20-30°C, it decreases on an average by 0.0007 per degree above 24°C and increases by the same amount per degree below 24°C. In the region of 40% (m/m), a change of 0.1% in calcium chloride concentration produces a change of 0.001 in specific gravity.

Specific gravity may be measured by pycnometer, hydrometer or other instrument capable of the

accuracy required. A convenient method is as follows:

- (i) Allow stoppered containers of calcium chloride solution and distilled water to stand for a number of hours (e.g. overnight) so that both reagents come to ambient temperature.
- (ii) Weigh a clean, dry 100 mL volumetric flask to 0.02 g. Fill to the mark with calcium chloride solution and weigh again to 0.02 g. Note the temperature of the solution to 0.1°C.
- (iii) Rinse out the flask and fill to the mark with distilled water at the same temperature as the calcium chloride solution, and weigh to 0.02 g. From the masses of the solution and water, and the temperature, calculate the specific gravity to 0.001.

- 4.2 **Acetic Acid (1 mol/L approx.).** Dilute 57 ± 1 mL of glacial acetic ($\rho_{20} = 1.049$ g/mL) acid to one litre with distilled water.

- 4.3 **Acetic Acid (0.033 mol/L approx.).** Dilute 3.0 ± 0.03 mL 1 mol/L acetic acid (4.2) to 100 mL with distilled water.

- 4.4 **Calcium Chloride-Acetic Acid Reagent.** Using a pH meter and stirrer, adjust the pH of a 100 mL aliquot of calcium chloride solution (4.1) to pH 3.0 ± 0.1 with 0.033 mol/L acetic acid. Because of the large salt concentration, allow sufficient time for the pH meter to equilibrate.

Adjust the pH of the bulk of the calcium chloride solution in the same proportion. Although the pH of a calcium chloride/acetic acid solution tends to alter on standing, do not readjust to pH 3.0 before using.

- 4.5 **Potassium Iodate Solution (0.0017 mol/L).** Dry about 0.5 g potassium iodate at 105-110°C for 1 hour. Dissolve 0.3566 ± 0.0002 g of the dried reagent in distilled water and make to 1 litre. Store in the dark in a brown, glass-stoppered bottle.

- 4.6 **Potassium Iodide 10% (m/v).** Dissolve 10.0 ± 0.1 g of potassium iodide in distilled water and dilute to 100 mL. Store in the dark in a brown, glass-stoppered bottle. Discard the solution if it becomes yellow.

- 4.7 **Potassium Iodide-Potassium Iodate Reagent.** This reagent must be prepared on the day it is to be used. Mix 10.0 ± 0.5 mL of potassium iodide solution (4.6), with 90.0 ± 0.5 mL of distilled water. To this solution add 100.0 ± 0.5 mL of potassium iodate reagent. Mix the reagents and keep in a brown, glass-stoppered bottle. This reagent must be discarded after 1 day.

- 4.8 **Standard Starch Solution (900 mg/L).** Use an approved batch of B.D.H. Laboratory Reagent Potato Starch, supplied by CSR Central Laboratory. Determine its moisture content, correct to 3 decimal places, by drying about 2 g (weighed to 0.0001 g) at 105-110°C for 2 hours.

- 4.8.1 Weigh into a 25 mL beaker $\frac{(0.9000 \times 100)}{(100 - \%H_2O)}$

METHOD 37 P.2.

± 0.0001 g of fresh starch, i.e. equivalent to 0.9000 ± 0.0002 g of anhydrous starch.

4.8.2 To the weighed quantity of starch, add 5 mL of cold distilled water and mix with a glass rod. Before the starch settles, transfer the mixture quantitatively to 500 mL of boiling water in a litre conical flask so that no slurry touches the wall of the flask.

4.8.3 With at least three additional 5 mL portions of distilled water, transfer all the starch to the flask. This operation should be completed within 1 minute.

4.8.4 Boil the starch for 3 minutes \pm 10 seconds, timed from the moment the first 5 mL of starch slurry enters the boiling water.

4.8.5 Rinse a 1 litre volumetric flask with hot distilled water. Quantitatively transfer the hot solution through a glass funnel to the 1 litre volumetric flask. Wash the conical flask at least twice with hot distilled water, by adding the water to the 25 mL beaker (4.8.1.) and then transferring it to the conical flask. Continue washing the conical flask with hot distilled water and transferring to the volumetric flask until the latter is filled to approximately 900 mL.

4.8.6 Swirl to mix the flask contents and cool under running water to room temperature. Make the solution to 1 litre, stopper and mix well. Store in a refrigerator.

NOTE: The solution will keep for one week, but if possible, the standardisation should be done on the same day as the solution is prepared, to eliminate all possibility of its deterioration.

4.9 **Standard Starch Solution (180 mg/L).** Using a Class A bulb pipette, pipette 20.00 mL of the 900 mg/L standard starch solution (4.8) into a 100 mL volumetric flask. Dilute to 100 mL with distilled water. Mix well by inverting and shaking.

This reagent will not keep and must be prepared on the day the standardisation is carried out.

4.10 **Starch Free Sucrose.** Use only sugar, tested to be free of starch. Suitable starch-free sugar is available from CSR Central Laboratory.

5.0 STANDARDISATION OF METHOD

A. PREPARATION OF STANDARDS

5.1 Weigh $3.60 \pm .02$ g of starch-free sucrose (4.10) into each of six 50 mL Kohlrausch volumetric flasks (3.4).

5.2 Pipette, using Class A bulb pipettes 0, 1.00, 2.00, 3.00, 4.00, 5.00 mL aliquots of the 180 mg/L standard starch solution (4.9) respectively into each of the six flasks. The final solutions in the flasks correspond to 0, 50, 100, 150, 200 and 250 mg/kg of starch in sugar.

5.3 Pipette, using a 25 ml graduated pipette, respectively into each of the six flasks 7.0 ± 0.1 , 6.0 ± 0.1 , 5.0 ± 0.1 , 4.0 ± 0.1 , 3.0 ± 0.1 , and 2.0 ± 0.1 mL of distilled water to make the liquid volume in each flask to 7.0 mL. Swirl each flask to completely dissolve the sugar.

5.4 Pipette, using an automatic dispenser or a 25 mL graduated pipette 15.0 ± 0.1 mL of the calcium chloride acetic acid reagent (4.4) into each flask. Mix well.

5.5 Stopper each flask loosely and place in a boiling water bath ($95-100^\circ\text{C}$) for 15 ± 1 minute. Commence timing from the moment the flasks are placed in the bath. Swirl each flask about 5 and 10 minutes after placing in the bath to aid dissolution of the starch.

NOTE: The flasks must be placed in the heating bath within 30 minutes after adding water to dissolve the sugar (5.3)

5.6 After 15 minutes, remove the flasks from the heating bath and cool to room temperature in a running water bath.

5.7 To each flask, add 15.00 ± 0.05 mL of 0.033 mol/L acetic acid (4.3). Use a 15 mL bulb pipette or an automatic pipette. Mix well.

B. MEASUREMENT OF ABSORBANCE

5.8 Determine the cell corrections for a pair of matched 2 cm cells using distilled water.

5.9 Add to the 0 mg/kg test solution 10.00 ± 0.05 mL of the potassium iodide-potassium iodate reagent (4.7) using a 10 mL barrel pipette or automatic dispenser. Mix well and make to the mark with distilled water. Stopper the flask and mix well.

5.10 *Immediately* rinse twice and fill a 2 cm cell with the 0 mg/kg test solution. Read the absorbance of the solution in a spectrophotometer at 700 nm against distilled water as the reference. Correct the absorbance reading for any cell correction (5.8). Record the absorbance to 0.001 units.

NOTE: The absorbance of the test solution must be read between two and five minutes after adding the iodide-iodate reagent to the test solution.

5.11 Repeat steps 5.9 and 5.10 for each concentration of starch involved in the standardisation (5.3).

NOTE: The reading of the 0 mg/kg starch standard should not exceed 0.010 absorbance for a 2 cm cell. As a guide the reading for the 200 mg/kg starch standard should be about 0.320 absorbance for a 2 cm cell at 700 nm.

C. PREPARATION OF GRAPH

5.12 Plot "mg/kg starch in sugar" against "absorbance" (5.2).

5.13 A straight line must be obtained for the graph. If three or more points lie off the line of best fit by more than 5% of the line's absorbance value at that particular starch concentration, the standardisation must be repeated.

5.14 The graph used by any laboratory for routine analysis should be constructed using the mean of independent standardisations by at least two analysts. Each set of results and the mean must comply with the 5% limits above (5.13). In addition, the slopes of the individual lines must not differ by more than 5%.

6.0 PROCEDURE

A. PREPARATION OF TEST SOLUTIONS

6.1 Weigh 3.60 ± 0.02 g of raw sugar into each of two 50 mL Kohlrausch volumetric flasks:

(a) sample blank solution

(b) sample test solution

6.2 Using an automatic dispenser or 25 mL calibrated pipette add 7.0 ± 0.1 mL of distilled water to each flask. Swirl the flasks to completely dissolve the sugar.

METHOD 37 P.3.

- 6.3 Add to each flask 15.00 ± 0.05 mL of calcium chloride/acetic acid reagent (4.4) using an automatic dispenser or a 15 mL bulb pipette. Mix well.
- 6.4 Stopper each flask loosely and place in boiling water bath at $95-100^{\circ}\text{C}$ for 15 ± 1 minutes. Commence timing from the moment the flask is placed in the bath. Swirl each flask after 5 and 10 minutes, to aid dissolution of starch.
- NOTE: The flasks must be placed in the boiling water bath within 30 minutes of adding water to dissolve the sugar (6.2).
- 6.5 Remove the flasks from the bath and cool in a running water bath to room temperature.
- 6.6 To each flask, add 15.00 ± 0.05 mL of 0.033 mol/L acetic acid (4.3). Use a 15 mL bulb pipette or automatic dispenser. Mix well.
- 6.7 Make the sample blank solution (6.1(a)) to the mark with distilled water and mix well.
- 6.8 Add 10.00 ± 0.04 mL of potassium iodide-iodate reagent (4.7) to the sample test solution (6.2(b)), and make to the mark with distilled water. Stopper the flask and mix well.

- 6.9 Read the absorbance of the sample test solution (6.8) in a 2 cm cell in a spectrophotometer at 700 nm, against the sample blank (6.7) as the reference solution. Record the absorbance to 0.001 units.

NOTE: The readings must be made between two and five minutes after the addition of the potassium iodide-iodate reagent to the sample test solution.

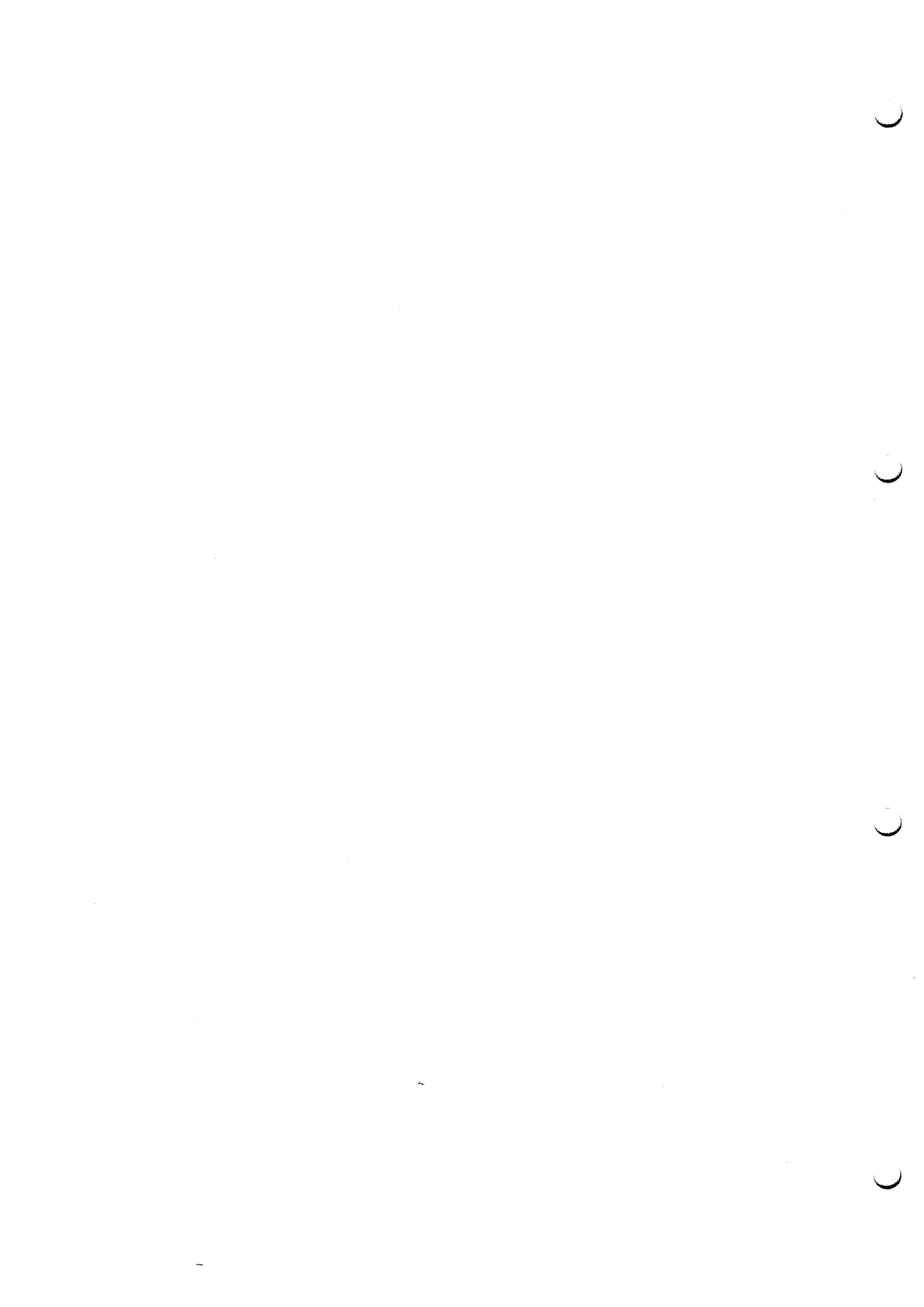
7.0 CALCULATIONS

Read the mg/kg starch in sugar directly from the calibration graph (5.12).

Record the result to 1 mg/kg starch in sugar.

8.0 PRECISION

Sample	Concentration Range (mg/kg Starch)	95% Repeatability r	95% Reproducibility R
Raw sugar	0-300	11	23



METHOD 38

ACID-SOLUBLE PHOSPHATE — DETERMINATION IN RAW SUGAR BY THE AMIDOL METHOD

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian Sugar Industry.

The phosphate content of cane juice is an important factor in efficient clarification. The phosphate result for a raw sugar is indicative of the efficiency of the mill clarification process.

2.0 PRINCIPLE OF METHOD

The sugar is dissolved in distilled water and the pH adjusted to between 3 and 4 with dilute sulphuric acid. The solution is filtered with Celite. Acid molybdate, amidol and sodium metabisulphite solutions are added to an aliquot of the filtrate and phosphate is measured as the absorbance, at a wavelength of 660 nm, of the blue coloration which develops. Interference from low concentrations of organic phosphates and from anions and cations found in sugar are not significant.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

- 3.1 **Spectrophotometer**, suitable for the measurement of absorbance at 660 nm with matched 2 cm cells.
- 3.2 **Bulb pipettes**, Class A (AS 2166) — 1 mL, 2 mL, 3 mL, 5 mL, 10 mL.
- 3.3 **Graduated straight pipette**, Class A (AS 2167) — 10 mL
- 3.4 **Automatic dispenser**, 10 mL

4.0 REAGENTS

During the analysis, unless otherwise stated, use only analytical grade reagents and only distilled water.

- 4.1 **Acid molybdate reagent**. Dissolve 16.6 ± 0.1 g ammonium molybdate tetrahydrate in about 600 mL of distilled water in a 1 litre beaker. Add 96 ± 1 mL of concentrated sulphuric acid. Add the acid slowly with stirring to avoid the generation of excessive heat. Cool, transfer to a 1 litre volumetric flask, make to the mark, and mix well.

Safety

Sulphuric acid is very corrosive. Safety glasses and gloves should be used when handling the acid. It must not be pipetted by mouth.

If in contact with skin, remove all contaminated clothing and wash affected area with copious quantities of water. If ingested, administer large quantities of aluminium hydroxide or milk of magnesia (magnesium hydroxide). Do not induce vomiting.

- 4.2 **Acid reagent**. Add 96 ± 1 mL of concentrated sulphuric acid (ρ_{20} 1.84 g/mL) to about 600 mL of distilled water in a 1 litre beaker, slowly as in 4.1. Cool, transfer to a 1 litre volumetric flask, make to the mark, and mix well.
- 4.3 **Amidol reagent**. Dissolve 1.00 ± 0.01 g of amidol (2,4-diaminophenol hydrochloride) and 20 ± 1 g of sodium metabisulphite in distilled water in a 100 mL volumetric flask, make to the mark and mix well. Filter under vacuum through a Whatman No. 3 filter

paper with a level teaspoon of acid washed Supercel (4.4).

Store in a dark bottle in a refrigerator and discard after one week.

Safety

Amidol-sodium metabisulphite reagent and its intermediate oxidation products are toxic and have a pungent odour. They may produce bronchial asthma, and hence should not be inhaled. Contact with the skin may give rise to an irritation leading to dermatitis; the reagent should be washed off the skin immediately. Use gloves when handling. Do not pipette by mouth.

- 4.4 **Acid washed Supercel**. Add 50 ± 5 g of Supercel to 1 litre of distilled water. Add 50 ± 5 mL of concentrated hydrochloric acid and stir for 5 minutes. Filter and wash Supercel cake free of acid with distilled water. Test washings with litmus paper. Dry Supercel for 6 hours at 90-100 °C and store in a closed container.

Safety

Concentrated hydrochloric acid is very corrosive and gives off pungent toxic fumes. It must be handled using safety glasses and gloves in a well ventilated atmosphere. It should not be pipetted by mouth.

First aid is the same as for sulphuric acid (4.1).

- 4.5 **Standard phosphate stock solution**, 1.0 mg P/mL. Dry about 1.5 g of potassium dihydrogen phosphate for 1 hour at 110 °C. Weigh out 1.0984 ± 0.0001 g of the dried salt, dissolve in freshly distilled water and make up to 250 mL in a volumetric flask. Do not keep for longer than one month.
- 4.6 **Working standard phosphate solution**, 0.01 mg P/mL. Dilute 10.00 ± 0.05 mL of the standard phosphate stock solution to 1 litre in a volumetric flask, and mix thoroughly.

5.0 STANDARDISATION OF METHOD

- 5.1 Using Class A pipettes, pipette aliquots of 0, 2.00, 4.00, 6.00, 8.00, 10.00, 12.00 mL of working solution (4.6) into separate 50 mL volumetric flasks. These solutions are equivalent to 0, 0.02, 0.04, 0.06, 0.08, 0.10, 0.12 mg phosphate.
- 5.2 To each flask, add 2 drops of acid reagent (4.2). Adjust the volume to about 30 mL with distilled water. Add 10.0 ± 0.2 mL of acid molybdate reagent (4.1) and 4.0 ± 0.2 mL of amidol reagent (4.3). Make each flask to 50 mL with distilled water, stopper and mix well.
- 5.3 Allow the solutions to stand for at least 10 minutes but not more than 30 minutes.

NOTE: If the temperature of the solutions is above 25 °C, do not allow to stand for more than 15 minutes.

- 5.4 Read the absorbance of each standard solution in a spectrophotometer at 660 nm, in a 2 cm cell using water as the reference solution. Record the absorbance to 0.001 units.

METHOD 38 P.2.

As a guide to performance of the method, the 0.06 mg P standard measured at 660 nm in a 2 cm cell should read about 0.28 absorbance. The reading for the zero P standard in a 2 cm cell against distilled water should not exceed 0.001 absorbance.

- 5.5 Plot the absorbance of each standard solution against the concentration of phosphate.

If two or more points lie off the line of best fit by more than 5% of the line's absorbance value at that particular phosphate concentration, the standardisation must be repeated.

The calibration graph should be constructed using the mean of independent standardisations by at least two analysts. Each set of results and the mean must comply with the limits above. In addition, the slopes of the individual lines must not differ by more than 2%.

Each time a new batch of reagents is prepared, check the calibration at the 0 point (reagent blank) and at one other point by repeating steps 5.2 to 5.4. If the results do not lie within the limits set out above repeat the calibration.

6.0 PROCEDURE

A. PREPARATION OF TEST SOLUTION

- 6.1 Weigh out 20.0 ± 0.1 g of sugar, wash into 100 mL volumetric flask with distilled water, and dissolve.
- 6.2 Acidify with acid reagent (4.2). Add 2 drops for high pol sugars and 3 drops for low pol sugars. (This should reduce the pH to between 3 and 4). The remainder of the analysis should be completed within 1 hour of adding acid. Make to mark, stopper and mix well.
- 6.3 Filter under vacuum through a Whatman No. 3 filter paper. Precoat the filter with a slurry consisting of a level teaspoon of acid washed Supercel (4.4) with about 10 mL of the solution. Use the filtrate from this precoat to rinse the filter flask.
- 6.4 Transfer 20.0 ± 0.1 mL of the filtrate (6.3) into each of two 50 mL volumetric flasks.

- 6.5 To one flask, add 10.0 ± 0.2 mL of acid molybdate reagent (4.1) and 4.0 ± 0.2 mL of amidol reagent (4.3). Make up to 50 mL with distilled water, stopper and mix well. Stand for at least 10 minutes, but not more than 30 minutes.

NOTE: If the temperature of the solution is above 25°C , read between 10 and 15 minutes.

B. PREPARATION OF BLANK SOLUTION

- 6.6 To the second 50 mL flask (6.5) add 10.0 ± 0.2 mL of acid reagent (4.2). Make up to 50 mL with distilled water, stopper and shake. Stand for at least 10 minutes, but not more than 30 minutes.

C. MEASUREMENT OF ABSORBANCE

- 6.7 Read the absorbance of the test solution (6.5) in a spectrophotometer at 660 nm, in a 2 cm cell using the corresponding test blank solution (6.6) as the reference solution. Record the absorbance to 0.001 units.

7.0 CALCULATIONS

- 7.1 Read from the standard graph "mg phosphate" in the test solution.

- 7.2 Calculate phosphate, (mg/kg) in the sample —

$$P \text{ (mg/kg)} = \frac{\text{mg P from graph}}{1} \times \frac{100}{20} \times \frac{1}{m} \times 1000$$

where: m is the mass of sample taken, normally 20 g.

$$P \text{ (mg/kg)} = 250 \times \text{mg P from graph (for a 20 g sample)}$$

- 7.3 Express results as P, (mg/kg) to the nearest mg/kg.

8.0 PRECISION

Sample	Concentration Range (mg/kg)	95% Repeatability r	95% Reproducibility R
Raw sugar	0-30	2	3

9.0 REFERENCE

Allen, R.J.L., Biochem. J.; Vol. 34 (1940).

METHOD 39

PARTICLE SIZE AND SIZE DISTRIBUTION (GRIST) — DETERMINATION IN RAW SUGAR

1.0 SCOPE

This method has been adopted officially within the Australian Sugar Industry. A financial incentive scheme, in relation to raw sugar delivered, is based on the percentage fine grain determined by this method. The size and uniformity of raw sugar crystals are important factors which have been shown to affect washing and purging characteristics during refinery affination. Results from this method are influenced by crystal shape.

2.0 DEFINITIONS

The % Fines is the per cent by mass of crystals which pass through a 600 micron sieve.

The Mean Aperture (M.A.) of a sugar sample is that sieve opening through which 50% of the crystals, by mass, will pass.

The Coefficient of Variation (C.V.) is the standard deviation of the grain size of the sample, expressed as a percentage of the mean aperture.

3.0 PRINCIPLE OF METHOD

A test portion of the raw sugar is washed with anhydrous methanol to remove the syrup film from the sugar crystals, followed by washing with iso-propanol to remove traces of methanol and any residual syrup. The washed crystals are dried in an oven at 80-90°C. The dried and washed sugar crystals are mechanically sieved through woven wire sieves and the separate fractions weighed.

The M.A.-C.V. terminology is a concise method of reporting the result of a sieving test on a sugar, eliminating the need to specify the sieves employed. Most samples of sugar have a normal size distribution and a plot of the cumulative percentages retained by a series of sieves, against sieve aperture will give a straight line on "arithmetic probability" paper.

4.0 APPARATUS

- 4.1 **Jar**, of metal or plastic, approximately 1L capacity and 100 mm dia. with tightly fitting lid.
- 4.2 **Rolling mill**, to rotate the jars at about 74 r/min.
- 4.3 **Metal lid** for jar, with centre cut out to form an opening of the same diameter as the jar. A 100 μ m wire mesh screen is soldered across the opening to form a filter.
- 4.4 **Buchner flask**, about 1L capacity fitted with a large (100 mm dia.) plastic filter funnel.
- 4.5 **Measuring cylinder**, 250 mL.
- 4.6 **Drying tray**, stainless steel, approximately 150 mm \times 150 mm \times 15 mm.
- 4.7 **Laboratory drying oven**, capable of being controlled at 80-90°C, with a flame-proof rating, in a fume hood or vented to outside air.
- 4.8 **Sieve shaker**, Rotap (115 taps per minute) or Pascall Inclyno (300 vibrations per minute) or equivalent, with automatic timer set to 10 minutes \pm 5 seconds.

- 4.9 **Set of woven**, wire test sieves, AS1152, with the following nominal aperture sizes : 850 μ m, 600 μ m, 425 μ m.
- 4.10 **Stiff-bristle brush**, for removing sugar crystals from sieve apertures.
- 4.11 **Soft-bristle brush**, to remove sugar from sieves and receiver.
- 4.12 **Sheet glazed paper**, about 400 mm \times 400 mm.
- 4.13 **One pad** Mathematical probability paper, for M.A. — C.V. calculations.
- 4.14 **Balance**, to weigh 200 g to 0.01 g.

5.0 REAGENTS

- 5.1 **Methanol**, (Technical, anhydrous, 99%) — n_D^{20} 1.329

Safety

Methanol is a dangerous cumulative poison if ingested, inhaled or absorbed through the skin. Symptoms of methanol poisoning are those of normal alcoholic intoxication accompanied by severe abdominal pain and visual disturbances. The symptoms may be delayed.

10 mL methanol is sufficient to cause permanent blindness and as little as 30 mL may be fatal.

If swallowed, administer large quantities of water or milk and induce vomiting. Follow with 2 teaspoonfuls of sodium bicarbonate in water. Seek medical attention.

Alcohol vapours are inflammable so precautions must be taken to eliminate any ignition sources. The laboratory should be well ventilated to ensure that concentrations of methyl alcohol in the air are kept below 200 mg/m³, the maximum allowable concentration for safe working over prolonged periods.

- 5.2 **Isopropanol**, (Technical, anhydrous, 99%) — n_D^{20} 1.377

6.0 STANDARDISATION

Working sieves should be certified to comply with AS 1152. The sieves should be checked once each year against a reference set of sieves using sugar samples typical of the samples analysed in the laboratory.

A reference set of test sieves is kept at CSR Central Laboratory.

7.0 PROCEDURE

A. PREPARATION OF WASHED SUGAR

- 7.1 Thoroughly mix the sample avoiding mechanical damage to the sugar crystals.
- 7.2 Weigh out 115 ± 1 g of sample and transfer to a clean, dry jar (4.1).
- 7.3 Add 250 ± 10 mL of methanol (5.1), close the jar carefully with a tight fitting lid and rotate for 3 minutes \pm 15 seconds on the jar rolling mill.
NOTE: Metal jars on the rollers should be separated by a piece of plastic or rubber to prevent sparks being struck.
- 7.4 Remove the lid and substitute the lid fitted with a wire screen (4.3). Invert the jar over the plastic funnel in the Buchner flask. Filter under vacuum to remove the alcohol. When filtration is complete remove the screen

METHOD 39 P.2.

lid and carefully return any crystals adhering to the screen, back to the jar.

- 7.5 Repeat steps 7.3 and 7.4 using isopropanol (5.2) instead of methanol.
- 7.6 For Brand JA sugar, repeat steps 7.3 to 7.5.
- 7.7 Spread the alcohol wet sugar on a stainless steel tray and place this in the oven.
- 7.8 Continue drying the sugar in the oven until the crystals are dry, free-flowing and free of lumps (normally about 1-1.5 hours). Allow to cool to room temperature, and transfer to a suitable sealed container.

B. SIEVE ANALYSIS

- 7.9 Assemble the nest of sieves in the order, top to bottom: lid, 850 micron, 600 micron, 425 micron and receiver.
- 7.10 Thoroughly mix the cooled sugar, *avoiding size segregation*, and weigh out 100.00 ± 0.01 g. Transfer the sugar to the top sieve of the nest of sieves.
- 7.11 Shake the nest of sieves on the mechanical shaker for 10 minutes \pm 5 seconds.
- 7.12 Transfer the sugar crystals caught on the 850 micron sieve on to a sheet of glazed paper. Brush out the sieve with the stiff brush taking care not to damage the screen.
- 7.13 Transfer the crystals from the glazed paper to a tared 100 mL beaker. Weigh to 0.01 g to find the mass of crystals caught on the sieve, and record to 0.01 g as C850 (Caught 850 microns).
- 7.14 Repeat steps 7.12 and 7.13 for the crystals caught respectively on the 600 micron and 425 micron sieves and in the receiver. Record the mass of the fractions to 0.01 g as C600, C425, T425 (through 425 microns).

NOTE: Where % fines is the only result required from the grist analysis, the 850 and 425 microns sieves need not be used. The nest of sieves is then made up of the lid, 600 micron sieve, and receiver. The % fines is then directly given by the % caught in the receiver. When M.A. and C.V., alone, are required, the 850 and 600 micron sieves only, need to be used.

8.0 CALCULATIONS

8.1 Per cent fines

Per cent fines = % by mass through 600 microns
Record the results to 1 per cent fines.

8.2 Full grist

Add up all the masses (7.13, 7.14). If the total is 100.0

± 0.2 g, round off the masses to one decimal place, so that the total is 100.0 g. If the total is outside the range, 100.0 ± 0.2 g, discard the results as unreliable, and repeat the analysis.

Convert the mass obtained to the “% caught” on each sieve expressed to 0.1% — this is the full grist, e.g.

Sieve (μm)	Symbol	Mass (g)	Caught %	Cumulative %
850	C850	36.77	36.8	36.8
600	C600	47.01	47.0	83.8
425	C425	11.52	11.5	95.3
—	T425	4.70	4.7	100.0

- 8.3 **Mean Aperture.** Convert the per cent caught by each sieve to cumulative percentages above each mesh size. Plot the values against the corresponding mesh aperture size (in mm) on arithmetic probability paper. Draw a straight line through all points with cumulative percentage in the range 10-90%. (or C850 and C600, if they are the only two sieves used).

From the graph:

Mean aperture (M.A.) = aperture size passing 50% of crystal

Report the mean aperture to the nearest 0.02 mm.

- 8.4 **Coefficient of Variation (C.V.)**

$$\text{C.V.} = \frac{\text{Size passing 16\%} - \text{Size passing 84\%}}{2 \times \text{M.A.}} \times \frac{100}{1}$$

Report the coefficient of variation to the nearest 0.2%.

9.0 PRECISION

The expected range for raw sugar is:

% Fines	0 to 50%
Mean Aperture	0.3 to 1.5 mm
Coefficient of Variation	10 to 50%

The precision, for the percentage mass caught in each sieve, that should normally be obtained by following the procedure is:

95% repeatability r	4.0%
95% reproducibility R	4.0%

10.0 REFERENCES

- 10.1 Powers, H.E.C.; International Sugar Journal; Vol. 50 (1948).
- 10.2 Payne, J.H.; Sugar Cane Factory Analytical Control; Elsevier (1968), p. 85.

METHOD 40

MEAN ELONGATION — DETERMINATION IN RAW SUGAR

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian Sugar Industry for the measurement of the mean elongation of raw sugar crystals.

Crystal elongation is a raw sugar quality parameter. It usually results from processing cane juices with high dextran concentrations.

The shape, size and uniformity of raw sugar crystals are important sugar quality criteria as they affect purging characteristics during refinery affination.

2.0 DEFINITIONS


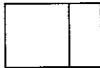

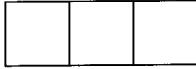
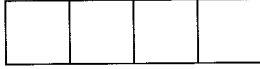
In crystallography, the terms 'length' and 'breadth' have specific well established meanings, namely the b and c axis dimensions respectively. The a:b:c axis ratio for a pure sucrose crystal is approximately 1.3:1.0:0.9, a b:c elongation of 1:1.1. Elongation can occur along the b axis or c axis with the latter being the most common.

However, given the difficulty in correctly recognising these axes in a routine laboratory situation, the above convention is not followed in this method.

For the purpose of this method, crystal elongation is taken as the ratio of the length of the crystal to the breadth of the crystal.

Mean elongation is the number average elongation of a minimum of 50 individual crystals.

Classification

Crystal Shape	Elongation Classification	Category
	1: 1.1	1
	1: 1.5	2
	1: 2.0	3
	1: 3.0	4
	1: 4.0	5

3.0 PRINCIPLES OF METHOD

A subsample of the raw sugar is washed with anhydrous methanol to remove the syrup film from the sugar crystals, followed by isopropanol to remove traces of methanol and any residual syrup. The washed crystals are dried in an oven at 80-90°C. The washed and dried sugar crystals are examined under a microscope and classified into five elongation categories. The mean elongation of the sugar is calculated from the number of crystals in each category of elongation.

4.0 APPARATUS (for analysis of one sample)

- 4.1 Jar, of metal or plastic, approximately 1L capacity and 100 mm diameter with tightly fitting lid.
- 4.2 Rolling mill to rotate the jar at approximately 74 r/min.
- 4.3 Metal or plastic lid for jar, with centre cut out and a 100 micron wire mesh screen permanently secured across the opening to form a filter.
- 4.4 Measuring cylinder, 250 mL capacity.
- 4.5 Buchner flask, about 1L capacity fitted with large plastic funnel.
- 4.6 Drying tray, stainless steel, approximately 150 mm × 150 mm × 15 mm.
- 4.7 Drying oven, controlled at 80-90°C, with flame-proof rating.
- 4.8 Balance, to weigh 115 ± 1 g.
- 4.9 Projection microscope, objective magnification × 20 (or similar), with 10 mm measuring grid on viewing screen, or microscope with graticule in eye-piece.
- 4.10 Microscope slides, glass or plastic, approximately 100 mm × 100 mm.

5.0 REAGENTS

- 5.1 Methanol (Technical, anhydrous, 99%) — n_D^{20} 1.329

Safety

Methanol is a dangerous cumulative poison if ingested, inhaled or absorbed through the skin. Symptoms of methanol poisoning are those of normal alcoholic intoxication accompanied by severe abdominal pain and visual disturbances. The symptoms may be delayed.

10 mL methanol is sufficient to cause permanent blindness and as little as 30 mL may be fatal.

If swallowed, administer large quantities of water or milk and induce vomiting. Follow with 2 teaspoonfuls of sodium bicarbonate in water. Seek medical attention.

Alcohol vapours are inflammable so precautions must be taken to eliminate any ignition sources. The laboratory should be well ventilated to ensure that concentrations of methyl alcohol in the air are kept below 200 mg/m³, the maximum allowable concentration for safe working over prolonged periods. The concentration of alcohol in the air can be determined by using a Drager test kit.

- 5.2 Isopropanol (Technical, anhydrous, 99%) — n_D^{20} 1.377

Safety. Treat as for Methanol.

6.0 PROCEDURE

A. PREPARATION OF WASHED SUGAR

- 6.1 Thoroughly mix the sample to be measured, avoiding mechanical damage to the sugar crystals.
- 6.2 Weigh out 115 ± 1 g of sample and transfer to a clean, dry jar (4.1).
- 6.3 Add 250 ± 10 mL of methanol (5.1), close the jar carefully with a tight fitting lid and rotate for 3 minutes ± 15 seconds on the jar rolling mill.

METHOD 40 P.2.

NOTE: Metal jars on the rollers should be separated by a piece of plastic or rubber to prevent sparks being struck.

- 6.4 Remove the lid and substitute the lid fitted with a wire screen (4.3). Invert the jar over the plastic funnel in the Buchner flask. Filter under vacuum to remove the alcohol. When filtration is complete remove the screen lid and carefully return any crystals adhering to the screen, back to the jar.
- 6.5 Repeat steps 6.3 and 6.4, using isopropanol (5.2) instead of methanol.
- 6.6 For Brand JA sugar, repeat steps 6.3 to 6.5.
- 6.7 Spread the alcohol wet sugar on a stainless steel tray and place this in the oven.
- 6.8 Continue drying the sugar in the oven until the crystals are dry, free-flowing and free of lumps (normally about 1 hour). Allow to cool to room temperature, and transfer to a suitable sealed container.

B. EXAMINATION OF CRYSTALS

- 6.9 Thoroughly mix the cooled sugar, avoiding size segregation and crystal breakage.
- 6.10 Distribute approximately 0.5 g of the sugar evenly over the clean, dry glass slide so that individual crystals can be easily read.
- 6.11 Position the slide on the microscope stage so that a crystal's image is superimposed on the scale on the microscope viewing screen. Rotate the slide until the grid lines run parallel to the sides of the crystal.

If using a microscope fitted with a graticule in the eye-piece, rotate the eye-piece until the graticule lines are aligned with the sides of the crystal.

- 6.12 Estimate the elongation of each crystal and record a tick on the worksheet in the appropriate box for the particular category encountered, i.e. 1:1.1, 1:1.5, 1:2.0, 1:3.0, 1:4.0. For classification purposes, round upwards or downwards to the nearest classification.
NOTE: Refer to page 3 for example work sheet.
- 6.13 Repeat steps 6.11 and 6.12 until a minimum of 50 crystals are read.
- 6.14 Move the slide up and down and left to right systematically to avoid re-reading previously observed crystals.
NOTE: Do not attempt to measure broken crystals or conglomerates.

