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HYGROSCOPIC WATER IN SUGAR CANE

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FOREWORD

At the 1989 Workshop on Cane Analysis conducted by the Sugar Research Council it emerged that the best technical method of cane analysis for the Australian industry was the direct cane analysis method (DCA) employing the wet disintegrator technique. This method involves comminuting a sample of prepared cane with a weighed amount of water and then analysing the resultant liquid extract. The analysis of cane is determined by calculation, making allowance for the degree of dilution by the added water. This calculation also makes allowance for some water in sugar cane which is not available for solution of the dissolved substances in juice. This water is commonly referred to as bound water or hygroscopic water.

The usual calculations with DCA involve the use of a value for hygroscopic water of 25 percent of the fibre percentage. The origin of this 25 percent figure cannot be determined but it is believed that it came about at the time of the method's development when it represented an approximate average of the data available at the time. Because early data showed a wide range of values reflecting possibly variations from one type of cane to the next and analytical errors, it was decided to attempt the rigorous determination of hygroscopic water in sugar cane with the benefit of today's better analytical instruments.

The author has found the same difficulties that were experienced by the earlier workers but an attempt has been made to show how much the variability in results can be attributed to the many causes. We think that the traditional 25 percent of fibre figure may be a little high for today's canes especially considering that the fibre as determined by traditional methods includes 1-2 percentage units of dirt. The hygroscopic water associated with dirt is much lower than that for true cane fibre. A more realistic value would probably lie between 15 and 20 percent of fibre.

M.R. Player
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Abstract

Hygroscopic water in sugar cane may be characterised as water strongly sorbed onto the cane fibre and being unavailable for the solution of the soluble components present in sugar cane. As a result, analytical methods utilising the wet disintegrator technique, produce higher pol and Brix results, than would be observed if all water present in cane fibre were available for solution of soluble substances.

Employing a method whereby dry, processed, essentially sucrose free cane fibre is contacted with a known quantity of sucrose solution for the measurement of hygroscopic water, this report aimed to improve on previous attempts in providing a repeatable, reproducible method for the determination of hygroscopic water in cane fibre, to investigate the effect of experimental variations, and to validate the currently used value of 25% on fibre.

In addition, absolute measurements of cane constituents via Soxhlet extraction under vacuum, controlled to maintain specified extraction conditions, were made in order to evaluate the effectiveness of this extraction technique in comparison to the currently used wet disintegrator technique.

Results indicated that the mean value of % hygroscopic water for several cane ranged between 17 and 22%, falling short of the currently used value of 25%, presenting a new value that may be incorporated in wet disintegrator results.

Soxhlet extraction results indicated that the possibility exists for using an extraction technique for Brix determinations in cane, however the quantity of cane used for the analysis would need to be increased in order to be representative of the gross sample. Overall, Soxhlet extraction results tended to be 0.5 to 1.0 % Brix on cane lower in comparison to pilot wet disintegrator results.

Summary

Hygroscopic water constitutes that portion of the water in cane not available for the solution of solubles in cane. Previous determinations of the constituent yielded values ranging from 10 to 50%, though a value of 25% is currently employed for convenience. The validity of this value, using a dry fibre/sucrose solution slurry, was tested, along with an attempt to develop a new absolute method for cane analysis via Soxhlet extraction techniques.

Wet disintegrator analyses performed at various dilutions yielded negative and large positive values for % hygroscopic water on fibre, with only 20% of the results falling in the 20-30% hygroscopic water region. Moisture loss via evaporation was also examined by cooling the disintegrator vessel, for which results indicated that any Brix (or pol) increase due to evaporation had no appreciable effect upon the determination. Consequently, another method was examined for use in the analysis.

Several attempts using wet (and dry) fibre were made in order to obtain a reproducible, repeatable method for use in the investigation. The dry fibre contact method, in which a weighed quantity of dry, essentially sucrose free fibre was mixed with a known quantity of sucrose solution, was adopted for the project owing to the reproducibility of results obtained.

An analysis monitoring the Brix change in a fibre/sucrose solution slurry as a function of time revealed that 30 minutes after preparing the slurry, no measurable change in Brix occurred, indicating equilibrium had been attained. As a result an equilibration time of 1.5 hours was adopted for all following analyses. In addition, hygroscopic water results obtained over 24 hour equilibration periods yielded results 1 to 2 units greater than the duplicate analysis performed over 1.5 hours, this difference being statistically significant.

The use of 4 and 10°Bx sucrose solutions for the analysis showed that the 10°Bx solutions gave rise to more precise results (% C.V. = 5.9) than were obtained using 4°Bx solutions (% C.V. = 15.8). For this reason, 10°Bx sucrose solutions were used for all dry fibre contact analyses throughout the project.

Samples prepared via Jeffco cutter-grinder, wet disintegrator (W.D.) and gyratory type machine grinder were analysed for hygroscopic water, with the finer machine ground fibre yielding results 4 to 5 units higher for % hygroscopic water in comparison to both wet disintegrator and Jeffco cutter material.

A comparison between % hygroscopic water results obtained at ambient temperature and 50°C revealed that results, on average, increased by 1 to 2 units for samples maintained at

the higher equilibration temperature, the difference being statistically insignificant.

Various components of the cane plant for the Herbert region varieties Q115 and Triton were analysed, both varieties showed root material to have a low hygroscopic water (5-8%) content, while cane tops exhibited high values (23-25%), in comparison to stalk and trash material which yielded values for hygroscopic water 4 to 5 units lower than that of tops material (20%). Pith and fibrous material separated from a sample of variety Q96 revealed that the soft pith yielded greater values for hygroscopic water (23%) than the fibrous material (19%).

Five soil samples collected from farms in the Herbert region indicated that soil yielded % hygroscopic water results in the order of 3 to 5%, with one soil type yielding results in the 6 to 9% range.

Several cane varieties representing approximately 80% of the cane milled in the Herbert region (i.e. Q96, Q119, Q115, Q117, Cassius and Triton) were analysed and gave % hygroscopic water values lying in the 17-20% range, the majority of samples yielding values closer to the higher end of this range. Two varieties (i.e. Q96 and Q117) representing approximately 80% of the cane milled in the Burdekin region indicated that regardless of crop location, a certain cane variety will yield similar results for hygroscopic water.

It was therefore concluded that the currently used value of 25% for hygroscopic water may be in error by as much as 8 units % hygroscopic water on fibre, as mean results for several cane varieties showed hygroscopic water to lie in the 17 to 20% region. Results also reflected the importance of a clean cane supply to the mill, as the inclusion of extraneous matter, if in sufficient quantity, could result in an inaccurate assessment of cane quality.

Soxhlet extraction work carried out, in conjunction with a small scale wet disintegrator system, yielded similar results for variety Q113, however variety Q124 produced higher than expected wet disintegrator results, possibly as a result of moisture loss in the sample received.

Hygroscopic Water in Sugar Cane

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A. Introduction

At a Cane Analysis workshop held in 1988 (WRIGHT, 1988), direct cane analysis (DCA) was chosen as the preferred method of cane analysis for miller/grower equity. Fundamental to the accuracy of this method is the value of hygroscopic water used for calculations.
Hygroscopic water is realised in wet disintegrator, direct cane analysis as water strongly sorbed onto the cane fibre and being unavailable for the solution of the soluble components. This has the effect of producing higher pol and Brix results in the disintegrator slurry, than would be observed if all water present in cane fibre were available for solution of soluble substances. The existence of hygroscopic water in cane fibre may be explained by inspecting the cross section of a sugar cane stalk, which reveals three distinct regions consisting of an outer peripheral region (or rind) and an inner soft pith section in which soft tissue is interspersed with fibre, each area differing mainly in the relative concentration of juice-containing (parenchyma) vacuoles and vascular bundles (see Figures 1, 2).
Vascular bundles, contributing greatly to the total fibre in cane, contain many small thick-walled cells which surround the large vessels and sieve tubes, with vessels transporting water from the roots and sieve tubes conveying sugary juices from the leaves. The thick walls of these fibre cells, which make up a large proportion of the vascular bundles, also constitute the major part of the weight of fibre in cane.