7.0 CALCULATIONS

7.1 Mean Elongation

$$ME = \frac{1.1 \times n_1 + 1.5 \times n_2 + 2.0 \times n_3 + 3.0 \times n_4 + 4.0 \times n_5}{n_1 + n_2 + n_3 + n_4 + n_5}$$

where: n_1 to n_5 = number of crystals observed in each category 1 to 5

8.0 PRECISION

Sample	Expected Range	95% Repeatability r	95% Reproducibility R
Raw sugar	1.1 - 4.0	Not known	Not known

**WORK SHEET
CRYSTAL EXAMINATION — MEAN ELONGATION**

Date:

Analyst:

Sample No.	No. of Crystals in each Category										Sum Category Products (a)	Total Crystals (b)	M.E. (a/b)
	1:1.1	Sum	1:1.5	Sum	1:2.0	Sum	1:3.0	Sum	1:4.0	Sum			

Note (a) Sum of Category Products = $1.1 \times n_1 + 1.5 \times n_2 + 2.0 \times n_3 + 3.0 \times n_4 + 4.0 \times n_5$
where n_j = No. of crystals in category j



METHOD 41

ALKALINITY, TOTAL ALKALINITY AND CAUSTIC ALKALINITY — DETERMINATION IN WATER

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry. It is applicable to raw and treated waters and the results are used for the calculation of chemical dosages in the treatment of water supplies.

2.0 PRINCIPLE OF METHOD

Alkalinity is determined by titration with standard acid solution to a designated pH with a colour indicator. For this method the phenolphthalein alkalinity end-point (P) is at pH 8.3 and the total alkalinity end-point (M) is at pH 4.5 (methyl orange). These methods are not suited to boiler waters where the presence of organic salts may cause serious interference and therefore the methyl orange end-point becomes meaningless.

In the determination of caustic alkalinity [P(BaCl₂)], carbonate ions, and most other organic salt ions, are removed by precipitation with barium chloride followed by titration with standard acid solution to pH 8.3 with phenolphthalein. This is the recommended method for the determination of caustic alkalinity in boiler waters (refer Laboratory Manual, Volume 1, p. 91) and caustic soda cleaning solutions.

NOTE: For a more precise determination, or when presence of colour or turbidity obscures the indicator end-points, or for boiler waters containing organic salts which interfere with the end-point, the potentiometric method described in Australian Standard 2449-1981 should be used.

3.0 REAGENTS

Unless otherwise stated, use only analytical grade reagents and only distilled water which has been freed of carbon dioxide by boiling for 15 minutes and cooled to room temperature immediately prior to use.

- 3.1 **Phenolphthalein indicator solution**, 1 g/100 mL. Dissolve 1 g phenolphthalein in 50 mL ethanol plus 50 mL distilled water.
- 3.2 **Methyl Orange indicator solution**, 500 mg/L. Dissolve 0.5 g methyl orange in 1L of distilled water.
- 3.3 **Mixed indicator solution**. Dissolve 0.02 g methyl red and 0.1 g bromocresol green in 50 mL ethanol plus 50 mL water.
- 3.4 **Sulphuric acid standard solution**, 0.01 mol/L. Prepare from a commercial concentrated volumetric solution.

NOTE: Commercial concentrated solutions may contain preservatives or other agents. For precise work standardise the sulphuric acid by titration against 0.025 mol/L standard sodium carbonate solution.

4.0 SAMPLING AND SAMPLES

4.1 **Sample containers**. Use borosilicate glass or polythene containers, prepared as follows :

- (a) Wash the container and cap with detergent and tap water,
- (b) Rinse thoroughly with tap water,
- (c) Rinse twice with distilled water,
- (d) Drain thoroughly and replace cap.

4.2 **Collection and preservation**. Rinse the container 2-3 times then completely fill with sample and seal. The determination should be performed immediately. If this is not possible store the sample at 4 °C and carry out the determination within 24 hours. As a check record the pH at sampling and again before analysis.

NOTE: Boiler waters collected under pressure should be cooled to near 20 °C while still under pressure to minimise the chance of losing dissolved gases.

5.0 PROCEDURE

A. ALKALINITY TO PHENOLPHTHALEIN, P

5.1 Pipette the sample aliquot (V) into a 250 mL conical flask keeping the tip of the pipette near the bottom of the flask. **For softened water** (boiler feed water) pipette 100 mL.

5.2 Add 3-4 drops of phenolphthalein indicator and if the sample turns pink, titrate over a white surface with 0.01 mol/L H₂SO₄ until the colour just disappears. Record the volume of H₂SO₄ used to 0.1 mL, (V₁).

B. ALKALINITY TO METHYL ORANGE, M (Total Alkalinity)

5.3 Add 5-6 drops of mixed indicator or 3-4 drops of methyl orange indicator to the test sample from 5.2 and continue titrating with standard 0.01 mol/L H₂SO₄. The colour change is from blue through grey to pink-grey for mixed indicator or yellow to orange for methyl orange. Record the total volume of H₂SO₄ used, (5.2 and 5.3) to 0.1 mL, (V₂).

C. ALKALINITY TO PHENOLPHTHALEIN AFTER ADDITION OF BARIUM CHLORIDE, P(BaCl₂) (Caustic Alkalinity)

5.4 Measure 100 mL of the sample into a 250 mL conical flask. Add 3-4 drops phenolphthalein indicator followed by about 1 g of BaCl₂. Swirl until dissolved and titrate over a white surface until the pink colour just disappears. Record the volume of H₂SO₄ used, to 0.1 mL, (V₃).

6.0 CALCULATION

6.1 **Calculate** the alkalinity to phenolphthalein, P

$$P = \frac{V_1}{V} \times 1000$$

where: V = volume of test sample, in mL (5.1)

V₁ = volume of standard acid used in titration to phenolphthalein end-point (5.2).

METHOD 41 P.2.

Record the result, expressed as mg/L CaCO₃ to 1 mg/L.

6.2 Calculate alkalinity to methyl orange, M

$$M = \frac{V_2}{V} \times 1000$$

where: V = volume of test sample, in mL (5.1)

V₂ = volume of standard acid used in titration to methyl orange end-point in mL (5.2 + 5.3).

Record the result, as mg/L CaCO₃ to 1 mg/L.

6.3 Calculate alkalinity to phenolphthalein after addition of barium chloride, P(BaCl₂)

$$P(\text{BaCl}_2) = \frac{V_3 \times 1000}{V}$$

where V = volume of test sample in mL (5.4)

V₃ = volume of standard acid used in titration to P(BaCl₂) end-point (5.4)

Record the result, expressed as mg/L CaCO₃ to 1 mg/L.

7.0 CALCULATION OF ALKALINITY RELATIONSHIPS

Calculate the hydroxide, carbonate and bicarbonate alkalinities from the table. All alkalinities are expressed as mg/L CaCO₃.

ALKALINITY RELATIONSHIPS

Results of titration	Hydroxide alkalinity as CaCO ₃	Carbonate alkalinity as CaCO ₃	Bicarbonate alkalinity as CaCO ₃
P = 0	0	0	M
P < 0.5M	0	2P	M - 2P
P = 0.5M	0	2P	0
P > 0.5M	2P - M	2(M - P)	0
P = M	M	0	0

These relationships do not apply to boiler waters containing organic salts which may cause significant interference.

8.0 PRECISION

No positive statement can be made about precision because of the wide variation in sample characteristics. The precision of titration is likely to be much greater than the uncertainties involved in sampling.

The expected range of results is:

Alkalinity to Methyl Orange (total alkalinity) —

10-500 mg/L CaCO₃

Caustic alkalinity —

10-500 mg/L CaCO₃

9.0 REFERENCES

- (1) Laboratory Manual for Australian Sugar Mills (Vol I, 1984) p91.
- (2) Australian Standard 2449-1981 Waters Determination of Alkalinity, Acidimetric Titration Method.
- (3) Standard Methods of Examination of Water and Waste Water; 16th edition; American Public Health Association (1985).
- (4) Australian Standard 2031.1-1986 Selection of Containers and Preservation of Water Samples for Chemical and Microbiological Analysis, Part 1, Chemical.
- (5) Ivin, P.C. (1982). Condensate Quality and Sugar Mill Boiler Operation. Proc. Aust. Soc. Sugar Cane Technol. 205-212.

METHOD 42

CHLORIDE — DETERMINATION IN CAUSTIC (NaOH) CLEANING SOLUTIONS

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry. Chloride ions accelerate the dissolution of most metals and can cause corrosion or pitting in evaporation tubes.

2.0 PRINCIPLE OF METHOD

This is based on the standard Volhard's titrimetric procedure. A test sample of the cleaning solution is acidified with nitric acid. An excess of standardised silver nitrate is added to precipitate chloride ions. The excess silver nitrate is back-titrated with standard ammonium thiocyanate solution with ferric alum as an indicator.

3.0 REAGENTS

Unless otherwise stated, use only analytical grade reagents and only distilled water.

3.1 Nitric acid, $\rho_{20} = 1.41$ g/mL.

3.2 Nitrobenzene.

3.3 Ferric ammonium sulphate, saturated solution.

3.4 Ammonium thiocyanate solution, 0.1 mol/L. Dissolve 8.5 g of ammonium thiocyanate in water and dilute to 1 litre in a volumetric flask. Standardise by titrating 25 mL of 0.1 mol/L silver nitrate, plus 2 mL nitric acid, $\rho_{20} = 1.41$ g/mL, and 1 mL of ferric ammonium sulphate (3.3) with the ammonium thiocyanate solution. A white precipitate forms which as the end-point approaches becomes flocculent and settles easily. Shake vigorously after each addition of thiocyanate solution. At the end-point, 1 drop of thiocyanate solution produces a faint red-brown colour which does not disappear on shaking.

Molarity of ammonium thiocyanate solution

$$= \frac{25 \times \text{molarity of Ag NO}_3}{\text{volume ammonium thiocyanate titre}}$$

3.5 Silver nitrate standard solution, 0.1 mol/L. Dissolve 8.496 g AgNO₃ in water and dilute to 500 mL in a volumetric flask. Standardise by titrating 25 mL of 0.1 mol/L NaCl solution (3.6) plus 1 mL potassium chromate indicator, with the AgNO₃ solution (ref. to Method 43 "Chloride — Determination in Water"). Store in a brown bottle.

Molarity of silver nitrate solution

$$= \frac{25 \times 0.1}{\text{volume AgNO}_3 \text{ titre}}$$

3.6 Sodium chloride standard solution, 0.1 mol/L. Dissolve 2.923 g of NaCl (dried at 100-110 °C for 3

hours) in water and dilute to exactly 500 mL in a volumetric flask.

4.0 PROCEDURE

4.1 Shake the sample well, and with a bulb pipette fitted with a safety bulb, pipette 20 mL into a 1 litre conical flask containing approximately 50 mL of distilled water.

4.2 Carefully add 25 mL of concentrated nitric acid. Test the solution with litmus paper and add a further 1 mL aliquot of nitric acid until the litmus turns red.

4.3 Pipette into the solution 10.00 ± 0.02 mL of the standard silver nitrate solution (3.5). Add 1-2 mL nitrobenzene. Shake the flask to help coagulate any turbidity, then add 10 drops of ferric ammonium sulphate (3.3). This is the test solution.

NOTE: The volume of silver nitrate solution added may need to be varied to give a reasonable back-titration with ammonium thiocyanate.

4.4 Titrate, over a white background, with standard ammonium thiocyanate solution (3.4), with frequent shaking, until a faint red-brown colour persists for 30 secs. Record the volume of ammonium thiocyanate used, V_1 , to 0.1 mL.

4.5 Repeat the titration (4.2-4.4) with 20 mL distilled water, instead of the test sample. This is the blank titration. Record the volume of ammonium thiocyanate used, V_b , to 0.1 mL.

5.0 CALCULATION

5.1 Calculate chloride, (mg/L) in the test sample —

$$\text{Chloride (mg/L)} = \frac{(V_b - V_1) \times c \times 35450}{V}$$

where: V = mL of test sample (4.1)

V_1 = mL of ammonium thiocyanate to titrate test sample (4.4)

V_b = mL of ammonium thiocyanate to titrate blank (4.5)

c = molarity of ammonium thiocyanate solution, mol/L (3.4)

6.0 PRECISION

The expected range of results is 200-8,000 mg/L chloride. The precision of the method has not been determined.

7.0 REFERENCE

- (1) Anon. (1980). Standard Test Method TM36-1.7-1. Chloride (as NaCl) ICI Aust. Ltd., 27/10/80
- (2) Ivin, P.C. and Doyle, C.D. (1978). Proc. Aust. Soc. of Sugar Cane Technol. 267-272.



METHOD 43

CHLORIDE — DETERMINATION IN WATER

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry. The presence of chloride ions indicates saline contamination in factory cooling water. Chlorides are relatively unreactive and the result can be used in a calculation of % blowdown.

2.0 PRINCIPLE OF METHOD

This method is based on the standard Mohr's filtration procedure.

The sample is adjusted to between 7 and 10 pH and titrated with silver nitrate in the presence of potassium chromate to indicate the end-point. Silver chloride is precipitated quantitatively before red silver chromate is formed.

Substances in the amounts normally found in boiler waters will not interfere. Sulphide, thiosulphate, and sulphite ions interfere and are removed by treatment with hydrogen peroxide.

3.0 REAGENTS

Unless otherwise stated, use only analytical grade reagents and only distilled or deionised water. (Distilled water that has been further treated by deionisation is preferred).

3.1 Potassium chromate indicator solution. Dissolve 50g K_2CrO_4 in a little water. Add $AgNO_3$ solution until a definite red precipitate is formed. Let stand 12 hours, filter and dilute to 1L with distilled water.

3.2 Standard silver nitrate solution, 0.02 mol/L. Dissolve 3.3980 g $AgNO_3$ in water and dilute to 1L. Standardize by titrating 25 mL 0.02 mol/L sodium chloride with the silver nitrate solution following the procedure in steps 5.1-5.5 below. Store in a brown bottle.

Calculate the concentration of the standard silver nitrate, c in moles per litre :

$$c = \frac{25 \times 0.02}{\text{Volume } AgNO_3 \text{ titre} - \text{Volume } AgNO_3 \text{ blank titre}}$$

3.3 Standard sodium chloride solution, 0.02 mol/L. Dissolve 1.1680 g NaCl (dried at 100-110 °C for 3 hours) in water and dilute to exactly 1L.

3.4 Sulphuric acid solution, 0.01 mol/L.

3.5 Hydrogen peroxide, 30%.

3.6 Phenolphthalein solution, 1 g/100 mL. Dissolve 1 g phenolphthalein in 50 mL ethanol plus 50 mL distilled water.

4.0 SAMPLING AND SAMPLES

4.1 Sample bottles. Use glass or polythene bottles prepared for sampling as follows —

- (a) Wash the bottle and cap with detergent and tap water,
- (b) Rinse thoroughly with tap water,
- (c) Rinse twice with distilled water,
- (d) Drain thoroughly and replace cap.

4.2 Sampling. Before sampling, thoroughly flush all distribution lines to ensure a representative sample. Rinse the sample bottle 2-3 times with the sample water then completely fill the container and replace cap. Boiler waters collected under pressure should be cooled to about 20 °C while still under pressure.

5.0 PROCEDURE

A. SAMPLE PREPARATION

- 5.1 Filter about 200 mL of the sample through a Whatman No. 41 paper. For boiler waters, pipette 50 mL of the filtered water into a 200 mL conical flask and add 50 mL of distilled water. For soft waters (boiler feed water), pipette 100 mL into a 250 mL conical flask.
- 5.2 If sulphite is present, add 1 mL H_2O_2 and stir for 1 minute.

B. TITRATION

- 5.3 Directly titrate samples in the pH range 7 to 10. Add 1-2 drops of phenolphthalein and adjust to pH 7-10 with 0.01 mol/L H_2SO_4 .
- 5.4 Add 1.0 mL of K_2CrO_4 indicator solution. Titrate (over a white surface) with standard 0.02 mol/L $AgNO_3$ solution to a persistent pinkish-yellow end-point. Record the volume of $AgNO_3$ used, (V_1) to 0.1 mL.
- 5.5 Determine a reagent blank by titrating 100 mL distilled water as in 5.3-5.4 above. Record the volume of $AgNO_3$ used, (V_b) to 0.1 mL.

6.0 CALCULATION

6.1 Calculate chloride, (mg/L) in sample.

$$\text{Chloride (mg/L)} = \frac{(V_1 - V_b) \times c \times 35450}{V}$$

where: V = mL of test sample (5.1)

V_1 = mL of $AgNO_3$ to titrate test solution (5.4)

V_b = mL of $AgNO_3$ to titrate blank (5.5)

c = Concentration (mol/L) of standard $AgNO_3$ solution (3.2)

Record the result as chloride in water to 1 mg/L.

7.0 PRECISION

Sample	Concentration Range (mg/L)	95% Repeatability r	95% Reproducibility R
Boiler water	20-50	8	25
Softened water	100-50	8	25

8.0 REFERENCES

1. Standard Methods for the Examination of Water and Waste Water; 16th Edition; American Public Health Association (1985).
2. Australian Standard 2031.1-1986; Selection of Containers and Preservation of Water Samples for Chemical and Microbiological Analysis — Part 1 — Chemical.



METHOD 44

SOLUBLE PHOSPHATE — DETERMINATION IN WATER BY THE AMIDOL METHOD

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use in the Australian Sugar Industry. Phosphate is present in water in three main forms, orthophosphate (soluble phosphate), condensed phosphate and organophosphorus compounds. This procedure measures soluble phosphate only.

2.0 PRINCIPLE OF METHOD

The test sample is reacted with ammonium molybdate, under acidic conditions, to form phosphomolybdic acid which is reduced to molybdenum blue by amidol and sodium metabisulphite. The absorbance of the blue colour is measured in a spectrophotometer at 660 nm.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

3.1 Spectrophotometer, suitable for the measurement of absorbance at 660 nm with matched 2 cm cells.

3.2 Automatic dispensers, 4 mL and 10 mL capacities.

4.0 REAGENTS

Unless otherwise stated, use only analytical grade reagents and only distilled or deionised water.

4.1 Acid molybdate reagent.

4.2 Acid reagent

4.3 Amidol reagent

NOTE: Directions for preparing these reagents is given in the method: "Acid Soluble Phosphate — Determination in Raw Sugar by the Amidol Method".

5.0 STANDARDISATION

The method is standardised against a standard solution prepared from dried potassium dihydrogen phosphate. The calibration graph, covering the range 0 to 0.12 mg phosphate, prepared for Method No. 38 "Acid Soluble Phosphate — Determination in Raw Sugar by the Amidol Method" is also used with this method.

6.0 SAMPLES AND SAMPLING

6.1 Sample containers. Glass bottle with stopper which has been prepared by washing with hot, dilute hydrochloric acid and rinsing well with distilled water. Do not use detergent which may contain phosphate.

6.2 Sampling. Before sampling, thoroughly flush all distribution lines to ensure a representative sample. Rinse the container 2-3 times then completely fill with sample and seal. If the sample is to be stored for more than 4 hours add 1 mL of concentrated hydrochloric acid ($\rho_{20} = 1.18$ g/mL), per litre.

7.0 PROCEDURE

NOTE: Wash all glassware with hot dilute HCl and rinse well with distilled water. Do not use detergents.

7.1 Pipette 5.0 mL of boiler water or 10.0 mL of softened water into each of two 50 mL volumetric flasks. Add 2-3 drops of phenolphthalein indicator and, if a pink colour develops, add acid reagent to discharge the colour.

7.2 To one flask, add with automatic dispensers 10 mL of acid molybdate reagent (4.1) and 4 mL of amidol reagent. Make to 50 mL with distilled water, stopper and mix well. This is the test solution.

7.3 To the second flask add 10 mL of acid reagent, dilute to 50 mL with distilled water, stopper and mix well. This is the blank solution.

7.4 After at least 10 minutes and no longer than 30 minutes read the absorbance of the test solution (7.2) in a 2 cm cell, in a spectrophotometer at 660 nm using the corresponding test blank solution (7.3) as the reference. Record the absorbance to 0.001 units.

8.0 CALCULATION

8.1 Read from the standard graph (5.0) mg of phosphate, P in the 50 mL of test solution (7.2).

8.2 Calculate soluble phosphate as P (mg/L) in the sample.

$$P(\text{mg/L}) = \frac{\text{mg P (from graph)}}{\text{sample aliquot}} \times 1000$$

8.3 Calculate soluble phosphate as PO_4 (mg/L)

$$\text{PO}_4 (\text{mg/L}) = P (\text{mg/L}) \times 3.1$$

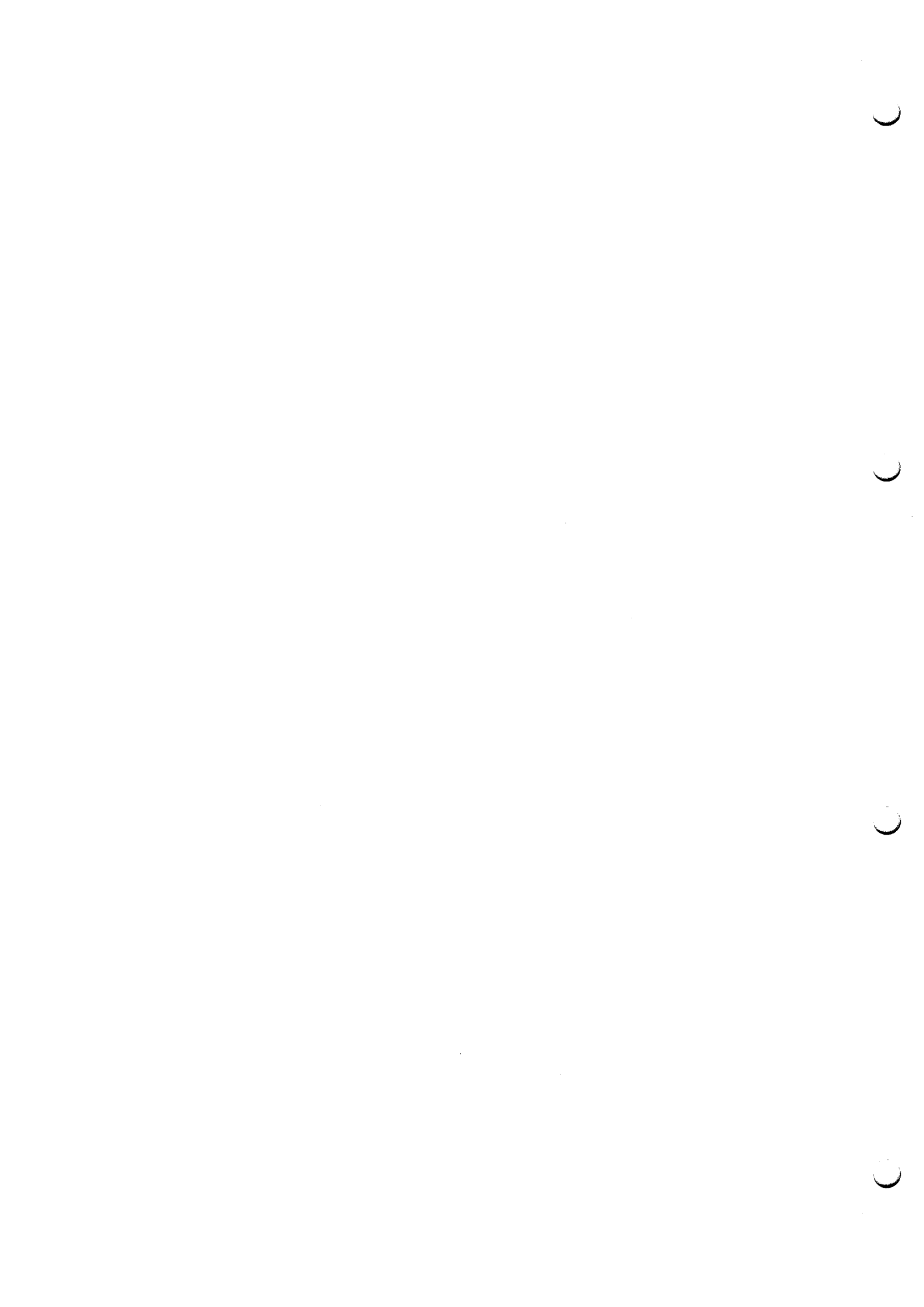
Record the result as soluble phosphate PO_4 (mg/L) to the nearest mg/L.

9.0 PRECISION

Sample	Concentration Range PO_4 (mg/L)	95%	95%
		Repeatability r	Reproducibility R
Boiler water	30 — 100	8	Not determined
Softened water	0 — 10	Not determined	Not determined

10.0 REFERENCES

- 10.1 British Standard Methods of Testing; Water used in Industry, Part 105. Soluble phosphate and organophosphorus compounds. BS 2690 : Part 105 : (1983).
- 10.2 Standard Methods for the Examination of Water and Waste Water; 16th Edition; American Public Health Association (1985).



METHOD 45

TOTAL DISSOLVED SOLIDS (TDS) — ESTIMATION IN WATER BY CONDUCTIVITY MEASUREMENT

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry. The results must be treated with some caution unless the conductivity meter is regularly standardised against solids by drying, as the relationship between conductivities and TDS will vary with differing composition of contaminants.

2.0 PRINCIPLE OF METHOD

The electrical conductivity of the test sample is measured under standard conditions using a conductivity meter calibrated in microsiemens per metre, S/m, (previously mhos/cm). Total dissolved solids is estimated by multiplying by an empirical factor. (Conductivity meters giving direct readout of total dissolved solids are available).

Total Dissolved Solids by Hydrometer, an alternative method, involves the use of a TDS hydrometer supplied by a commercial firm. Results from the hydrometer should also be checked regularly against solids by drying.

3.0 APPARATUS

3.1 **Conductivity meter**, with direct readout of total dissolved solids (mg/L) and with automatic temperature compensation.

4.0 REAGENTS

Unless otherwise stated use only analytical grade reagents. Distilled water which has been further purified by passing through a mixed bed deioniser is preferred. Conductivity of the distilled water should be less than 0.01 μ S/m (1 μ mho/cm).

4.1 **Phenolphthalein indicator** solution, 1 g/100 mL.

Dissolve 1 g phenolphthalein in 50 mL ethanol plus 50 mL of distilled water.

4.2 Acetic acid $\rho_{20} = 1.06$ g/mL.

5.0 SAMPLES and SAMPLING

5.1 Sample bottles. Use polythene bottles prepared for sampling as follows:

- (a) Wash bottle and cap with detergent and tap water.
- (b) Rinse thoroughly with tap water.
- (c) Rinse twice with distilled water.
- (d) Drain thoroughly and replace cap.

5.2 Sampling. Before sampling, thoroughly flush all distribution lines to ensure a representative sample. Rinse the sample bottle 2-3 times with the sample water then completely fill the container and replace cap. Boiler water collected under pressure should be cooled to about 20 °C while still under pressure. Conductivity should be measured within 4 hours of sampling.

6.0 PROCEDURE

6.1 Filter the cooled sample through a Whatman No. 1 filter paper.

6.2 Add 1-2 drops of phenolphthalein solution and if the water turns pink neutralise by adding acetic acid dropwise until the colour disappears.

6.3 Rinse and then fill the conductivity cell with sample.

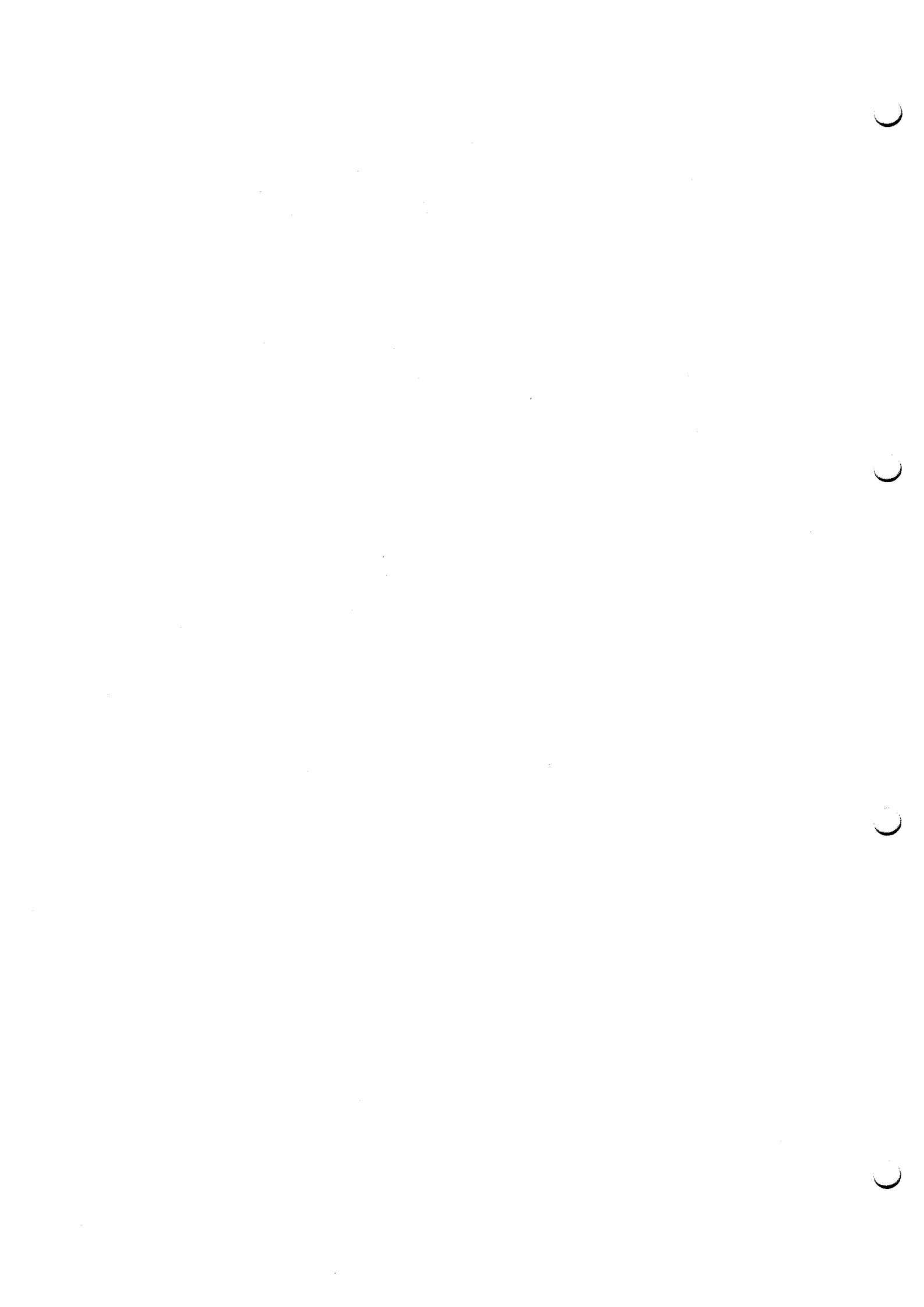
6.4 Set the meter to read total dissolved solids and record the reading. (Refer to the operating instructions for the conductivity meter).

6.5 Record the total dissolved solids to 1 mg/L.

7.0 PRECISION

The expected range of results is 100-15,000 mg/L.

The precision of the method has not been determined.



METHOD 46

SULPHITE — DETERMINATION IN WATER

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry. Sulphite ions (SO_3^{2-}) may occur in boiler and boiler feed water treated with sulphite for dissolved oxygen control.

2.0 PRINCIPLE OF METHOD

A test sample of the water is acidified and titrated with standardised potassium iodide-iodate reagent. Free iodine liberated by the iodide-iodate reagent reacts with sulphite ions. When all the sulphite is oxidised an excess of free iodine occurs which forms a blue colour with water soluble starch indicator.

3.0 REAGENTS

Unless otherwise stated, use only analytical grade reagents and only distilled or deionised water.

3.1 **Standard potassium iodide-iodate solution**, 0.0125 mol/L. Dissolve 0.4458 g of anhydrous potassium iodate (dried for 4 hours at 120 °C), 4.35 g potassium iodide and 0.31 g sodium bicarbonate in distilled water and dilute to 1 litre.

3.2 **Starch indicator**. Use a commercial soluble starch indicator (e.g. Vitex).

4.0 SAMPLING AND SAMPLES

4.1 **Sample containers**. Use borosilicate glass containers prepared as follows :

- (a) wash the container and cap with detergent and tap water,
- (b) rinse thoroughly with tap water,
- (c) rinse twice with distilled water,
- (d) drain thoroughly and replace cap.

4.2 Before sampling, thoroughly flush all distribution lines to ensure a representative sample. Rinse the sample containers 2-3 times with the sample water then completely fill the containers and replace the cap. Take care to minimise contact with the air.

Boiler waters collected under pressure should be cooled to about 20 °C while still under pressure to minimise the loss of dissolved gases.

5.0 PROCEDURE

5.1 Measure 100 mL of sample into a 250 mL conical flask.

5.2 Add 0.5 mL of concentrated hydrochloric acid, $\rho_{20} = 1.16$ g/mL. Use a graduated (barrel) pipette, with safety bulb.

5.3 Add, with a spatula, about 0.5 g of soluble starch indicator.

5.4 Titrate, over a white background, with potassium iodide-iodate reagent until a permanent blue tinge appears. Record the volume of iodide-iodate reagent used (V_1) to 0.1 mL.

5.5 Carry out a blank titration with 100 mL of distilled water following steps 5.2-5.4. Record the volume of iodide-iodate reagent used, V_b , to 0.1 mL.

6.0 CALCULATION

Calculate the sulphite in water as mg/L SO_3^{2-}

$$\text{SO}_3^{2-} \text{ (mg/L)} = \frac{(V_1 - V_b) \times c \times 40\,000}{V}$$

where: V = mL sample taken

V_1 = mL iodide-iodate reagent to titrate sample (5.4)

V_b = mL iodide-iodate reagent to titrate blank (5.5)

c = conc. (mol/L) of iodide-iodate reagent (3.1)

For a 100 mL sample aliquot

$$\text{SO}_3^{2-} \text{ (mg/L)} = (V_1 - V_b) \times 5$$

Record the result as mg/L, SO_3^{2-} to 1 mg/L

7.0 PRECISION

Sample	Concentration Range SO_3^{2-} (mg/L)	95% Repeatability r	95% Reproducibility R
Boiler waters	3-30	5	8



METHOD 47

TOTAL HARDNESS (CALCIUM AND MAGNESIUM) — DETERMINATION IN WATER BY EDTA TITRATION

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry. Calcium and magnesium ions contribute to the formation of sludge and scale in water systems, and are removed by water treatment.

2.0 PRINCIPLE OF METHOD

The pH of the test sample is adjusted to a value of 10 by adding buffer solution, and the sample titrated with EDTA, using Eriochrome Black T indicator. The method measures the total content of calcium plus magnesium in the water.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

3.1 **Porcelain Basin** (evaporating basin), 25-30 cm dia.

3.2 **Magnetic stirrer**

4.0 REAGENTS

Unless otherwise stated, use only analytical grade reagents and only distilled or deionised water.

4.1 **Ammonium buffer solution**, dissolve 16.9 g ammonium chloride in 143 mL of concentrated ammonium hydroxide, $\rho_{20} = 0.88$ g/mL. Add 1.25 g magnesium salt of EDTA and dilute to 250 mL with water.

If the magnesium salt of EDTA is unavailable, dissolve 1.129 g of disodium salt of EDTA and 0.780 g magnesium sulphate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) in 50 mL water. Add this solution to 16.9 g ammonium chloride and 143 mL concentrated ammonium hydroxide, $\rho_{20} = 0.88$ g/mL. Mix well and dilute to 250 mL with distilled water.

Store the solution in a tightly sealed plastic bottle. Discard after 1 month.

4.2 **Eriochrome Black T** indicator. Dissolve 0.50 g eriochrome black in about 2 mL water, add 10 g sodium chloride and mix well. Dry for 1-2 hours at 100-105 °C. Add 90 g sodium chloride and grind until thoroughly mixed.

NOTE: Prepared dry indicator is available commercially (total hardness indicator tablets).

4.3 **Standard EDTA solution**, 0.01 mol/L. Dissolve 3.723 g EDTA in water and dilute to 1 litre in a volumetric flask. Store in a sealed polythene container. Standardise against standard calcium solution (4.4) by titrating 10.0 mL standard calcium solution, plus 80 mL distilled water, 2 mL ammonium buffer solution and 0.2 g of total hardness indicator with EDTA as described in steps 6.2-6.4 below.

Calculate the concentration, c of EDTA solution in moles per litre.

$$c = \frac{0.1}{\text{volume EDTA}}$$

4.4 **Standard Calcium Solution**, 1 mg/mL CaCO_3 . Weigh 1.000 g anhydrous calcium carbonate into a 500 mL

conical flask. Place a funnel in the neck of the flask and add 5 mol/L hydrochloric acid slowly until all the CaCO_3 is dissolved. Add about 200 mL water and boil 3-5 minutes to expel CO_2 . Cool, add 2-3 drops methyl red indicator and adjust the pH to the intermediate orange colour by adding 5 mol/L HCl or 3 mol/L NH_4OH as required. Transfer quantitatively to a 1L volumetric flask and dilute to volume with water.

5.0 SAMPLING AND SAMPLES

5.1 **Sample containers**, use polythene containers prepared as follows :

(a) Wash the container and cap with detergent and tap water,

(b) Rinse thoroughly with tap water,

(c) Rinse twice with distilled water,

(d) Drain thoroughly and replace cap.

5.2 **Collection and preservation**. Before sampling thoroughly flush all distribution lines to ensure a representative sample. Rinse the sample container 2-3 times with the sample then completely fill with sample and seal. Immediately store between 1 °C and 4 °C or acidify by adding 1 drop of concentrated nitric acid, $\rho_{20} = 1.42$ g/mL.

6.0 PROCEDURE

6.1 With a measuring cylinder, transfer an aliquot (V) of the test sample to a porcelain dish

For softened waters, transfer 250 to 500 mL

For raw waters, transfer 50 to 100 mL

NOTE: The sample volume selected should require less than 15 mL of EDTA titrant and the titration should be completed within 5 minutes of adding buffer.

6.2 Add 2.0 mL of the ammonium buffer (4.1) for raw waters and 5.0 mL for softened waters. Mix well.

6.3 Add 0.2 g of total hardness indicator (4.2) or 0.4 g of indicator for softened waters. Mix well.

NOTE: For samples larger than 250 mL, increase the amounts of buffer and indicator proportionately.

6.4 Add EDTA (4.3) slowly with continuous stirring, until the last reddish tinge disappears [at the end-point the indicator changes from wine red to pale blue]. Record the volume of EDTA used, (V₁) to 0.1 mL.

7.0 CALCULATION

7.1 Calculate the total hardness of the sample expressed as mg/L CaCO_3 .

$$\text{Hardness as } \text{CaCO}_3(\text{mg/L}) = \frac{V_1 \times c \times 100 \times 1000}{V}$$

where: V = mL of sample (6.1)

V₁ = mL of EDTA titrant (6.4)

c = concentration of standard EDTA solution, moles/L (4.3)

METHOD 47 P.2.

8.0 PRECISION

Sample	Concentration Range (mg/L, as CaCO ₃)	95% Repeatability r	95% Reproducibility R
Softened water			
Boiler feed water	0-50	8	40

9.0 REFERENCE

Standard methods for the Examination of Water and Waste Water; 16th Edition; American Public Health Association (1985).

METHOD 48

DISSOLVED OXYGEN (D.O.) — DETERMINATION IN WATER BY THE AZIDE MODIFICATION

1.0 SCOPE AND FIELD OF APPLICATION

This method is recommended for use within the Australian Sugar Industry. The analysis for dissolved oxygen is a key test in water pollution and waste treatment process control. This procedure is used also to determine dissolved oxygen, before and after incubation, in the determination of biological oxygen demand (B.O.D.).

2.0 PRINCIPLE OF METHOD

A divalent manganese solution, followed by a strongly alkaline solution, is added to the test sample. Dissolved oxygen oxidises an equivalent amount of the divalent manganous hydroxide precipitate to hydroxides of higher valency states. In the presence of iodide ions in acid solution, the oxidised manganese reverts to the divalent state, liberating iodine equivalent to the D.O. content. The iodine is titrated with standard thiosulphate solution.

Sodium azide is added if the sample contains more than 0.1 mg/L of nitrite N and not more than 1 mg/L of ferrous Fe. Other reducing or oxidising materials should be absent.

3.0 REAGENTS

3.1 Manganous sulfate solution. Dissolve 480 g $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$, or 400 g $\text{MnSO}_4 \cdot 2\text{H}_2\text{O}$, or 364 g $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ in distilled water, filter, and dilute to 1 L. The MnSO_4 solution should not give a colour with starch when added to an acidified potassium iodide (KI) solution.

3.2 Alkaline iodide-sodium azide solution. Dissolve 500 g NaOH (or 700 g KOH) and 135 g NaI (or, 150 g KI) in water, dilute to about 950 mL and cool. Slowly with stirring add a solution of 10 g NaN_3 in 40 mL water. Store in a dark bottle.

This reagent should not give a colour with starch indicator when diluted and acidified.

Safety

Avoid contact with skin and eyes. Toxic if swallowed. Wear gloves and safety glasses. In cases of contact with skin or eyes wash with large quantities of cold water. If swallowed, give about 250 mL of cold water. Seek immediate medical advice. Contact with acids liberates a very toxic gas.

3.3 Sulphuric acid, concentrated $\rho_{20} = 1.84 \text{ g/mL}$.

Safety

Concentrated sulphuric acid is very corrosive. Safety glasses and gloves must be worn when handling the acid. It must not be pipetted by mouth.

3.4 Sodium thiosulfate standard solution, 0.025 mol/L. Dissolve 6.205 g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in water. Add 0.4 g solid NaOH and dilute to 1 L in a volumetric flask. Standardise against 0.0021 mol/L potassium bi-iodate solution (3.5). Dissolve about 2 g KI in 100-150 mL water in a conical flask. Add carefully, 2-3 drops of concentrated H_2SO_4 and 20.00 mL potassium bi-

iodate solution. Titrate with sodium thiosulfate solution, to a pale straw yellow colour. Add starch indicator and continue titration to disappearance of blue colour.

Calculate the concentration of the sodium thiosulfate solution (c), in moles/L —

$$c = \frac{0.0021 \times 12 \times 20}{\text{volume Na}_2\text{S}_2\text{O}_3}$$

NOTE: It is convenient to adjust the concentration of thiosulfate solution to exactly 0.025 mol/L.

3.5 Potassium bi-iodate standard solution, 0.0021 mol/L. Dissolve 0.8125 g $\text{KH}(\text{IO}_3)_2$ in water in a 1 L volumetric flask and dilute to exactly 1 litre.

3.6 Starch indicator. Use an aqueous solution or a soluble starch powder.

3.7 Potassium fluoride, 40 g/100 mL solution of $\text{KF} \cdot 2\text{H}_2\text{O}$.

Safety

Toxic by inhalation, contact with skin and if swallowed. Wear gloves and safety glasses. If in contact with skin or eyes wash with large quantities of cold water. If swallowed, give about 250 mL water to dilute in stomach. Do not induce vomiting. Seek immediate medical advice.

4.0 SAMPLES AND SAMPLING

4.1 Sample bottles. Use narrow neck, glass stoppered, glass bottles, 250-300 mL, prepared for sampling as follows:

- (a) Wash bottle and stopper with detergent and tap water,
- (b) Rinse thoroughly with tap water,
- (c) Rinse with nitric acid (1+1),
- (d) Rinse twice with distilled water,
- (e) Drain and replace stopper.

4.2 Sampling. Collect samples very carefully. Do not let sample be agitated or remain in contact with air. Avoid entraining air or dissolving atmospheric oxygen.

Rinse the bottle by filling 2-3 times with sample. When sampling from a line under pressure attach a rubber tube and extend to the bottom of the bottle and allow the bottle to overflow 2-3 times its volume. Immediately replace stopper to exclude air.

Samples should be analysed immediately or stored for up to 4 hours after fixing by adding manganous sulphate solution, alkali iodide solution and H_2SO_4 as in 5.1 below.

5.0 PROCEDURE

NOTE: Add all reagents, except H_2SO_4 , well below surface of sample from 10 mL graduated pipettes. Rinse pipettes before returning to reagent bottles.

5.1 Add 1 mL MnSO_4 solution (3.1) and 1 mL alkali-iodide-azide reagent (3.2) to the sample collected in the 250-300 mL bottle (4.2).

5.2 Stopper carefully to exclude air and mix by inverting several times.

METHOD 48 P.2.

- 5.3 After the precipitate has settled, leaving about 100 mm clear supernatant, remove stopper and add 1 mL of H_2SO_4 (3.3) down neck of bottle [If >100 mg/L ferric, (Fe^{3+}), iron is present add 1.0 mL KF solution (3.7) before acidifying].
- 5.4 Re-stopper and mix by inverting until precipitate is completely dissolved.
- 5.5 Immediately titrate 201 mL (1 mL is allowance for added reagents) with 0.025 mol/L $\text{Na}_2\text{S}_2\text{O}_3$ solution (3.4) to a pale straw colour. Add starch indicator (3.6) and continue titration to disappearance of blue colour. Record the volume of $\text{Na}_2\text{S}_2\text{O}_3$ used to 0.1 mL.

6.0 CALCULATION

$$\text{Dissolved oxygen, (mg/L)} = V \times \frac{c}{0.025}$$

where: V = mL, $\text{Na}_2\text{S}_2\text{O}_3$ used in titration (5.5)
c = conc. (moles/L) of $\text{Na}_2\text{S}_2\text{O}_3$ solution (3.4)

Record dissolved oxygen in sample to 1 mg/L.

7.0 PRECISION

The precision of the method has not been determined. As a guide the standard deviation for D.O. in waste waters is about 60 mg/L but can be much higher, due to bad sampling, suspended organic solids or heavy pollution.

8.0 REFERENCES

1. Standard Methods for the Examination of Water and Waste Water, 16th Edition, (1985); American Public Health Association.
2. Official Methods of Analysis, 14th Edition, (1984); Association of Official Analytical Chemists; U.S.A.

METHOD 49

SUCROSE SOLUBLE ALKALI — DETERMINATION IN LIME

1.0 SCOPE AND FIELD OF APPLICATION

This method has been adopted officially within the Australian Sugar Industry. The method is used to determine the alkali (expressed as CaO) in a sample of rock lime or slaked lime, which is available to react with sucrose to form a soluble calcium saccharate. It is a measure of the quality of lime purchased for use in juice clarification in mills or carbonation in refineries.

2.0 PRINCIPLE

The sample is completely ground to less than 150 microns size. A small amount of ethanol is used to transfer the weighed sample into a flask, where it is mixed with a known volume of 10% sucrose solution. The flask is then shaken for 30 minutes and the suspended matter is allowed to settle out. The solution is coarse filtered, and an aliquot titrated with hydrochloric acid solution to a methyl orange end-point.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

3.1 Mortar and pestle, porcelain.

3.2 Standard sieve, 150 micron.

3.3 Mechanical flask shaker, to take 200 mL volumetric flask.

4.0 REAGENTS

Unless otherwise specified, use only analytical grade reagents and only distilled water which has been freed of carbon dioxide by boiling for 15 minutes and cooled to room temperature immediately before use.

4.1 Sucrose solution, 100 g/L. Dissolve 100 g sucrose in CO₂ free water and dilute to exactly 1 litre in a volumetric flask. Prepare the solution daily.

4.2 Methyl Orange Indicator Solution, 500 mg/L. Dissolve 0.5 g methyl orange in 1L of distilled water. Filter if necessary.

4.3 Hydrochloric Acid Standard Solution 0.2 mol/L. Prepare by diluting commercial concentrated volumetric solution with CO₂ free distilled water. Standardise weekly against sodium carbonate.

Dry about 2 g Na₂CO₃ at 260 °C for 30 minutes, and cool in a dessicator. Accurately weigh about 0.4 g dried Na₂CO₃ and transfer to a 250 mL conical flask. Record the weight of Na₂CO₃ to 0.1 mg. Add 3 drops of methyl orange, and titrate with the 0.2 mol/L hydrochloric acid. Record the titre to 0.1 mL.

Calculate the concentration, (c) in moles/L of the HCl

$$c = \frac{\text{g Na}_2\text{CO}_3 \times 1000}{\text{mL Acid} \times 52.994}$$

4.4 95% Ethyl alcohol. Dilute 95.0 ± 0.1 mL ethyl alcohol in a 100 mL volumetric flask to 100 mL with water and mix thoroughly.

5.0 PREPARATION OF SAMPLE

The grinding operating should be carried out as

quickly as possible to minimise reaction with atmospheric carbon dioxide.

5.1 For rock lime or slaked lime, grind approximately 20 g of well mixed sample using a mortar and pestle. Mix the sample thoroughly using a flat-bladed spatula. Cone and quarter, mix the two diagonally opposite portions, and discard the remaining sample.

5.2 Place the sample into a 150 micron sieve (with lid and base) and shake by hand until all material smaller than 150 micron has passed through.

5.3 Regrind sample larger than 150 micron and shake again. Repeat until all sample passes through mesh. Mix thoroughly and store in a clean, dry, well sealed container.

6.0 PROCEDURE

The analysis should be performed in duplicate using separately prepared lime samples. The titration of each sample should also be done in duplicate.

6.1 Weigh exactly 0.800 g of the finely ground lime and wash it into a clean, dry 200 mL volumetric flask with 4.00 ± 0.05 mL of 95% ethyl alcohol. To prevent lumping mix the sample and alcohol carefully, so that the sample is completely wet with alcohol before transferring to the flask.

6.2 Add 160 ± 1 mL of 10% sucrose solution (4.1), stopper and shake on a mechanical flask shaker for 30 minutes ± 30 secs. Let the flask stand for a few minutes to allow suspended matter to settle out. Dilute to the mark with 10% sucrose solution and mix thoroughly.

6.3 Filter through a coarse filter paper and reject the first 20 mLs of filtrate. Use the filtrate to rinse the filter flask.

6.4 Pipette a 50.0 mL aliquot of the filtrate into a 250 mL conical flask, without delay to avoid reaction with CO₂.

6.5 Add 4 drops of methyl orange indicator, and titrate with 0.2 mol/L HCl from a 50 mL burette until the clear yellow solution changes to a salmon colour. Over-titration leads to an orange colour.

Record the volume, (v) of 0.2 mol/L HCl used to 0.1 mL.

7.0 CALCULATIONS

1 mL of 1 mol/L HCl is equivalent to 0.02804 g of CaO.

$$\begin{aligned} & \% \text{ sucrose soluble alkali, as CaO} \\ &= \frac{v \times c \times 0.02804 \times 200 \times 100}{50 \times 0.8000} \\ &= v \times c \times 14.02 \end{aligned}$$

where v = volume of HCl used in the titration (6.5)

c = concentration of HCl, (moles/L) (4.3)

Record the % sucrose soluble alkali as CaO to 0.01% sucrose.

METHOD 49 P.2.

8.0 PRECISION

Sample	Concentration Range (%)	95% Repeatability r	95% Reproducibility R
Quicklime	70-97	0.33	not determined
Slaked lime	60-70	0.33	not determined

METHOD 50

SUCROSE — DETERMINATION IN EFFLUENTS BY THE PHENOL-SULPHURIC ACID METHOD

1.0 SCOPE AND FIELD OF APPLICATION

This method gives a semi-quantitative measure of sugar and sugar degradation products in factory effluents. The prime objective of the analysis is a measure of sucrose loss by leakage or entrainment into condensate streams and cooling waters in the factory.

A limitation imposed on the method is that it also effectively measures furfuraldehyde and its homologues. Furthermore, it has been established that α - and β - keto acids and aliphatic aldehydes and ketones give a yellow colour with this reagent and they will interfere to a varying degree in the determination of carbohydrates.

2.0 PRINCIPLE

An aliquot of the test solution is mixed with the phenol reagent in a test tube and a measured volume of concentrated sulphuric acid added. The acid reacts with sucrose to give a furfural derivative which then combines with phenol to give an orange condensation product. The concentration of sucrose in the test solution is obtained by measuring the absorbance of light at 488 nm and then reading off the concentration from a previously prepared graph of absorbance versus sucrose concentration.

3.0 APPARATUS

Ordinary laboratory apparatus and glassware and

- 3.1 Pyrex test tubes, 20 mm x 150 mm
- 3.2 Volumetric flasks, Class A, 100 mL, 1L
- 3.3 Bulb pipettes, Class A, 0.5 mL, 1 mL, 2 mL, 3 mL, 4 mL, 6 mL, 8 mL
- 3.4 Automatic dispenser, to dispense 5.0 ± 0.03 mL sulphuric acid
- 3.5 Vortex mixer
- 3.6 Spectrophotometer, suitable for the measurement of absorbance at 488 nm, and 1 cm matched cuvettes.

4.0 REAGENTS

Unless otherwise specified, use only analytical grade reagents and only distilled water.

- 4.1 Sulphuric Acid, conc. ($\rho_{20} = 1.84$ g/mL)

Safety

Safety glasses and gloves must be worn. Sulphuric acid is best dispensed behind a perspex safety shield.

- 4.2 Phenol Reagent, 50 ± 1 g/L solution in water*. (This should be stored in a dark bottle).

Safety

Avoid contact with the skin and breathing the vapour.

- 4.3 Standard Sucrose, 1000 mg/L solution.
Dissolve 1.000 ± 0.001 g sucrose in water and dilute to 1 litre with water.

- 4.4 Working Standard Sucrose Solution. Pipette 0.50, 1.0, 2.0, 3.0, 4.0, 6.0 and 8.0 mL of the 1000 mg/L standard sucrose solution into separate 100 mL volumetric flasks and make to the mark with water. This will give standard dilutions of 5, 10, 20, 30, 40, 60 and 80 mg/L of sucrose.

5.0 STANDARDISATION OF METHOD

- 5.1 Pipette 1.00 ± 0.015 mL of water into a test tube. This will be the 0 mg/L standard or blank required for zeroing the spectrophotometer.
- 5.2 Pipette 1.00 ± 0.015 mL of each of the working standards respectively into seven test tubes. These will correspond to 5, 10, 20, 30, 40 and 80 mg/L of sucrose.
- 5.3 Add 1.00 ± 0.015 mL of the phenol reagent (4.2) to each tube and mix.

Safety

Avoid contact with the skin and breathing the vapour.

- 5.4 Add, with an automatic dispenser, 5.0 mL of concentrated sulphuric acid to each test tube. Add the acid in a continuous jet into the centre of the liquid, while holding the test tube in a vertical position. Do not allow the acid to run down the sides of the tube.

Safety

Safety glasses and gloves must be worn.

- 5.5 Using a vortex mixer, mix the contents of each test tube immediately, then stand the solutions for at least 30 minutes to allow the colour to develop.
After addition of the acid, the temperature rises spontaneously to approximately 110 °C and the heat so generated completes the development of the colour. The colour is stable for several hours.
- 5.6 Determine the absorbance of each standard solution in 1 cm cells in a spectrophotometer at 488 nm, with the 0 mg/L standard (blank) as reference.
- 5.7 Plot the absorbance for each standard against its sucrose content (mg/L) and draw a straight line of best fit. It should pass through the origin. If two or more points lie off the line of best fit by more than 5% of the line's absorbance value at that particular sugar concentration, the standardisation must be repeated.

6.0 PROCEDURE

In collecting samples, care must be taken to avoid contamination. Samples should be tested as soon as possible after being taken to avoid the risk of decomposition of sucrose in these dilute solutions. Determination in duplicate reduces errors and allows exclusion of gross anomalies resulting from accidental contamination.

- 6.1 Pipette 1.00 ± 0.015 mL of the test sample into a test tube.
- 6.2 Add 1.00 ± 0.015 mL of the phenol reagent (4.2) to the test tube and mix.

Safety

Avoid contact with skin and breathing the vapour.

* If solid phenol is not available, liquified phenol (commercial product, 30% phenol in water) can be diluted to 9.2% phenol (w/w), distilled as a two phase azeotrope (bp 99.5 °C), and diluted to 5%. This operation must be carried out in an efficient fume hood.

METHOD 50 P.2.

- 6.3 Add, with an automatic dispenser, 5.0 mL of concentrated sulphuric acid. Add the acid in a continuous jet into the centre of the liquid, while holding the test tube in a vertical position. Do not allow the acid to run down the sides of the test tube.

Safety

Safety glasses and gloves must be worn.

- 6.4 Using a vortex mixer, mix the contents of the test tube immediately and allow to stand for 30 minutes for colour to develop.
- 6.5 Determine the absorbance of the sample at 488 nm relative to the 0 mg/L standard.
A new 0 mg/L standard (blank) may have to be prepared if the old standard is more than a day old.
- 6.6 Estimate the sucrose content of the test sample from the plot of absorbance versus sucrose concentration. Record the result as mg/L sucrose in the test sample.
- 6.7 If the absorbance of the test solution exceeds that of

the highest standard, dilute an aliquot of the test sample with water and repeat the test.

7.0 CALCULATION

$$\text{Sucrose (mg/L)} = \frac{\text{estimated sucrose (mg/L)}}{\text{x dilution factor}} \quad (\text{see 6.6})$$

where:

$$\text{dilution factor} = \frac{\text{diluted volume}}{\text{test sample aliquot}} \quad (\text{see 6.7})$$

8.0 PRECISION

Expected range of results: 0-100 mg/L
95% repeatability (r): 2.3 ppm

9.0 REFERENCE

M. Dubois, K.A. Gilles, J.K. Hamilton, P.A. Rebers and F. Smith. *Anal. Chem.*, Vol. 28, No. 3, 1956, p. 350.

METHOD 51

BIOCHEMICAL OXYGEN DEMAND (BOD₅) — DETERMINATION IN LIQUID EFFLUENTS

1.0 SCOPE AND FIELD OF APPLICATION

This method is a standard method for BOD analysis and is prescribed by the Water Quality Council of Queensland for the determination of the quality of effluents discharged to a water-course.

It is applied also to a broad-spectrum assessment of the level of organic matter in other water discharges e.g. discharge to an effluent disposal plant or from a cooling tower. However, the test will under-estimate to varying degrees the level of organic matter and the result in these instances is best interpreted in comparison with an analysis of Chemical Oxygen Demand (COD).

2.0 PRINCIPLE OF METHOD

The consumption of oxygen or oxygen-uptake by microorganisms metabolising organic matter follows roughly a sigmoidal curve with respect to time. The rate of growth of microorganisms is temperature-dependent. Therefore, by convention, a five-day time period and temperature of 20 °C have been accepted as standard. The five-day BOD is about 70% of the ultimate oxygen demand which takes 20 to 30 days to be exerted.

The test requires a determination of the dissolved oxygen content of a sample at the time of preparation and after five days incubation. The determination of dissolved oxygen is done by the azide modification method (Method No. 48). The saturation level of dissolved oxygen in water at 20 °C is only 9.2 mg/L. Therefore, samples contaminated with high levels of organic matter must be diluted sufficiently to obtain a titratable level of oxygen after five days. Also, as nitrifying microorganisms could oxidise the ammonium ion present in the dilution water, inhibition of nitrification is recommended for samples likely to contain large numbers of these organisms. Such wastewaters are secondary effluents, aerobic ponds, polluted streams and any of these samples can be when used as seed (see 5.2).

3.0 APPARATUS

Ordinary laboratory apparatus and glassware (Brown ground-glass-stoppered reagent bottles are preferable for incubated samples) and

3.1 A refrigerator or water-bath, controlled at 20 °C which permits incubation of samples in the dark.

(NOTE: Minute quantities of sugar or molasses or bio-degradable detergents will result in inflated BOD readings. BOD glassware should be separate from normal laboratory materials and not washed with detergent, only rinsed thoroughly with tap water followed by distilled water).

4.0 REAGENTS

Unless otherwise specified, use only analytical grade reagents and only distilled water.

4.1 **Phosphate buffer solution.** Dissolve 8.5 g potassium dihydrogen phosphate, 21.75 g dipotassium hydrogen phosphate, 33.4 g disodium hydrogen phosphate heptahydrate, and 1.7 g ammonium chloride in about 500 mL distilled water and dilute to 1L. The pH should be 7.2.

4.2 **Magnesium sulphate solution.** Dissolve 22.5 g magnesium sulphate heptahydrate in distilled water and dilute to 1L.

4.3 **Calcium chloride solution.** Dissolve 27.5 g anhydrous calcium chloride in distilled water and dilute to 1L.

4.4 **Ferric chloride solution.** Dissolve 0.25 g of ferric chloride hexahydrate in distilled water and dilute to 1L.

4.5 **Sulphuric acid,** 0.1 mol/L solution.

4.6 **Sodium hydroxide,** 0.1 mol/L solution.

4.7 **Sodium sulphite solution,** 0.013 mol/L. 1.575 g sodium sulphite per L distilled water. Prepare daily.

4.8 **Nitrification inhibitor.** Reagent grade 2-chloro-6-(trichloromethyl) pyridine, (e.g. N-Serve, Dow Chemical Co., or Nitrification inhibitor 25-33, Hach Chemical Co.)

4.9 **Glucose-Glutamic acid solution.** Dry at 105 °C for one hour reagent grade glucose and glutamic acid. Add 150 mg glucose and 150 mg glutamic acid to distilled water and dilute to 1L. Prepare fresh immediately before use when required as a check solution.

5.0 PROCEDURE

(a) BOD₅

5.1 **Dilution Water.** Store the distilled water in cotton wool-plugged bottles at 20 °C for sufficient time to allow the water to become saturated with dissolved oxygen. Alternatively, aerate overnight with filtered air at 20 °C. Add 1 mL of phosphate buffer (4.1), magnesium sulphate (4.2), calcium chloride (4.3) and ferric chloride (4.4) solutions per L.

5.2 **The Addition of Seed.** In many samples, e.g. wastewater from a treatment plant or pond or the overflow from a cooling tower, and in river water, there are sufficient numbers of microorganisms to initiate oxidation of organic matter. However, frozen samples or samples with extreme pH or that were very hot will require 'seeding'. Suitable sources of 'seed' are a river water taken from below a waste discharge point or an effluent plant overflow. A suitable seed concentration is 1 to 5 mL/L. The 'seed' can be added to the sample or to dilution water for a large number of samples but must be analysed for BOD and corrected for. The DO uptake of the seeded dilution water should be between 0.7 and 1.0 mg/L.

5.3 **Sample Pre-Treatment.** Samples containing caustic alkalinity or acidity should be adjusted to pH 7.0, without diluting the sample by more than 0.5%. Samples supersaturated with oxygen e.g. due to algal activity should be brought to 20 °C and agitated by vigorous shaking. If the sample has been chlorinated, seed the dilution water. If the sample contains residual

METHOD 51 P.2.

chlorine, dechlorinate or preferably sample ahead of the chlorination process. Chlorine may dissipate on standing the sample for 1 to 2 hours in the light. If not, destroy the chlorine residual by adding Na_2SO_3 solution to the neutralised sample. Determine the required volume of Na_2SO_3 solution on a 100 to 1000 mL portion of neutralised sample, by adding 10 mL of 1 + 1 acetic acid or 1 + 50 sulphuric acid, 10 mL potassium iodide solution (10 g/100 mL) and titrating with 0.025 mol/L thiosulphate titrant to the starch-iodine end-point. Add the required volume of Na_2SO_3 to the sample and check for residual chlorine after 10 to 20 minutes.

If the nitrification inhibitor is desired, add 10 mg 2-chloro-6 (trichloro methyl) pyridine per litre of dilution water at the same time as adding nutrient and buffer solutions. Samples that may require nitrification inhibition include, but are not limited to, biologically treated effluents, samples seeded with such effluents and river waters. Note the use of the nitrification inhibitor when reporting results.

- 5.4 **Sample Dilution.** A sample has to be diluted sufficiently to allow about 5 mg/L drop in DO in five days. Without prior knowledge of the BOD, a range of dilutions for a particular sample will be required. As a guide, unpolluted river water is not diluted, a treatment plant discharging near 20 mg/L BOD₅ would be diluted 1/4 or 1/5 and a cooling tower overflow 1/200 or 1/400. A useful rule of thumb is to divide the anticipated BOD₅ by five to obtain the dilution required.

- 5.5 **Procedure.** Add the desired quantity of sample to a 1L graduated cylinder or volumetric flask and dilute to 1L with standard dilution water. Mix well with a plunger-type mixing rod taking care to avoid excessive shaking or air entrainment. Siphon the sample into three 250 mL brown reagent bottles, fill to their brim and stopper. One sample is analysed for initial DO and the remaining two are stopped tightly, water-sealed and incubated for 5 days at 20°C.

Include seed controls, dilution water blanks (unseeded) and from time to time, glucose-glutamic acid solution checks. Their DO uptakes should be 0.6 to 1.0, <0.1 and 200 ± 37 mg/L respectively.

The dilution selected for BOD determination is one which gives closest to a 5 mg/L drop in dissolved

oxygen. Grossly over-diluted samples should not be used for BOD calculation. Similarly, the 'seed' correction should be determined from a normal determination of BOD on the 'seed' sample and not one where the analysis is based on the 'seed' dilution used in the main sample. Grossly over-diluted samples are subject to erratic oxidation.

(b) Oxygen Determination

The initial sample, 5 day incubated samples, seed controls, dilution water blanks and glucose-glutamic acid solution checks are analysed for dissolved oxygen by Method No. 48 'Dissolved Oxygen (DO) — Determination in Water by the Azide Modification'.

6.0 CALCULATIONS

For 200 mL of original sample,

1 mL 0.0250 mol/L sodium thiosulphate = 1mg/L DO.

When seeding is not required

$$\text{mg/L BOD} = \frac{D_1 - D_2}{P}$$

where: D_1 = DO of diluted sample after 15 minutes

D_2 = DO of diluted sample after incubation

P = decimal fraction of sample used

When seeding is required

$$\text{mg/L BOD} = \frac{(D_1 - D_2) - (B_1 - B_2)f}{P}$$

where: B_1 = DO of diluted seed control after 15 minutes

B_2 = DO of diluted seed control after incubation

f = ratio of seed in sample to seed in control

7.0 PRECISION

The BOD test is a bioassay and not a precise chemical analysis. A check on the method can be obtained by analysis of a solution containing 150 mg/L of glucose and 150 mg/L of glutamic acid. The BOD₅ should be in the range 200 ± 37 mg/L.

8.0 REFERENCES

American Public Health Association, American Water Works Association and Water Pollution Council Federation (1980). In, *Standard Methods for the Examination of Water and Wastewater*, 15th Edition, APHA, Washington, DC, pp. 390-393, 483-489.

TABLE I

Temperature Corrections to Readings of Brix Hydrometers (Calibrated at 20°C)

Temperature °C	Observed per cent of sugar																		
	0	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75	80	85	90
0	0.30	0.49	0.65	0.77	0.89	0.99	1.08	1.16	1.24	1.31	1.37	1.41	1.44	1.47	1.49	1.50	1.50	1.51	1.51
5	0.36	0.47	0.56	0.65	0.73	0.80	0.86	0.91	0.97	1.01	1.05	1.08	1.10	1.12	1.14	1.16	1.17	1.18	1.19
10	0.32	0.38	0.43	0.48	0.52	0.57	0.60	0.64	0.67	0.70	0.72	0.74	0.75	0.76	0.77	0.78	0.79	0.80	0.81
11	0.31	0.35	0.40	0.44	0.48	0.51	0.55	0.58	0.60	0.63	0.65	0.66	0.68	0.69	0.70	0.71	0.72	0.73	0.74
12	0.29	0.32	0.36	0.40	0.43	0.46	0.50	0.52	0.54	0.56	0.58	0.59	0.60	0.61	0.62	0.63	0.64	0.64	0.65
13	0.26	0.29	0.32	0.35	0.38	0.41	0.44	0.46	0.48	0.49	0.51	0.52	0.53	0.54	0.55	0.56	0.56	0.57	0.57
14	0.24	0.26	0.29	0.31	0.34	0.36	0.38	0.40	0.41	0.42	0.44	0.45	0.46	0.46	0.47	0.47	0.48	0.48	0.48
15	0.20	0.22	0.24	0.26	0.28	0.30	0.32	0.33	0.34	0.36	0.36	0.37	0.38	0.38	0.39	0.39	0.40	0.40	0.41
16	0.17	0.18	0.20	0.22	0.23	0.25	0.26	0.27	0.28	0.28	0.29	0.30	0.31	0.31	0.32	0.32	0.33	0.33	0.34
17	0.13	0.14	0.15	0.16	0.18	0.19	0.20	0.20	0.21	0.21	0.22	0.23	0.23	0.24	0.24	0.25	0.25	0.25	0.26
18	0.09	0.10	0.10	0.11	0.12	0.13	0.13	0.14	0.14	0.14	0.15	0.15	0.15	0.16	0.16	0.16	0.16	0.17	0.17
19	0.05	0.05	0.05	0.06	0.06	0.06	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.09	0.09	0.09	0.09
20																			
21	0.04	0.05	0.06	0.06	0.06	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.09	0.09	0.09	0.09	0.09
22	0.10	0.10	0.11	0.12	0.12	0.13	0.14	0.14	0.14	0.15	0.16	0.16	0.16	0.16	0.16	0.17	0.17	0.17	0.17
23	0.16	0.16	0.17	0.17	0.19	0.20	0.21	0.21	0.22	0.23	0.24	0.24	0.24	0.24	0.24	0.25	0.25	0.25	0.25
24	0.21	0.22	0.23	0.24	0.26	0.27	0.28	0.29	0.30	0.31	0.32	0.32	0.32	0.32	0.32	0.33	0.33	0.33	0.33
25	0.27	0.28	0.30	0.31	0.32	0.34	0.35	0.36	0.38	0.38	0.39	0.39	0.40	0.40	0.39	0.39	0.39	0.38	0.38
26	0.33	0.34	0.36	0.37	0.40	0.40	0.42	0.44	0.46	0.47	0.47	0.48	0.48	0.48	0.48	0.49	0.49	0.48	0.48
27	0.40	0.41	0.42	0.44	0.46	0.48	0.50	0.52	0.54	0.54	0.55	0.56	0.56	0.56	0.57	0.57	0.57	0.56	0.56
28	0.46	0.47	0.49	0.51	0.54	0.56	0.58	0.60	0.61	0.62	0.63	0.64	0.64	0.64	0.65	0.65	0.65	0.64	0.64
29	0.54	0.55	0.56	0.59	0.61	0.63	0.66	0.68	0.70	0.70	0.71	0.72	0.72	0.72	0.73	0.73	0.73	0.72	0.72
30	0.61	0.62	0.63	0.66	0.68	0.70	0.73	0.76	0.78	0.78	0.79	0.80	0.80	0.80	0.81	0.81	0.81	0.81	0.81
31	0.69	0.70	0.71	0.74	0.76	0.79	0.82	0.84	0.86	0.87	0.88	0.88	0.89	0.89	0.89	0.89	0.89	0.89	0.89
32	0.76	0.78	0.79	0.82	0.85	0.87	0.90	0.93	0.95	0.95	0.96	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97
33	0.84	0.85	0.87	0.90	0.93	0.96	0.99	1.01	1.03	1.04	1.05	1.05	1.06	1.06	1.06	1.06	1.06	1.06	1.05
34	0.91	0.93	0.95	0.98	1.02	1.04	1.07	1.10	1.12	1.12	1.13	1.14	1.14	1.14	1.14	1.14	1.14	1.14	1.13
35	0.99	1.01	1.02	1.06	1.10	1.13	1.16	1.18	1.20	1.21	1.22	1.22	1.23	1.23	1.22	1.22	1.22	1.22	1.21

Subtract from observed per cent.

Add to observed per cent.

TABLE I P.2.

Table I—continued

Temperature °C	Observed per cent of sugar																			
	0	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75	80	85	90	
36	1.07	1.09	1.12	1.15	1.19	1.22	1.25	1.27	1.29	1.30	1.31	1.32	1.32	1.32	1.31	1.31	1.30	1.30	1.30	1.29
37	1.15	1.17	1.21	1.24	1.28	1.31	1.34	1.36	1.38	1.39	1.39	1.40	1.40	1.40	1.39	1.39	1.39	1.39	1.39	1.38
38	1.24	1.26	1.29	1.33	1.36	1.39	1.42	1.44	1.46	1.47	1.48	1.49	1.49	1.49	1.48	1.48	1.47	1.47	1.47	1.46
39	1.33	1.35	1.38	1.42	1.45	1.48	1.51	1.53	1.55	1.56	1.56	1.57	1.57	1.57	1.56	1.56	1.56	1.56	1.56	1.55
40	1.42	1.45	1.47	1.51	1.54	1.57	1.62	1.62	1.64	1.65	1.65	1.66	1.66	1.66	1.65	1.65	1.64	1.64	1.64	1.63
41	1.51	1.54	1.56	1.60	1.63	1.67	1.69	1.71	1.73	1.74	1.74	1.75	1.75	1.75	1.74	1.73	1.72	1.72	1.72	1.71
42	1.61	1.64	1.66	1.70	1.73	1.76	1.79	1.81	1.82	1.83	1.83	1.84	1.84	1.83	1.82	1.82	1.81	1.80	1.80	1.79
43	1.71	1.74	1.76	1.80	1.83	1.86	1.88	1.90	1.92	1.92	1.92	1.92	1.92	1.92	1.91	1.90	1.89	1.89	1.89	1.88
44	1.81	1.84	1.86	1.90	1.93	1.95	1.98	2.00	2.01	2.01	2.01	2.01	2.01	2.01	1.99	1.99	1.98	1.97	1.96	1.96
45	1.91	1.94	1.96	2.00	2.03	2.05	2.07	2.09	2.10	2.10	2.10	2.10	2.10	2.09	2.08	2.07	2.06	2.05	2.04	2.04
46	2.01	2.05	2.07	2.11	2.14	2.15	2.17	2.19	2.20	2.20	2.20	2.19	2.19	2.18	2.17	2.16	2.14	2.13	2.12	2.12
47	2.12	2.16	2.18	2.21	2.24	2.26	2.27	2.29	2.30	2.29	2.29	2.28	2.28	2.27	2.26	2.24	2.23	2.21	2.20	2.20
48	2.23	2.26	2.28	2.32	2.35	2.36	2.38	2.39	2.39	2.39	2.39	2.38	2.38	2.36	2.34	2.33	2.31	2.30	2.28	2.28
49	2.35	2.37	2.39	2.42	2.45	2.47	2.48	2.49	2.49	2.48	2.48	2.47	2.47	2.45	2.43	2.41	2.40	2.38	2.36	2.36
50	2.46	2.48	2.50	2.53	2.56	2.57	2.58	2.59	2.59	2.58	2.58	2.56	2.56	2.54	2.52	2.50	2.48	2.46	2.44	2.44
51	2.58	2.60	2.62	2.64	2.67	2.68	2.69	2.69	2.69	2.68	2.68	2.65	2.65	2.63	2.61	2.59	2.57	2.54	2.52	2.52
52	2.70	2.72	2.74	2.76	2.78	2.79	2.80	2.80	2.79	2.78	2.78	2.75	2.75	2.72	2.70	2.68	2.65	2.63	2.60	2.60
53	2.81	2.83	2.85	2.87	2.90	2.90	2.90	2.90	2.90	2.88	2.87	2.84	2.84	2.82	2.79	2.76	2.74	2.71	2.69	2.69
54	2.93	2.95	2.97	2.99	3.01	3.01	3.01	3.01	3.00	2.98	2.97	2.94	2.94	2.91	2.88	2.85	2.82	2.80	2.77	2.77
55	3.05	3.07	3.09	3.12	3.12	3.12	3.12	3.11	3.10	3.08	3.07	3.03	3.03	3.00	2.97	2.94	2.91	2.88	2.85	2.85
56	3.18	3.20	3.22	3.23	3.24	3.24	3.23	3.22	3.20	3.18	3.17	3.12	3.12	3.09	3.06	3.03	3.00	2.97	2.93	2.93
57	3.31	3.33	3.35	3.35	3.36	3.35	3.34	3.33	3.31	3.29	3.27	3.22	3.22	3.19	3.15	3.12	3.09	3.05	3.02	3.02
58	3.43	3.46	3.47	3.48	3.48	3.47	3.45	3.43	3.41	3.39	3.37	3.34	3.31	3.28	3.25	3.21	3.17	3.14	3.10	3.10
59	3.56	3.59	3.60	3.60	3.60	3.58	3.56	3.54	3.52	3.50	3.47	3.44	3.41	3.38	3.34	3.30	3.26	3.22	3.19	3.19
60	3.69	3.72	3.73	3.73	3.72	3.70	3.67	3.65	3.62	3.60	3.57	3.54	3.50	3.47	3.43	3.39	3.35	3.31	3.27	3.27

This table is calculated using data on thermal expansion of sugar solutions by Plato assuming the instrument to be of Jena 16¹¹¹ glass. The table should be used with caution and only for approximate results when the temperature differs much from the standard temperature or from the temperature of the surrounding air.