Long chain cellulose molecules forming the basis of cell wall material (see Figure 3), occasionally pack together in parallel rows giving rise to a crystalline structure. In other areas, these cellulose chains have a random arrangement leading to the formation of micro-capillaries in the cell structure, contributing greatly to the water sorbing properties of the fibre.

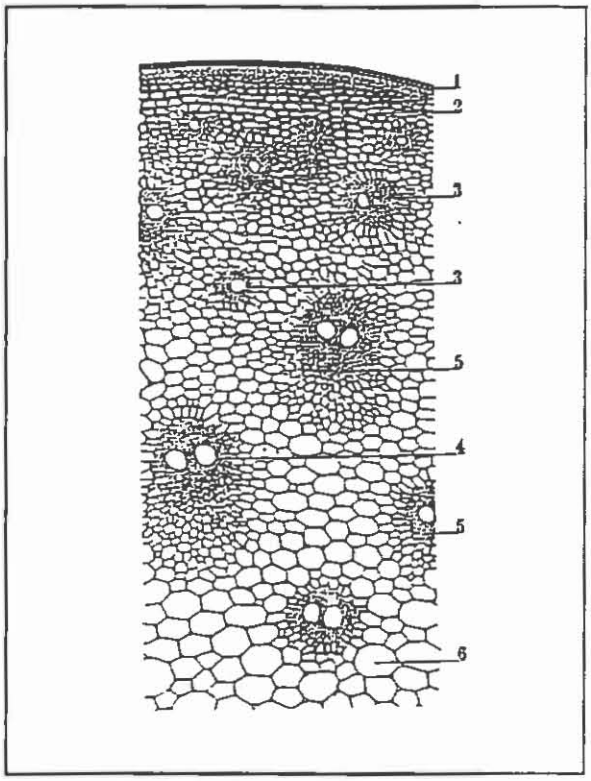


FIGURE 1 : Cross Section through Internode.
1. Epidermis; 2. Thick walled cells; 3,4. Vascular bundles; 5. Sclerenchyma; 6. Ground tissue.
VAN DILLEWIJN (1952)

The majority of the water present in fibre is held by two processes:-

a) Surface Adsorption

b) Capillary condensation.

Surface adsorbed water is thought to be held by weak chemical bonds onto the surfaces of the fibre substance, including the interior surfaces of the micro-capillaries, consisting of a single molecular layer (monolayer) only and being more strongly bound than the capillary condensed water (surface tension phenomena). However, sigmoidal adsorption isotherms of cellulose materials indicate the presence of multilayer adsorption (Cote and Kollmann, 1968).

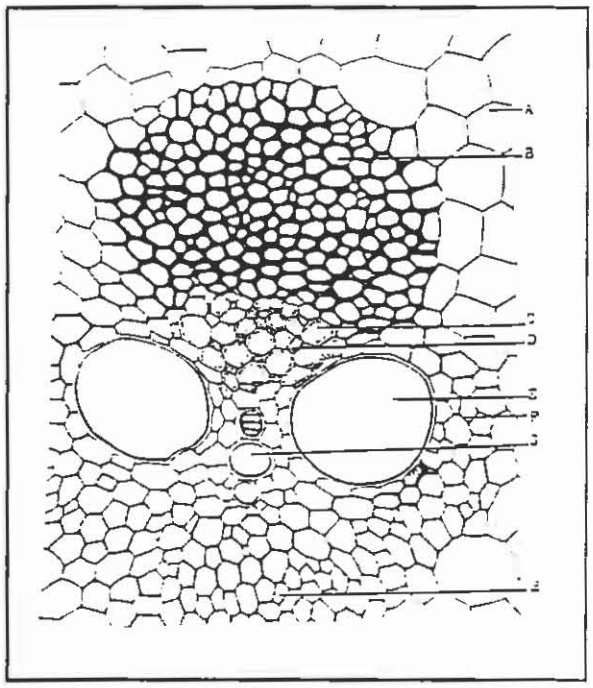


FIGURE 2 : Bundle cross section.
A. Outer bundle parenchyma; B.Sclerenchyma cap; C. Sieve tube; D. Companion cell; E. Large pitted vessel; F. Bundle sheath; G. Protoxylem; H. Sclerechyma cap of xylem pole of bundle.
VAN DILLEWIJN (1952)

Previous attempts to measure this hygroscopic water component in cane fibre followed several paths. Steuerwald (1912) approached the problem in two ways,

(i) by measuring the increase in concentration of a known weight of sucrose solution after remaining in contact with a known weight of dry fibre, yielding a value of 22.4% for adsorbed water,

(ii) by analysing the sugar content of a residue obtained after pressing cane fibre with a quantity of pure sugar solution, producing a result of 16.5% for adsorbed water with even some negative results being recorded.

Foster (1962), amending Steuerwald's press method (ii), returned residue from pressings back to the fibre several times, resulting in values of 10-18% hygroscopic water, suggesting that the varied results were obtained due to the expression of hygroscopic water from within the cane fibre.

Again in 1963, Foster attempted the measurement of hygroscopic water employing another approach, where by Brix changes in disintegrated water/sugar cane slurries were used to calculate % hygroscopic water values ranging from 25 to 40%. Later, CSR staff officers (RICHARDSON, 1970) utilising the dry fibre contact method, recorded hygroscopic water results ranging from 15 to 29% and having an average of 24.8%.
Currently, a value of 25% hygroscopic water on fibre is employed, however it is thought that its value has no more validity than being the mean result of previous determinations whose range varied from 10-50%, the majority falling in the 20-30% region.

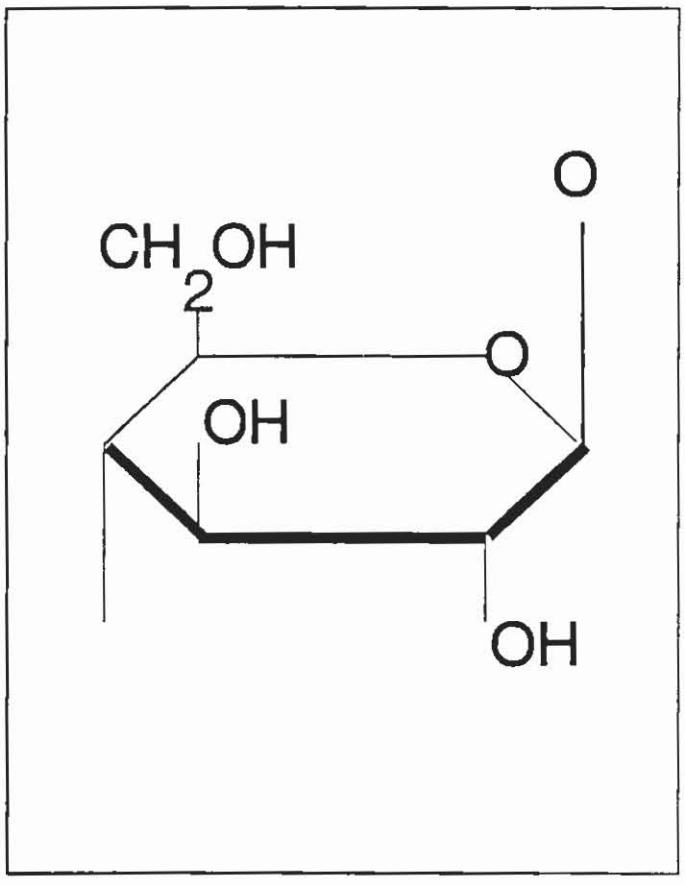


FIGURE 3 : Cellulose repeat unit - $(C_6H_{10}O_5)_n$.

B. Theory and Methodology

Wet Disintegrator

The wet disintegrator method employed for this project involved combining a known mass of cane with a known mass of water in a wet disintegrator, and comminuting for a fixed period of time. Following disintegration the resulting slurry was analysed for Brix and/or pol prior to dilution with additional water. Following each dilution, the slurry was mixed and again analysed for Brix and/or pol.

From the relationship between change in slurry concentration and solvent (water) added, the amount of unbound water present in the original sample was determined, allowing the % hygroscopic (bound) water for that fibre to be calculated.

Theory

The wet disintegrator method of analysis permits the analysis of cane for Brix and Pol by comminuting milled cane in the presence of a known weight of water and by measuring the concentration of Brix and Pol in the resulting liquor. From the Bureau of Sugar Experiment stations Laboratory Manual (1970), the following calculation is used for wet disintegrator analyses :-

$$Bx\ Cane = \frac{Bx\ Extract \times (\theta + w)}{(\phi - Bx\ Extract)} \quad \ldots\ldots\ldots (1)$$

where :

$$\theta = \frac{\frac{(X+Y)}{Y} - (1+\frac{z}{100})}{1+\frac{z}{100}} \quad \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

and

y = weight cane
x = weight water
z = hygroscopic water

$$\phi = \frac{100}{(1+\frac{z}{100})} \quad \ldots\ldots\ldots\ldots\ldots\ldots (3)$$

w = moisture % cane

Note : Hygroscopic water currently holds the value of 25% on fibre (i.e. z = 25).

In addition to the conventional wet disintegrator analysis, dilution of the initial slurry created a means by which, theoretically, hygroscopic water in fibre could be determined from the relationship between change in slurry concentration and diluent added (i.e standard addition approach). The following method was adopted to investigate the multiple dilution, wet disintegrator technique.

Two methods of calculation were used to determine unbound water in fibre, as the following equations show :-

A. Two Point (Point by Point) Calculation

$$Q = \frac{1}{C_1 - C_2}\left[C_2 W_2 - C_1 W_1 + \left(V_1 \frac{C_1}{1+\frac{C_1}{100}}\right) - \frac{C_2 V_1}{100}\left(100 - \frac{C_1}{1+\frac{C_1}{100}}\right)\right] \ldots . (4)$$

where :-

Q = unbound water

W_i = dilution ratio i (water/cane)

C_i = concentration of extract after dilution i

$$V_i = \frac{S_i}{M} \times 100 = \frac{(grams\ sample\ withdrawn)}{(mass\ cane\ used)} \times 100 \ldots . (5)$$

NOTE : i) "i" represents stage of analysis, i.e. at the start of the analysis, i=1 after the first dilution, i=2 and so on.

B. Graphical Representation - Regression

$$\frac{1}{C_i} = \frac{W_i}{100BC} + \frac{Q}{100BC} \quad \ldots\ldots\ldots (6)$$

where BC = Brix of cane
Q = unbound water

$$\frac{1}{100BC} = Slope$$

$$\frac{Q}{100BC} = Intercept$$

C_i = concentration of extract after dilution "i"

W_i = dilution ratio (water/cane)

i = stage of analysis.
At start of analysis, i=1,
after first dilution, i=2 etc.

A plot of $1/C_i$ vs W_i for i = 1,2,3...n yields a linear relationship, from which unbound water "Q" may be calculated by dividing the intercept by the slope.

Method

1. Weigh accurately 2 kilograms of milled cane into the wet disintegrator vessel.

2. Add to this 6 kilograms of distilled water and seal the wet disintegrator vessel.

3. Disintegrate the vessel contents for 45 minutes, after which time start the peristaltic pump to begin solution circulation from within the disintegration vessel (see Figure 4).

4. Once the disintegrator liquor has circulated for approximately 2 minutes, sample the liquor in a preweighed collection tube (50ml is sufficient).

5. Having sampled the liquor, stop the peristaltic pump and add a further kilogram (1kg) of distilled water to the disintegration vessel via the external inlet (see Figure 4), mixing the resultant slurry for approximately 2 minutes.

6. Repeat steps 4. and 5. until a further 3 kilograms of distilled water have been added, sampling the disintegrator liquor after each kilogram (1kg) dilution.

7. On completing the disintegration, samples taken at each dilution were weighed and analysed for Brix and/or pol.
(NOTE : See section C for Equipment and Instrumentation)

Calculations

Unbound Water

Unbound water may be calculated using equation (4) above.

If the amount of sucrose present was measured as Brix, Brix was converted to concentration via the following equation :-

$$Conc = \frac{Bx}{1-\frac{Bx}{100}} \quad \ldots\ldots\ldots\ldots (7)$$

% Hygroscopic Water on Fibre

If the total moisture in cane is represented by "W" then % hygroscopic water on fibre may be calculated viz :-

$$Z = \frac{W-Q}{F} \times 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots (8)$$

where :- Z = % Hygroscopic water
Q = unbound water
F = % fibre in cane

Fibre Contacting - Method Development

In order to investigate the suitability of the fibre contact method for the determination of hygroscopic water in sugar cane fibre, a series of analyses were performed which involved mixing wet (or dry) sucrose free fibre with sucrose solutions of known concentration. In the case of wet fibre, moisture determinations were required in order to calculate % hygroscopic water.

Once fibre and sucrose solution had been mixed, the mixture was left for a period of time allowing the migration of water molecules to the fibre surface to attain equilibrium. On equilibrating, the resultant slurry was analysed for sucrose concentration, the change in concentration reflecting the fibres ability to adsorb water from solution.

Method

In the course of the investigation, several variables were examined on the basis of :-

a. Practicality - total volume required for the analysis needed to fit into a sealable reaction vessel. This was considered necessary so as to avoid evaporative water losses producing erroneous results.

b. Minimising Error - errors involved with taking accurate Brix measurements led to the use of higher sucrose concentrations (ca. 10°Bx) than were obtained via wet disintegrator trials (ca. 4-5°Bx).

Of the several systems trialled, the method yielding reproducible results and meeting the above mentioned criteria involved using :-

Fibre Drying Time = 3 hours ± 5 min @ 80°C, 625mm Hg Vac

Fibre = 8.00 ± 0.01 grams washed to [sucrose] < 5ppm

Sucrose = 150.00 ± 0.01 grams 10°Bx aqueous solution

Mixing time = 2 minutes, hand mixed

Equilibration time = 1.5 hours

Reaction vessel = sealed Agee jar

Dry Fibre Contacting - Analysis

Hygroscopic water can be thought of as strongly adsorbed water not available for solution of soluble components within cane, but may be driven off at elevated temperatures, as is the case in fibre analysis.

The method employed involves drying to ensure that all sorbed water is removed from a pre-washed, essentially sucrose free fibre sample, followed by contacting with a sucrose solution of known concentration.
Consequently, the resultant sucrose solution, having lost moisture to the fibre, increases in concentration. From the change in sucrose concentration, the percent hygroscopic moisture on fibre may be calculated.

Method

The following method was developed for use in all fibre analyses.

1. For a duplicate analysis, dry approximately 17 grams of processed fibre per sample type in a vacuum oven for 3 hours (625mm Hg, 80°C), and cool in desiccator for 40 minutes (constant weight).