Table II—continued

Polariscope reading	Degrees Brix															Polariscope reading		
	11.5	12.0	12.5	13.0	13.5	14.0	14.5	15.0	15.5	16.0	16.5	17.0	17.5	18.0	18.5		19.0	19.5
41	10.22	10.20	10.18	10.16	10.14	10.12	10.09	10.07	10.05	10.03	10.01	9.99	9.97	9.95	9.93	9.91	9.89	41
42	10.47	10.45	10.42	10.40	10.38	10.36	10.34	10.32	10.30	10.28	10.26	10.24	10.22	10.20	10.18	10.16	10.15	42
43	10.71	10.69	10.67	10.65	10.63	10.61	10.59	10.57	10.54	10.52	10.50	10.48	10.46	10.44	10.42	10.40	10.37	43
44	10.96	10.94	10.92	10.90	10.88	10.86	10.83	10.81	10.79	10.77	10.75	10.72	10.70	10.68	10.66	10.64	10.62	44
45	11.21	11.19	11.17	11.15	11.12	11.10	11.08	11.06	11.04	11.01	10.99	10.97	10.95	10.92	10.90	10.88	10.86	45
46	11.46	11.44	11.42	11.39	11.37	11.35	11.33	11.30	11.28	11.26	11.23	11.21	11.19	11.17	11.14	11.12	11.10	46
47	..	11.69	11.67	11.64	11.62	11.60	11.57	11.55	11.53	11.50	11.48	11.46	11.43	11.41	11.39	11.36	11.34	47
48	..	11.94	11.91	11.89	11.87	11.84	11.82	11.79	11.77	11.75	11.72	11.70	11.68	11.65	11.63	11.60	11.58	48
49	12.16	12.14	12.11	12.09	12.06	12.04	12.02	11.99	11.97	11.94	11.92	11.89	11.87	11.85	11.82	49
50	12.39	12.36	12.34	12.31	12.29	12.26	12.24	12.21	12.19	12.16	12.14	12.11	12.09	12.06	50
51	12.63	12.61	12.58	12.56	12.53	12.51	12.48	12.46	12.43	12.41	12.38	12.35	12.33	12.30	51
52	13.10	12.86	12.80	12.78	12.75	12.73	12.70	12.67	12.65	12.62	12.60	12.57	12.55	52
53	13.08	13.03	13.02	13.00	12.97	12.94	12.92	12.89	12.87	12.84	12.81	12.79	53
54	13.32	13.30	13.27	13.24	13.22	13.19	13.16	13.13	13.11	13.08	13.05	13.03	54
55	13.57	13.54	13.51	13.49	13.46	13.43	13.41	13.38	13.35	13.32	13.30	13.27	55
56	13.79	13.76	13.73	13.70	13.68	13.65	13.62	13.59	13.57	13.54	13.51	56
57	14.03	14.01	13.98	13.95	13.92	13.89	13.86	13.84	13.81	13.78	13.75	57
58	14.28	14.25	14.22	14.19	14.17	14.14	14.11	14.08	14.05	14.02	13.99	58
59	14.50	14.47	14.44	14.41	14.38	14.35	14.32	14.29	14.26	14.23	59
60	14.74	14.71	14.68	14.65	14.62	14.59	14.56	14.53	14.51	14.48	60
61	14.96	14.93	14.90	14.87	14.84	14.81	14.78	14.75	14.72	61
62	15.17	15.14	15.11	15.08	15.05	15.02	14.99	14.96	62
63	15.42	15.39	15.36	15.32	15.29	15.26	15.23	15.20	63
64	15.66	15.63	15.60	15.57	15.54	15.50	15.47	15.44	64
65	15.91	15.87	15.84	15.81	15.78	15.75	15.71	15.68	65
66	16.12	16.09	16.05	16.02	15.99	15.96	15.92	66
67	16.36	16.33	16.30	16.26	16.23	16.20	16.16	67
68	16.57	16.54	16.51	16.47	16.44	16.41	68
69	16.82	16.78	16.75	16.71	16.68	16.65	69
70	17.03	16.99	16.96	16.92	16.89	70
71	17.27	17.23	17.20	17.16	17.13	71
72	17.48	17.44	17.41	17.37	72
73	17.72	17.68	17.65	17.61	73
74	17.96	17.93	17.89	17.85	74
75	18.17	18.13	18.09	75
76	0.1	0.2	0.02	0.02	0.6	0.6	0.15	76
77	0.2	0.05	0.05	0.05	0.7	0.7	0.17	77
78	0.3	0.07	0.07	0.07	0.8	0.8	0.20	78
79	0.4	0.10	0.10	0.10	0.9	0.9	0.22	79
80	0.5	0.12	0.12	0.12	80

Brix 10.0 to 28.5.

Table II—continued

Polariscope reading	Degrees Brix															Polariscope reading		
	20.5	21.0	21.5	22.0	22.5	23.0	23.5	24.0	24.5	25.0	25.5	26.0	26.5	27.0	27.5		28.0	28.5
81	19.46	19.42	19.38	19.34	19.30	19.26	19.22	19.18	19.14	19.10	19.06	19.02	18.98	18.94	18.90	18.86	18.82	81
82	19.70	19.66	19.62	19.58	19.54	19.50	19.46	19.42	19.38	19.34	19.30	19.26	19.22	19.18	19.14	19.10	19.06	82
83	19.94	19.90	19.86	19.82	19.78	19.74	19.70	19.66	19.61	19.57	19.53	19.49	19.45	19.41	19.37	19.33	19.29	83
84	20.18	20.14	20.10	20.06	20.02	19.97	19.93	19.89	19.85	19.81	19.77	19.73	19.69	19.64	19.60	19.56	19.52	84
85	..	20.38	20.34	20.30	20.25	20.21	20.17	20.13	20.09	20.05	20.00	19.96	19.92	19.88	19.84	19.79	19.75	85
86	..	20.62	20.58	20.54	20.49	20.45	20.41	20.37	20.32	20.28	20.24	20.20	20.15	20.11	20.07	20.03	19.99	86
87	20.82	20.77	20.73	20.69	20.65	20.60	20.56	20.52	20.47	20.43	20.39	20.35	20.30	20.26	20.22	87
88	21.06	21.01	20.97	20.93	20.88	20.84	20.80	20.75	20.71	20.67	20.62	20.58	20.54	20.49	20.45	88
89	21.25	21.21	21.16	21.12	21.08	21.03	20.99	20.94	20.90	20.86	20.81	20.77	20.73	20.68	89
90	21.49	21.45	21.40	21.36	21.31	21.27	21.22	21.18	21.14	21.09	21.05	21.00	20.96	20.91	90
91	21.73	21.68	21.64	21.59	21.55	21.50	21.46	21.42	21.37	21.33	21.28	21.24	21.19	21.15	91
92	21.92	21.88	21.83	21.79	21.74	21.70	21.65	21.61	21.56	21.52	21.47	21.42	21.38	92
93	22.16	22.12	22.07	22.02	21.98	21.93	21.89	21.84	21.79	21.75	21.70	21.66	21.61	93
94	22.35	22.31	22.26	22.21	22.17	22.12	22.08	22.03	21.98	21.94	21.89	21.84	94
95	22.59	22.54	22.50	22.45	22.40	22.36	22.31	22.26	22.22	22.17	22.12	22.08	95
96	22.83	22.78	22.73	22.69	22.64	22.59	22.54	22.50	22.45	22.40	22.36	22.31	96
97	23.02	22.97	22.92	22.88	22.83	22.78	22.73	22.68	22.64	22.59	22.54	97
98	23.21	23.16	23.11	23.06	23.01	22.97	22.92	22.87	22.82	22.77	98
99	23.44	23.40	23.35	23.30	23.25	23.20	23.15	23.10	23.05	23.01	99
100	23.68	23.63	23.58	23.53	23.48	23.44	23.39	23.34	23.29	23.24	100
101	23.92	23.87	23.82	23.77	23.72	23.67	23.62	23.57	23.52	23.47	101
102	24.10	24.05	24.00	23.95	23.90	23.85	23.80	23.75	23.70	102
103	24.34	24.29	24.24	24.19	24.14	24.09	24.04	23.99	23.94	103
104	24.53	24.47	24.42	24.37	24.32	24.27	24.22	24.17	104
105	24.76	24.71	24.66	24.61	24.56	24.50	24.45	24.40	105
106	0.1	0.2	0.02	0.6	0.6	0.15	24.95	24.89	24.84	24.79	24.74	24.68	24.63	106
107	0.3	0.07	0.05	0.7	0.7	0.17	25.13	25.08	25.02	24.97	24.92	24.87	107
108	0.4	0.10	0.07	0.8	0.8	0.20	25.31	25.26	25.20	25.15	25.10	108
109	0.5	0.12	0.10	0.9	0.9	0.22	25.49	25.44	25.38	25.33	109
110	0.12	25.72	25.67	25.62	25.56	110

Brix 10.0 to 28.5

Tenths of the polariscope reading	Per cent sucrose	Tenths of the polariscope reading	Per cent sucrose
0.1	0.02	0.6	0.15
0.2	0.05	0.7	0.17
0.3	0.07	0.8	0.20
0.4	0.10	0.9	0.22
0.5	0.12

TABLE III

Table of Factors for the Calculation of Pol Per Cent Juice from Pol Reading for Use in the Dry Lead Method with Undiluted Solutions

$$\text{Pol per cent juice} = \frac{\text{Pol Reading}}{\text{Pol Factor}}$$

Degrees Brix	Factor	Degrees Brix	Factor
0.5	3.84273	16.5	4.09450
1.0	3.85023	17.0	4.10285
1.5	3.85769	17.5	4.11119
2.0	3.86519	18.0	4.11962
2.5	3.87273	18.5	4.12804
3.0	3.88027	19.0	4.13650
3.5	3.88785	19.5	4.14496
4.0	3.89546	20.0	4.15350
4.5	3.90308	20.5	4.16204
5.0	3.91077	21.0	4.17062
5.5	3.91842	21.5	4.17923
6.0	3.92615	22.0	4.18788
6.5	3.93388	22.5	4.19658
7.0	3.94165	23.0	4.20527
7.5	3.94942	23.5	4.21400
8.0	3.95723	24.0	4.22277
8.5	3.96512	24.5	4.23158
9.0	3.97296	25.0	4.24042
9.5	3.98088	25.5	4.24931
10.0	3.98881	26.0	4.25819
10.5	3.99677	26.5	4.26712
11.0	4.00473	27.0	4.27608
11.5	4.01277	27.5	4.28508
12.0	4.02081	28.0	4.29412
12.5	4.02885	28.5	4.30315
13.0	4.03696	29.0	4.31227
13.5	4.04508	29.5	4.32138
14.0	4.05327	30.0	4.33054
14.5	4.06146	30.5	4.33973
15.0	4.06965	31.0	4.34896
15.5	4.07792	31.5	4.35823
16.0	4.08619	32.0	4.36750

NOTE 1.—These factors have been calculated from data in column 2 of Table XVI using the formula—

$$\text{Pol factor} = \frac{100 \times \text{apparent density at } 20^{\circ}\text{C}}{26.000}$$

The values have been calculated to sixteen significant figures and rounded to six significant figures using the rounding rule in British Standards 1957.

NOTE 2.—Due to rounding errors and differences in original data there may be discrepancies in the second decimal place of pol between values calculated using these factors and those obtained from Table II. Providing sufficient significant figures are used in the calculation the values obtained using the pol factors of this table are to be considered the correct results.

This Table has been calculated on the mass-based 'old' millilitre. To convert to values calculated on the 'new' millilitre (based on volume), the values in the Table should be divided by 1.000028, where such precision is required.

TABLE IV

Factors for the Calculation of Sucrose according to
Jackson and Gillis Method 2 of Double Polarisation

$(P - I) \times \text{Normality of}$ Original Solution (i.e. $\times 1$ or $\times 1/2$)	Factor
133.34	133.34
133	133.34
130	133.32
125	133.29
120	133.25
115	133.22
110	133.18
105	133.15
100	133.12
95	133.09
90	133.06
85	133.02
80	132.99
75	132.95
70	132.92
65	132.89
60	132.86
55	132.82
50	132.79
45	132.75
40	132.72
35	132.69
30	132.66
25	132.63
20	132.60
15	132.56
10	132.53
5	132.49

NOTE: (i) $(P - I)$ is the algebraic difference between the actual readings, corrected for normality and dilution. This value is used in the numerator of the formula.

(ii) The concentration of sucrose in the original solution (measured by $(P - I) \times$ normality as weighed out) is used in the table to give the appropriate factor.

Refer: Jackson, R.F. and Gillis, C.L., U.S. Bureau of Standards, Scientific Paper No. 375 (1920), p. 185

TABLE V P.1.

TABLE V

Temperature Corrections for Plain Polarisation Reading
in Determination of Sucrose by Double Polarisation

NOTE: For plain pol solution normality of 0.25 only

$t_p - t_i$ (°C)	Approximate % Reducing Sugars in Product			
	10	15	20	25
	Correction to be ADDED (°Z)			
-10.0	+0.10	+0.15	+0.20	+0.25
-9.0	0.09	0.14	0.18	0.23
-8.0	0.08	0.12	0.16	0.20
-7.0	0.07	0.11	0.14	0.18
-6.0	0.06	0.09	0.12	0.15
-5.0	0.05	0.08	0.10	0.13
-4.0	0.04	0.06	0.08	0.10
-3.0	0.03	0.05	0.06	0.08
-2.0	0.02	0.03	0.04	0.05
-1.0	0.01	0.02	0.02	0.03
0.0	0.00	0.00	0.00	0.00
1.0	-0.01	-0.02	-0.02	-0.03
2.0	-0.02	-0.03	-0.04	-0.05
3.0	-0.03	-0.05	-0.06	-0.08
4.0	-0.04	-0.06	-0.08	-0.10
5.0	-0.05	-0.08	-0.10	-0.13
6.0	-0.06	-0.09	-0.12	-0.15
7.0	-0.07	-0.11	-0.14	-0.18
8.0	-0.08	-0.12	-0.16	-0.20
9.0	-0.09	-0.14	-0.18	-0.23
10.0	-0.10	-0.15	-0.20	-0.25

NOTE: For plain pol solution normality of 0.5 only

$t_p - t_i$ (°C)	Approximate % Reducing Sugars in Product			
	10	15	20	25
	Correction to be ADDED (°Z)			
-10.0	+0.20	0.30	0.40	0.50
-9.0	0.18	0.27	0.36	0.45
-8.0	0.16	0.24	0.32	0.40
-7.0	0.14	0.21	0.28	0.35
-6.0	0.12	0.18	0.24	0.30
-5.0	0.10	0.15	0.20	0.25
-4.0	0.08	0.12	0.16	0.20
-3.0	0.06	0.09	0.12	0.15
-2.0	0.04	0.06	0.08	0.10
-1.0	0.02	0.03	0.04	0.05
0.0	+0.00	0.00	0.00	0.00
1.0	-0.02	-0.03	-0.04	-0.05
2.0	-0.04	-0.06	-0.08	-0.10
3.0	-0.06	-0.09	-0.12	-0.15
4.0	-0.08	-0.12	-0.16	-0.20
5.0	-0.10	-0.15	-0.20	-0.25
6.0	-0.12	-0.18	-0.24	-0.30
7.0	-0.14	-0.21	-0.28	-0.35
8.0	-0.16	-0.24	-0.32	-0.40
9.0	-0.18	-0.27	-0.36	-0.45
10.0	-0.20	-0.30	-0.40	-0.50

FORMULA:

$$P_{t_i} - P_{t_p} = -0.004 \text{ N.R. } (t_p - t_i)$$

where:

P_{t_i} = Polarisation of invert solution (°Z) at
temperature t_i

P_{t_p} = Polarisation of plain solution (°Z) at
temperature t_p

t_i = Temperature of reading of invert solution

t_p = Temperature of reading of plain solution

N = Normality of plain solution at reading

R = % Reducing Sugars

Refer: Wilson, R.A.M., International Sugar Journal,
Vol. 67 (1965), pp 234-6 and 265-8

TABLE VII P.1.

TABLE VII

% POL IN BAGASSE

This Tables gives the % Pol in Bagasse
(at various bagasse moistures)
corresponding to the polarimeter reading of wet
disintegrator fluid (1 part bagasse to 10 parts water)
when read in a 200 mm tube on a sugar polarimeter
for wet disintegrator fluid purity of 75-80

Pol Reading in 200 mm Tube	% Water in Bagasse							Pol Reading in 200 mm Tube	% Water in Bagasse						
	35	40	45	50	55	60	65		35	40	45	50	55	60	65
	% Pol in Bagasse								% Pol in Bagasse						
3.0	8.0	8.1	8.1	8.2	8.2	8.3	8.3	0.5	1.33	1.34	1.34	1.35	1.36	1.37	1.38
3.1	8.3	8.4	8.4	8.5	8.5	8.6	8.6	0.6	1.59	1.60	1.61	1.62	1.63	1.64	1.65
3.2	8.6	8.6	8.7	8.7	8.8	8.8	8.9	0.7	1.86	1.87	1.88	1.89	1.90	1.92	1.93
3.3	8.8	8.9	8.9	9.0	9.1	9.1	9.2	0.8	2.13	2.14	2.15	2.16	2.18	2.19	2.20
3.4	9.1	9.2	9.2	9.3	9.3	9.4	9.4	0.9	2.39	2.41	2.42	2.44	2.45	2.47	2.48
3.5	9.4	9.4	9.5	9.6	9.6	9.7	9.8	1.0	2.66	2.68	2.69	2.71	2.72	2.74	2.76
3.6	9.7	9.7	9.8	9.8	9.9	10.0	10.0	1.1	2.93	2.94	2.96	2.98	3.00	3.02	3.03
3.7	9.9	10.0	10.0	10.1	10.2	10.2	10.3	1.2	3.19	3.21	3.23	3.25	3.27	3.29	3.31
3.8	10.2	10.3	10.3	10.4	10.4	10.5	10.6	1.3	3.46	3.48	3.50	3.53	3.55	3.57	3.59
3.9	10.5	10.5	10.6	10.7	10.7	10.8	10.8	1.4	3.73	3.75	3.77	3.80	3.82	3.84	3.87
4.0	10.7	10.8	10.9	10.9	11.0	11.1	11.2	1.5	4.0	4.0	4.0	4.1	4.1	4.1	4.1
4.1	11.0	11.1	11.1	11.2	11.3	11.3	11.4	1.6	4.3	4.3	4.3	4.3	4.4	4.4	4.4
4.2	11.3	11.4	11.4	11.5	11.6	11.6	11.7	1.7	4.5	4.6	4.6	4.6	4.6	4.7	4.7
4.3	11.6	11.6	11.7	11.8	11.8	11.9	12.0	1.8	4.8	4.8	4.9	4.9	4.9	5.0	5.0
4.4	11.8	11.9	12.0	12.1	12.1	12.2	12.3	1.9	5.1	5.1	5.1	5.2	5.2	5.2	5.3
4.5	12.1	12.2	12.2	12.3	12.4	12.5	12.6	2.0	5.3	5.4	5.4	5.4	5.5	5.5	5.5
4.6	12.4	12.4	12.5	12.6	12.7	12.8	12.8	2.1	5.6	5.6	5.7	5.7	5.7	5.8	5.8
4.7	12.6	12.7	12.8	12.9	12.9	13.0	13.1	2.2	5.9	5.9	6.0	6.0	6.0	6.1	6.1
4.8	12.9	13.0	13.1	13.2	13.2	13.3	13.4	2.3	6.1	6.2	6.2	6.3	6.3	6.3	6.4
4.9	13.2	13.3	13.4	13.4	13.5	13.6	13.7	2.4	6.4	6.5	6.5	6.6	6.6	6.6	6.7
5.0	13.5	13.5	13.6	13.7	13.8	13.9	14.0	2.5	6.7	6.7	6.8	6.8	6.8	6.9	6.9
5.1	13.7	13.8	13.9	14.0	14.1	14.2	14.2	2.6	7.0	7.0	7.0	7.1	7.1	7.2	7.2
5.2	14.0	14.1	14.2	14.3	14.4	14.4	14.5	2.7	7.2	7.3	7.3	7.4	7.4	7.4	7.5
5.3	14.3	14.4	14.4	14.5	14.6	14.7	14.8	2.8	7.5	7.5	7.6	7.6	7.7	7.7	7.8
5.4	14.6	14.6	14.7	14.8	14.9	15.0	15.1	2.9	7.8	7.8	7.9	7.9	8.0	8.0	8.0

This Table shows the % pol in bagasses of various water contents, for polarimeter readings of wet disintegrator extracts, when read in a 200 mm tube in a sugar polarimeter, of 26 g normal weight.

The calculation falls into 2 parts:

(a) Obtaining % pol in W.D. extract from the polarimeter reading,

(b) Obtaining % pol in bagasse from % pol in W.D. extract, (10:1 water:bagasse).

(a) % Pol in W.D. extract is obtained from the expression:
Pol reading in 200 mm tube

$$\times \frac{26 \times 1.001057}{\text{Wt. in air of 100 mL of W.D. extract}}$$

Since the brix of the W.D. extract is not known, the pol is first calculated approximately and the brix obtained by assuming a purity of 80 for 1st bagasse and 75 for others. The corresponding "weight in air of 100 mL" is then inserted in the expression and the % pol recalculated. (The influence of this is small).

(b) Calculation of % pol in bagasse from % pol in W.D. extract

% pol (or brix)
in bagasse

$$= \frac{\% \text{ pol (or brix) in W.D. extract}}{\text{Wt. of bagasse taken}} \times \frac{\text{Wt. of W.D. extract}}{\text{Wt. of bagasse taken}} \times 100 \quad (1)$$

So it is necessary to calculate the factor:

$$\frac{\text{Wt. of W.D. extract}}{\text{Wt. of bagasse taken}}$$

Let B be °brix, W be % water, F be % fibre, and X be the weight of bagasse taken; Let b be °brix and H be the mass of wet disintegrator extract. Assume the hygroscopic water is 25% on dry fibre, and let the water:bagasse ratio be 10:1 (i.e. 10 kg:1 kg).

Then Wt. of bagasse + Wt. of water = Wt. of W.D. extract + Wt. of hydrated fibre, i.e.

$$X + 10X = H + X \times \frac{F}{100} \times \frac{125}{100}, \text{ so that } H = X \left(11 - \frac{1.25F}{100} \right) \quad (2)$$

From the equations (1) and (2) above:

$$B = b \times \frac{H}{X} = b \left(11 - \frac{1.25F}{100} \right)$$

$$\begin{aligned} \text{But } F &= (100 - W - B), \text{ so } B = b \left[11 - \frac{1.25}{100} (100 - W - B) \right] \\ &= \frac{b(780 + W)}{80 - b} \end{aligned}$$

and the factor $\frac{H}{X} = \frac{780 + W}{80 - b}$

Thus % pol in bagasse =

$$\% \text{ pol in W.D. extract} \times \frac{780 + \% \text{ water in bagasse}}{80 - \% \text{ brix of W.D. extract}}$$

As stated previously, the °brix of W.D. extract is calculated from % pol of W.D. extract assuming the purity to be 80 for 1st bagasse and 75 for other bagasses.

TABLE VIII

**Refractive Indices of Sugar Solutions at 20°C in Air at
20°C, 760 mm Pressure and 50 per cent Relative Humidity**

The following values are according to the smoothed measured values of the Physikalisch-Technische Bundesanstalt in West Germany, and have been computed from the polynomial adopted by the ICUMSA 1966.

$$n_{D \text{ air}} = 1.33298731 + 0.142935392 \times 10^{-2} \times P_{\text{air}} \\ + 0.517183404 \times 10^{-5} P_{\text{air}}^2 + 0.23320854 \times 10^{-7} \times P_{\text{air}}^3 \\ - 0.410680669 \times 10^{-10} \times P_{\text{air}}^4$$

P = sugar concentration as percentage by weight in air at 20°C 760 mm pressure and 50 per cent relative humidity.

% Sucrose	Refractive index (n_D^{20})	% Sucrose	Refractive index (n_D^{20})	% Sucrose	Refractive index (n_D^{20})
0.0	1.332987	30.0	1.381119	60.0	1.441872
1.0	1.334422	31.0	1.382924	61.0	1.444147
2.0	1.335867	32.0	1.384744	62.0	1.446439
3.0	1.337323	33.0	1.386578	63.0	1.448748
4.0	1.338789	34.0	1.388426	64.0	1.451074
5.0	1.340266	35.0	1.390288	65.0	1.453418
6.0	1.341755	36.0	1.392166	66.0	1.45577
7.0	1.343254	37.0	1.394058	67.0	1.45815
8.0	1.344765	38.0	1.395965	68.0	1.46055
9.0	1.346287	39.0	1.397887	69.0	1.46296
10.0	1.347821	40.0	1.399824	70.0	1.46539
11.0	1.349366	41.0	1.401776	71.0	1.46784
12.0	1.350924	42.0	1.403743	72.0	1.47031
13.0	1.352493	43.0	1.405726	73.0	1.47279
14.0	1.354074	44.0	1.407724	74.0	1.47529
15.0	1.355668	45.0	1.409738	75.0	1.47782
16.0	1.357274	46.0	1.411767	76.0	1.48035
17.0	1.358892	47.0	1.413812	77.0	1.48291
18.0	1.360523	48.0	1.415873	78.0	1.48548
19.0	1.362168	49.0	1.417950	79.0	1.48808
20.0	1.363823	50.0	1.420043	80.0	1.49069
21.0	1.365493	51.0	1.422152	81.0	1.49332
22.0	1.367175	52.0	1.424277	82.0	1.49597
23.0	1.368871	53.0	1.426419	83.0	1.49863
24.0	1.370580	54.0	1.428577	84.0	1.50132
25.0	1.372302	55.0	1.430751	85.0	1.50402
26.0	1.374038	56.0	1.432942		
27.0	1.375787	57.0	1.435149		
28.0	1.377551	58.0	1.437373		
29.0	1.379328	59.0	1.439614		

TABLE IX

Table of Temperature Corrections for the Abbé Refractometer Calibrated at 20°C

Temp °C	Percent sucrose										Temp °C
	0	5	10	15	20	30	40	50	60	70	
Subtract from the percent sucrose											
10	0.50	0.54	0.58	0.61	0.64	0.68	0.72	0.74	0.76	0.79	10
11	0.46	0.49	0.53	0.55	0.58	0.62	0.65	0.67	0.69	0.71	11
12	0.42	0.45	0.48	0.50	0.52	0.56	0.58	0.60	0.61	0.63	12
13	0.37	0.40	0.42	0.44	0.46	0.49	0.51	0.53	0.54	0.55	13
14	0.33	0.35	0.37	0.39	0.40	0.42	0.44	0.45	0.46	0.48	14
15	0.27	0.29	0.31	0.33	0.34	0.35	0.37	0.38	0.39	0.40	15
16	0.22	0.24	0.25	0.26	0.27	0.28	0.30	0.30	0.31	0.32	16
17	0.17	0.18	0.19	0.20	0.21	0.21	0.22	0.23	0.23	0.24	17
18	0.12	0.13	0.13	0.14	0.14	0.14	0.15	0.15	0.16	0.16	18
19	0.06	0.06	0.06	0.07	0.07	0.07	0.08	0.08	0.08	0.08	19
Add to the percent sucrose											
21	0.06	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	21
22	0.13	0.13	0.14	0.14	0.15	0.15	0.15	0.16	0.16	0.16	22
23	0.19	0.20	0.21	0.22	0.22	0.23	0.23	0.24	0.24	0.24	23
24	0.26	0.27	0.28	0.29	0.30	0.31	0.31	0.31	0.32	0.32	24
25	0.33	0.35	0.36	0.37	0.38	0.39	0.40	0.40	0.40	0.40	25
26	0.40	0.42	0.43	0.44	0.45	0.47	0.48	0.48	0.48	0.48	26
27	0.48	0.50	0.52	0.53	0.54	0.55	0.56	0.56	0.56	0.56	27
28	0.56	0.57	0.60	0.61	0.62	0.63	0.64	0.64	0.64	0.64	28
29	0.64	0.66	0.68	0.69	0.71	0.72	0.73	0.73	0.73	0.73	29
30	0.72	0.74	0.77	0.78	0.79	0.80	0.81	0.81	0.81	0.81	30

TABLE X

The Refractive Index of Pure Water at
Various Temperatures, Relative to Air
(Sodium Line)

Temperature (°C)	Refractive Index	Temperature (°C)	Refractive Index
10	1.3337	23	1.3327
11	1.3336	24	1.3326
12	1.3336	25	1.3325
13	1.3335	26	1.3324
14	1.3335	27	1.3323
15	1.3334	28	1.3322
16	1.3333	29	1.3321
17	1.3332	30	1.3319
18	1.3332	31	1.3318
19	1.3331	32	1.3317
20	1.3330	33	1.3316
21	1.3329	34	1.3314
22	1.3328	35	1.3313

SOURCE:

Tilton, Leroy, W. and Taylor, John K. Research Paper,
RP 1085; J. Research of the National Bureau Standards,
Vol. 20, April (1938), P449, "Table 6. Sodium-lines index
of refraction of Distilled Water".

TABLE XI

Temperature Corrections to be Applied to Refractometer Readings of Sucrose Solutions of Various Concentrations, to obtain the Refractive Index at 20 °C

Refrac Index Reading	1.333	1.340	1.350	1.360	1.370	1.380	1.390	1.400	1.410	Refrac Index Reading	1.420	1.430	1.440	1.450	1.460	1.470	1.480	1.490	1.500	
Approx Brix	0	5	11	18	24	29	35	40	45	Approx Brix	50	55	59	64	68	72	76	80	84	
Temp (°C)	SUBTRACT from Refractometer Reading:									Temp (°C)	SUBTRACT from Refractometer Reading:									
10	.0008	.0008	.0009	.0010	.0011	.0012	.0013	.0014	.0015	10	.0016	.0016	.0017	.0017	.0018	.0019	.0019	.0019	.0019	.0020
11	.0007	.0008	.0008	.0009	.0010	.0011	.0012	.0013	.0013	11	.0014	.0015	.0015	.0016	.0016	.0017	.0017	.0017	.0017	.0018
12	.0006	.0007	.0008	.0008	.0009	.0010	.0011	.0011	.0012	12	.0013	.0013	.0014	.0014	.0014	.0015	.0015	.0015	.0016	.0016
13	.0006	.0006	.0007	.0007	.0008	.0009	.0009	.0010	.0011	13	.0011	.0012	.0012	.0012	.0013	.0013	.0013	.0013	.0014	.0014
14	.0005	.0005	.0006	.0006	.0007	.0008	.0008	.0009	.0009	14	.0010	.0010	.0010	.0011	.0011	.0011	.0011	.0011	.0012	.0012
15	.0004	.0005	.0005	.0005	.0006	.0006	.0007	.0007	.0008	15	.0008	.0008	.0009	.0009	.0009	.0009	.0010	.0010	.0010	.0010
16	.0003	.0004	.0004	.0004	.0005	.0005	.0006	.0006	.0006	16	.0006	.0007	.0007	.0007	.0007	.0008	.0008	.0008	.0008	.0008
17	.0003	.0003	.0003	.0003	.0004	.0004	.0004	.0004	.0005	17	.0005	.0005	.0005	.0005	.0006	.0006	.0006	.0006	.0006	.0006
18	.0002	.0002	.0002	.0002	.0002	.0003	.0003	.0003	.0003	18	.0003	.0003	.0003	.0004	.0004	.0004	.0004	.0004	.0004	.0004
19	.0001	.0001	.0001	.0001	.0001	.0001	.0001	.0001	.0002	19	.0002	.0002	.0002	.0002	.0002	.0002	.0002	.0002	.0002	.0002
	ADD TO Refractometer Reading:										ADD TO Refractometer Reading:									
21	.0001	.0001	.0001	.0001	.0001	.0001	.0001	.0002	.0002	21	.0002	.0002	.0002	.0002	.0002	.0002	.0002	.0002	.0002	.0002
22	.0002	.0002	.0002	.0002	.0003	.0003	.0003	.0003	.0003	22	.0003	.0003	.0004	.0004	.0004	.0004	.0004	.0004	.0004	.0004
23	.0003	.0003	.0003	.0004	.0004	.0004	.0004	.0005	.0005	23	.0005	.0005	.0005	.0005	.0006	.0006	.0006	.0006	.0006	.0006
24	.0004	.0004	.0004	.0005	.0005	.0006	.0006	.0006	.0006	24	.0007	.0007	.0007	.0007	.0008	.0008	.0008	.0008	.0008	.0008
25	.0005	.0005	.0006	.0006	.0007	.0007	.0007	.0008	.0008	25	.0008	.0009	.0009	.0009	.0009	.0010	.0010	.0010	.0010	.0010
26	.0006	.0006	.0007	.0007	.0008	.0008	.0009	.0009	.0010	26	.0010	.0010	.0011	.0011	.0011	.0012	.0012	.0012	.0012	.0012
27	.0007	.0008	.0008	.0009	.0009	.0010	.0010	.0011	.0011	27	.0012	.0012	.0013	.0013	.0013	.0013	.0013	.0014	.0014	.0014
28	.0008	.0009	.0009	.0010	.0011	.0011	.0012	.0013	.0013	28	.0014	.0014	.0014	.0015	.0015	.0015	.0016	.0016	.0016	.0016
29	.0009	.0010	.0011	.0011	.0012	.0013	.0014	.0014	.0015	29	.0015	.0016	.0016	.0017	.0017	.0017	.0018	.0018	.0018	.0018
30	.0011	.0011	.0012	.0013	.0014	.0014	.0015	.0016	.0017	30	.0017	.0018	.0018	.0019	.0019	.0019	.0020	.0020	.0020	.0020
31	.0012	.0013	.0013	.0014	.0015	.0016	.0017	.0018	.0018	31	.0019	.0020	.0020	.0021	.0021	.0021	.0022	.0022	.0022	.0022
32	.0013	.0014	.0015	.0016	.0017	.0018	.0019	.0019	.0020	32	.0021	.0021	.0022	.0023	.0023	.0023	.0024	.0024	.0024	.0024
33	.0014	.0015	.0016	.0017	.0018	.0019	.0020	.0021	.0022	33	.0023	.0023	.0024	.0024	.0025	.0025	.0026	.0026	.0026	.0026
34	.0016	.0017	.0018	.0019	.0020	.0021	.0022	.0023	.0024	34	.0024	.0025	.0026	.0026	.0027	.0027	.0028	.0028	.0028	.0028
35	.0017	.0018	.0019	.0020	.0021	.0023	.0024	.0025	.0025	35	.0026	.0027	.0028	.0028	.0029	.0029	.0030	.0030	.0030	.0030

SOURCE: ICUMSA Proceedings, 1978, p166, Equation 1

Assuming —

Wavelength of sodium light in vacuum = 589.3 nm

Refractive index of 'normal' air = 1.00027189

(20 °C, 760 nm Hg pressure, 50% relative humidity)

(Derived from: 'Wavelength data and correction tables for length interferometry', National Measurements Laboratory, Australia)

TABLE XII P.7.