2. Prepare one (1) x 500 ml aqueous sucrose solution of 10.00g/100g concentration per sample type.

3. Weigh 8 grams of the dried fibre to the nearest 0.01 grams into a clean, dry, preweighed Agee jar.

4. To this fibre add 150 grams of 10°Bx sucrose solution (from 3.2.2) to the nearest 0.01 grams, and mix intimately for 2 minutes ensuring all the fibre is covered by the sucrose solution, sealing the jar once mixing is complete.

5. Repeat steps (3.) and (4.) until all samples and control are prepared. Note that the extra sample represents a control sample to which no fibre is added.

6. Once all samples and control have been prepared the following instruments are used to determine the Brix of each solution :-

6.1 Dipping Refractometer

Used to approximate the initial Brix prior to determination via differential refractometer. See section C - Equipment and Instrumentation for further detail.

6.2 Differential Refractometer

Used to accurately determine the Brix of solutions. See section C - Equipment and Instrumentation for further detail.

Note :

1. Both instruments were equilibrated to 20.0 ± 0.1°C during analyses.

2. All hygroscopic water calculations including statistical calculations were performed using differential refractometer results unless otherwise stated.

7. After allowing samples to equilibrate for one and a half hours, all samples including the control were filtered through Whatman No91 filter paper prior to Brix measurement.

Calculation of Hygroscopic Water

$$\% \textit{ Hygroscopic Water} = W\left(1 - \frac{p^0}{p^1}\right) \ldots\ldots\ldots (9)$$

where :

$$W = \frac{\textit{weight sucrose solution}}{\textit{weight of fibre}} \times 100 \ldots\ldots\ldots (10)$$

p^0 = Brix of sucrose solution prior mixing with fibre

P^1 = Brix of sucrose solution after mixing with fibre

Dry Fibre Contacting - Investigation

Sample Equilibration Time

In order to determine the equilibration time required when contacting dry fibre with a sucrose solution of known concentration, trials were conducted which involved measuring the Brix of the solution in the slurry (i.e. dry fibre + sucrose solution) as a function of time.

Previous determinations (Harris, 1962) investigated equilibrium times involving 1 and 24 hour periods, in which case no significant difference was observed.

Extended Equilibration Times

Having analysed cane fibre over a 1.5 hour equilibration period, it was decided to examine the effect of longer equilibration times on hygroscopic water results.

Previously, equilibration times of 1 and 24 hour periods were examined, the conclusion being that no significant difference existed between hygroscopic water results obtained over these two equilibration time periods.

In an attempt to reproduce and improve on results of past investigations, dry fibre was mixed with sucrose solutions and allowed to equilibrate for 1.5 and 24 hour periods.

Sucrose Concentration

In an attempt to highlight the effect of sucrose concentration on fibre's ability to adsorb water, sucrose solutions of two distinct concentrations (namely 4 and 10°Bx) were used for the analysis. For each run a duplicate sample was analysed using both 4°Bx and 10°Bx solutions, with control solutions containing no fibre being included also.

Sample Preparation

Of the various ways in which sugar cane may be processed for routine analysis, this investigation examined three modes of preparation of which the first two forms, namely wet disintegrator and Jeffco cutter-grinder preparations, are currently used in mill operations.

a. Wet Disintegrator

The wet disintegrator system (see Figure 4) consisted of a disintegrating vessel with a centrally located bladed shaft, all being sealed by the vessel lid.

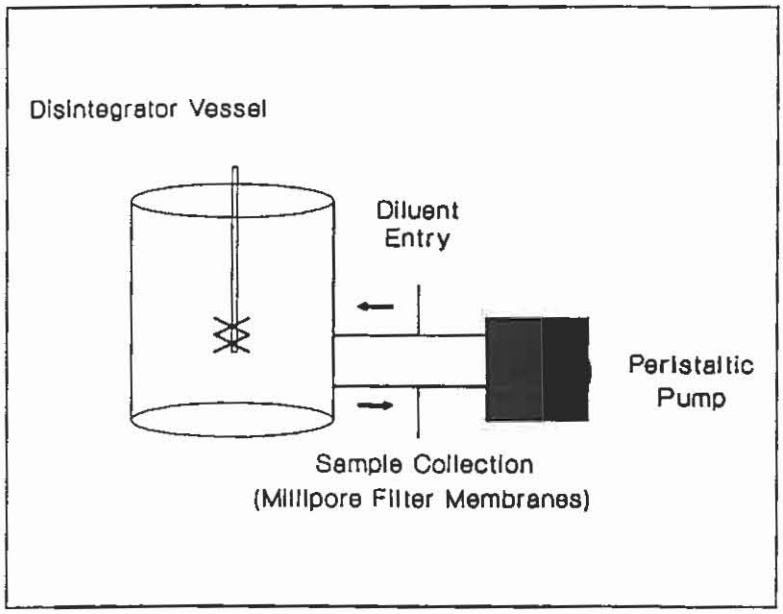


FIGURE 4 : Wet disintegrator system

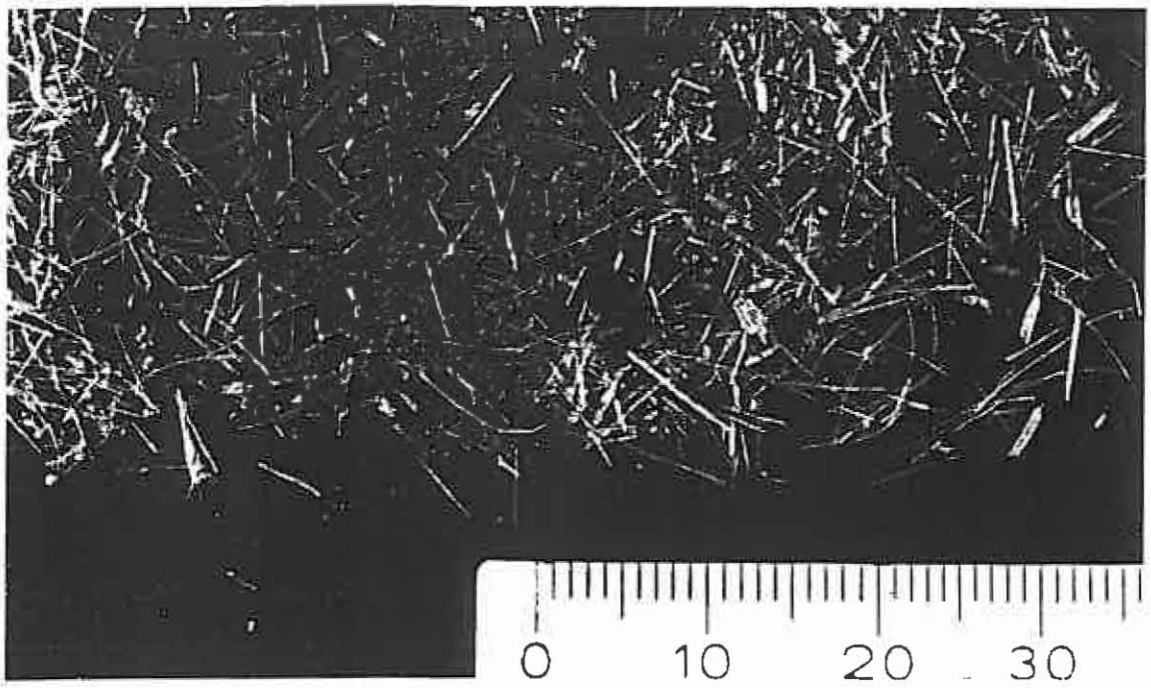
Hammer milled cane (2kg) was combined with 6kg of distilled water and placed in the vessel followed by comminuting for 45 minutes. The fibre was then washed with hot and cold water to remove enough sucrose until thymol testing indicated sucrose concentration of washings to be less than 5ppm. This treatment was followed by drying the fibre under vacuum at 80°C/625mm Hg until constant weight was achieved prior analysis (See Figure 5, a).

b. Jeffco Cutter-Grinder

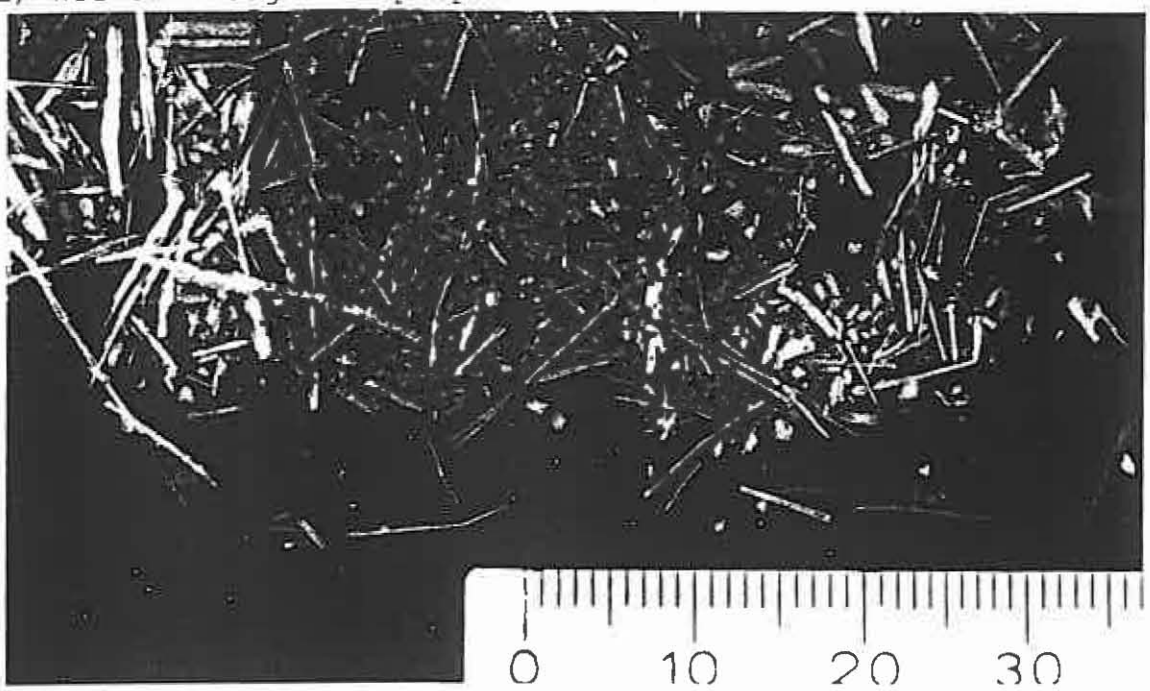
The Jeffco Cutter-Grinder material was obtained by passing billets of cane through the unit resulting in a roughly ground product, in which pith and long fibre were easily separable. Once ground, the fibre was washed and dried as per the wet disintegrator material prior analysis. Of the three modes of preparation, the Jeffco cutter-grinder produced the coarsest material (See Figure 5, b).

c. Machine Gyratory Grinder

Machine ground fibre consisted of dried, washed wet disintegrator fibre ground in a gyratory type grinder consisting of three concentric steel rings. Following this preparation the fibre had the consistency of flour, with both pith and rind components of the fibre being intimately mixed.



(a) Wet disintegrator prepared fibre



(b) Jeffco cutter-grinder prepared fibre

FIGURE 5 : Photographic representation of cane fibe prepared in two ways, scale shown is in millimetres.

Of the three modes of preparation, machine ground fibre represented the finest material in terms of particle size. An idea of the degree if finess obtained for machine machine ground fibre was determined via physical separation through sieving (see Table 1).

TABLE 1
Machine ground fibre size separation sieving results

Sample	% > 150 µ (100 mesh)	% > 45 µ (325 mesh)	% < 45 µ (325 mesh)
Q115	38.1	42.6	17.6
Q96	40.5	41.4	16.3

Equilibration Temperature

In order to assess the effect of hygroscopic water in wet disintegrator analyses, mixed fibre/sucrose solution slurries for hygroscopic water determinations were placed in an oven at elevated temperatures. A temperature of 50 degrees centigrade was chosen for the trial, this being the observed temperature reached by wet disintegrator analyses both in pilot plant and routine mill analyses.

In each case, a duplicate sample maintained at ambient conditions was analysed for comparative purposes. Also, as with all fibre analyses performed for this project, a control sample containing only sucrose solution was analysed during each analysis at both temperatures, in order to account for any Brix increase eventuating from evaporation.

Cane Plant Characteristics

The Sectioned Plant

The macro-structure of a growing cane plant consists essentially of four portions, these being tops, stalk, root and trash material.
Each portion identified above (see Figure 6) serves the growing plant in its own unique way :-

a. Tops and Leaves - provide a means by which the plant, via photosynthesis, produces carbohydrates for the production of sucrose and other sugars.

b. Stalk - provides mechanical support; contains a fibrovascular system for the transportation of water and

nutrients; provides storage for the sugary juices in parenchymatous cells.

c. Roots - providing a two-fold function, the root hairs envelop soil particles in order to maintain plant stability in the soil, and secondly, root hairs adsorb water and nutrients from the soil, transporting them to other parts of the growing plant.

d. Trash - ripening stalks lead to the termination of leaf function, in which leaves die and either remain attached to the stalk or fall to the base of the stalk as trash.

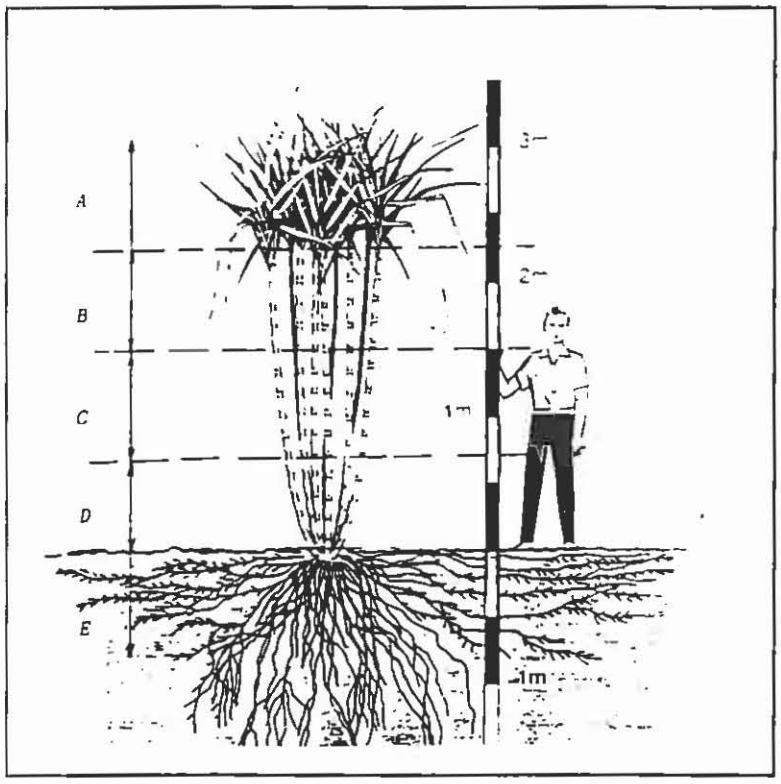


FIGURE 6 : Cane Plant Schematic
A. Tops, B. 1/3 Top, C. 1/3 Middle, D. 1/3 Bottom, E. Roots and Soil.
SUGAR INDUSTRY INFORMATION SERVICE (1989)

Once sectioned, each portion collected was processed via standard mill wet disintegrator method (see Appendix b) followed by washing to remove excess sucrose prior hygroscopic water analysis.
(Note : Tops, trash and root material were processed via Jeffco cutter-grinder prior wet disintegrator processing).