R.I.	Degrees Brix	True Density (g/mL)	Conc. (g/mL)	R.I.	Degrees Brix	True Density (g/mL)	Conc. (g/mL)
1.4908	80.04	1.4119	1.1301	1.4961	82.05	1.4254	1.1696
1.4909	80.07	1.4122	1.1308	1.4962	82.09	1.4257	1.1703
1.4910	80.11	1.4125	1.1315	1.4963	82.13	1.4259	1.1711
1.4911	80.15	1.4127	1.1323	1.4964	82.16	1.4262	1.1718
1.4912	80.19	1.4130	1.1330	1.4965	82.20	1.4264	1.1725
1.4913	80.23	1.4132	1.1338	1.4966	82.24	1.4267	1.1733
1.4914	80.26	1.4135	1.1345	1.4967	82.28	1.4269	1.1740
1.4915	80.30	1.4137	1.1353	1.4968	82.31	1.4272	1.1748
1.4916	80.34	1.4140	1.1360	1.4969	82.35	1.4274	1.1755
1.4917	80.38	1.4142	1.1367	1.4970	82.39	1.4277	1.1763
1.4918	80.42	1.4145	1.1375	1.4971	82.43	1.4279	1.1770
1.4919	80.46	1.4147	1.1382	1.4972	82.47	1.4282	1.1778
1.4920	80.49	1.4150	1.1390	1.4973	82.50	1.4285	1.1785
1.4921	80.53	1.4152	1.1397	1.4974	82.54	1.4287	1.1793
1.4922	80.57	1.4155	1.1405	1.4975	82.58	1.4290	1.1800
1.4923	80.61	1.4158	1.1412	1.4976	82.62	1.4292	1.1808
1.4924	80.65	1.4160	1.1420	1.4977	82.65	1.4295	1.1815
1.4925	80.68	1.4163	1.1427	1.4978	82.69	1.4297	1.1823
1.4926	80.72	1.4165	1.1435	1.4979	82.73	1.4300	1.1830
1.4927	80.76	1.4168	1.1442	1.4980	82.77	1.4302	1.1838
1.4928	80.80	1.4170	1.1449	1.4981	82.80	1.4305	1.1845
1.4929	80.84	1.4173	1.1457	1.4982	82.84	1.4307	1.1853
1.4930	80.88	1.4175	1.1464	1.4983	82.88	1.4310	1.1860
1.4931	80.91	1.4178	1.1472	1.4984	82.92	1.4312	1.1867
1.4932	80.95	1.4180	1.1479	1.4985	82.95	1.4315	1.1875
1.4933	80.99	1.4183	1.1487	1.4986	82.99	1.4318	1.1882
1.4934	81.03	1.4186	1.1494	1.4987	83.03	1.4320	1.1890
1.4935	81.07	1.4188	1.1502	1.4988	83.07	1.4323	1.1897
1.4936	81.10	1.4191	1.1509	1.4989	83.11	1.4325	1.1905
1.4937	81.14	1.4193	1.1516	1.4990	83.14	1.4328	1.1912
1.4938	81.18	1.4196	1.1524	1.4991	83.18	1.4330	1.1920
1.4939	81.22	1.4198	1.1531	1.4992	83.22	1.4333	1.1927
1.4940	81.26	1.4201	1.1539	1.4993	83.26	1.4335	1.1935
1.4941	81.29	1.4203	1.1546	1.4994	83.29	1.4338	1.1942
1.4942	81.33	1.4206	1.1554	1.4995	83.33	1.4340	1.1950
1.4943	81.37	1.4208	1.1561	1.4996	83.37	1.4343	1.1957
1.4944	81.41	1.4211	1.1569	1.4997	83.40	1.4345	1.1965
1.4945	81.45	1.4213	1.1576	1.4998	83.44	1.4348	1.1972
1.4946	81.48	1.4216	1.1584	1.4999	83.48	1.4350	1.1980
1.4947	81.52	1.4219	1.1591				
1.4948	81.56	1.4221	1.1599	1.5000	83.52	1.4353	1.1987
1.4949	81.60	1.4224	1.1606	1.5001	83.55	1.4356	1.1995
1.4950	81.63	1.4226	1.1613	1.5002	83.59	1.4358	1.2002
1.4951	81.67	1.4229	1.1621	1.5003	83.63	1.4361	1.2010
1.4952	81.71	1.4231	1.1628	1.5004	83.67	1.4363	1.2017
1.4953	81.75	1.4234	1.1636	1.5005	83.70	1.4366	1.2025
1.4954	81.79	1.4236	1.1643	1.5006	83.74	1.4368	1.2032
1.4955	81.82	1.4239	1.1651	1.5007	83.78	1.4371	1.2040
1.4956	81.86	1.4241	1.1658	1.5008	83.82	1.4373	1.2047
1.4957	81.90	1.4244	1.1666	1.5009	83.85	1.4376	1.2055
1.4958	81.94	1.4246	1.1673	1.5010	83.89	1.4378	1.2062
1.4959	81.98	1.4249	1.1681	1.5011	83.93	1.4381	1.2070
1.4960	82.01	1.4252	1.1688	1.5012	83.97	1.4383	1.2077

R.I.	Degrees Brix	True Density (g/mL)	Conc. (g/mL)	R.I.	Degrees Brix	True Density (g/mL)	Conc. (g/mL)
1.5013	84.00	1.4386	1.2085	1.5027	84.52	1.4421	1.2190
1.5014	84.04	1.4388	1.2092	1.5028	84.56	1.4424	1.2197
1.5015	84.08	1.4391	1.2100	1.5029	84.60	1.4426	1.2205
1.5016	84.11	1.4394	1.2107	1.5030	84.64	1.4429	1.2212
1.5017	84.15	1.4396	1.2115	1.5031	84.67	1.4432	1.2220
1.5018	84.19	1.4399	1.2122	1.5032	84.71	1.4434	1.2227
1.5019	84.23	1.4401	1.2130	1.5033	84.75	1.4437	1.2235
1.5020	84.26	1.4404	1.2137	1.5034	84.78	1.4439	1.2242
1.5021	84.30	1.4406	1.2145	1.5035	84.82	1.4442	1.2250
1.5022	84.34	1.4409	1.2152	1.5036	84.86	1.4444	1.2257
1.5023	84.38	1.4411	1.2160	1.5037	84.90	1.4447	1.2265
1.5024	84.41	1.4414	1.2167	1.5038	84.93	1.4449	1.2272
1.5025	84.45	1.4416	1.2175	1.5039	84.97	1.4452	1.2280
1.5026	84.49	1.4419	1.2182	1.5040	85.01	1.4454	1.2287

SOURCE:

1. **RI to Brix conversion:**

ICUMSA 1978, p166, Equation 1.

This table was generated by solving Equation 1, using a computer iteration.

where: Refractive index is relative to standard air (20 °C, 760 mm pressure, 50% relative humidity)

$$RI_{air} = 1.00027189 \text{ at } 589.3 \text{ nm}$$

2. **Brix to true density conversion:** Regression equation based on Brown, C.A. and Zerban, F.W., Physical and Chemical Methods of Sugar Analysis, Ed. 3; 1941.

$$\begin{aligned} \text{True Density} = & 0.99822719 + 3.8612869 \cdot 10^{-3} \cdot Bx + \\ & 1.246674818 \cdot 10^{-5} \cdot Bx^2 \\ & + 7.09611659 \cdot 10^{-8} \cdot Bx^3 \\ & 2.8173378 \cdot 10^{-10} \cdot Bx^4 \end{aligned}$$

(NB — True Density = mass/vol where mass is weighed in vacuum)

3. **Concentration (g/mL) = Brix x Density/1000**

TABLE XIV

Solubility of Sucrose in Water in g Sucrose (S) per 100 g Solution* (Charles)

°C	S	°C	S	°C	S	°C	S	°C	S
0	64.41	19	66.47	37	69.45	55	73.11	73	77.15
1	64.48	20	66.61	38	69.64	56	73.33	74	77.38
2	64.56	21	66.75	39	69.83	57	73.55	75	77.60
3	64.64	22	66.90	40	70.02	58	73.77	76	77.83
4	64.73	23	67.05	41	70.22	59	73.99	77	78.06
5	64.82	24	67.21	42	70.41	60	74.21	78	78.29
6	64.92	25	67.36	43	70.61	61	74.43	79	78.52
7	65.02	26	67.52	44	70.81	62	74.66	80	78.75
8	65.11	27	67.69	45	71.01	63	74.88	81	78.97
9	65.22	28	67.85	46	71.21	64	75.10	82	79.20
10	65.33	29	68.02	47	71.42	65	75.33	83	79.43
11	65.44	30	68.19	48	71.63	66	75.56	84	79.66
12	65.56	31	68.36	49	71.84	67	75.78	85	79.88
13	65.68	32	68.54	50	72.05	68	76.01	86	80.11
14	65.80	33	68.72	51	72.26	69	76.24	87	80.33
15	65.93	34	68.89	52	72.47	70	76.46	88	80.56
16	66.06	35	69.08	53	72.68	71	76.69	89	80.78
17	66.19	36	69.26	54	72.90	72	76.92	90	81.01
18	66.33								

*Beware of confusion between this Table and Table XIII.

TABLE XV

Densities of Solutions of Cane Sugar at 20 °C in g/ml
(This table is the basis for standardizing hydrometers indicating per cent of sugar at 20 °C).

Per cent sugar	Tenths of per cent										Per cent sugar
	.0	.1	.2	.3	.4	.5	.6	.7	.8	.9	
0	0.998234	0.998622	0.999010	0.999398	0.999786	1.000174	1.000563	1.000952	1.001342	1.001731	0
1	1.002120	1.002509	1.002897	1.003286	1.003675	1.004064	1.004453	1.004844	1.005234	1.005624	1
2	1.006015	1.006405	1.006796	1.007188	1.007580	1.007972	1.008363	1.008755	1.009148	1.009541	2
3	1.009934	1.010327	1.010721	1.011115	1.011510	1.011904	1.012298	1.012694	1.013089	1.013485	3
4	1.013881	1.014277	1.014673	1.015070	1.015467	1.015864	1.016261	1.016659	1.017058	1.017456	4
5	1.017854	1.018253	1.018652	1.019052	1.019451	1.019851	1.020251	1.020651	1.021053	1.021454	5
6	1.021855	1.022257	1.022659	1.023061	1.023463	1.023867	1.024270	1.024673	1.025077	1.025481	6
7	1.025885	1.026289	1.026694	1.027099	1.027504	1.027910	1.028316	1.028722	1.029128	1.029535	7
8	1.029942	1.030349	1.030757	1.031165	1.031573	1.031982	1.032391	1.032800	1.033209	1.033619	8
9	1.034029	1.034439	1.034850	1.035260	1.035671	1.036082	1.036494	1.036906	1.037318	1.037730	9
10	1.038143	1.038556	1.038970	1.039383	1.039797	1.040212	1.040626	1.041041	1.041456	1.041872	10
11	1.042288	1.042704	1.043121	1.043537	1.043954	1.044370	1.044788	1.045206	1.045625	1.046043	11
12	1.046462	1.046881	1.047300	1.047720	1.048140	1.048559	1.048980	1.049401	1.049823	1.050243	12
13	1.050665	1.051087	1.051510	1.051933	1.052356	1.052778	1.053202	1.053626	1.054050	1.054475	13
14	1.054900	1.055325	1.055751	1.056176	1.056602	1.057029	1.057455	1.057882	1.058310	1.058737	14
15	1.059165	1.059593	1.060022	1.060451	1.060880	1.061308	1.061738	1.062168	1.062598	1.063029	15
16	1.063460	1.063892	1.064324	1.064756	1.065188	1.065621	1.066054	1.066487	1.066921	1.067355	16
17	1.067789	1.068223	1.068658	1.069093	1.069529	1.069964	1.070400	1.070836	1.071273	1.071710	17
18	1.072147	1.072585	1.073023	1.073461	1.073900	1.074338	1.074777	1.075217	1.075657	1.076097	18
19	1.076537	1.076978	1.077419	1.077860	1.078302	1.078744	1.079187	1.079629	1.080072	1.080515	19
20	1.080959	1.081403	1.081848	1.082292	1.082737	1.083182	1.083628	1.084074	1.084520	1.084967	20
21	1.085414	1.085861	1.086309	1.086757	1.087205	1.087652	1.088101	1.088550	1.089000	1.089450	21
22	1.089900	1.090351	1.090802	1.091253	1.091704	1.092155	1.092607	1.093060	1.093513	1.093966	22
23	1.094420	1.094874	1.095328	1.095782	1.096236	1.096691	1.097147	1.097603	1.098053	1.098514	23
24	1.098971	1.099428	1.099886	1.100344	1.100802	1.101259	1.101718	1.102177	1.102637	1.103097	24
25	1.103557	1.104017	1.104478	1.104938	1.105400	1.105862	1.106324	1.106786	1.107248	1.107711	25
26	1.108175	1.108639	1.109103	1.109568	1.110033	1.110497	1.110963	1.111429	1.111895	1.112361	26
27	1.112828	1.113295	1.113763	1.114229	1.114697	1.115166	1.115635	1.116104	1.116572	1.117042	27
28	1.117512	1.117982	1.118453	1.118923	1.119395	1.119867	1.120339	1.120812	1.121284	1.121757	28
29	1.122231	1.122705	1.123179	1.123653	1.124128	1.124603	1.125079	1.125555	1.126030	1.126507	29

*All weights in vacuo—International Critical Tables 2, 343.

TABLE XV P.2.

Per cent sugar	Tenths of per cent										Per cent sugar
	0	1	2	3	4	5	6	7	8	9	
30	1.126984	1.127461	1.127939	1.128417	1.128896	1.129374	1.129853	1.130332	1.130812	1.131292	30
31	1.131773	1.132254	1.132735	1.133216	1.133698	1.134180	1.134663	1.135146	1.135628	1.136112	31
32	1.136596	1.137080	1.137565	1.138049	1.138534	1.139020	1.139506	1.139993	1.140479	1.140966	32
33	1.141453	1.141941	1.142429	1.142916	1.143405	1.143894	1.144384	1.144874	1.145363	1.145854	33
34	1.146345	1.146836	1.147328	1.147820	1.148313	1.148805	1.149298	1.149792	1.150286	1.150780	34
35	1.151275	1.151770	1.152265	1.152760	1.153256	1.153752	1.154249	1.154746	1.155242	1.155740	35
36	1.156238	1.156736	1.157235	1.157733	1.158233	1.158733	1.159233	1.159733	1.160233	1.160734	36
37	1.161236	1.161738	1.162240	1.162742	1.163245	1.163748	1.164252	1.164756	1.165259	1.165764	37
38	1.166269	1.166775	1.167281	1.167786	1.168293	1.168800	1.169307	1.169815	1.170322	1.170831	38
39	1.171340	1.171849	1.172359	1.172869	1.173379	1.173889	1.174400	1.174911	1.175423	1.175935	39
40	1.176447	1.176960	1.177473	1.177987	1.178501	1.179014	1.179527	1.180044	1.180560	1.181076	40
41	1.181592	1.182108	1.182625	1.183142	1.183660	1.184178	1.184696	1.185215	1.185734	1.186253	41
42	1.186773	1.187293	1.187814	1.188335	1.188856	1.189379	1.189901	1.190423	1.190946	1.191469	42
43	1.191993	1.192517	1.193041	1.193565	1.194090	1.194616	1.195141	1.195667	1.196193	1.196720	43
44	1.197247	1.197775	1.198303	1.198832	1.199360	1.199890	1.200420	1.200950	1.201480	1.202010	44
45	1.202540	1.203071	1.203603	1.204136	1.204668	1.205200	1.205733	1.206266	1.206801	1.207335	45
46	1.207870	1.208400	1.208940	1.209477	1.210013	1.210549	1.211086	1.211623	1.212162	1.212700	46
47	1.213238	1.213777	1.214317	1.214856	1.215395	1.215936	1.216476	1.217017	1.217559	1.218101	47
48	1.218643	1.219185	1.219729	1.220272	1.220815	1.221360	1.221904	1.222449	1.222995	1.223540	48
49	1.224086	1.224632	1.225180	1.225727	1.226274	1.226823	1.227371	1.227919	1.228469	1.229018	49
50	1.229567	1.230117	1.230668	1.231219	1.231770	1.232322	1.232874	1.233426	1.233979	1.234532	50
51	1.235085	1.235639	1.236194	1.236748	1.237303	1.237859	1.238414	1.238970	1.239527	1.240084	51
52	1.240641	1.241198	1.241757	1.242315	1.242873	1.243433	1.243992	1.244552	1.245113	1.245673	52
53	1.246234	1.246795	1.247358	1.247920	1.248482	1.249046	1.249609	1.250172	1.250737	1.251301	53
54	1.251866	1.252431	1.252997	1.253563	1.254129	1.254697	1.255264	1.255831	1.256400	1.256967	54
55	1.257535	1.258104	1.258674	1.259244	1.259815	1.260385	1.260955	1.261527	1.262099	1.262671	55
56	1.263243	1.263816	1.264390	1.264963	1.265537	1.266112	1.266686	1.267261	1.267837	1.268413	56
57	1.268989	1.269565	1.270143	1.270720	1.271299	1.271877	1.272455	1.273035	1.273614	1.274194	57
58	1.274774	1.275353	1.275936	1.276517	1.277098	1.277680	1.278262	1.278844	1.279428	1.280011	58
59	1.280595	1.281179	1.281764	1.282349	1.282935	1.283521	1.284107	1.284694	1.285281	1.285869	59
60	1.286456	1.287044	1.287633	1.288222	1.288811	1.289401	1.289991	1.290581	1.291172	1.291763	60
61	1.292354	1.292946	1.293539	1.294131	1.294725	1.295318	1.295911	1.296506	1.297100	1.297696	61
62	1.298291	1.298886	1.299483	1.300079	1.300677	1.301274	1.301871	1.302470	1.303068	1.303668	62
63	1.304267	1.304867	1.305467	1.306068	1.306669	1.307271	1.307872	1.308475	1.309077	1.309680	63
64	1.310282	1.310885	1.311489	1.312093	1.312699	1.313304	1.313909	1.314515	1.315121	1.315728	64

TABLE XV P.3.

Per cent sugar	Tenths of per cent										Per cent sugar
	0	1	2	3	4	5	6	7	8	9	
65	1.316334	1.316941	1.317549	1.318157	1.318766	1.319374	1.319983	1.320593	1.321203	1.321814	65
66	1.322425	1.323036	1.323648	1.324259	1.324872	1.325484	1.326097	1.326711	1.327325	1.327940	66
67	1.328554	1.329170	1.329785	1.330401	1.331017	1.331633	1.332250	1.332868	1.333485	1.334103	67
68	1.334722	1.335342	1.335961	1.336581	1.337200	1.337821	1.338441	1.339063	1.339684	1.340306	68
69	1.340928	1.341551	1.342174	1.342798	1.343421	1.344046	1.344671	1.345296	1.345922	1.346547	69
70	1.347174	1.347801	1.348427	1.349055	1.349682	1.350311	1.350939	1.351568	1.352197	1.352827	70
71	1.353456	1.354087	1.354717	1.355349	1.355980	1.356612	1.357245	1.357877	1.358511	1.359144	71
72	1.359778	1.360413	1.361047	1.361682	1.362317	1.362953	1.363590	1.364226	1.364864	1.365501	72
73	1.366139	1.366777	1.367415	1.368054	1.368693	1.369333	1.369973	1.370613	1.371254	1.371894	73
74	1.372536	1.373178	1.373820	1.374463	1.375105	1.375749	1.376392	1.377036	1.377680	1.378326	74
75	1.378971	1.379617	1.380262	1.380909	1.381555	1.382203	1.382851	1.383499	1.384148	1.384796	75
76	1.385446	1.386096	1.386745	1.387396	1.388045	1.388696	1.389347	1.389999	1.390651	1.391303	76
77	1.391956	1.392610	1.393263	1.393917	1.394571	1.395226	1.395881	1.396536	1.397192	1.397848	77
78	1.398505	1.399162	1.399819	1.400477	1.401134	1.401793	1.402452	1.403111	1.403771	1.404430	78
79	1.405091	1.405752	1.406412	1.407074	1.407735	1.408398	1.409061	1.409723	1.410387	1.411051	79
80	1.411715	1.412380	1.413044	1.413709	1.414374	1.415040	1.415706	1.416373	1.417039	1.417707	80
81	1.418374	1.419043	1.419711	1.420380	1.421049	1.421719	1.422390	1.423059	1.423730	1.424400	81
82	1.425072	1.425744	1.426416	1.427089	1.427761	1.428435	1.429109	1.429782	1.430457	1.431131	82
83	1.431807	1.432483	1.433158	1.433835	1.434511	1.435188	1.435866	1.436543	1.437222	1.437900	83
84	1.438579	1.439259	1.439938	1.440619	1.441299	1.441980	1.442661	1.443342	1.444024	1.444705	84
85	1.445388	1.446071	1.446754	1.447438	1.448121	1.448806	1.449491	1.450175	1.450860	1.451545	85
86	1.452232	1.452919	1.453605	1.454292	1.454980	1.455668	1.456357	1.457045	1.457735	1.458424	86
87	1.459114	1.459805	1.460495	1.461186	1.461877	1.462568	1.463260	1.463953	1.464645	1.465338	87
88	1.466032	1.466726	1.467420	1.468115	1.468810	1.469504	1.470200	1.470896	1.471592	1.472289	88
89	1.472986	1.473684	1.474381	1.475080	1.475779	1.476477	1.477176	1.477876	1.478575	1.479275	89
90	1.479976	1.480677	1.481378	1.482080	1.482782	1.483484	1.484187	1.484890	1.485593	1.486297	90
91	1.487002	1.487707	1.488411	1.489117	1.489823	1.490528	1.491234	1.491941	1.492647	1.493355	91
92	1.494063	1.494771	1.495479	1.496188	1.496897	1.497606	1.498316	1.499026	1.499736	1.500447	92
93	1.501158	1.501870	1.502582	1.503293	1.504006	1.504719	1.505432	1.506146	1.506859	1.507574	93
94	1.508289	1.509004	1.509720	1.510435	1.511151	1.511868	1.512585	1.513302	1.514019	1.514737	94
95	1.515455	1.516174	1.516893	1.517612	1.518332	1.519051	1.519771	1.520492	1.521212	1.521934	95
96	1.522656	1.523378	1.524102	1.524823	1.525546	1.526269	1.526993	1.527717	1.528441	1.529166	96
97	1.529891	1.530616	1.531342	1.532068	1.532794	1.533521	1.534248	1.534976	1.535704	1.536432	97
98	1.537161	1.537889	1.538618	1.539347	1.540076	1.540806	1.541536	1.542267	1.542998	1.543730	98
99	1.544462	1.545194	1.545926	1.546659	1.547392	1.548127	1.548861	1.549595	1.550329	1.551064	99
100	1.551800	100

This Table has been calculated on the mass-based 'old' millilitre. To convert to values calculated on the 'new' millilitre (based on volume), the values in the Table should be divided by 1.000028, where such precision is required.

TABLE XVI

Table XVI—Brix, Apparent Density, Apparent Specific Gravity, and Grams of Sucrose per 100 ml of Sugar Solutions

(NBS—C440, 1942, p. 632)

Column 1 gives Brix or percentage of sucrose in the solution.

Column 2 gives apparent density, that is, the weight in air with brass weights of 1 ml of solution at 20°C. The values in this column correspond to the values of true density (table XV), having been obtained by means of the formula

$$M = W \left[1 + \frac{\rho}{d_2} \left(\frac{d_2 - d_1}{d_1 - \rho} \right) \right] = W \left(1 + \frac{k}{1000} \right),$$

which may be utilized for converting apparent density into true density, and vice versa, by considering that M , the weight in vacuo, and W , the apparent weight, refer to 1 ml, since true density is defined as the weight in vacuo of 1 ml, and the apparent density as the weight of 1 ml of substance in air with brass weights. ρ is the density of air, which has been taken as 0.0012046; d_1 the density of the solution, d_2 the density of the weights, which has been taken as 8.4 g/ml.

Column 3 gives the apparent specific gravity at 20°C. The values in this column were obtained by dividing the apparent density in column 2 by the apparent density of water at 20°C, which was taken as 0.997174.

Column 4 gives the grams sucrose (weighed in vacuo) per 100 ml of solution.

The values in the table were calculated in three sections by different individuals; thus from 40 to 60 Brix by Peters and Phelps (BS Tech. Paper T338, 1927); 60 to 83.9 Brix by Brewster and Phelps (NBS Research Paper RP536, 1933); and the remaining values 0 to 40 and 84 to 93 Brix by Snyder, Saunders, and Golden of the National Bureau of Standards. After the computations were completed, the tabulations were made by rounding off the values to the last figure given. The values are considered exact to ± 1 in the fifth decimal.

Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo	Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo
1	2	3	4	1	2	3	4
0.0	0.99717	1.00000	0.000	2.0	1.00495	1.00780	2.012
.1	.99756	.00039	.100	.1	.00534	.00819	.113
.2	.99795	.00078	.200	.2	.00574	.00859	.215
.3	.99834	.00117	.300	.3	.00613	.00898	.317
.4	.99872	.00156	.400	.4	.00652	.00937	.418
.5	.99911	.00194	.500	.5	.00691	.00977	.520
.6	.99950	.00233	.600	.6	.00730	.01016	.622
.7	.99989	.00272	.701	.7	.00769	.01055	.724
.8	1.00028	.00312	.801	.8	.00809	.01094	.826
.9	.00067	.00351	.902	.9	.00848	.01134	.928
1.0	1.00106	1.00390	1.002	3.0	1.00887	1.01173	3.030
.1	.00145	.00429	.103	.1	.00927	.01213	.132
.2	.00184	.00468	.203	.2	.00966	.01252	.234
.3	.00223	.00507	.304	.3	.01006	.01292	.337
.4	.00261	.00546	.405	.4	.01045	.01331	.439
.5	.00300	.00585	.506	.5	.01084	.01371	.542
.6	.00339	.00624	.607	.6	.01124	.01410	.644
.7	.00378	.00663	.708	.7	.01163	.01450	.747
.8	.00417	.00702	.809	.8	.01203	.01490	.850
.9	.00456	.00741	.911	.9	.01243	.01529	.953

TABLE XVI P.2.

Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo	Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo
1	2	3	4	1	2	3	4
4.0	1.01282	1.01569	4.056	9.0	1.03297	1.03590	9.306
.1	.01322	.01609	.159	.1	.03338	.03631	.413
.2	.01361	.01649	.262	.2	.03379	.03672	.521
.3	.01401	.01688	.365	.3	.03420	.03713	.628
.4	.01441	.01728	.468	.4	.03461	.03755	.735
.5	.01480	.01768	.571	.5	.03503	.03796	.843
.6	.01520	.01808	.675	.6	.03544	.03837	.950
.7	.01560	.01848	.778	.7	.03585	.03879	10.058
.8	.01600	.01888	.882	.8	.03626	.03920	.166
.9	.01640	.01928	.986	.9	.03667	.03961	.274
5.0	1.01680	1.01968	5.089	10.0	1.03709	1.04003	10.381
.1	.01719	.02008	.193	.1	.03750	.04044	.489
.2	.01759	.02048	.297	.2	.03791	.04086	.597
.3	.01799	.02088	.401	.3	.03833	.04127	.706
.4	.01839	.02128	.506	.4	.03874	.04169	.814
.5	.01879	.02168	.609	.5	.03916	.04210	.922
.6	.01919	.02208	.713	.6	.03957	.04252	11.031
.7	.01959	.02248	.818	.7	.03999	.04293	.139
.8	.01999	.02289	.922	.8	1.04040	.04335	.248
.9	.02040	.02329	6.027	.9	.04082	.04377	.356
6.0	1.02080	1.02369	6.131	11.0	1.04123	1.04418	11.465
.1	.02120	.02409	.236	.1	.04165	.04460	.574
.2	.02160	.02450	.340	.2	.04207	.04502	.683
.3	.02200	.02490	.445	.3	.04248	.04544	.792
.4	.02241	.02530	.550	.4	.04290	.04585	.901
.5	.02281	.02571	.655	.5	.04332	.04627	12.010
.6	.02321	.02611	.760	.6	.04373	.04669	.120
.7	.02362	.02652	.865	.7	.04415	.04711	.229
.8	.02402	.02692	.971	.8	.04457	.04753	.338
.9	.02442	.02733	7.076	.9	.04499	.04795	.448
7.0	1.02483	1.02773	7.181	12.0	1.04541	1.04837	12.558
.1	.02523	.02814	.287	.1	.04583	.04879	.667
.2	.02564	.02854	.392	.2	.04625	.04921	.777
.3	.02604	.02895	.498	.3	.04667	.04963	.887
.4	.02645	.02936	.604	.4	.04709	1.05005	.997
.5	.02685	.02976	.709	.5	.04750	.05047	13.107
.6	.02726	.03017	.815	.6	.04793	.05090	.217
.7	.02766	.03058	.921	.7	.04835	.05132	.327
.8	.02807	.03098	8.027	.8	.04877	.05174	.438
.9	.02848	.03139	.133	.9	.04919	.05216	.548
8.0	1.02888	1.03180	8.240	13.0	1.04961	1.05259	13.659
.1	.02929	.03221	.346	.1	1.05003	.05301	.769
.2	.02970	.03262	.452	.2	.05046	.05343	.880
.3	.03011	.03303	.559	.3	.05088	.05386	.991
.4	.03052	.03344	.665	.4	.05130	.05428	14.102
.5	.03093	.03385	.772	.5	.05172	.05470	.213
.6	.03133	.03426	.879	.6	.05215	.05513	.324
.7	.03174	.03467	.985	.7	.05257	.05556	.435
.8	.03215	.03508	9.092	.8	.05300	.05598	.546
.9	.03256	.03549	.199	.9	.05342	.05641	.657

TABLE XVI P. 3.

Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo	Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo
1	2	3	4	1	2	3	4
14.0	1.05385	1.05683	14.769	19.0	1.07549	1.07853	20.454
.1	.05427	.05726	.880	.1	.07593	.07898	.570
.2	.05470	.05769	.992	.2	.07637	.07942	.686
.3	.05512	.05811	15.103	.3	.07681	.07986	.803
.4	.05555	.05854	.215	.4	.07725	1.08030	.919
.5	.05598	.05897	.327	.5	.07769	.08075	21.036
.6	.05640	.05940	.439	.6	.07814	.08119	.152
.7	.05683	.05982	.551	.7	.07858	.08164	.269
.8	.05726	1.06025	.663	.8	.07902	.08208	.385
.9	.05768	.06068	.775	.9	.07947	.08252	.502
15.0	1.05811	1.06111	15.887	20.0	1.07991	1.08297	21.619
.1	.05854	.06154	16.000	.1	1.08035	.08342	.736
.2	.05897	.06197	.112	.2	.08080	.08386	.853
.3	.05940	.06240	.225	.3	.08124	.08431	.971
.4	.05983	.06283	.338	.4	.08169	.08475	22.088
.5	1.06026	.06326	.450	.5	.08213	.08520	.205
.6	.06069	.06369	.563	.6	.08258	.08565	.323
.7	.06112	.06412	.676	.7	.08302	.08609	.440
.8	.06155	.06455	.789	.8	.08347	.08654	.558
.9	.06198	.06499	.902	.9	.08392	.08699	.676
16.0	1.06241	1.06542	17.015	21.0	1.08436	1.08744	22.794
.1	.06284	.06585	.129	.1	.08481	.08789	.912
.2	.06327	.06629	.242	.2	.08526	.08834	23.030
.3	.06370	.06672	.356	.3	.08571	.08879	.148
.4	.06414	.06715	.469	.4	.08616	.08923	.266
.5	.06457	.06759	.583	.5	.08660	.08968	.385
.6	.06500	.06802	.697	.6	.08705	1.09013	.503
.7	.06544	.06845	.810	.7	.08750	.09058	.622
.8	.06587	.06889	.924	.8	.08795	.09103	.740
.9	.06630	.06933	18.038	.9	.08840	.09149	.859
17.0	1.06674	1.06976	18.152	22.0	1.08885	1.09194	23.978
.1	.06717	1.07020	.267	.1	.08930	.09239	24.097
.2	.06761	.07063	.381	.2	.08975	.09284	.216
.3	.06804	.07107	.495	.3	1.09020	.09329	.335
.4	.06848	.07151	.610	.4	.09066	.09375	.454
.5	.06891	.07194	.724	.5	.09111	.09420	.573
.6	.06935	.07238	.839	.6	.09156	.09465	.693
.7	.06978	.07282	.954	.7	.09201	.09511	.812
.8	1.07022	.07325	19.069	.8	.09247	.09556	.932
.9	.07066	.07369	.184	.9	.09292	.09602	25.052
18.0	1.07110	1.07413	19.299	23.0	1.09337	1.09647	25.172
.1	.07153	.07457	.414	.1	.09383	.09693	.292
.2	.07197	.07501	.529	.2	.09428	.09738	.412
.3	.07241	.07545	.644	.3	.09473	.09784	.532
.4	.07285	.07589	.760	.4	.09519	.09829	.652
.5	.07329	.07633	.875	.5	.09564	.09875	.772
.6	.07373	.07677	.991	.6	.09610	.09921	.893
.7	.07417	.07721	20.107	.7	.09656	1.09966	26.013
.8	.07461	.07765	.222	.8	.09701	1.10012	.134
.9	.07505	.07809	.338	.9	.09747	.10058	.255

TABLE XVI P.4.

Percentage of sucrose by weight (Brix)	Apparent density at 20 C	Apparent specific gravity at 20 C/20 C	Grams of sucrose per 100 ml weight in vacuo	Percentage of sucrose by weight (Brix)	Apparent density at 20 C	Apparent specific gravity at 20 C/20 C	Grams of sucrose per 100 ml weight in vacuo
1	2	3	4	1	2	3	4
24.0	1.09792	1.10104	26.375	29.0	1.12119	1.12436	32.545
.1	.09838	.10149	.496	.1	.12166	.12484	.671
.2	.09884	.10195	.617	.2	.12214	.12532	.797
.3	.09930	.10241	.738	.3	.12261	.12579	.923
.4	.09976	.10287	.860	.4	.12308	.12627	33.049
.5	1.10021	.10333	.981	.5	.12356	.12674	.176
.6	.10067	.10379	27.102	.6	.12404	.12722	.302
.7	.10113	.10425	.224	.7	.12451	.12770	.429
.8	.10159	.10471	.345	.8	.12499	.12817	.556
.9	.10205	.10517	.467	.9	.12546	.12865	.683
25.0	1.10251	1.10564	27.589	30.0	1.12594	1.12913	33.810
.1	.10297	.10610	.710	.1	.12642	.12961	.937
.2	.10343	.10656	.833	.2	.12690	1.13009	34.064
.3	.10389	.10702	.955	.3	.12737	.13057	.191
.4	.10435	.10748	28.077	.4	.12785	.13105	.318
.5	.10482	.10795	.199	.5	.12833	.13153	.446
.6	.10528	.10841	.322	.6	.12881	.13201	.574
.7	.10574	.10887	.444	.7	.12929	.13249	.701
.8	.10620	.10934	.567	.8	.12977	.13297	.829
.9	.10667	.10980	.690	.9	1.13025	.13345	.957
26.0	1.10713	1.11027	28.813	31.0	1.13073	1.13394	35.085
.1	.10759	.11073	.935	.1	.13121	.13442	.213
.2	.10806	.11120	29.059	.2	.13169	.13490	.341
.3	.10852	.11166	.182	.3	.13217	.13538	.470
.4	.10899	.11213	.305	.4	.13266	.13587	.598
.5	.10945	.11260	.428	.5	.13314	.13635	.727
.6	.10992	.11306	.552	.6	.13362	.13683	.855
.7	1.11038	.11353	.675	.7	.13410	.13732	.984
.8	.11085	.11400	.799	.8	.13459	.13780	36.113
.9	.11131	.11447	.923	.9	.13507	.13829	.242
27.0	1.11178	1.11493	30.046	32.0	1.13555	1.13877	36.371
.1	.11225	.11540	.170	.1	.13604	.13926	.500
.2	.11272	.11587	.294	.2	.13652	.13974	.630
.3	.11318	.11634	.418	.3	.13701	1.14023	.759
.4	.11365	.11681	.543	.4	.13749	.14072	.889
.5	.11412	.11728	.667	.5	.13798	.14120	37.018
.6	.11459	.11775	.792	.6	.13846	.14169	.148
.7	.11506	.11822	.916	.7	.13895	.14218	.278
.8	.11553	.11869	31.041	.8	.13944	.14267	.408
.9	.11600	.11916	.165	.9	.13992	.14316	.538
28.0	1.11647	1.11963	31.290	33.0	1.14041	1.14364	37.668
.1	.11694	1.12010	.415	.1	.14090	.14413	.798
.2	.11741	.12058	.540	.2	.14139	.14462	.929
.3	.11788	.12105	.666	.3	.14188	.14511	38.059
.4	.11835	.12152	.791	.4	.14236	.14560	.190
.5	.11882	.12199	.916	.5	.14285	.14609	.320
.6	.11929	.12247	32.042	.6	.14334	.14658	.451
.7	.11977	.12294	.167	.7	.14383	.14708	.582
.8	1.12024	.12341	.293	.8	.14432	.14757	.713
.9	.12071	.12389	.419	.9	.14481	.14806	.844

TABLE XVI P.5.

Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo	Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo
1	2	3	4	1	2	3	4
34.0	1.14530	1.14855	38.976	39.0	1.17030	1.17362	45.682
.1	.14580	.14904	39.107	.1	.17081	.17413	.819
.2	.14629	.14954	.239	.2	.17132	.17464	.956
.3	.14678	1.15003	.370	.3	.17183	.17515	46.094
.4	.14727	.15052	.502	.4	.17234	.17566	.231
.5	.14776	.15102	.634	.5	.17285	.17618	.369
.6	.14826	.15151	.767	.6	.17336	.17669	.506
.7	.14875	.15201	.898	.7	.17387	.17720	.644
.8	.14925	.15250	40.030	.8	.17439	.17772	.782
.9	.14974	.15300	.162	.9	.17490	.17823	.920
35.0	1.15024	1.15350	40.295	40.0	1.17541	1.17874	47.058
.1	.15073	.15399	.427	40.1	.593	.926	.196
.2	.15123	.15449	.560	40.2	.644	1.17977	.334
.3	.15172	.15498	.692	40.3	.695	1.18029	.473
.4	.15222	.15548	.825	40.4	.747	.080	.611
.5	.15271	.15598	.958	40.5	1.17798	1.18132	.750
.6	.15321	.15648	41.091	40.6	.849	.183	47.889
.7	.15371	.15698	.224	40.7	.901	.235	48.028
.8	.15420	.15747	.358	40.8	1.17953	.287	.167
.9	.15470	.15797	.491	40.9	1.18004	.339	.306
36.0	1.15520	1.15847	41.625	41.0	1.18056	1.18390	48.445
.1	.15570	.15897	.758	41.1	.107	.442	.585
.2	.15620	.15947	.892	41.2	.159	.494	.724
.3	.15669	.15997	42.026	41.3	.211	.546	.864
.4	.15719	1.16047	.160	41.4	.263	.598	49.004
.5	.15769	.16098	.294	41.5	1.18314	1.18650	.143
.6	.15819	.16148	.428	41.6	.356	.702	.283
.7	.15869	.16198	.562	41.7	.418	.754	.424
.8	.15919	.16248	.697	41.8	.470	.806	.564
.9	.15970	.16298	.831	41.9	.522	.858	.704
37.0	1.16020	1.16349	42.966	42.0	1.18574	1.18910	49.845
.1	.16070	.16399	43.100	42.1	.626	1.18962	49.985
.2	.16120	.16449	.235	42.2	.678	1.19014	50.126
.3	.16170	.16500	.370	42.3	.730	.062	.267
.4	.16221	.16550	.505	42.4	.782	.119	50.408
.5	.16271	.16601	.641	42.5	1.18835	1.19171	.549
.6	.16321	.16651	.776	42.6	.887	.224	.690
.7	.16372	.16702	.911	42.7	.939	.276	.831
.8	.16422	.16752	44.047	42.8	.991	.329	50.973
.9	.16473	.16803	.182	42.9	1.19044	.381	51.114
38.0	1.16523	1.16853	44.318	43.0	1.19096	1.19434	51.256
.1	.16574	.16904	.454	43.1	.148	.486	.398
.2	.16624	.16955	.590	43.2	.201	.539	.539
.3	.16675	1.17006	.726	43.3	.253	.591	.681
.4	.16726	.17056	.862	43.4	.306	.644	.824
.5	.16776	.17107	.999	43.5	1.19358	1.19697	51.966
.6	.16827	.17158	45.135	43.6	.411	.749	52.108
.7	.16878	.17209	.272	43.7	.483	.802	.251
.8	.16929	.17260	.408	43.8	.516	.855	.393
.9	.16979	.17311	.545	43.9	.569	.908	.536

TABLE XVI P.6.

Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo	Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo
1	2	3	4	1	2	3	4
44.0	1.19622	1.19961	52.679	49.0	1.22306	1.22652	59.980
44.1	674	1.20013	.822	49.1	360	707	60.129
44.2	727	066	52.965	49.2	415	762	.279
44.3	780	119	53.108	49.3	470	817	.428
44.4	833	172	.252	49.4	525	872	.578
44.5	1.19886	1.20226	.395	49.5	1.22580	1.22927	.728
44.6	939	279	.539	49.6	634	1.22982	60.878
44.7	992	332	.683	49.7	689	1.23037	61.028
44.8	1.20045	385	.826	49.8	744	092	.178
44.9	098	438	53.970	49.9	799	147	61.328
45.0	1.20151	1.20491	54.114	50.0	1.22854	1.23202	61.478
45.1	204	545	.259	50.1	909	1.23257	.629
45.2	257	598	.403	50.2	1.22964	313	.780
45.3	311	651	.547	50.3	1.23019	368	.930
45.4	364	705	.692	50.4	074	423	62.081
45.5	1.20417	1.20758	.837	50.5	1.23130	1.23478	.232
45.6	470	812	54.981	50.6	185	534	.383
45.7	524	865	55.126	50.7	240	589	.535
45.8	577	919	.272	50.8	295	645	.686
45.9	630	1.20972	.417	50.9	351	700	.838
46.0	1.20684	1.21026	55.562	51.0	1.23406	1.23756	62.989
46.1	737	080	.708	51.1	461	811	63.141
46.2	791	133	.853	51.2	517	867	.293
46.3	845	187	55.999	51.3	572	922	.445
46.4	898	241	56.145	51.4	628	1.23978	.597
46.5	1.20952	1.21295	.291	51.5	1.23683	1.24034	.750
46.6	1.21006	349	.437	51.6	739	089	.902
46.7	059	402	.583	51.7	794	145	64.055
46.8	113	456	.729	51.8	850	201	.208
46.9	167	510	56.876	51.9	906	257	.360
47.0	1.21221	1.21564	57.022	52.0	1.23962	1.24313	64.513
47.1	275	618	.169	52.1	1.24017	369	.666
47.2	329	673	.316	52.2	073	425	.820
47.3	383	727	.463	52.3	129	481	.973
47.4	437	781	57.610	52.4	185	537	65.127
47.5	1.21491	1.21835	57.757	52.5	1.24241	1.24593	65.280
47.6	545	889	57.904	52.6	297	649	.433
47.7	599	943	58.052	52.7	353	705	.588
47.8	653	1.21998	.199	52.8	409	761	.742
47.9	707	1.22052	.347	52.9	465	818	.896
48.0	1.21761	1.22106	58.495	53.0	1.24521	1.24874	66.050
48.1	816	161	.643	53.1	577	930	.205
48.2	870	215	.791	53.2	633	987	.359
48.3	924	270	58.939	53.3	690	1.25043	.514
48.4	979	324	59.087	53.4	746	099	.669
48.5	1.22033	1.22379	.236	53.5	1.24802	1.25156	.824
48.6	088	434	.385	53.6	858	212	.979
48.7	142	488	.533	53.7	915	269	67.134
48.8	197	543	.682	53.8	971	325	.290
48.9	251	598	.831	53.9	1.25028	382	.445

TABLE XVI P.7.

Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo	Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo
1	2	3	4	1	2	3	4
54.0	1.25084	1.25439	67.601	59.0	1.27958	1.28320	75.555
54.1	141	495	.757	59.1	1.28017	379	.718
54.2	197	552	.912	59.2	075	437	.880
54.3	254	609	68.069	59.3	134	497	76.043
54.4	311	666	.225	59.4	193	556	.207
54.5	1.25367	1.25723	.381	59.5	251	614	.369
54.6	424	780	.537	59.6	309	672	.533
54.7	481	836	.694	59.7	367	731	.696
54.8	538	893	.851	59.8	426	789	.860
54.9	594	950	69.008	59.9	485	849	77.024
55.0	1.25651	1.26007	69.164	60.0	1.28544	1.28908	77.188
55.1	708	064	.322	60.1	602	966	.351
55.2	765	122	.479	60.2	661	1.29025	.515
55.3	822	179	.636	60.3	720	084	.680
55.4	879	236	.794	60.4	779	143	.844
55.5	1.25936	1.26293	69.951	60.5	838	203	78.009
55.6	1.25993	350	70.109	60.6	897	262	.173
55.7	1.26050	408	.267	60.7	956	321	.338
55.8	108	465	.425	60.8	1.29015	380	.503
55.9	165	522	.583	60.9	074	439	.668
56.0	1.26222	1.26580	70.742	61.0	1.29133	1.29498	78.833
56.1	279	637	70.900	61.1	193	559	.999
56.2	337	695	71.059	61.2	252	618	79.165
56.3	394	752	.217	61.3	311	677	.330
56.4	452	810	.376	61.4	370	736	.496
56.5	1.26509	1.26868	.535	61.5	430	796	.662
56.6	566	925	.694	61.6	489	855	.828
56.7	624	1.26983	71.854	61.7	548	915	.995
56.8	682	1.27041	72.013	61.8	608	975	80.161
56.9	739	098	.173	61.9	667	1.30034	.328
57.0	1.26797	1.27156	72.332	62.0	1.29726	1.30093	80.494
57.1	854	214	.492	62.1	786	153	.661
57.2	912	272	.652	62.2	845	212	.828
57.3	970	330	.812	62.3	905	273	.995
57.4	1.27028	388	72.973	62.4	966	334	81.162
57.5	1.27086	1.27446	73.133	62.5	1.30025	393	.329
57.6	143	504	.293	62.6	085	453	.497
57.7	201	562	.454	62.7	145	513	.665
57.8	259	620	.615	62.8	205	573	.833
57.9	317	678	.776	62.9	265	633	82.001
58.0	1.27375	1.27736	73.937	63.0	1.30325	1.30694	82.169
58.1	433	794	74.098	63.1	385	754	.337
58.2	492	853	.260	63.2	446	815	.506
58.3	550	911	.421	63.3	506	875	.674
58.4	608	1.27969	.583	63.4	566	936	.843
58.5	1.27664	1.28028	.744	63.5	626	994	83.012
58.6	724	086	74.906	63.6	686	1.31055	.180
58.7	782	145	75.068	63.7	747	117	.350
58.8	841	203	.230	63.8	807	177	.519
58.9	899	262	.393	63.9	867	237	.688

TABLE XVI P.8.

Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo	Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo
1	2	3	4	1	2	3	4
64.0	1.30927	1.31297	83.858	69.0	1.33992	1.34371	92.524
64.1	988	359	84.028	69.1	1.34054	433	.701
64.2	1.31048	418	.198	69.2	116	495	.878
64.3	108	479	.367	69.3	179	558	93.056
64.4	169	540	.538	69.4	241	621	.233
64.5	229	600	.708	69.5	304	684	.411
64.6	290	661	.879	69.6	366	746	.589
64.7	350	723	85.049	69.7	429	809	.767
64.8	412	784	.220	69.8	491	871	.945
64.9	473	845	.391	69.9	554	934	94.123
65.0	1.31533	1.31905	85.561	70.0	1.34616	1.34997	94.302
65.1	594	966	.733	70.1	679	1.35060	.481
65.2	655	1.32028	.904	70.2	742	123	.660
65.3	716	089	86.076	70.3	805	186	.839
65.4	777	150	.248	70.4	867	248	95.017
65.5	837	210	.419	70.5	930	311	.197
65.6	898	271	.591	70.6	993	375	.376
65.7	959	332	.763	70.7	1.35056	438	.556
65.8	1.32019	393	.935	70.8	119	501	.736
65.9	081	455	87.107	70.9	182	564	.916
66.0	1.32142	1.32516	87.280	71.0	1.35245	1.35627	96.096
66.1	203	577	.453	71.1	308	691	.276
66.2	264	638	.626	71.2	371	754	.456
66.3	325	699	.798	71.3	434	817	.636
66.4	385	759	.971	71.4	498	881	.817
66.5	446	820	88.142	71.5	561	944	.998
66.6	509	884	.318	71.6	625	1.36008	97.179
66.7	570	945	.492	71.7	688	072	.360
66.8	632	1.33007	.666	71.8	751	135	.541
66.9	693	068	.839	71.9	814	198	.722
67.0	1.32754	1.33129	89.012	72.0	1.35877	1.36261	97.904
67.1	816	192	.187	72.1	940	324	98.085
67.2	878	254	.361	72.2	1.36004	389	.268
67.3	939	315	.536	72.3	067	452	.449
67.4	1.33001	377	.711	72.4	131	516	.632
67.5	062	438	.885	72.5	194	579	.814
67.6	124	500	90.060	72.6	258	643	.997
67.7	186	562	.235	72.7	322	707	99.179
67.8	248	625	.411	72.8	385	771	.362
67.9	309	686	.585	72.9	450	836	.545
68.0	1.33371	1.33748	90.761	73.0	1.36514	1.36900	99.728
68.1	433	810	.937	73.1	578	964	.912
68.2	495	872	91.112	73.2	642	1.37028	100.095
68.3	557	935	.288	73.3	705	092	.278
68.4	619	997	.464	73.4	769	156	.462
68.5	681	1.34059	.641	73.5	833	220	.646
68.6	743	121	.817	73.6	896	283	.827
68.7	805	183	.993	73.7	960	347	101.014
68.8	867	245	92.169	73.8	1.37024	411	.198
68.9	930	309	.347	73.9	088	476	.383

TABLE XVI P.9.

Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo	Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo
1	2	3	4	1	2	3	4
74.0	1.37153	1.37541	101.568	79.0	1.40409	1.40806	111.002
74.1	217	605	.753	79.1	475	872	.195
74.2	281	669	.937	79.2	541	938	.388
74.3	345	733	102.122	79.3	607	1.41005	.581
74.4	410	798	.308	79.4	674	072	.775
74.5	475	864	.493	79.5	740	138	.968
74.6	539	928	.679	79.6	806	204	112.161
74.7	604	993	.865	79.7	872	270	.354
74.8	668	1.38057	103.050	79.8	939	337	.549
74.9	733	122	.237	79.9	1.41005	404	.743
75.0	1.37797	1.38187	103.423	80.0	1.41072	1.41471	112.938
75.1	862	252	.609	80.1	138	537	113.131
75.2	926	316	.796	80.2	204	603	.326
75.3	991	381	.983	80.3	271	670	.521
75.4	1.38055	445	104.170	80.4	337	737	.715
75.5	119	1.38510	104.356	80.5	404	804	.911
75.6	184	575	.543	80.6	472	872	114.106
75.7	249	640	.731	80.7	537	937	.301
75.8	314	705	.919	80.8	604	1.42004	.497
75.9	379	770	105.106	80.9	671	072	.692
76.0	1.38444	1.38835	105.294	81.0	1.41737	1.42138	114.888
76.1	510	902	.482	81.1	804	205	115.084
76.2	575	967	.670	81.2	871	272	.280
76.3	640	1.39032	.859	81.3	938	339	.477
76.4	705	097	106.047	81.4	1.42005	406	.673
76.5	770	162	.236	81.5	072	474	.870
76.6	835	228	.424	81.6	139	541	116.067
76.7	900	293	.613	81.7	206	608	.264
76.8	965	358	.802	81.8	273	675	.461
76.9	1.39030	423	.991	81.9	340	742	.658
77.0	1.39096	1.39489	107.181	82.0	1.42407	1.42810	116.856
77.1	161	554	.370	82.1	475	878	117.053
77.2	225	619	.560	82.2	543	946	.252
77.3	291	685	.750	82.3	610	1.43013	.449
77.4	356	750	.940	82.4	677	080	.647
77.5	422	816	108.130	82.5	744	148	.845
77.6	488	882	.320	82.6	811	214	118.044
77.7	554	949	.511	82.7	878	282	.243
77.8	619	1.40014	.701	82.8	946	350	.442
77.9	685	080	.892	82.9	1.43013	417	.641
78.0	1.39751	1.40146	109.084	83.0	1.43081	1.43486	118.840
78.1	816	211	.274	83.1	148	553	119.039
78.2	882	277	.466	83.2	216	621	.239
78.3	948	344	.657	83.3	283	688	.438
78.4	1.40013	409	.848	83.4	351	756	.638
78.5	079	475	110.041	83.5	419	824	.838
78.6	145	541	.232	83.6	488	894	120.039
78.7	211	607	.425	83.7	555	961	.238
78.8	277	674	.617	83.8	623	1.44029	.439
78.9	343	740	.809	83.9	691	097	.640

TABLE XVI P.10.

Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo	Percentage of sucrose by weight (Brix)	Apparent density at 20°C	Apparent specific gravity at 20°C/20°C	Grams of sucrose per 100 ml weight in vacuo
1	2	3	4	1	2	3	4
84.0	1.43758	1.44165	120.841	89.0	1.47199	1.47616	131.096
.1	.43826	.44234	121.042	.1	.47269	.47686	.305
.2	.43894	.44302	.243	.2	.47339	.47756	.515
.3	.43962	.44370	.444	.3	.47409	.47826	.725
.4	1.44030	.44438	.646	.4	.47479	.47897	.935
.5	.44098	.44507	.847	.5	.47548	.47967	132.145
.6	.44166	.44575	122.049	.6	.47618	1.48037	.355
.7	.44234	.44643	.251	.7	.47688	.48107	.565
.8	.44303	.44712	.453	.8	.47758	.48177	.776
.9	.44371	.44780	.655	.9	.47828	.48247	.987
85.0	1.44439	1.44848	122.858	90.0	1.47898	1.48317	133.198
.1	.44507	.44917	123.061	.1	.47968	.48388	.409
.2	.44576	.44985	.263	.2	1.48039	.48458	.620
.3	.44644	1.45054	.466	.3	.48109	.48529	.832
.4	.44712	.45123	.670	.4	.48179	.48599	134.043
.5	.44781	.45191	.873	.5	.48249	.48669	.255
.6	.44849	.45260	124.076	.6	.48320	.48740	.467
.7	.44918	.45329	.280	.7	.48390	.48810	.680
.8	.44986	.45397	.484	.8	.48460	.48881	.892
.9	1.45055	.45466	.688	.9	.48531	.48951	135.104
86.0	1.45124	1.45535	124.892	91.0	1.48601	1.49022	135.317
.1	.45192	.45604	125.096	.1	.48672	.49093	.530
.2	.45261	.45673	.301	.2	.48742	.49164	.743
.3	.45330	.45741	.505	.3	.48813	.49234	.956
.4	.45398	.45810	.710	.4	.48883	.49305	136.170
.5	.45467	.45879	.915	.5	.48954	.49376	.383
.6	.45536	.45949	126.121	.6	1.49024	.49447	.597
.7	.45605	1.46018	.326	.7	.49095	.49518	.811
.8	.45674	.46087	.531	.8	.49166	.49588	137.025
.9	.45743	.46156	.737	.9	.49236	.49659	.239
87.0	1.45812	1.46225	126.943	92.0	1.49307	1.49730	137.454
.1	.45881	.46294	127.149	.1	.49378	.49801	.668
.2	.45950	.46364	.355	.2	.49449	.49872	.883
.3	1.46019	.46433	.562	.3	.49520	.49944	138.098
.4	.46088	.46502	.768	.4	.49591	1.50015	.313
.5	.46157	.46572	.975	.5	.49662	.50086	.529
.6	.46227	.46641	128.182	.6	.49733	.50157	.744
.7	.46296	.46710	.389	.7	.49804	.50228	.960
.8	.46365	.46780	.596	.8	.49875	.50299	139.176
.9	.46434	.46849	.803	.9	.49946	.50371	.392
88.0	1.46504	1.46919	129.011	93.0	1.50017	1.50442	139.608
.1	.46573	.46989	.219	.1	.50088	.50513	.824
.2	.46643	1.47058	.426	.2	.50159	.50585	140.041
.3	.46712	.47128	.635	.3	.50230	.50656	.257
.4	.46782	.47198	.843	.4	.50302	.50728	.474
.5	.46851	.47267	130.051	.5	.50373	.50799	.691
.6	.46921	.47337	.260	.6	.50444	.50871	.908
.7	.46990	.47407	.468	.7	.50516	.50942	141.126
.8	1.47060	.47477	.677	.8	.50587	1.51014	.343
.9	.47130	.47547	.886	.9	.50659	.51086	.561

This Table has been calculated on the mass-based 'old' millilitre. To convert to values calculated on the 'new' millilitre (based on volume), the values in the Table should be divided by 1.000028, where such precision is required.

TABLE XVII

Celite 505 Standard Filter Aid
Issued in May 1987

Weights of Pure Sugar Syrup filtered (between 2 and 7 minutes after application of pressure) at various final temperatures, under the standard conditions of the filtrability test.

NOTE:

This table is to be used only for Celite 505 filter aid issued May 1987. Use 0.95% by mass on solids, i.e. 1.425 ± 0.020 g of filter aid in the "150 g method" or 1.900 ± 0.020 g of filter aid in the "200 g method".

Final Temperature (°C)	Mass of Filtrate (g)	Final Temperature (°C)	Mass of Filtrate (g)	Final Temperature (°C)	Mass of Filtrate (g)	Final Temperature (°C)	Mass of Filtrate (g)	Final Temperature (°C)	Mass of Filtrate (g)	Final Temperature (°C)	Mass of Filtrate (g)
15.0	105	18.0	115	21.0	125	24.0	136	27.0	147	30.0	160
.1	105	.1	115	.1	125	.1	136	.1	148	.1	160
.2	106	.2	115	.2	126	.2	137	.2	148	.2	161
.3	106	.3	116	.3	126	.3	137	.3	149	.3	161
.4	106	.4	116	.4	126	.4	137	.4	149	.4	162
.5	107	.5	116	.5	127	.5	138	.5	149	.5	162
.6	107	.6	117	.6	127	.6	138	.6	150	.6	162
.7	107	.7	117	.7	127	.7	138	.7	150	.7	163
.8	107	.8	117	.8	128	.8	139	.8	151	.8	163
.9	108	.9	118	.9	128	.9	139	.9	151	.9	164
16.0	108	19.0	118	22.0	128	25.0	140	28.0	152	31.0	164
.1	108	.1	118	.1	129	.1	140	.1	152		
.2	109	.2	119	.2	129	.2	140	.2	152		
.3	109	.3	119	.3	129	.3	141	.3	153		
.4	109	.4	119	.4	130	.4	141	.4	153		
.5	110	.5	120	.5	130	.5	142	.5	154		
.6	110	.6	120	.6	131	.6	142	.6	154		
.7	110	.7	120	.7	131	.7	142	.7	154		
.8	111	.8	121	.8	131	.8	143	.8	155		
.9	111	.9	121	.9	132	.9	143	.9	155		
17.0	111	20.0	121	23.0	132	26.0	143	29.0	156		
.1	112	.1	122	.1	132	.1	144	.1	156		
.2	112	.2	122	.2	133	.2	144	.2	156		
.3	112	.3	122	.3	133	.3	145	.3	157		
.4	113	.4	123	.4	134	.4	145	.4	157		
.5	113	.5	123	.5	134	.5	145	.5	158		
.6	113	.6	123	.6	134	.6	146	.6	158		
.7	114	.7	124	.7	135	.7	146	.7	159		
.8	114	.8	124	.8	135	.8	147	.8	159		
.9	114	.9	124	.9	135	.9	147	.9	159		

The mathematical relationship is:

$$m = 67.8123 + 1.8876t + 0.0393t^2$$

where m = Mass of standard sugar filtrate in g

t = Temperature in °C

TABLE XVIII

Specific Rotation of Sugars

Sugar	Formula	Specific rotation in water $[\alpha]_{\text{D}}^{20}$
d—Glucose	$\text{C}_6\text{H}_{12}\text{O}_6$	+ 52.5
d—Galactose	$\text{C}_6\text{H}_{12}\text{O}_6$	+ 80.5
d—Mannose	$\text{C}_6\text{H}_{12}\text{O}_6$	+ 14.6
d—Fructose	$\text{C}_6\text{H}_{12}\text{O}_6$	- 92.5
d—Xylose	$\text{C}_5\text{H}_{10}\text{O}_5$	+ 19
d—Lyxose	$\text{C}_5\text{H}_{10}\text{O}_5$	- 14
D—Arabinose	$\text{C}_5\text{H}_{10}\text{O}_5$	- 105
I—Rhamnose	$\text{C}_6\text{H}_{12}\text{O}_6$	+ 8.9
a—Glucoheptose	$\text{C}_7\text{H}_{14}\text{O}_7$	- 20
Lactose	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$	+ 55.3
Maltose	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$	+136
Melibiose	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$	+142.5
Cellobiose	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$	+ 35
Sucrose	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$	+ 66.5
Raffinose	$\text{C}_{18}\text{H}_{32}\text{O}_{16}$	+105.5
1—Kestose	$\text{C}_{18}\text{H}_{32}\text{O}_{16}$	+ 28.5
6—Kestose	$\text{C}_{18}\text{H}_{32}\text{O}_{16}$	+ 27.3
Neo—Kestose	$\text{C}_{18}\text{H}_{32}\text{O}_{16}$	+ 21
Nystose	$\text{C}_{24}\text{H}_{42}\text{O}_{21}$	+ 10.7

TABLE XIX

Crystal Content of Massecuities*

Mass. purity	Purity drop										
	15	16	17	18	19	20	21	22	23	24	25
90	60.0	61.5	63.0	64.3	65.5	66.7
89	57.7	59.3	60.7	62.1	63.3	64.5	65.6
88	55.6	57.1	58.6	60.0	61.3	62.5	63.6	64.7
87	53.6	55.2	56.7	58.1	59.4	60.6	61.8	62.9	63.9
86	51.7	53.3	54.8	56.3	57.6	58.8	60.0	61.1	62.1	63.2	..
85	50.0	51.6	53.1	54.5	55.9	57.1	58.3	59.4	60.5	61.5	62.5
84	48.4	50.0	51.5	52.9	54.3	55.6	56.8	57.9	59.0	60.0	61.0
83	46.9	48.5	50.0	51.4	52.8	54.1	55.3	56.4	57.5	58.5	59.5
82	45.5	47.1	48.6	50.0	51.4	52.6	53.8	55.0	56.1	57.1	58.1
81	44.1	45.8	47.2	48.6	50.0	51.3	52.5	53.7	54.8	55.8	56.8
80	42.9	44.4	45.9	47.4	48.7	50.0	51.2	52.4	53.5	54.5	55.6
79	41.7	43.2	44.7	46.2	47.5	48.8	50.0	51.2	52.3	53.3	54.3
78	40.5	42.1	43.6	45.0	46.3	47.6	48.8	50.0	51.1	52.2	53.2
77	39.5	41.0	42.5	43.9	45.2	46.5	47.7	48.9	50.0	51.1	52.1
76	38.5	40.0	41.5	42.9	44.2	45.5	46.7	47.8	48.9	50.0	51.0
75	37.5	39.0	40.5	41.9	43.2	44.4	45.7	46.8	47.9	49.0	50.0
74	36.6	38.1	39.5	40.9	42.2	43.5	44.7	45.8	46.9	48.0	49.0
73	35.7	37.2	38.6	40.0	41.3	42.6	43.7	44.9	46.0	47.1	48.1
72	34.9	36.4	37.8	39.1	40.4	41.7	42.9	44.0	45.1	46.2	47.2
71	34.1	35.6	37.0	38.3	39.6	40.8	42.0	43.1	44.2	45.3	46.3
70	33.3	34.8	36.2	37.5	38.8	40.0	41.2	42.3	43.4	44.4	45.5

	15	16	17	18	19	20	22	24	26	28	30
69	32.6	34.0	35.4	36.7	38.0	39.2	41.5	43.6	45.6	47.5	49.2
68	31.9	33.3	34.7	36.0	37.3	38.5	40.7	42.9	44.8	46.7	48.4
67	31.2	32.7	34.0	35.3	36.5	37.7	40.0	42.1	44.1	45.9	47.6
66	30.6	32.0	33.3	34.6	35.8	37.0	39.3	41.4	43.3	45.2	46.9
65	30.0	31.4	32.7	34.0	35.2	36.4	38.6	40.7	42.6	44.4	46.2
64	29.4	30.8	32.1	33.3	34.5	35.7	37.9	40.0	41.9	43.8	45.5
63	28.8	30.2	31.5	32.7	33.9	35.1	37.3	39.3	41.3	43.1	44.8
62	28.3	29.6	30.9	32.1	33.3	34.5	36.7	38.7	40.6	42.4	44.1
61	27.8	29.1	30.4	31.6	32.8	33.9	36.1	38.1	40.0	41.8	43.5
60	27.3	28.6	29.8	31.0	32.2	33.3	35.5	37.5	39.4	41.2	42.9
59	26.8	28.1	29.3	30.5	31.7	32.8	34.9	36.9	38.8	40.6	42.3
58	26.3	27.6	28.8	30.0	31.1	32.3	34.4	36.4	38.2	40.0	41.7
57	25.9	27.1	28.3	29.5	30.6	31.7	33.8	35.8	37.7	39.4	41.1
56	25.4	26.7	27.9	29.0	30.2	31.3	33.3	35.3	37.1	38.9	40.5
55	25.0	26.2	27.4	28.6	29.7	30.8	32.8	34.8	36.6	38.3	40.0

*With apparent purities the crystal content per cent Brix is derived. The use of true purities gives crystal per cent dry substance. To obtain crystal per cent massecuite multiply by Brix or dry substance per unit of massecuite.

TABLE XX

Degree of Supersaturation—All Values Being Prefixed by 1

Actual temp. °C	Saturation temperature °C																																			
	40	42	44	46	48	50	52	54	56	58	60	62	64	66	68	70	72	74	76	78	80	82	84	86	88	90	92	94	96	98	100					
	30	.08	.10	.12	.14	.16	.19	.21	.23	.26	.28	.31	.34	.37	.39	.43	.46	.49	.53	.57	.61	.65	.69	.73	.77	.81	.85	.89	.93	.97	.101	.105				
32	.07	.09	.11	.13	.15	.17	.19	.21	.24	.26	.29	.32	.34	.37	.40	.44	.47	.51	.54	.58	.62	.66	.70	.74	.78	.82	.86	.90	.94	.98	.102	.106				
34	.05	.07	.09	.11	.13	.15	.17	.19	.22	.24	.27	.29	.32	.35	.38	.41	.45	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104				
36	.03	.05	.07	.09	.11	.13	.15	.17	.20	.22	.25	.27	.30	.33	.36	.39	.42	.46	.49	.53	.57	.61	.65	.69	.73	.77	.81	.85	.89	.93	.97	.101	.105			
38	.02	.04	.05	.07	.09	.11	.13	.16	.18	.20	.23	.25	.28	.31	.34	.37	.40	.44	.47	.51	.55	.59	.63	.67	.71	.75	.79	.83	.87	.91	.95	.99	.103	.107		
40	.00	.02	.04	.05	.07	.09	.11	.14	.16	.18	.21	.23	.26	.29	.32	.35	.38	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104		
42	.00	.02	.04	.05	.07	.09	.10	.12	.14	.16	.19	.21	.24	.26	.29	.32	.35	.39	.42	.46	.50	.54	.58	.62	.66	.70	.74	.78	.82	.86	.90	.94	.98	.102	.106	
44	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.17	.19	.22	.24	.27	.30	.33	.36	.40	.43	.47	.51	.55	.59	.63	.67	.71	.75	.79	.83	.87	.91	.95	.99	.103	.107
46	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
48	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
50	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
52	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
54	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
56	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
58	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
60	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
62	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
64	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
66	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
68	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
70	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
72	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
74	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
76	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
78	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104
80	.00	.02	.04	.05	.07	.08	.10	.12	.14	.16	.15	.17	.19	.22	.25	.28	.31	.34	.37	.41	.44	.48	.52	.56	.60	.64	.68	.72	.76	.80	.84	.88	.92	.96	.100	.104

TABLE XXI P.1.

TABLE XXI

Stock Recovery

Total Pol and Recoverable Pol in tonnes per cubic metre of stock, when the apparent purity of final molasses is 30.

Brix of Product	Total Pol (T)	Apparent Purity of Product									
	Recov. Pol (R)	45	50	55	60	65	70	75	80	85	90
64	T	.378	.418	.460	.503	.543	.586	.628	.671	.711	.753
	R	.179	.239	.299	.358	.418	.478	.539	.597	.657	.717
66	T	.391	.436	.478	.523	.565	.610	.653	.697	.740	.784
	R	.188	.248	.311	.373	.436	.498	.561	.621	.684	.746
68	T	.407	.454	.498	.543	.588	.635	.679	.724	.771	.816
	R	.194	.259	.324	.389	.454	.519	.583	.648	.713	.776
70	T	.425	.472	.519	.565	.612	.659	.706	.753	.800	.847
	R	.201	.268	.335	.402	.472	.539	.606	.673	.740	.805
72	T	.440	.489	.536	.586	.635	.684	.733	.782	.831	.878
	R	.210	.277	.349	.418	.489	.559	.628	.697	.767	.838
74	T	.456	.507	.557	.608	.659	.708	.760	.811	.860	.912
	R	.217	.291	.362	.434	.507	.579	.653	.724	.796	.869
76	T	.474	.525	.579	.630	.684	.735	.789	.840	.894	.945
	R	.226	.299	.375	.451	.525	.601	.675	.751	.825	.901
78	T	.489	.545	.599	.653	.708	.762	.816	.872	.925	.979
	R	.232	.311	.389	.467	.545	.621	.700	.778	.856	.932
80	T	.507	.563	.619	.677	.733	.789	.845	.901	.959	1.015
	R	.241	.322	.402	.483	.563	.644	.724	.805	.885	.966
82	T	.525	.583	.641	.700	.758	.816	.874	.932	.990	1.050
	R	.250	.333	.416	.501	.583	.666	.749	.834	.916	.999
84	T	.543	.603	.664	.722	.784	.845	.905	.966	1.026	1.086
	R	.259	.344	.431	.516	.603	.688	.776	.860	.948	1.033
86	T	.561	.624	.686	.749	.809	.872	.934	.997	1.059	1.122
	R	.268	.355	.445	.534	.624	.713	.800	.890	.979	1.068
88	T	.579	.644	.708	.773	.836	.901	.966	1.030	1.095	1.158
	R	.275	.369	.460	.552	.644	.735	.827	.919	1.012	1.104
90	T	.599	.664	.731	.798	.865	.930	.997	1.064	1.129	1.196
	R	.284	.380	.474	.570	.664	.760	.854	.950	1.044	1.138
92	T	.617	.686	.755	.822	.892	.961	1.028	1.097	1.167	1.234
	R	.295	.391	.489	.588	.686	.784	.881	.979	1.077	1.176
94	T	.637	.708	.778	.849	.919	.990	1.062	1.131	1.202	1.274
	R	.304	.405	.505	.606	.706	.809	.910	1.010	1.111	1.214
96	T	.657	.729	.802	.876	.948	1.021	1.095	1.167	1.240	1.312
	R	.313	.416	.521	.626	.729	.834	.939	1.042	1.147	1.249
98	T	.677	.751	.827	.903	.977	1.053	1.126	1.202	1.278	1.352
	R	.322	.429	.536	.644	.751	.858	.966	1.073	1.180	1.290
100	T	.697	.773	.852	.930	1.006	1.084	1.162	1.238	1.316	1.395
	R	.333	.443	.552	.664	.773	.885	.995	1.106	1.216	1.328

Stock Recovery

Total Pol and Recoverable Pol in tonnes per cubic metre of stock, when the apparent purity of final molasses is 35.

Brix of Product	Total Pol(T)	Apparent Purity of Product									
	Recov. pol (R)	45	50	55	60	65	70	75	80	85	90
64	T	.378	.418	.460	.503	.543	.586	.628	.671	.711	.753
	R	.130	.192	.257	.322	.387	.451	.514	.579	.644	.708
66	T	.391	.436	.478	.523	.565	.610	.653	.697	.740	.784
	R	.134	.201	.268	.335	.402	.469	.536	.603	.671	.738
68	T	.407	.454	.498	.543	.588	.635	.679	.724	.771	.816
	R	.139	.210	.279	.349	.418	.487	.557	.628	.697	.767
70	T	.425	.472	.519	.565	.612	.659	.706	.753	.800	.847
	R	.145	.217	.291	.362	.434	.507	.579	.650	.724	.796
72	T	.440	.489	.536	.586	.635	.684	.733	.782	.831	.878
	R	.150	.226	.302	.375	.451	.525	.601	.677	.751	.827
74	T	.456	.507	.557	.608	.659	.708	.760	.811	.860	.912
	R	.156	.235	.313	.389	.467	.545	.624	.702	.780	.858
76	T	.474	.525	.579	.630	.684	.735	.789	.840	.894	.945
	R	.161	.241	.324	.405	.485	.565	.646	.726	.809	.890
78	T	.489	.545	.599	.653	.708	.762	.816	.872	.925	.979
	R	.168	.250	.335	.418	.503	.586	.671	.753	.838	.921
80	T	.507	.563	.619	.677	.733	.789	.845	.901	.959	1.015
	R	.172	.259	.346	.434	.521	.606	.693	.780	.867	.954
82	T	.525	.583	.641	.700	.758	.816	.874	.932	.990	1.050
	R	.179	.268	.358	.449	.539	.628	.717	.807	.896	.986
84	T	.543	.603	.664	.722	.784	.845	.905	.966	1.026	1.086
	R	.186	.277	.371	.463	.557	.648	.742	.834	.928	1.019
86	T	.561	.624	.686	.749	.809	.872	.934	.997	1.059	1.122
	R	.192	.288	.387	.478	.574	.671	.767	.863	.959	1.055
88	T	.579	.644	.708	.773	.836	.901	.966	1.030	1.095	1.158
	R	.199	.297	.396	.496	.595	.693	.791	.892	.990	1.088
90	T	.599	.664	.731	.798	.865	.930	.997	1.064	1.129	1.196
	R	.203	.306	.409	.512	.612	.715	.818	.921	1.021	1.124
92	T	.617	.686	.755	.822	.892	.961	1.028	1.097	1.167	1.234
	R	.208	.317	.422	.527	.633	.738	.845	.950	1.055	1.160
94	T	.637	.708	.778	.849	.919	.990	1.062	1.131	1.202	1.274
	R	.219	.326	.436	.543	.653	.760	.869	.979	1.088	1.198
96	T	.657	.729	.802	.876	.948	1.021	1.095	1.167	1.240	1.312
	R	.224	.337	.449	.561	.673	.784	.898	1.010	1.120	1.234
98	T	.677	.751	.827	.903	.977	1.053	1.126	1.202	1.278	1.352
	R	.230	.346	.463	.579	.693	.809	.925	1.042	1.155	1.272
100	T	.697	.773	.852	.930	1.006	1.084	1.162	1.238	1.316	1.395
	R	.239	.358	.476	.595	.715	.834	.952	1.073	1.191	1.310

TABLE XXI P.3.

Stock Recovery

Total Pol and Recoverable Pol in tonnes per cubic metre of stock, when the apparent purity of final molasses is 40.

Brix of Product	Total Pol(T) Recov. Pol(R)	Apparent Purity of Product									
		45	50	55	60	65	70	75	80	85	90
64	T	.378	.418	.460	.503	.543	.586	.628	.671	.711	.753
	R	.069	.139	.210	.279	.349	.427	.487	.559	.628	.697
66	T	.391	.436	.478	.523	.565	.610	.653	.697	.740	.784
	R	.072	.145	.217	.288	.362	.434	.507	.579	.653	.725
68	T	.407	.454	.498	.543	.588	.635	.679	.724	.771	.816
	R	.076	.152	.226	.302	.378	.451	.527	.603	.677	.755
70	T	.425	.472	.519	.565	.612	.659	.706	.753	.800	.847
	R	.078	.156	.235	.313	.391	.469	.550	.626	.704	.784
72	T	.440	.489	.536	.586	.635	.684	.733	.782	.831	.878
	R	.080	.163	.244	.324	.407	.487	.570	.650	.731	.814
74	T	.456	.507	.557	.608	.659	.708	.760	.811	.860	.912
	R	.085	.170	.253	.337	.422	.507	.592	.675	.760	.845
76	T	.474	.525	.579	.630	.684	.735	.789	.840	.894	.945
	R	.087	.174	.264	.351	.438	.525	.612	.702	.789	.878
78	T	.489	.545	.599	.653	.708	.762	.816	.872	.925	.979
	R	.092	.181	.273	.364	.454	.545	.635	.726	.818	.907
80	T	.507	.563	.619	.677	.733	.789	.845	.901	.959	1.015
	R	.094	.188	.282	.375	.469	.563	.657	.751	.847	.939
82	T	.525	.583	.641	.700	.758	.816	.874	.932	.990	1.050
	R	.096	.194	.291	.387	.485	.581	.679	.776	.876	.972
84	T	.543	.603	.662	.724	.784	.845	.905	.966	1.026	1.086
	R	.101	.201	.302	.402	.503	.601	.704	.802	.905	1.006
86	T	.561	.624	.686	.749	.809	.872	.934	.997	1.059	1.122
	R	.103	.208	.311	.416	.519	.624	.726	.829	.934	1.039
88	T	.579	.644	.708	.773	.836	.901	.966	1.030	1.095	1.158
	R	.107	.215	.322	.429	.536	.644	.751	.858	.966	1.073
90	T	.599	.664	.731	.798	.865	.930	.997	1.064	1.129	1.196
	R	.112	.221	.333	.445	.554	.666	.776	.887	.999	1.106
92	T	.617	.686	.755	.822	.892	.961	1.028	1.097	1.167	1.234
	R	.114	.228	.342	.458	.570	.686	.800	.912	1.028	1.142
94	T	.637	.708	.778	.849	.919	.990	1.062	1.131	1.202	1.274
	R	.118	.235	.353	.472	.588	.706	.825	.941	1.059	1.178
96	T	.657	.729	.802	.876	.948	1.021	1.095	1.167	1.240	1.312
	R	.121	.244	.364	.485	.608	.729	.852	.975	1.093	1.216
98	T	.677	.751	.827	.903	.977	1.053	1.126	1.202	1.278	1.352
	R	.125	.250	.375	.501	.626	.751	.876	1.001	1.126	1.252
100	T	.697	.773	.852	.930	1.006	1.084	1.162	1.238	1.316	1.395
	R	.130	.257	.387	.516	.644	.773	.903	1.033	1.160	1.290

TABLE XXII

Density (g/ml) of Water at Temperatures from 0 to 102°C

According to M. Thiesen, Wiss. Abh. der Physikalisch-Technischen Reichsanstalt, 4, No. 1; 1904

Temp. °C	Density	Temp. °C	Density	Temp. °C	Density
0	0.99987	35	0.99406	70	0.97781
1	0.99993	36	0.99371	71	0.97723
2	0.99997	37	0.99336	72	0.97666
3	0.99999	38	0.99299	73	0.97607
4	1.00000	39	0.99262	74	0.97548
5	0.99999	40	0.99225	75	0.97489
6	0.99997	41	0.99186	76	0.97428
7	0.99993	42	0.99147	77	0.97368
8	0.99988	43	0.99107	78	0.97307
9	0.99981	44	0.99066	79	0.97245
10	0.99973	45	0.99024	80	0.97183
11	0.99963	46	0.98982	81	0.97120
12	0.99952	47	0.98940	82	0.97057
13	0.99940	48	0.98896	83	0.96994
14	0.99927	49	0.98852	84	0.96930
15	0.99913	50	0.98807	85	0.96865
16	0.99897	51	0.98762	86	0.96800
17	0.99880	52	0.98715	87	0.96734
18	0.99862	53	0.98669	88	0.96668
19	0.99843	54	0.98621	89	0.96601
20	0.99823	55	0.98573	90	0.96534
21	0.99802	56	0.98524	91	0.96467
22	0.99780	57	0.98478	92	0.96399
23	0.99756	58	0.98425	93	0.96330
24	0.99732	59	0.98375	94	0.96261
25	0.99707	60	0.98324	95	0.96192
26	0.99681	61	0.98272	96	0.96122
27	0.99654	62	0.98220	97	0.96051
28	0.99626	63	0.98167	98	0.95981
29	0.99597	64	0.98113	99	0.95909
30	0.99567	65	0.98059	100	0.95838
31	0.99537	66	0.98005	101	0.95765
32	0.99505	67	0.97950	102	0.95693
33	0.99473	68	0.97894
34	0.99440	69	0.97838

This Table has been calculated on the mass-based 'old' millilitre. To convert to values calculated on the 'new' millilitre (based on volume), the values in the Table should be divided by 1.000028, where such precision is required.