Pith and Long Fibre

In a mechanically processed cane sample, as produced via Jeffco cutter-grinder or wet disintegrator preparation, the pith and long fibre components are separable making the investigation of each component possible.
As the relative proportions of pith and long fibre may vary in certain portions of a growing sugar cane plant, this section of the report aimed to identify whether these two distinguishable components have different affinities for hygroscopic moisture.

Pith and long fibre components used for the analysis were separated via shaking the mixed disintegrated sample in a 600 micron sieve for a period of 20 minutes. All material retained by the sieve was analysed as "long fibre", while material smaller than 600 microns was analysed as "pith".

Variation between Cane Varieties

Variation between sugar cane varieties may be seen in a multitude of consistuents ranging from % sucrose to ppm levels of silica, and of characteristics ranging from leaf tensile strength to pest immunity.

The fact that no two varieties of cane are identical in all aspects led this project to investigate % hygroscopic water as a function cane variety.

During November 1989, cane was sampled at Victoria, Pioneer and Kalamia mills and processed for subsequent analysis via the dry fibre contact method (as per section 3.0 of this report), samples being identified in terms of crop region, mill, sample type, mill number, ratoon number, farm number and farm owner for the 1989 season. (see Appendix G for Ingham Cane land area).

Variation with Crop Location

Two cane varieties, namely Q96 and Q117, representing approximately 80% of the cane milled in the Burdekin region, were collected from both Pioneer and Kalamia mills in order to assess whether crop location for a particular cane variety will alter its hygroscopic water capacity.
In addition, similar cane varieties sampled from the Herbert region at Victoria mill were analysed for hygroscopic water, giving a broad crop area to investigate.

Variation in Soil Samples

Soils contain a broad variety of constituents which contribute to the varying properties of different soils. Sandy soils may be characterised by their relatively high silica (as SiO_2) content in comparison to certain clay soils that exhibit low silica levels.

In terms of hygroscopic moisture, the quantity held by a particular soil will vary approximately with the colloidal content of the soil and the nature of that colloidal material present. For example, some sandy soils low in organic matter content may contain a very small percentage of colloids leading to low hygroscopic water capacity, whereas some clay soils containing large quantities of this constituent may return higher hygroscopic water readings.

Water held in this condition does not move in the soil, is not used by plants, and may even exist in other than the liquid state.

Any water held in excess of the hygroscopic moisture is defined as capillary moisture, this being susceptible to movement in the soil, may be adsorbed by plant roots, and exists in the liquid state.

In order to analyse soil samples for hygroscopic water, some modifications to the methodology applied to fibre samples were required as initial values calculated fluctuated greatly. The following represents alterations made to the method employed for fibre samples.

Alterations

a. Mass of adsorbate (soil) = 16 grams, twice the amount used for fibre.

b. Mass of sucrose solution = 100 grams not 150 grams.

c. Filtration of samples = Celite + Whatman 91 initially, followed by fine filtration via Millipore 0.8µm filter in order to obtain a clear, colourless solution.

Soxhlet Extraction

The Soxhlet extraction system (see Figure 7) provides continuous washing and removal of soluble substances combined with insoluble solids, allowing a quantitative analysis of the soluble portion present in the total mass.

In the analysis of cane for soluble substances, a very fine preparation was required to ensure all solubles are removed from the cane in a reasonable length of time. Of the various food/material processing tools available on a small

scale basis, the Tecator 1094 homogeniser (see Figure 8) proved to be most satisfactory for the processing required.

Having sampled the same variety of cane in both Tecator processed and billet form, a comparative trial was conducted to evaluate the effectiveness of Soxhlet extraction versus conventional disintegration techniques for the analysis of Brix in cane. The following method was developed for use in all extractions performed.

1. Into a preweighed soxhlet extraction thimble (Whatman - cellulose 30mm x 100mm) add Tecator processed cane, slightly tapping the thimble to ensure efficient packing. Determine by difference the mass of cane used (approx 30 g) and cover the thimble opening with glass wool.

2. Into the preweighed solvent reservoir (containing glass beads), add approximately 400 g distilled water.

3. Assemble the extraction system as per Figure 7, using the following settings -

   a. Vacuum - 25" (625mm)

   b. Temperature - controlled to 70 ± 1 °C

   c. Condensor - cold tap water

4. Allow the process to extract for 4 hours from the first full extraction, after which time cool and reweigh the solvent reservoir containing the extracted solubles/solvent mixture.

5. Determine the % solids (Brix) of the reservoir solution via Dipping refractometer

Calculation

The Brix % cane was calculated using the following formula -

$$Brix\ \%\ Cane = \frac{BE \times \frac{M_E}{100}}{M_C} \times 100$$

where

BE = Brix of extract

M_E = Mass of extract (g)

M_C = Original mass of cane

Methods of Analysis \ Sample Preparation

Wet Disintegrator - Small scale

For comparative purposes, a small scale, bench-top wet disintegrator was setup, consisting of a suitably sized disintegrating vessel and a high speed Ultra Turrax homogeniser.

Samples of sliced cane were disintegrated via conventional food processor followed by wet disintegration with distilled water for 4 one minute periods. Tests revealed that a disintegration time of 4 minutes extracted the maximum quantity of solubles in the processed cane.

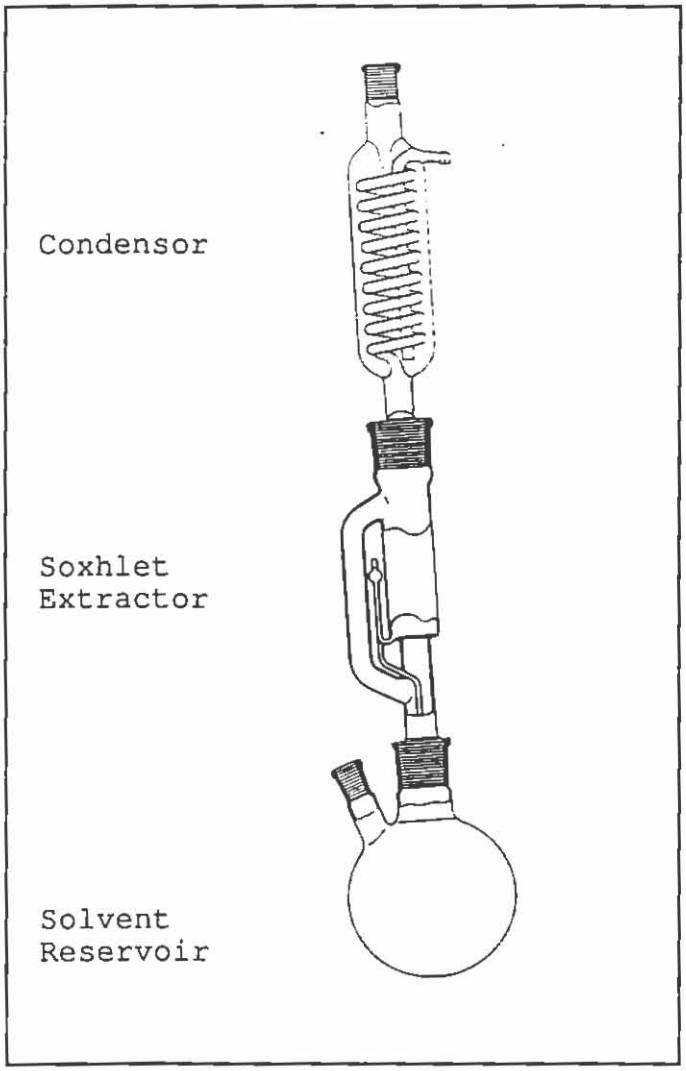


FIGURE 7 : Soxhlet Extraction System

Tecator 1094 Homogeniser

The Tecator 1094 Homogeniser, designed for macerating a variety of high-moisture, high-fat and fibrous samples, consists of a stainless steel bowl into which sits angled, steel blades (see Figure 8).

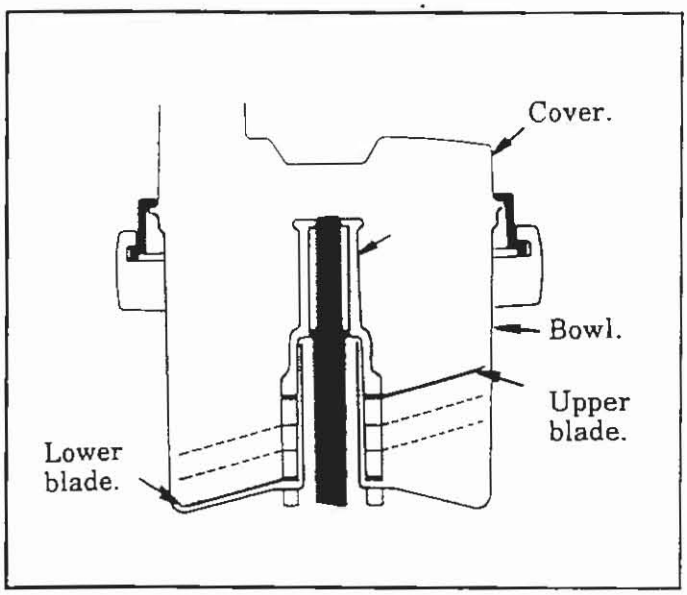


FIGURE 8 : Tecator Homogeniser 1094 - Schematic

In order to prepare sugar cane to the required consistency, the following approach was adopted, samples being prepared by staff at BSES, Meringa.

Containers of Jeffco cutter-grinder material were frozen at approximately -18°C immediately after preparation. Several days later, each frozen portion (approximately 200 grams) was quartered and placed in the homogeniser. Solid CO_2 was added via the nozzle for 5 seconds before the motor was started. CO_2 addition continued for 20 seconds, the homogeniser running for a total of 1 minute. Following preparation, the cane samples had a consistency comparable to fine, moist bread crumbs.

C. Experimental Equipment and Instrumentation

Dipping Refractometer

The Dipping Refractometer consists essentially of a measuring prism (1, see Figure 9), compensator (2), objective (3), compensator adjusting ring (4), scale plate (5), vernier scale for 1/10th scale readings (6) and eyepiece (7).

In order to achieve the maximum accuracy, careful temperature control of the measuring prism was essential, with measurements being made by observing the boundary line position of the incident beam on a scale reading from 5 to 105 units when viewed through the eyepiece.

Prism adjustments required in order to obtain repeatable, reproducible results were performed as per instruction manual (Zeiss).

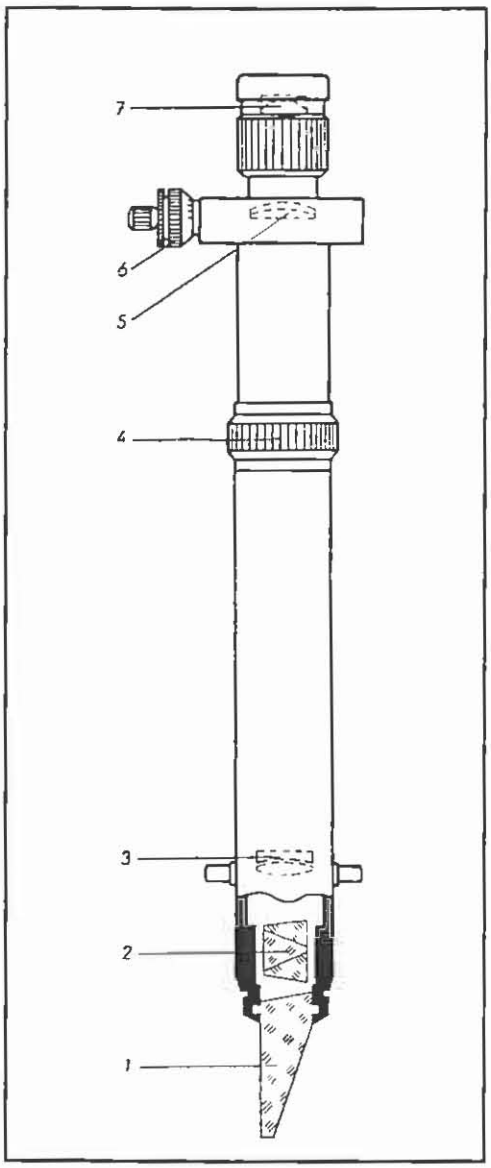


FIGURE 9 : Dipping Refractometer Schematic (Zeiss)

Measurements and Calculations

Refractive index measurements made via the Dipping Refractometer involved placing several drops of the liquid in question on the prism and recording three consecutive refractometer scale readings.
Once recorded, the average scale reading was converted to refractive index as per instruction manual (Zeiss).

For example water readings were calculated as follows :-

Scale Readings = 85.51, .50, .48

Average = 85.50

In Tables (Zeiss) -> 85 = 1.33320 RI

$$.50 = \frac{0.50 \times 39}{1 \times 10^5} = 1.95 \times 10^{-4}\ RI$$

Therefore Sample RI = $1.33320 - (1.95 \times 10^{-4})$

$= 1.333005$

All Refractive Indices were then converted to Brix using the Chebyshev series (Rosenbruch, 1982), see Appendix D.

From the Chebyshev relationship :-

1.333005 RI $= 0.0126°$Bx

a value which was then subtracted from each sample Brix in order to obtain true Brix for each solution.

Differential Refractometer

The Waters Associates R-401 refractometer used for the investigation utilises the conventional differential technique which measures the deflection of a light beam as it passes through a partition at an angle to the source.

Since the deflection phenomena takes place at the surface of a 10µL cell partition, detection of any changes that occur in small volumes is possible.

Quoted (Differential Refractometer Manual, 1976) specifications for the R-401 refractometer are as follows :-

Maximum Sensitivity (¼x) = 6×10^{-6} RI Full Scale (F.S.)

Minimum Sensitivity (128x) = 3×10^{-3} RI F.S.

Noise Level = Less than ± 2% F.S.

Ambient temperature change of 10°F produces less than a 4×10^{-6} RI change.

NOTE : Refractometer unit was temperature controlled to 20 ± 0.1°C during all analyses.

Measurements and Calculations

In order to determine Brix differences via differential refractometry, a relationship had to be established between voltage output and Brix difference between sample and reference cells.

Calibration Graph

A calibration graph plotting voltage output versus Brix difference was constructed using standard sucrose solutions ranging from 0.01 to 0.03 Brix in 0.01°Bx gradations (see Appendix E for standard graph data collected over the course of the investigation).
Therefore, any Brix difference equivalent to ± 0.03°Bx was converted from voltage to Brix via the calibration graph.