TABLE XXIII

Corrections for temperature (in g) to be added to weight of water contained to obtain
 volume (in ml) of vessel at 20°C*
 Nominal Capacity 1,000 ml
 (For Vessels made of Soda Glass)

Temp. of water t°C	.0	.1	.2	.3	.4	.5	.6	.7	.8	.9	Temp. of water t°C
5	1.49	1.49	1.49	1.49	1.49	1.49	1.49	1.49	1.49	1.49	5
6	1.49	1.49	1.49	1.49	1.49	1.49	1.49	1.49	1.50	1.50	6
7	1.50	1.50	1.50	1.50	1.51	1.51	1.51	1.52	1.52	1.52	7
8	1.52	1.53	1.53	1.54	1.54	1.54	1.55	1.55	1.56	1.56	8
9	1.57	1.57	1.57	1.58	1.59	1.59	1.60	1.60	1.61	1.61	9
10	1.62	1.63	1.63	1.64	1.64	1.65	1.66	1.67	1.67	1.68	10
11	1.69	1.69	1.70	1.71	1.72	1.73	1.73	1.74	1.75	1.76	11
12	1.77	1.78	1.78	1.79	1.80	1.81	1.82	1.83	1.84	1.85	12
13	1.86	1.87	1.88	1.89	1.90	1.91	1.92	1.93	1.94	1.96	13
14	1.97	1.98	1.99	2.00	2.01	2.02	2.04	2.05	2.06	2.07	14
15	2.08	2.10	2.11	2.12	2.13	2.15	2.16	2.17	2.19	2.20	15
16	2.21	2.23	2.24	2.25	2.27	2.28	2.30	2.31	2.33	2.34	16
17	2.35	2.37	2.38	2.40	2.41	2.43	2.44	2.46	2.48	2.49	17
18	2.51	2.52	2.54	2.55	2.57	2.59	2.60	2.62	2.64	2.65	18
19	2.67	2.69	2.70	2.72	2.74	2.76	2.77	2.79	2.81	2.83	19
20	2.84	2.86	2.88	2.90	2.92	2.94	2.95	2.97	2.99	3.01	20
21	3.03	3.05	3.07	3.09	3.11	3.13	3.14	3.16	3.18	3.20	21
22	3.22	3.24	3.26	3.28	3.30	3.33	3.35	3.37	3.39	3.41	22
23	3.43	3.45	3.47	3.49	3.51	3.54	3.56	3.58	3.60	3.62	23
24	3.64	3.67	3.69	3.71	3.73	3.75	3.78	3.80	3.82	3.85	24
25	3.87	3.89	3.91	3.94	3.96	3.98	4.01	4.03	4.06	4.08	25
26	4.10	4.13	4.15	4.17	4.20	4.22	4.25	4.27	4.30	4.32	26
27	4.35	4.37	4.40	4.42	4.45	4.47	4.50	4.52	4.55	4.57	27
28	4.60	4.62	4.65	4.68	4.70	4.73	4.75	4.78	4.81	4.83	28
29	4.86	4.89	4.91	4.94	4.97	4.99	5.02	5.05	5.08	5.10	29
30	5.13	5.16	5.19	5.21	5.24	5.27	5.30	5.33	5.35	5.38	30
31	5.41	5.44	5.47	5.50	5.52	5.55	5.58	5.61	5.64	5.67	31
32	5.70	5.73	5.76	5.79	5.82	5.85	5.88	5.91	5.94	5.97	32
33	6.00	6.03	6.06	6.09	6.12	6.15	6.18	6.21	6.24	6.27	33
34	6.30	6.33	6.36	6.39	6.42	6.46	6.49	6.52	6.55	6.58	34
35	6.61										35

*From B.S. 1797:1968.

NOTE.— For vessels of other size the correction to be applied bears the same ratio to those listed as the nominal capacity bears to 1000 ml

TABLE XXIV

Corrections for Atmospheric Pressure (in g) to be Added to or Subtracted from the Weight of Water Contained to Obtain Volume (in ml) of Vessel at Standard Temperature and Pressure*

Temp. of air °C	Pressure in mm of mercury at 0 °C														
	730	735	740	745	750	755	760	765	770	775	780	785	790		
5	+0.02	+0.02	+0.03	+0.04	+0.04	+0.05	+0.06	+0.07	+0.08	+0.08	+0.09	+0.10	+0.10		
6	+0.01	+0.02	+0.03	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08	+0.08	+0.09	+0.10		
7		+0.02	+0.02	+0.03	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08	+0.08	+0.09		
8		+0.01	+0.02	+0.02	+0.03	+0.04	+0.04	+0.05	+0.06	+0.07	+0.07	+0.08	+0.09		
9		+0.01	+0.01	+0.02	+0.02	+0.03	+0.04	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
10	-0.01			+0.01	+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
11	-0.01			+0.01	+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
12	-0.02			+0.01	+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
13	-0.02			+0.01	+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
14	-0.02			+0.01	+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
15	-0.02			+0.01	+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
16	-0.03				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
17	-0.03				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
18	-0.03				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
19	-0.04				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
20	-0.04				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
21	-0.05				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
22	-0.05				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
23	-0.05				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
24	-0.06				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
25	-0.06				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
26	-0.06				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
27	-0.07				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
28	-0.07				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
29	-0.08				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
30	-0.08				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
31	-0.08				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
32	-0.09				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
33	-0.09				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
34	-0.09				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		
35	-0.10				+0.02	+0.02	+0.03	+0.04	+0.05	+0.06	+0.06	+0.07	+0.08		

* From B.S. 1797:1968.
NOTE.—For vessels of other size the correction to be applied bears the same ratio to those listed as the nominal capacity bears to 1000 ml.

TABLE XXV

Requirements for Apparatus for Use in the Analysis of Cane for Payment Purposes

When apparatus is used for the analysis of cane for payment purposes it must conform either to a specification from a recognised Standards authority or to the following requirements.

Brix Hydrometers

The hydrometer must be of an approved shape, size and construction. The scale shall correspond to one of the following ranges: 0 to 10, 10 to 20, 15 to 25, 20 to 30. It shall be calibrated to read degrees Brix at 20°C and the range shall be divided in intervals of one tenth of one degree with full numbering at each unit graduation mark. The graduation lines shall be fine, of uniform thickness and at right angles to the axis of the hydrometer. The scale shall be firmly secured inside the stem and without twist. The readings must conform to a tolerance of $\pm 0.1^\circ$ Brix at any point of the scale. The following inscriptions shall be clearly marked on the scale within the stem and shall not encroach on the scale or numbering.

- (a) The makers name
- (b) Serial number
- (c) Brix or per cent of sugar by weight
- (d) Temp. 20°C.

Polarimeter or Saccharimeter tubes

The tube must be straight. The length of the tube at 20°C shall be within ± 0.03 per cent of the nominal lengths of 100 and 200 mm. The ends of the tube must be parallel and ground flat in a plane at right angles to the axis of the tube and no detectable change in reading should be observed on rotating the tube.

Each end must project beyond the ferrule or threaded collar to a distance not exceeding 1 mm, such that a cover glass placed over the end of the tube does not touch any other part of the tube.

Cover glasses

Cover glasses for polarimeter or saccharimeter tubes must be made of clear optical glass and free from strain. They must have plane parallel surfaces free from scratches. The edges should be slightly bevelled to prevent chipping. A thickness of 1.5 to 2 mm is desirable for tubes of 200 mm length.

Polarimeters and Saccharimeters

These must be in a satisfactory condition mechanically and optically. The error at any point of the scale must not exceed ± 0.1 scale degrees. It is recommended that they should be calibrated in terms of the International Sugar Scale corresponding to a normal weight of 26.000 grammes.

Thermometers

Thermometers are to be of mercury in glass, solid stem, or of an approved enclosed scale type. All ranges up to a maximum of 110°C to include zero. The maximum error allowed is 1.0°C. Total immersion thermometers are preferred. Inscriptions should include the maker or vendors name or mark and the immersion for which the thermometer is calibrated.

Refractometers

These must be in satisfactory condition mechanically and optically. The maximum error at any point of the scale should be the equivalent of 0.2 degrees Brix.

Balances

These should be within accepted tolerances for sensitivity and reproducibility corresponding to the maximum capacity of the balance. Efficient damping is required for rapid weighing.

Weights

Weights to 100g should conform to Class B tolerances as specified by the National Standards Laboratory Australia.

Weights of nominal values from 100g to 1kg should conform to tolerances of 15 parts in 100,000.

The tolerances shown in this Table have been compiled from specifications issued by the British Standards Institution. They are recommended as being suitable for apparatus for general use.

Flasks—One mark volumetric

Nominal capacity ml	5	10	25	50	100	200	250	500	1000	2000
Tolerance \pm ml	0.04	0.04	0.06	0.10	0.15	0.30	0.30	0.50	0.80	1.20

(British Standard 1792:1960 endorsed as Australian Standard R.20-1961)

Sugar Flasks

Type 1—Two graduation marks.

Type 2—Single graduation mark for polarization of sugars.

Nominal capacity ml Tolerance \pm ml	Type 1			Type 2
	50/55 0.1	100/110 0.15	200/220 0.25	100 0.03

(British Standard 675:1953)

Burettes

Nominal capacity ml	Subdivision ml	Tolerance on capacity \pm ml	Delivery times	
			min.	max.
1	0.01	0.01	20	50
2	0.02	0.02	20	50
5	0.02	0.02	50	120
5	0.05	0.04	20	50
10	0.02	0.02	100	200
10	0.1	0.05	15	40
25	0.05	0.05	85	170
25	0.1	0.1	35	70
50	0.1	0.1	75	150
100	0.2	0.2	65	130

(British Standard 846:1962 endorsed as Australian Standard R.10-1964)

TABLE XXV P.3.

Pipettes—One mark bulb

Nominal capacity ml..	..	1	2	5	10	15	20	25	50	100
Tolerance \pm ml	..	·015	·02	·03	·04	·05	·06	·06	·08	·12
Delivery times (seconds)										
minimum	..	5	5	10	10	15	20	20	20	30
maximum	..	15	15	25	25	30	40	40	50	60

(British Standard 1583:1961 endorsed as Australian Standard R.16-1962)

Graduated Pipettes

Type 1—for delivery from zero mark to graduation marks.

Type 2—for delivery down to jet.

Nominal capacity ml	1	2	5	10	25
Subdivisions ml	·01	·02	·05	·10	·10
Tolerance \pm ml	·01	·02	·05	·10	·20

Delivery times, all sizes

Type 1 Minimum 15 s Maximum 30 s

Type 2 Minimum 10 s Maximum 25 s

(British Standard 700:1962, amendment No. 1 published 7/5/1963)

Measuring Cylinders—unstoppered

Nominal capacity ml	5	10	25	50	100	250	500	1000	2000
Tolerance \pm ml	·08	·15	·25	·5	·8	1·5	2	4	8

(British Standard 604:1952 endorsed as Australian Standard R.6-1953)

Thermometers—Mercury in glass type

Range °C	British Standard	Graduation interval deg C	Tolerance \pm °C	
			Total immersion	Partial immersion
−5 to +100	593	0·1	0·2	0·4
−20 to +60	593	0·2	0·3	0·4
50 to 110	593	0·2	0·3	0·6
99 to 160	593	0·2	0·4	0·8
150 to 210	593	0·2	0·6	1·2
−5 to +105	1704	0·5	0·5	0·6
−5 to +105	593	1·0	0·3	0·6
−5 to +105	1704	1·0	1·0	1·0
−5 to +250	1704	1·0	1·0	1·0
−5 to +360	1704	1·0	2·0	3·0
95 to 205	593	1·0	0·5	1·0

Metric Weights

Nominal value kg	5	3	2	1
Tolerance \pm mg	250	150	100	50

TABLE XXV P.4.

Nominal value g	500	300	200	100	50	30	20	10 to 0.1	0.05 to 0.001
Tolerance \pm mg	25	15	10	5	2.5	1.5	1.0	0.5	0.2

For values not tabulated the tolerances are the same as those given for the next larger tabulated value. The tolerances for burettes, graduated pipettes, graduated cylinders, and thermometers apply to the whole of the graduated portion or to any fraction of it.

TABLE XXVI

Corrections to Quartz Plate Polarisation Readings for
the Temperature of the Quartz Plate

Temperature (°C)	Correction to be Added (°Z)
15.0	+ .07
15.5	+ .06
16.0	+ .06
16.5	+ .05
17.0	+ .04
17.5	+ .04
18.0	+ .03
18.5	+ .02
19.0	+ .01
19.5	+ .01
20.0	.00
20.5	- .01
21.0	- .01
21.5	- .02
22.0	- .03
22.5	- .04
23.0	- .04
23.5	- .05
24.0	- .06
24.5	- .06
25.0	- .07
25.5	- .08
26.0	- .09
26.5	- .09
27.0	- .10
27.5	- .11
28.0	- .12
28.5	- .12
29.0	- .13
29.5	- .14
30.0	- .14

FORMULA:

$$C_{t_q} = - 0.000144 Q_{20} (t_q - 20)$$

C_{t_q} = correction to be added to the observed quartz plate polarisation for the effect of quartz plate temperature.

Q_{20} = certified polarisation of the quartz plate at 20°C.
Table II has been calculated on the basis of $Q_{20} = 100^\circ Z$.

t_q = temperature of quartz plate.

REFERENCE

ICUMSA 18th Session, 1982, Subject 11, page 179.

TABLE XXVII

Corrections to Polarisation for Temperatures
of Making to the Mark — (C_{tm})

TEMP (°C)	POLARISATION (°Z)				TEMP (°C)	POLARISATION (°Z)			
	100	99	98	97		100	99	98	97
	CORRECTIONS TO BE ADDED (°Z)					CORRECTIONS TO BE ADDED (°Z)			
15.0	+.13	+.13	+.12	+.12	19.0	+.03	+.03	+.03	+.03
15.1	+.13	+.12	+.12	+.12	19.1	+.02	+.02	+.02	+.02
15.2	+.12	+.12	+.12	+.12	19.2	+.02	+.02	+.02	+.02
15.3	+.12	+.12	+.12	+.12	19.3	+.02	+.02	+.02	+.02
15.4	+.12	+.12	+.12	+.11	19.4	+.02	+.02	+.02	+.02
15.5	+.12	+.11	+.11	+.11	19.5	+.01	+.01	+.01	+.01
15.6	+.11	+.11	+.11	+.11	19.6	+.01	+.01	+.01	+.01
15.7	+.11	+.11	+.11	+.11	19.7	+.01	+.01	+.01	+.01
15.8	+.11	+.11	+.11	+.10	19.8	+.01	+.01	+.01	+.01
15.9	+.11	+.10	+.10	+.10	19.9	+.00	+.00	+.00	+.00
16.0	+.10	+.10	+.10	+.10	20.0	+.00	+.00	+.00	+.00
16.1	+.10	+.10	+.10	+.10	20.1	+.00	+.00	+.00	+.00
16.2	+.10	+.10	+.10	+.10	20.2	-.01	-.01	-.01	-.01
16.3	+.10	+.09	+.09	+.09	20.3	-.01	-.01	-.01	-.01
16.4	+.09	+.09	+.09	+.09	20.4	-.01	-.01	-.01	-.01
16.5	+.09	+.09	+.09	+.09	20.5	-.01	-.01	-.01	-.01
16.6	+.09	+.09	+.09	+.09	20.6	-.02	-.02	-.02	-.02
16.7	+.09	+.08	+.08	+.08	20.7	-.02	-.02	-.02	-.02
16.8	+.08	+.08	+.08	+.08	20.8	-.02	-.02	-.02	-.02
16.9	+.08	+.08	+.08	+.08	20.9	-.02	-.02	-.02	-.02
17.0	+.08	+.08	+.08	+.08	21.0	-.03	-.03	-.03	-.03
17.1	+.08	+.08	+.07	+.07	21.1	-.03	-.03	-.03	-.03
17.2	+.07	+.07	+.07	+.07	21.2	-.03	-.03	-.03	-.03
17.3	+.07	+.07	+.07	+.07	21.3	-.04	-.04	-.03	-.03
17.4	+.07	+.07	+.07	+.07	21.4	-.04	-.04	-.04	-.04
17.5	+.07	+.06	+.06	+.06	21.5	-.04	-.04	-.04	-.04
17.6	+.06	+.06	+.06	+.06	21.6	-.04	-.04	-.04	-.04
17.7	+.06	+.06	+.06	+.06	21.7	-.05	-.05	-.05	-.05
17.8	+.06	+.06	+.06	+.06	21.8	-.05	-.05	-.05	-.05
17.9	+.06	+.05	+.05	+.05	21.9	-.05	-.05	-.05	-.05
18.0	+.05	+.05	+.05	+.05	22.0	-.06	-.05	-.05	-.05
18.1	+.05	+.05	+.05	+.05	22.1	-.06	-.06	-.06	-.06
18.2	+.05	+.05	+.05	+.05	22.2	-.06	-.06	-.06	-.06
18.3	+.05	+.04	+.04	+.04	22.3	-.06	-.06	-.06	-.06
18.4	+.04	+.04	+.04	+.04	22.4	-.07	-.07	-.07	-.06
18.5	+.04	+.04	+.04	+.04	22.5	-.07	-.07	-.07	-.07
18.6	+.04	+.04	+.04	+.04	22.6	-.07	-.07	-.07	-.07
18.7	+.03	+.03	+.03	+.03	22.7	-.08	-.07	-.07	-.07
18.8	+.03	+.03	+.03	+.03	22.8	-.08	-.08	-.08	-.08
18.9	+.03	+.03	+.03	+.03	22.9	-.08	-.08	-.08	-.08

TABLE XXVII P.2.

TEMP (°C)	POLARISATION (°Z)				TEMP (°C)	POLARISATION (°Z)			
	100	99	98	97		100	99	98	97
	CORRECTIONS TO BE ADDED (°Z)					CORRECTIONS TO BE ADDED (°Z)			
23.0	-.08	-.08	-.08	-.08	27.0	-.20	-.20	-.20	-.20
23.1	-.09	-.09	-.08	-.08	27.1	-.21	-.20	-.20	-.20
23.2	-.09	-.09	-.09	-.09	27.2	-.21	-.21	-.21	-.20
23.3	-.09	-.09	-.09	-.09	27.3	-.21	-.21	-.21	-.21
23.4	-.10	-.09	-.09	-.09	27.4	-.22	-.21	-.21	-.21
23.5	-.10	-.10	-.10	-.10	27.5	-.22	-.22	-.21	-.21
23.6	-.10	-.10	-.10	-.10	27.6	-.22	-.22	-.22	-.22
23.7	-.10	-.10	-.10	-.10	27.7	-.23	-.22	-.22	-.22
23.8	-.11	-.11	-.10	-.10	27.8	-.23	-.23	-.22	-.22
23.9	-.11	-.11	-.11	-.11	27.9	-.23	-.23	-.23	-.23
24.0	-.11	-.11	-.11	-.11	28.0	-.24	-.23	-.23	-.23
24.1	-.12	-.11	-.11	-.11	28.1	-.24	-.24	-.23	-.23
24.2	-.12	-.12	-.12	-.12	28.2	-.24	-.24	-.24	-.23
24.3	-.12	-.12	-.12	-.12	28.3	-.24	-.24	-.24	-.24
24.4	-.12	-.12	-.12	-.12	28.4	-.25	-.25	-.24	-.24
24.5	-.13	-.13	-.13	-.12	28.5	-.25	-.25	-.25	-.24
24.6	-.13	-.13	-.13	-.13	28.6	-.25	-.25	-.25	-.25
24.7	-.13	-.13	-.13	-.13	28.7	-.26	-.26	-.25	-.25
24.8	-.14	-.14	-.13	-.13	28.8	-.26	-.26	-.26	-.25
24.9	-.14	-.14	-.14	-.14	28.9	-.26	-.26	-.26	-.26
25.0	-.14	-.14	-.14	-.14	29.0	-.27	-.26	-.26	-.26
25.1	-.15	-.14	-.14	-.14	29.1	-.27	-.27	-.27	-.26
25.2	-.15	-.15	-.15	-.14	29.2	-.27	-.27	-.27	-.27
25.3	-.15	-.15	-.15	-.15	29.3	-.28	-.27	-.27	-.27
25.4	-.15	-.15	-.15	-.15	29.4	-.28	-.28	-.27	-.27
25.5	-.16	-.16	-.15	-.15	29.5	-.28	-.28	-.28	-.28
25.6	-.16	-.16	-.16	-.16	29.6	-.29	-.28	-.28	-.28
25.7	-.16	-.16	-.16	-.16	29.7	-.29	-.29	-.28	-.28
25.8	-.17	-.17	-.16	-.16	29.8	-.29	-.29	-.29	-.28
25.9	-.17	-.17	-.17	-.16	29.9	-.30	-.29	-.29	-.29
26.0	-.17	-.17	-.17	-.17	30.0	-.30	-.30	-.29	-.29
26.1	-.18	-.17	-.17	-.17					
26.2	-.18	-.18	-.18	-.17					
26.3	-.18	-.18	-.18	-.18					
26.4	-.19	-.18	-.18	-.18					
26.5	-.19	-.19	-.18	-.18					
26.6	-.19	-.19	-.19	-.19					
26.7	-.19	-.19	-.19	-.19					
26.8	-.20	-.20	-.19	-.19					
26.9	-.20	-.20	-.20	-.19					

FORMULA:

$$C_{tm} = - 0.000270 P_{tr} (t_m - 20) - 0.0000030 P_{tr} (t_m - 20)^2$$

C_{tm} = correction to the polarisation reading of the test solution for temperature of making to the mark

P_{tr} = polarisation of the test solution at the temperature of reading

t_m = temperature of making to the mark.

REFERENCE

ICUMSA, 17th Session (1978) Subject 11 pages 153-156

TABLE XXVIII

Corrections to Polarisation for Temperatures
of Reading — (C_{tr})

POL (°Z) R.S. (%)	100 0.0	99 0.0-0.3	98 0.3-0.6	97 0.5-1.0	POL (°Z) R.S. (%)	100 0.0	99 0.0-0.3	98 0.3-0.6	97 0.5-1.0
TEMP (°C)	CORRECTIONS TO BE ADDED (°Z)				TEMP (°C)	CORRECTIONS TO BE ADDED (°Z)			
15.0	-.23	-.23	-.22	-.21	19.0	-.05	-.05	-.04	-.04
15.1	-.23	-.22	-.22	-.21	19.1	-.04	-.04	-.04	-.04
15.2	-.22	-.22	-.21	-.20	19.2	-.04	-.04	-.04	-.03
15.3	-.22	-.21	-.21	-.20	19.3	-.03	-.03	-.03	-.03
15.4	-.21	-.21	-.20	-.19	19.4	-.03	-.03	-.03	-.03
15.5	-.21	-.21	-.20	-.19	19.5	-.02	-.02	-.02	-.02
15.6	-.21	-.20	-.19	-.19	19.6	-.02	-.02	-.02	-.02
15.7	-.20	-.20	-.19	-.18	19.7	-.01	-.01	-.01	-.01
15.8	-.20	-.19	-.18	-.18	19.8	-.01	-.01	-.01	-.01
15.9	-.19	-.19	-.18	-.17	19.9	+.00	+.00	+.00	+.00
16.0	-.19	-.18	-.18	-.17	20.0	+.00	+.00	+.00	+.00
16.1	-.18	-.18	-.17	-.16	20.1	+.00	+.00	+.00	+.00
16.2	-.18	-.17	-.17	-.16	20.2	+.01	+.01	+.01	+.01
16.3	-.17	-.17	-.16	-.16	20.3	+.01	+.01	+.01	+.01
16.4	-.17	-.16	-.16	-.15	20.4	+.02	+.02	+.02	+.02
16.5	-.16	-.16	-.15	-.15	20.5	+.02	+.02	+.02	+.02
16.6	-.16	-.16	-.15	-.14	20.6	+.03	+.03	+.03	+.03
16.7	-.15	-.15	-.15	-.14	20.7	+.03	+.03	+.03	+.03
16.8	-.15	-.15	-.14	-.14	20.8	+.04	+.04	+.04	+.03
16.9	-.14	-.14	-.14	-.13	20.9	+.04	+.04	+.04	+.04
17.0	-.14	-.14	-.13	-.13	21.0	+.05	+.05	+.04	+.04
17.1	-.14	-.13	-.13	-.12	21.1	+.05	+.05	+.05	+.05
17.2	-.13	-.13	-.12	-.12	21.2	+.06	+.05	+.05	+.05
17.3	-.13	-.12	-.12	-.11	21.3	+.06	+.06	+.06	+.05
17.4	-.12	-.12	-.11	-.11	21.4	+.07	+.06	+.06	+.06
17.5	-.12	-.11	-.11	-.11	21.5	+.07	+.07	+.07	+.06
17.6	-.11	-.11	-.11	-.10	21.6	+.07	+.07	+.07	+.07
17.7	-.11	-.10	-.10	-.10	21.7	+.08	+.08	+.07	+.07
17.8	-.10	-.10	-.10	-.09	21.8	+.08	+.08	+.08	+.08
17.9	-.10	-.10	-.09	-.09	21.9	+.09	+.09	+.08	+.08
18.0	-.09	-.09	-.09	-.08	22.0	+.09	+.09	+.09	+.08
18.1	-.09	-.09	-.08	-.08	22.1	+.10	+.10	+.09	+.09
18.2	-.08	-.08	-.08	-.08	22.2	+.10	+.10	+.10	+.09
18.3	-.08	-.08	-.07	-.07	22.3	+.11	+.10	+.10	+.10
18.4	-.07	-.07	-.07	-.07	22.4	+.11	+.11	+.11	+.10
18.5	-.07	-.07	-.07	-.06	22.5	+.12	+.11	+.11	+.11
18.6	-.07	-.06	-.06	-.06	22.6	+.12	+.12	+.11	+.11
18.7	-.06	-.06	-.06	-.05	22.7	+.13	+.12	+.12	+.11
18.8	-.06	-.05	-.05	-.05	22.8	+.13	+.13	+.12	+.12
18.9	-.05	-.05	-.05	-.05	22.9	+.14	+.13	+.13	+.12

TABLE XXVIII P.2.

POL (°Z) R.S. (%)	100 0.0	99 0.0-0.3	98 0.3-0.6	97 0.5-1.0	POL (°Z) R.S. (%)	100 0.0	99 0.0-0.3	98 0.3-0.6	97 0.5-1.0
TEMP (°C)	CORRECTIONS TO BE ADDED (°Z)				TEMP (°C)	CORRECTIONS TO BE ADDED (°Z)			
23.0	+.14	+.14	+.13	+.13	27.0	+.33	+.32	+.31	+.30
23.1	+.14	+.14	+.14	+.13	27.1	+.33	+.32	+.31	+.30
23.2	+.15	+.15	+.14	+.14	27.2	+.34	+.33	+.32	+.30
23.3	+.15	+.15	+.15	+.14	27.3	+.34	+.33	+.32	+.31
23.4	+.16	+.16	+.15	+.14	27.4	+.35	+.34	+.33	+.31
23.5	+.16	+.16	+.15	+.15	27.5	+.35	+.34	+.33	+.32
23.6	+.17	+.16	+.16	+.15	27.6	+.35	+.35	+.33	+.32
23.7	+.17	+.17	+.16	+.16	27.7	+.36	+.35	+.34	+.33
23.8	+.18	+.17	+.17	+.16	27.8	+.36	+.36	+.34	+.33
23.9	+.18	+.18	+.17	+.16	27.9	+.37	+.36	+.35	+.33
24.0	+.19	+.18	+.18	+.17	28.0	+.37	+.37	+.35	+.34
24.1	+.19	+.19	+.18	+.17	28.1	+.38	+.37	+.36	+.34
24.2	+.20	+.19	+.18	+.18	28.2	+.38	+.37	+.36	+.35
24.3	+.20	+.20	+.19	+.18	28.3	+.39	+.38	+.36	+.35
24.4	+.21	+.20	+.19	+.19	28.4	+.39	+.38	+.37	+.36
24.5	+.21	+.21	+.20	+.19	28.5	+.40	+.39	+.37	+.36
24.6	+.21	+.21	+.20	+.19	28.6	+.40	+.39	+.38	+.36
24.7	+.22	+.21	+.21	+.20	28.7	+.41	+.40	+.38	+.37
24.8	+.22	+.22	+.21	+.20	28.8	+.41	+.40	+.39	+.37
24.9	+.23	+.22	+.22	+.21	28.9	+.42	+.41	+.39	+.38
25.0	+.23	+.23	+.22	+.21	29.0	+.42	+.41	+.40	+.38
25.1	+.24	+.23	+.22	+.22	29.1	+.42	+.42	+.40	+.38
25.2	+.24	+.24	+.23	+.22	29.2	+.43	+.42	+.40	+.39
25.3	+.25	+.24	+.23	+.22	29.3	+.43	+.42	+.41	+.39
25.4	+.25	+.25	+.24	+.23	29.4	+.44	+.43	+.41	+.40
25.5	+.26	+.25	+.24	+.23	29.5	+.44	+.43	+.42	+.40
25.6	+.26	+.26	+.25	+.24	29.6	+.45	+.44	+.42	+.41
25.7	+.27	+.26	+.25	+.24	29.7	+.45	+.44	+.43	+.41
25.8	+.27	+.26	+.26	+.25	29.8	+.46	+.45	+.43	+.41
25.9	+.28	+.27	+.26	+.25	29.9	+.46	+.45	+.44	+.42
26.0	+.28	+.27	+.26	+.25	30.0	+.47	+.46	+.44	+.42
26.1	+.28	+.28	+.27	+.26					
26.2	+.29	+.28	+.27	+.26					
26.3	+.29	+.29	+.28	+.27					
26.4	+.30	+.29	+.28	+.27					
26.5	+.30	+.30	+.29	+.27					
26.6	+.31	+.30	+.29	+.28					
26.7	+.31	+.31	+.29	+.28					
26.8	+.32	+.31	+.30	+.29					
26.9	+.32	+.31	+.30	+.29					

FORMULA:

$$C_{tr} = 0.000467 P_{tr} (t_r - 20) - 0.004 R (t_r - 20)$$

C_{tr} = correction to the polarisation reading of the test solution for temperature of reading.

P_{tr} = polarisation of the test solution at the temperature of reading.

t_r = temperature of reading.

R = % reducing sugars.

REFERENCE

ICUMSA, 18th Session (1982) Subject 11 pages 177-180

TABLE XXIX

Corrections to Polarisation Reading for the Effect of
Polarimeter Temperature

Corrections to be Added (°Z)

Temperature (°C)	Autopol IIS	Polartronic Universal	Saccharomat IV
15.0	-.04	-.03	-.07
15.5	-.03	-.02	-.06
16.0	-.03	-.02	-.06
16.5	-.03	-.02	-.05
17.0	-.02	-.02	-.04
17.5	-.02	-.01	-.04
18.0	-.02	-.01	-.03
18.5	-.01	-.01	-.02
19.0	-.01	-.01	-.01
19.5	-.00	-.00	-.01
20.0	+.00	+.00	+.00
20.5	+.00	+.00	+.01
21.0	+.01	+.01	+.01
21.5	+.01	+.01	+.02
22.0	+.02	+.01	+.03
22.5	+.02	+.01	+.04
23.0	+.02	+.02	+.04
23.5	+.03	+.02	+.05
24.0	+.03	+.02	+.06
24.5	+.03	+.02	+.06
25.0	+.04	+.03	+.07
25.5	+.04	+.03	+.08
26.0	+.05	+.03	+.09
26.5	+.05	+.03	+.09
27.0	+.05	+.04	+.10
27.5	+.06	+.04	+.11
28.0	+.06	+.04	+.12
28.5	+.06	+.04	+.12
29.0	+.07	+.05	+.13
29.5	+.07	+.05	+.14
30.0	+.08	+.05	+.14

FORMULA

1. AUTOPOL IIS (Rudolf)

$$C_{tp} = 0.000076 P_{tr} (t_p - 20)$$

2. POLARTRONIC UNIVERSAL
(Schmidt & Haensch)

$$C_{tp} = 0.00005 P_{tr} (t_p - 20)$$

3. SACCHAROMAT IV (Schmidt & Haensch)

$$C_{tp} = 0.000144 P_{tr} (t_p - 20)$$

C_{tp} = Correction to polarisation reading of the test solution for the effect of polarimeter temperature.

P_{tr} = Observed polarisation of the test solution at the temperature of reading. This Table has been calculated on the basis of $P_{tr} = 100^\circ Z$.

t_p = Temperature of polarimeter.

TABLE XXX

Milligrams of Reducing Sugars Required to Reduce 10 ml
Fehling's Solution (Lane and Eynon Method)

ml sugar solution required	mg reducing sugar per 100 ml solution, when concentration of sucrose is—												
	0	0.5g	1g	2g	3g	4g	5g	10g	25g	50g*			
15.	336	335	333	329	325	322	317	307	289	275
16.	316	314	312	309	305	301	297	288	271	257
17.	298	296	295	291	287	284	280	271	255	241
18.	282	280	278	274	271	268	264	256	240	227
19.	267	265	264	260	257	254	250	243	227	215
20.	255	253	251	248	245	242	238	231	216	204
21.	243	241	239	236	233	230	227	220	206	194
22.	232	230	228	225	222	220	216	210	196	185
23.	222	220	219	216	213	210	207	200	187	176
24.	213	211	210	207	204	202	198	192	179	168
25.	205	203	202	198	196	194	190	184	171	161
26.	197	195	194	191	189	186	183	177	164	155
27.	190	189	187	184	182	179	176	170	158	149
28.	184	182	180	178	175	173	170	164	152	143
29.	178	176	174	171	169	167	165	159	147	138
30.	172	170	168	166	164	161	159	153	142	133
31.	166	165	163	161	159	157	154	148	137	129
32.	161	160	158	156	154	152	149	143	132	125
33.	157	155	153	151	149	147	145	139	128	121
34.	152	151	149	147	145	143	140	135	124	117
35.	148	147	145	143	141	139	136	131	121	113
36.	144	143	141	139	137	135	133	127	117	109
37.	140	139	137	135	133	131	129	124	114	106
38.	137	135	134	131	130	128	126	120	111	103
39.	133	132	130	128	126	124	122	117	107	100
40.	130	129	127	125	123	121	119	114	104	97
41.	127	125	124	122	120	118	116	111	102	95
42.	124	123	121	119	117	116	114	109	99	92
43.	121	120	118	116	115	113	111	106	97	90
44.	119	117	116	114	112	110	108	103	94	88
45.	116	114	113	111	110	108	106	101	92	86
46.	114	112	111	109	107	105	104	99	90	84
47.	111	110	108	106	105	103	102	96	88	82
48.	109	108	106	104	103	101	99	94	86	81
49.	107	106	104	102	102	100	97	92	84	79
50.	105	103	102	100	100	98	95	90	82	77

*Calculated by extrapolation.

TABLE XXXI

Showing Grams of Product to be weighed out per 100 mL
of Titrating Solution, for the Determination of Reducing
Sugars in Products of Various Sucrose and Reducing
Sugar Contents

Approx % Reducing Sugars in Product	Approximate Percentage of Sucrose in Product									
	0-5	5-10	10-25	25-40	40-50	50-60	60-70	70-80	80-90	90-100
0.00- 0.15	D	D	D	D	D	D	D	D	D	D
0.15- 0.22	100	100	100	D	D	D	D	D	D	D
0.22- 0.26	100	100	60	60	D	D	D	D	D	D
0.26- 0.30	60	60	60	60	D	D	D	D	D	D
0.30- 0.35	50	50	50	50	50	D	D	D	D	D
0.35- 0.40	50	50	50	50	50	40	D	D	D	D
0.40- 0.50	40	40	40	40	40	40	35	30	D	D
0.50- 0.60	40	40	35	35	30	30	30	30	D	D
0.60- 0.75	30	30	30	30	30	30	30	30	25	25
0.75- 0.90	20	20	20	20	20	20	20	20	20	20
0.90- 1.00	20	20	20	20	20	20	20	20	20	20
1.00- 2.00	15	15	15	15	15	15	15	15	15	15
2.00- 3.00	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5
3.00- 4.00	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
4.00- 5.00	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0
5.00- 7.00	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0
7.00- 9.00	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
9.00-11.00	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
11.0-16.00	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
16.0-24.00	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
24.0-30.00	0.75	0.75	0.75	0.75	0.75	0.75	0.75	0.75	—	—
30.0-60.00	0.50	0.50	0.50	0.50	0.50	0.50	0.50	—	—	—
60.0-100.0	0.25	0.25	0.25	0.25	0.25	—	—	—	—	—

Note: (i) Those weights apply for the straight method only.
(ii) D = differential method.

TABLE XXXII

Grams of Reducing Sugars (as Invert Sugar)
per 100 mL of Titrating Solution for Various
End-Points and Sucrose Concentrations of
25 g/100 mL

End Point (mL)	g RS/100 mL Titration Solution	End Point (mL)	g RS/100 mL Titration Solution
15.0	0.289	30.0	0.142
.5	.280	.5	.139
16.0	.271	31.0	.137
.5	.263	.5	.134
17.0	.255	32.0	.132
.5	.248	.5	.130
18.0	.241	33.0	.128
.5	.234	.5	.126
19.0	.228	34.0	.124
.5	.222	.5	.122
20.0	0.216	35.0	0.120
.5	.211	.5	.118
21.0	.206	36.0	.117
.5	.201	.5	.115
22.0	.196	37.0	.113
.5	.191	.5	.112
23.0	.187	38.0	.110
.5	.183	.5	.109
24.0	.179	39.0	.107
.5	.175	.5	.106
25.0	0.171	40.0	0.105
.5	.168	.5	.103
26.0	.165	41.0	.102
.5	.162	.5	.101
27.0	.159	42.0	.099
.5	.156	.5	.098
28.0	.153	43.0	.097
.5	.150	.5	.095
29.0	.147	44.0	.094
.5	.144	.5	.093
		45.0	.092

Refer: Lane, J.H. and Eynon L.; J.Soc.Chem.Ind.,
Vol 42 (1923), p 32T.

TABLE XXXIII

Grams of Reducing Sugars (as Invert Sugar) per 100 mL Titrating Solution for Various End-Points, and
Sucrose Concentrations of 0-3 g/100 mL

End Point (mL)	Grams of Sucrose present in 100 mL of Titrating Solution					End Point (mL)	End Point (mL)	Grams of Sucrose present in 100 mL of Titrating Solution					End Point (mL)
	Nil	0.5	1	2	3			Nil	0.5	1	2	3	
Grams RS per 100 mL Titrating Solution						Grams RS per 100 mL Titrating Solution							
15.0	0.337	0.335	0.332	0.329	0.325	15.0	20.0	0.255	0.253	0.251	0.248	0.245	20.0
.1	.335	.333	.330	.327	.323	.1	.1	.254	.251	.250	.247	.244	.1
.2	.333	.331	.328	.325	.321	.2	.2	.252	.250	.248	.245	.242	.2
.3	.331	.328	.326	.323	.319	.3	.3	.251	.249	.247	.244	.241	.3
.4	.329	.326	.324	.321	.317	.4	.4	.250	.248	.246	.243	.240	.4
.5	.326	.324	.322	.319	.315	.5	.5	.249	.247	.245	.242	.239	.5
.6	.324	.322	.320	.317	.313	.6	.6	.248	.246	.244	.241	.238	.6
.7	.322	.320	.318	.315	.311	.7	.7	.247	.244	.243	.240	.237	.7
.8	.320	.318	.316	.313	.309	.8	.8	.245	.243	.241	.238	.236	.8
.9	.318	.316	.315	.311	.307	.9	.9	.244	.242	.240	.237	.234	.9
16.0	0.316	0.315	0.313	0.309	0.305	16.0	21.0	0.243	0.241	0.239	0.236	0.233	21.0
.1	.314	.313	.311	.307	.304	.1	.1	.242	.240	.238	.235	.232	.1
.2	.313	.311	.309	.306	.302	.2	.2	.241	.239	.237	.234	.231	.2
.3	.311	.309	.307	.304	.300	.3	.3	.240	.237	.236	.233	.230	.3
.4	.309	.307	.305	.302	.298	.4	.4	.238	.236	.235	.232	.229	.4
.5	.307	.305	.304	.300	.296	.5	.5	.237	.235	.234	.231	.228	.5
.6	.305	.304	.302	.298	.295	.6	.6	.236	.234	.233	.229	.226	.6
.7	.304	.302	.300	.297	.293	.7	.7	.235	.233	.232	.228	.225	.7
.8	.302	.300	.298	.295	.291	.8	.8	.234	.232	.231	.227	.224	.8
.9	.300	.298	.297	.293	.289	.9	.9	.233	.231	.230	.226	.223	.9
17.0	0.298	0.297	0.295	0.291	0.288	17.0	22.0	0.232	0.230	0.229	0.225	0.222	22.0
.1	.297	.295	.293	.290	.286	.1	.1	.231	.229	.228	.224	.221	.1
.2	.295	.293	.292	.288	.284	.2	.2	.230	.228	.227	.223	.220	.2
.3	.293	.292	.290	.286	.283	.3	.3	.229	.227	.226	.222	.219	.3
.4	.292	.290	.288	.285	.281	.4	.4	.228	.226	.225	.221	.218	.4
.5	.290	.288	.287	.283	.279	.5	.5	.227	.225	.224	.220	.217	.5
.6	.288	.287	.285	.281	.278	.6	.6	.226	.224	.223	.219	.216	.6
.7	.287	.285	.283	.280	.276	.7	.7	.225	.223	.222	.218	.216	.7
.8	.285	.283	.282	.278	.275	.8	.8	.224	.222	.221	.217	.215	.8
.9	.284	.282	.280	.277	.273	.9	.9	.223	.221	.220	.216	.214	.9
18.0	0.282	0.280	0.278	0.275	0.272	18.0	23.0	0.222	0.220	0.219	0.216	0.213	23.0
.1	.281	.279	.277	.274	.270	.1	.1	.221	.220	.218	.215	.212	.1
.2	.279	.277	.275	.272	.269	.2	.2	.220	.219	.217	.214	.211	.2
.3	.278	.276	.274	.271	.267	.3	.3	.219	.218	.216	.213	.210	.3
.4	.276	.274	.273	.269	.266	.4	.4	.218	.217	.215	.212	.209	.4
.5	.275	.273	.271	.268	.265	.5	.5	.218	.216	.214	.211	.208	.5
.6	.273	.271	.270	.266	.263	.6	.6	.217	.215	.213	.210	.208	.6
.7	.272	.270	.268	.265	.262	.7	.7	.216	.214	.212	.209	.207	.7
.8	.270	.268	.267	.264	.260	.8	.8	.215	.213	.212	.208	.206	.8
.9	.269	.267	.266	.262	.259	.9	.9	.214	.212	.211	.208	.205	.9
19.0	0.268	0.266	0.264	0.261	0.258	19.0	24.0	0.213	0.212	0.210	0.207	0.204	24.0
.1	.266	.264	.263	.259	.256	.1	.1	.212	.211	.209	.206	.203	.1
.2	.265	.263	.261	.258	.255	.2	.2	.211	.210	.208	.205	.202	.2
.3	.264	.262	.260	.257	.254	.3	.3	.210	.209	.207	.204	.202	.3
.4	.262	.260	.259	.255	.253	.4	.4	.210	.208	.206	.203	.201	.4
.5	.261	.259	.257	.254	.251	.5	.5	.209	.207	.206	.202	.200	.5
.6	.260	.258	.256	.253	.250	.6	.6	.208	.207	.205	.202	.199	.6
.7	.258	.257	.255	.251	.249	.7	.7	.207	.206	.204	.201	.198	.7
.8	.257	.255	.253	.250	.247	.8	.8	.206	.205	.203	.200	.198	.8

TABLE XXXIII P.2.

End Point (mL)	Grams of Sucrose present in 100 mL of Titrating Solution					End Point (mL)	End Point (mL)	Grams of Sucrose present in 100 mL of Titrating Solution					End Point (mL)
	Nil	0.5	1	2	3			Nil	0.5	1	2	3	
Grams RS per 100 mL Titrating Solution						Grams RS per 100 mL Titrating Solution							
25.0	0.205	0.203	0.202	0.198	0.196	25.0	30.0	0.172	0.170	0.168	0.166	0.164	30.0
.1	.204	.203	.201	.198	.195	.1	.1	.171	.169	.168	.165	.163	.1
.2	.203	.202	.200	.197	.195	.2	.2	.171	.169	.167	.165	.163	.2
.3	.203	.201	.199	.196	.194	.3	.3	.170	.168	.167	.164	.162	.3
.4	.202	.200	.198	.195	.193	.4	.4	.170	.168	.166	.164	.162	.4
.5	.201	.199	.198	.195	.192	.5	.5	.169	.167	.166	.163	.161	.5
.6	.200	.199	.197	.194	.192	.6	.6	.168	.167	.165	.163	.160	.6
.7	.200	.198	.196	.193	.191	.7	.7	.168	.166	.165	.162	.160	.7
.8	.199	.197	.195	.192	.190	.8	.8	.167	.166	.164	.162	.159	.8
.9	.198	.196	.195	.192	.189	.9	.9	.167	.165	.164	.161	.159	.9
26.0	0.197	0.196	0.194	0.191	0.189	26.0	31.0	0.166	0.165	0.163	0.161	0.158	31.0
.1	.197	.195	.193	.190	.188	.1	.1	.166	.164	.163	.160	.158	.1
.2	.196	.194	.193	.190	.187	.2	.2	.165	.164	.162	.160	.157	.2
.3	.195	.194	.192	.189	.187	.3	.3	.165	.163	.162	.159	.157	.3
.4	.195	.193	.191	.188	.186	.4	.4	.164	.163	.161	.159	.156	.4
.5	.194	.192	.190	.188	.185	.5	.5	.164	.162	.161	.158	.156	.5
.6	.193	.191	.190	.187	.184	.6	.6	.163	.162	.160	.158	.155	.6
.7	.193	.191	.189	.186	.184	.7	.7	.163	.161	.160	.157	.155	.7
.8	.192	.190	.188	.185	.183	.8	.8	.162	.161	.159	.157	.154	.8
.9	.191	.189	.188	.185	.182	.9	.9	.162	.160	.159	.156	.154	.9
27.0	0.190	0.189	0.187	0.184	0.182	27.0	32.0	0.161	0.160	0.158	0.156	0.154	32.0
.1	.190	.188	.186	.183	.181	.1	.1	.161	.159	.158	.155	.153	.1
.2	.189	.187	.186	.183	.180	.2	.2	.160	.159	.157	.155	.153	.2
.3	.188	.187	.185	.182	.180	.3	.3	.160	.158	.157	.154	.152	.3
.4	.188	.186	.184	.181	.179	.4	.4	.159	.158	.156	.154	.152	.4
.5	.187	.185	.183	.181	.179	.5	.5	.159	.157	.156	.153	.151	.5
.6	.186	.185	.183	.180	.178	.6	.6	.158	.157	.155	.153	.151	.6
.7	.186	.184	.182	.179	.177	.7	.7	.158	.156	.155	.152	.150	.7
.8	.185	.183	.182	.179	.177	.8	.8	.157	.156	.154	.152	.150	.8
.9	.184	.183	.181	.178	.176	.9	.9	.157	.156	.154	.151	.150	.9
28.0	0.184	0.182	0.180	0.177	0.175	28.0	33.0	0.157	0.155	0.153	0.151	0.149	33.0
.1	.183	.181	.180	.177	.175	.1	.1	.156	.155	.153	.151	.149	.1
.2	.182	.181	.179	.176	.174	.2	.2	.156	.154	.153	.150	.148	.2
.3	.182	.180	.178	.176	.174	.3	.3	.155	.154	.152	.150	.148	.3
.4	.181	.180	.178	.175	.173	.4	.4	.155	.153	.152	.149	.147	.4
.5	.181	.179	.177	.175	.172	.5	.5	.154	.153	.151	.149	.147	.5
.6	.180	.178	.177	.174	.172	.6	.6	.154	.152	.151	.149	.146	.6
.7	.179	.178	.176	.173	.171	.7	.7	.153	.152	.150	.148	.146	.7
.8	.179	.177	.175	.173	.171	.8	.8	.153	.151	.150	.148	.146	.8
.9	.178	.176	.175	.172	.170	.9	.9	.153	.151	.150	.147	.145	.9
29.0	0.178	0.176	0.174	0.171	0.169	29.0	34.0	0.152	0.151	0.149	0.147	0.145	34.0
.1	.177	.175	.173	.171	.169	.1	.1	.152	.150	.149	.146	.144	.1
.2	.176	.174	.173	.171	.168	.2	.2	.151	.150	.148	.146	.144	.2
.3	.176	.174	.172	.170	.168	.3	.3	.151	.149	.148	.146	.144	.3
.4	.175	.173	.172	.169	.167	.4	.4	.151	.149	.147	.145	.143	.4
.5	.175	.173	.171	.169	.166	.5	.5	.150	.148	.147	.145	.143	.5
.6	.174	.172	.171	.168	.166	.6	.6	.150	.148	.147	.144	.142	.6
.7	.173	.172	.170	.168	.165	.7	.7	.149	.147	.146	.144	.142	.7
.8	.173	.171	.169	.167	.165	.8	.8	.149	.147	.146	.143	.142	.8
.9	.172	.170	.169	.166	.164	.9	.9	.148	.147	.145	.143	.141	.9

TABLE XXXIII P.3.

End Point (mL)	Grams of Sucrose present in 100 mL of Titrating Solution					End Point (mL)
	Nil	0.5	1	2	3	
Grams RS per 100 mL Titrating Solution						
35.0	0.148	0.146	0.145	0.143	0.141	35.0
.1	.148	.146	.144	.142	.140	.1
.2	.147	.145	.144	.142	.140	.2
.3	.147	.145	.144	.141	.139	.3
.4	.146	.145	.143	.141	.139	.4
.5	.146	.144	.143	.141	.139	.5
.6	.146	.144	.142	.140	.138	.6
.7	.145	.144	.142	.140	.138	.7
.8	.145	.143	.142	.140	.137	.8
.9	.144	.143	.141	.139	.137	.9
36.0	0.144	0.142	0.141	0.139	0.137	36.0
.1	.144	.142	.140	.138	.136	.1
.2	.143	.142	.140	.138	.136	.2
.3	.143	.141	.140	.138	.136	.3
.4	.143	.141	.139	.137	.135	.4
.5	.142	.140	.139	.137	.135	.5
.6	.142	.140	.139	.137	.134	.6
.7	.141	.140	.138	.136	.134	.7
.8	.141	.139	.138	.136	.134	.8
.9	.141	.139	.138	.135	.133	.9
37.0	0.140	0.139	0.137	0.135	0.133	37.0
.1	.140	.138	.137	.135	.133	.1
.2	.139	.138	.136	.134	.132	.2
.3	.139	.137	.136	.134	.132	.3
.4	.139	.137	.136	.134	.132	.4
.5	.138	.137	.135	.133	.131	.5
.6	.138	.136	.135	.133	.131	.6
.7	.138	.136	.135	.133	.131	.7
.8	.137	.136	.134	.132	.130	.8
.9	.137	.135	.134	.132	.130	.9

Refer: Lane, J.H. and Eynon, L.; J.Soc.Chem.Ind.; Vol. 42 (1923), p.32T.

The Table is interpolated from values in this paper.

TABLE XXXIV

Properties of Saturated Steam

Pressure p (kPa)		Temp. °C t	Specific Enthalpy (kJ/kg)			Specific Volume (l/kg)	
Absolute p	Gauge		Saturated liquid h _f	Evapora- tion h _{fg}	Saturated vapour h _g	Saturated liquid v _f	Saturated vapour v _g
1.0	—100.325	6.983	29.34	2485.0	2514.4	1.0001	129209.0
1.5	—99.825	13.036	54.71	2470.7	2525.5	1.0006	87982.1
2.0	—99.325	17.513	73.46	2460.2	2533.6	1.0012	67006.1
2.5	—98.825	21.096	88.45	2451.7	2540.2	1.0019	54256.2
3.0	—98.325	24.100	101.0	2444.6	2545.6	1.0027	45667.3
3.5	—97.825	26.694	111.8	2438.5	2550.4	1.0033	39478.7
4.0	—97.325	28.983	121.4	2433.1	2554.5	1.0040	34802.2
4.5	—96.825	31.035	130.0	2428.2	2558.2	1.0046	31140.8
5.0	—96.325	32.898	137.8	2423.8	2561.6	1.0052	28194.3
6.0	—95.325	36.183	151.5	2416.0	2567.5	1.0064	23741.0
7.0	—94.325	39.025	163.4	2409.2	2572.6	1.0074	20531.0
8.0	—93.325	41.534	173.9	2403.2	2577.1	1.0084	18104.6
9.0	—92.325	43.787	183.3	2397.9	2581.1	1.0093	16204.3
10	—91.325	45.833	191.8	2392.9	2584.8	1.0102	14674.6
12	—89.325	49.446	206.9	2384.3	2591.2	1.0118	12361.9
14	—87.325	52.574	220.0	2376.7	2596.7	1.0133	10694.2
16	—85.325	55.341	231.6	2370.0	2601.6	1.0147	9433.1
18	—83.325	57.826	242.0	2363.9	2605.9	1.0160	8445.2
20	—81.325	60.086	251.5	2358.4	2609.9	1.0172	7649.8
25	—76.325	64.992	272.0	2346.4	2618.3	1.0199	6204.5
30	—71.325	69.124	289.3	2336.1	2625.4	1.0223	5229.3
40	—61.325	75.886	317.7	2319.2	2636.9	1.0265	3993.4
50	—51.325	81.345	340.6	2305.4	2646.0	1.0301	3240.2
60	—41.325	85.954	359.9	2293.6	2653.6	1.0333	2731.8
70	—31.325	89.959	376.8	2283.3	2660.1	1.0361	2364.7
80	—21.325	93.512	391.7	2274.0	2665.8	1.0387	2087.0
90	—11.325	96.713	405.2	2265.6	2670.9	1.0411	1869.2
100	—1.325	99.632	417.5	2257.9	2675.4	1.0434	1693.7
101.325	0	100.0	418.98	2256.8	2675.6	1.0437	1672.9
110	8.675	102.317	428.8	2250.8	2679.6	1.0455	1549.2
120	18.675	104.808	439.4	2244.1	2683.4	1.0475	1428.1
130	28.675	107.133	449.2	2237.8	2687.0	1.0495	1325.1
140	38.675	109.315	458.4	2231.9	2690.3	1.0513	1236.3
150	48.675	111.372	467.1	2226.2	2693.4	1.0530	1159.0
160	58.675	113.320	475.4	2220.9	2696.2	1.0547	1091.1
170	68.675	115.170	483.2	2215.7	2699.0	1.0563	1030.9
180	78.675	116.933	490.7	2210.8	2701.5	1.0579	977.23
190	88.675	118.617	497.8	2206.1	2704.0	1.0594	929.00
200	98.675	120.231	504.7	2201.6	2706.3	1.0608	885.44
220	118.675	123.270	517.6	2193.0	2710.6	1.0636	809.84
240	138.675	126.091	529.6	2184.9	2714.5	1.0663	746.45
260	158.675	128.727	540.9	2177.3	2718.2	1.0688	692.51
280	178.675	131.203	551.4	2170.1	2721.5	1.0712	646.04
300	198.675	133.540	561.4	2163.2	2724.7	1.0735	605.56
320	218.675	135.753	570.9	2156.7	2727.6	1.0757	569.99
340	238.675	137.858	579.9	2150.4	2730.3	1.0778	538.46
360	258.675	139.865	588.5	2144.4	2732.9	1.0799	510.32
380	278.675	141.784	596.8	2138.6	2735.3	1.0819	485.05
400	298.675	143.623	604.7	2133.0	2737.6	1.0839	462.22
420	318.675	145.390	612.3	2127.5	2739.8	1.0857	441.50
440	338.675	147.090	619.6	2122.3	2741.9	1.0876	422.60
460	358.675	148.729	626.7	2117.2	2743.9	1.0894	405.28
480	378.675	150.313	633.5	2112.2	2745.7	1.0911	389.36
500	398.675	151.844	640.1	2107.4	2747.5	1.0928	374.68
520	418.675	153.327	646.5	2102.7	2749.3	1.0945	361.08
540	438.675	154.765	652.8	2098.1	2750.9	1.0961	348.46
560	458.675	156.161	658.8	2093.7	2752.5	1.0977	336.71
580	478.675	157.518	664.7	2089.3	2754.0	1.0993	325.74
600	498.675	158.838	670.4	2085.0	2755.5	1.1009	315.47

TABLE XXXIV P.2.

Pressure p (kPa)		Temp. °C t	Specific Enthalpy (kJ/kg)			Specific Volume (l/kg)	
Absolute p	Gauge		Saturated liquid h _f	Evapora- tion h _{fg}	Saturated vapour h _g	Saturated liquid v _f	Saturated vapour v _g
620	518.675	160.123	676.0	2080.9	2756.9	1.1024	305.85
640	538.675	161.376	681.5	2076.8	2758.2	1.1039	296.81
660	558.675	162.598	686.8	2072.7	2759.5	1.1053	288.30
680	578.675	163.791	692.0	2068.8	2760.8	1.1068	280.27
700	598.675	164.956	697.1	2064.9	2762.0	1.1082	272.68
720	618.675	166.095	702.0	2061.1	2763.2	1.1096	265.50
740	638.675	167.209	706.9	2057.4	2764.3	1.1110	258.70
760	658.675	168.300	711.7	2053.7	2765.4	1.1123	252.24
780	678.675	169.368	716.3	2050.1	2766.4	1.1137	246.10
800	698.675	170.415	720.9	2046.5	2767.5	1.1150	240.26
820	718.675	171.441	725.4	2043.0	2768.5	1.1163	234.69
840	738.675	172.448	729.9	2039.6	2769.4	1.1176	229.38
860	758.675	173.436	734.2	2036.2	2770.4	1.1188	224.30
880	778.675	174.405	738.5	2032.8	2771.3	1.1201	219.45
900	798.675	175.358	742.6	2029.5	2772.1	1.1213	214.81
920	818.675	176.294	746.8	2026.2	2773.0	1.1226	210.36
940	838.675	177.214	750.8	2023.0	2773.8	1.1238	206.10
960	858.675	178.119	754.8	2019.8	2774.6	1.1250	202.01
980	878.675	179.009	758.7	2016.7	2775.4	1.1262	198.07
1000	898.675	179.884	762.6	2013.6	2776.2	1.1274	194.29
1050	948.675	182.015	772.0	2005.9	2778.0	1.1303	185.45
1100	998.675	184.067	781.1	1998.5	2779.7	1.1331	177.38
1150	1048.675	186.048	789.9	1991.3	2781.3	1.1359	169.99
1200	1098.675	187.961	798.4	1984.3	2782.7	1.1386	163.20
1250	1148.675	189.814	806.7	1977.4	2784.1	1.1412	156.93
1300	1198.675	191.609	814.7	1970.7	2785.4	1.1438	151.13
1350	1248.675	193.350	822.5	1964.2	2786.6	1.1464	145.74
1400	1298.675	195.042	830.1	1957.7	2787.8	1.1489	140.72
1450	1348.675	196.688	837.5	1951.4	2788.9	1.1514	136.04
1500	1398.675	198.289	844.7	1945.2	2789.9	1.1538	131.66
1550	1448.675	199.850	851.7	1939.2	2790.8	1.1563	127.55
1600	1498.675	201.372	858.6	1933.2	2791.7	1.1586	123.69
1650	1548.675	202.857	865.3	1927.3	2792.6	1.1610	120.05
1700	1598.675	204.307	871.8	1921.5	2793.4	1.1633	116.62
1750	1648.675	205.725	878.3	1915.9	2794.1	1.1656	113.38
1800	1698.675	207.111	884.6	1910.3	2794.8	1.1678	110.32
1850	1748.675	208.468	890.7	1904.7	2795.5	1.1700	107.41
1900	1798.675	209.797	896.8	1899.3	2796.1	1.1723	104.65
1950	1848.675	211.099	902.8	1893.9	2796.7	1.1744	102.03
2000	1898.675	212.375	908.6	1888.6	2797.2	1.1766	99.536
2050	1948.675	213.626	914.3	1883.4	2797.7	1.1787	97.158
2100	1998.675	214.855	920.0	1878.2	2798.2	1.1809	94.890
2150	2048.675	216.060	925.5	1873.1	2798.6	1.1830	92.723
2200	2098.675	217.244	931.0	1868.1	2799.1	1.1850	90.652
2250	2148.675	218.408	936.3	1863.1	2799.4	1.1871	88.669
2300	2198.675	219.552	941.6	1858.2	2799.8	1.1891	86.769
2350	2248.675	220.676	946.8	1853.3	2800.1	1.1912	84.948
2400	2298.675	221.783	951.9	1848.5	2800.4	1.1932	83.199
2450	2348.675	222.871	957.0	1843.7	2800.7	1.1952	81.520
2500	2398.675	223.943	962.0	1839.0	2800.9	1.1972	79.905
2550	2448.675	224.998	966.9	1834.3	2801.2	1.1991	78.352
2600	2498.675	226.037	971.7	1829.6	2801.4	1.2011	76.856
2650	2548.675	227.061	976.5	1825.1	2801.6	1.2030	75.415
2700	2598.675	228.071	981.2	1820.5	2801.7	1.2050	74.025
2750	2648.675	229.066	985.9	1816.0	2801.9	1.2069	72.684
2800	2698.675	230.047	990.5	1811.5	2802.0	1.2088	71.389
2850	2748.675	231.014	995.0	1807.1	2802.1	1.2107	70.138
2900	2798.675	231.969	999.5	1802.6	2802.2	1.2126	68.928
2950	2848.675	232.911	1004.0	1798.3	2802.2	1.2145	67.758
3000	2898.675	233.841	1008.4	1793.9	2802.3	1.2163	66.626

TABLE XXXV

Equivalents
Volume and Capacity Equivalents

in ³	ft ³	UK gal	US gal	litres	m ³
1	0.0005787	0.00360	0.00433	0.01639	1.639 x 10 ⁻⁵
1728	1	6.229	7.481	28.32	0.02832
277.42	0.1605	1	1.2	4.546	4.546 x 10 ⁻³
231	0.1337	0.833	1	3.785	3.785 x 10 ⁻³
61.02	0.03531	0.22	0.2642	1	1 x 10 ⁻³

Mass Equivalents

kg	oz	lb	Long ton	Short ton	Tonne
1	35.27	2.205	0.0009842	0.001102	0.001
0.02835	1	0.0625	0.0000279	0.00003125	0.00002835
0.4536	16	1	0.0004464	0.0005	0.0004536
1 016	35 840	2 240	1	1.12	1.016
907.2	32 000	2 000	0.8929	1	0.9072
1 000	35 274	2 205	0.9842	1.102	1

Density Equivalents

kg/m ³	g/mL	lb/ft ³	lb/UK gal
1	0.001	0.06243	0.01
1000	1	62.43	10
16.02	0.01602	1	0.1605

Linear Measure Equivalents

km	m	in	ft	yd	mile	micro-metre
1	10 ³	39.370	3 280.84	1 093.61	0.62137	10 ⁹
10 ⁻³	1	39.37	3.2808	1.0936	0.621 x 10 ⁻³	10 ⁶
2.54 x 10 ⁻⁵	0.0254	1	0.0833	0.02778	0.158 x 10 ⁻⁴	25 400
3.048 x 10 ⁻⁴	0.3048	12	1	0.3333	0.1894 x 10 ⁻³	304 800
9.144 x 10 ⁻⁴	0.9144	36	3	1	0.5682 x 10 ⁻³	914 400

Surface and Area Equivalents

m ²	in ²	ft ²	yd ²	acre	hectare
1	1 550	10.76	1.196	0.0002471	0.0001
0.0006452	1	0.006944	0.0007716	0.0000001594	0.6452 x 10 ⁻⁷
0.0929	144	1	0.1111	0.00002296	0.929 x 10 ⁻⁵
0.8361	1 296	9	1	0.0002066	0.8361 x 10 ⁻⁴
4 047	6 272 640	43 560	4 840	1	0.4047
10 ⁴	15 500 031	107 639	11 960	2.471	1

Pressure Equivalents

lb/in ²	Columns of Mercury at 0°C		Columns of Water at 15°C		kg/cm ²	kPa
	mm	in	mm	in		
	1	51.715	2.0360	703.78		
19.337	1 000	39.37	13 609	535.77	1.3595	133.322
0.49115	25.4	1	345.667	13.609	0.03453	3.38638
0.00142	0.07348	0.00289	1	0.03938	0.00010	0.00980
0.03609	1.8665	0.07348	25.4	1	0.00254	0.24884
14.223	735.56	28.959	10 010	394.09	1	98.0665
0.14504	7.5006	0.2953	102.07	4.0186	0.01020	1

Heat, Energy and Work Equivalents

Joules	ft lbf	kWh	hph	cal	Btu
1	0.7376	0.2778×10^{-6}	0.3725×10^{-6}	0.2388	0.0009478
1.356	1	0.3766×10^{-6}	0.505×10^{-6}	0.3238	0.001285
3.6×10^6	2.655×10^6	1	1.341	859 845	3412.1
2.6845×10^6	1.98×10^6	0.7457	1	641 186	2544.4
4.1868	3.088	0.1163×10^{-5}	0.15596×10^{-5}	1	0.003968
1 055	778.2	0.000293	0.000393	252	1

Heat Flow Equivalents

cal/sec cm ²	cal/h cm ²	Btu/h ft ²	W/m ²
1	3 600	13 272	4.1868×10^4
0.0002778	1	3.687	11.631
0.0000753	0.2712	1	3.1546
0.0000239	0.0860	0.3170	1

TABLE XXXVI

Mensuration of Surfaces and Solids

- The circumference of a **circle** = diameter $\times \frac{22}{7}$ (or 3.1416).
 The area of **circle** = square of radius $\times 3.1416$.
 The area of **circle** = square of diameter $\times 0.7854$.
 Area of sector of **circle** = length of arc $\times \frac{1}{2}$ radius.
 Area of segment of **circle** = area of sector less area of triangle.
 Area of **square, rhombus or rhomboid** = base \times height.
 Area of **triangle** = base $\times \frac{1}{2}$ perpendicular height.
 Area of **equilateral triangle** = square of side $\times 0.433$.
 Area of **trapezoid** = $\frac{1}{2}$ sum of the two parallel sides \times height.
 Area of **any rectilinear figure** that has four or more unequal sides is found by dividing it into triangles, finding the area of each and adding them together.
 Area of any **regular polygon** = $\frac{1}{2}$ radius of the inscribed circle \times length of one side \times number of sides.
 Area of **parabola** = base $\times \frac{2}{3}$ height.
 Area of **ellipse** = major axis \times minor axis $\times 0.7854$.
 Surface area of **prism or cylinder** = area of 2 ends + length \times perimeter.
 Volume of **prism or cylinder** = area of base \times height.
 Surface area of **cone or pyramid** = $\frac{1}{2}$ slant height \times perimeter of base + area of base.
 Volume of **cone or pyramid** = $\frac{1}{3}$ area of base \times perpendicular height.
 Area of surface of **cube or parallelepiped** = sum of the areas of the sides.
 Volume of **cube or parallelepiped** = length \times breadth \times depth.
 Surface area of **sphere** = square of diameter $\times 3.1416$.
 Volume of **sphere** = cube of diameter $\times 0.5236$.
 Volume of **hexagonal prism** = square of side \times height $\times 2.598$.
 Volume of **paraboloid** = $\frac{1}{2}$ volume of circumscribing cylinder.
 Volume of **ring** (circular section) = mean diameter of ring $\times 2.47 \times$ square of diameter of section.
 Side of **square** or area equal to **circle** = diameter $\times 0.8862$.
 Diameter of **circle** equal in area to **square** = side $\times 1.1284$.
 Side of **square** inscribed in **circle** = diameter $\times 0.707$.
 Diameter of **circle** circumscribing **square** = side $\times 1.414$.

TABLE XXXVII

Capacity of Horizontal Cylindrical Tanks at Varying Levels

i = depth of liquid
d = diameter of vessel.

i/d	fraction of total	i/d	fraction of total	i/d	fraction of total	i/d	fraction of total
.01	.0017	.26	.2066	.51	.5127	.76	.8155
.02	.0048	.27	.2178	.52	.5255	.77	.8263
.03	.0087	.28	.2292	.53	.5382	.78	.8369
.04	.0134	.29	.2407	.54	.5509	.79	.8473
.05	.0187	.30	.2523	.55	.5636	.80	.8576
.06	.0245	.31	.2640	.56	.5762	.81	.8677
.07	.0308	.32	.2759	.57	.5888	.82	.8776
.08	.0375	.33	.2878	.58	.6014	.83	.8873
.09	.0446	.34	.2998	.59	.6140	.84	.8967
.10	.0520	.35	.3119	.60	.6265	.85	.9059
.11	.0598	.36	.3241	.61	.6389	.86	.9149
.12	.0680	.37	.3364	.62	.6513	.87	.9236
.13	.0764	.38	.3487	.63	.6636	.88	.9320
.14	.0851	.39	.3611	.64	.6759	.89	.9402
.15	.0941	.40	.3735	.65	.6881	.90	.9480
.16	.1033	.41	.3860	.66	.7002	.91	.9554
.17	.1127	.42	.3986	.67	.7122	.92	.9625
.18	.1224	.43	.4112	.68	.7241	.93	.9692
.19	.1323	.44	.4238	.69	.7360	.94	.9755
.20	.1424	.45	.4364	.70	.7477	.95	.9813
.21	.1527	.46	.4491	.71	.7593	.96	.9866
.22	.1631	.47	.4618	.72	.7708	.97	.9913
.23	.1737	.48	.4745	.73	.7822	.98	.9952
.24	.1845	.49	.4873	.74	.7934	.99	.9983
.25	.1955	.50	.5000	.75	.8045	1.00	1.0000

TABLE XXXVIII

Boiling Point Elevation of Sugar Solutions and Cane Juices (°C)
at 760 mm pressure

Brix	Purity						
	100	90	80	70	60	50	40
10	0.1	0.1	0.1	0.1	0.2	0.2	0.2
15	0.2	0.2	0.2	0.2	0.3	0.3	0.4
20	0.3	0.3	0.3	0.4	0.4	0.5	0.6
25	0.4	0.5	0.5	0.6	0.7	0.8	0.9
30	0.6	0.7	0.7	0.8	1.0	1.1	1.2
35	0.8	0.9	1.0	1.1	1.3	1.4	1.6
40	1.0	1.1	1.3	1.5	1.7	1.9	2.1
45	1.4	1.5	1.8	2.0	2.2	2.4	2.7
50	1.8	1.9	2.2	2.5	2.8	3.1	3.4
55	2.3	2.5	2.8	3.1	3.5	3.9	4.3
60	3.0	3.2	3.6	4.0	4.4	4.9	5.4
65	3.8	4.1	4.5	4.9	5.3	6.0	6.5
70	5.1	5.5	6.0	6.5	7.1	7.7	8.3
75	7.0	7.5	8.0	8.6	9.4	10.1	10.8
80	9.4	10.0	10.5	11.3	12.3	13.1	14.1
85	13.0	13.7	14.4	15.3	16.4	17.4	19.1
90	19.6	20.5	21.2	22.4	23.7	25.3	
94	30.5						

TABLE XXXIX

International Atomic Weights, 1971
(Published by Perry and Chilton, "Chemical Engineer's Handbook" Fifth Edition.)

Element	Symbol	Atomic Number	Atomic Weight	Element	Symbol	Atomic Number	Atomic Weight
Actinium	Ac	89	..	Mercury	Hg	80	200.59
Aluminium	Al	13	26.982	Molybdenum	Mo	42	95.94
Americium	Am	95	..	Neodymium	Nd	60	144.24
Antimony	Sb	51	121.75	Neon	Ne	10	20.183
Argon	Ar	18	39.948	Neptunium	Np	93	..
Arsenic	As	33	74.922	Nickel	Ni	28	58.71
Astatine	At	85	..	Niobium	Nb	41	92.906
Barium	Ba	56	137.34	Nitrogen	N	7	14.007
Berkelium	Bk	97	..	Nobelium	No	102	..
Beryllium	Be	4	9.012	Osmium	Os	76	190.2
Bismuth	Bi	83	208.98	Oxygen	O	8	15.999
Boron	B	5	10.811	Palladium	Pd	46	106.4
Bromine	Br	35	79.909	Phosphorus	P	15	30.974
Cadmium	Cd	48	112.40	Platinum	Pt	78	195.09
Calcium	Ca	20	40.08	Plutonium	Pu	94	..
Californium	Cf	98	..	Polonium	Po	84	..
Carbon	C	6	12.011	Potassium	K	19	39.098
Cerium	Ce	58	140.12	Praseodymium	Pr	59	140.907
Cesium	Cs	55	132.905	Promethium	Pm	61	..
Chlorine	Cl	17	35.453	Protactinium	Pa	91	..
Chromium	Cr	24	51.996	Radium	Ra	88	..
Cobalt	Co	27	58.933	Radon	Rn	86	..
Copper	Cu	29	63.54	Rhenium	Re	75	186.20
Curium	Cm	96	..	Rhodium	Rh	45	102.905
Dysprosium	Dy	66	162.50	Rubidium	Rb	37	85.47
Einsteinium	Es	99	..	Ruthenium	Ru	44	101.07
Erbium	Er	68	167.26	Samarium	Sm	62	150.35
Europium	Eu	63	151.96	Scandium	Sc	21	44.956
Fermium	Fm	100	..	Selenium	Se	34	78.96
Fluorine	F	9	19.00	Silicon	Si	14	28.086
Francium	Fr	87	..	Silver	Ag	47	107.870
Gadolinium	Gd	64	157.25	Sodium	Na	11	22.990
Gallium	Ga	31	69.72	Strontium	Sr	38	87.62
Germanium	Ge	32	72.59	Sulphur	S	16	32.064
Gold	Au	79	196.967	Tantalum	Ta	73	180.948
Hafnium	Hf	72	178.49	Technetium	Tc	43	..
Helium	He	2	4.003	Tellurium	Te	52	127.60
Holmium	Ho	67	164.930	Terbium	Tb	65	158.924
Hydrogen	H	1	1.008	Thallium	Tl	81	204.37
Indium	In	49	114.82	Thorium	Th	90	232.038
Iodine	I	53	126.904	Thulium	Tm	69	168.934
Iridium	Ir	77	192.2	Tin	Sn	50	118.69
Iron	Fe	26	55.847	Titanium	Ti	22	47.90
Krypton	Kr	36	83.80	Tungsten	W	74	183.85
Lanthanum	La	57	138.91	Uranium	U	92	238.03
Lawrencium	Lw	103	..	Vanadium	V	23	50.942
Lead	Pb	82	207.19	Xenon	Xe	54	131.30
Lithium	Li	3	6.939	Ytterbium	Yb	70	173.04
Lutetium	Lu	71	174.97	Yttrium	Y	39	88.905
Magnesium	Mg	12	24.312	Zinc	Zn	30	65.38
Manganese	Mn	25	54.938	Zirconium	Zr	40	91.22
Mendelevium	Md	101	..				

Note: The above atomic weights are based on the isotope C12.

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