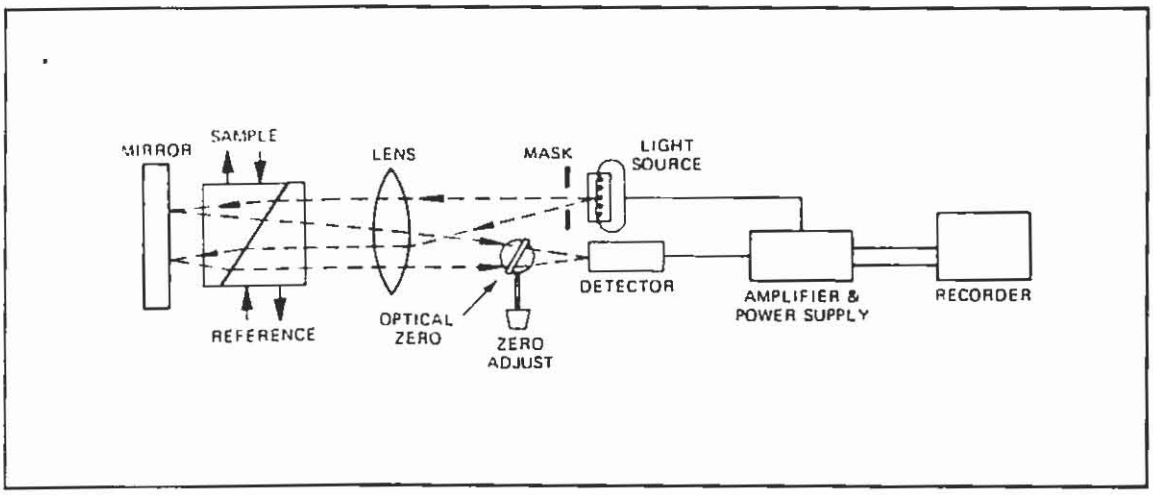


FIGURE 10 : Differential Refractometer Schematic

Comparison Standards

In order to obtain Brix differences between reference and sample cells in the differential refractometer within the ± 0.03°Bx range, comparison standards matching unknown solution Brix were prepared for use as reference solutions.
Having prepared the comparison solution, both reference and sample cells of the differential refractometer were filled with the solution, at which time the Brix difference is zero (i.e. voltage reading adjusted via optical zero to read 0 volts).
Once the unit had stabilised thermally after introducing the comparison solution, the sample cell of the instrument was filled with the unknown solution.

Any difference in Brix between the standard and unknown solutions was then reflected as either a positive or negative voltage, this voltage being converted to Brix via the calibration graph produced above.

D. Results

Wet Disintegrator

Having performed disintegrations involving multiple dilutions (in some cases up to five dilutions), factors associated with the system analysed gave rise to a broad range data points (see Table 2, Figure 11), producing even some negative values for hygroscopic water in fibre. As can be seen from Figure 11, most values for unbound water were positive, in the 50 to 100% region .

In order to obtain a value for % hygroscopic water on fibre in the 20 to 30% region, unbound water must lie in the 63 to 69% range when using values for total moisture and fibre in cane of 69-71% and 10-12% respectively. Of the data generated, only 20% of unbound water values fell in this 63-69% region (see bold type figures in Table 2), which indicated the unsuitability of the wet disintegrator multiple dilution technique, in its present form, for the determination of hygroscopic water in cane fibre.

TABLE 2
Wet Disintegrator Results
% Unbound Water

Calculation Method		
Run	Point by Point	Regression
1	92.0	71.4
2	**69.4**	**69.5**
3	102.6	102.6
4	87.9	86.9
5	73.3	79.3
6	**68.2**	33.0
7	112.7	253.7
8	75.0	73.8
9	-32.9	-4.1
10	**69.4**	77.9
11	88.9	93.2
12	76.8	91.5
13	70.1	**67.8**
14	78.4	92.5
15	**68.1**	73.3

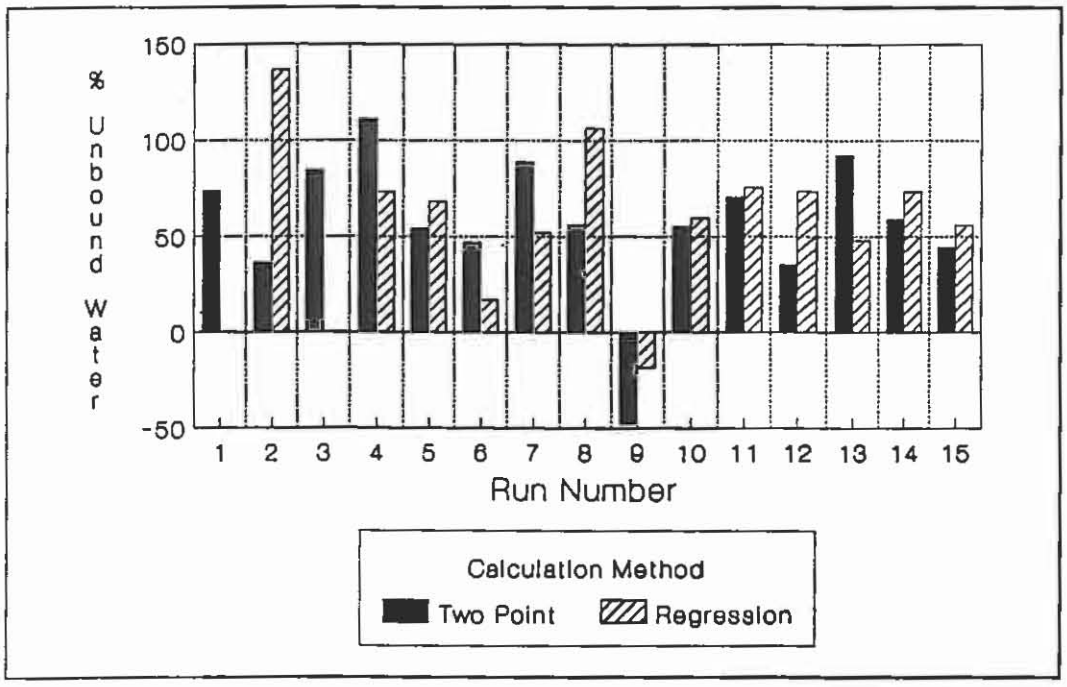


FIGURE 11 : Wet Disintegrator Unbound Water Results calculated two ways.

Factors held constant during disintegrations included :-

i) disintegration time of 45 minutes, with additional water being mixed into the system for 2 minute periods,

ii) initial disintegration constituents of 6kg distilled water combined with 2kg of hammer milled cane,

iii) filtration of sample prior analysis, consisting of 2, 0.8 and 0.45µ Millipore disposable filter membranes connected in series, directly to the disintegration vessel for sampling purposes (see Figure 4 - wet disintegrator schematic).

One area of concern investigated in previous wet disintegrator determinations (CSR Mill Chemical Dept, 1971), involved cooling the contents of the wet disintegrator vessel in order to avoid attaining slurry temperatures in the region of 50 to 60 degrees centigrade, a possible cause for loss of water by evaporation.

In an attempt to reproduce wet disintegrator conditions of past trials, a copper cooling coil was attached to the outside of the disintegrator vessel, through which cooled water (ca. 5 degrees centigrade) was circulated (refer to Table 2, bold

type run numbers indicate use of cooling system). As with previous determinations, this approach did not improve results even though slurry temperatures were approximately halved (temperatures ranged from 25 - 37°C), indicating that any possible water loss via evaporation had no significant or measurable effect on wet disintegrator hygroscopic water results.
Consequently, a method of contacting fibre with sucrose solutions of known concentrations, as used by Steuerwald (1912), was adopted in an attempt to obtain data for hygroscopic water in cane fibre.

Fibre Contact - Method Development

Prior to obtaining a suitable reproducible method for the investigation, % hygroscopic water values varied considerably with results being mainly positive and in the vicinity of 40-50% (see Table 3, results reported in order of development). Having analysed primarily wet fibre at the outset of the project with poor reproducibility, dry fibre contacting was adopted.

The major problem with both wet and dry fibre was in obtaining a sample containing little sucrose, for which a value of 5ppm via thymol or less in fibre washings was deemed acceptable, with the possibility that hydrolysis of fibre compounds during sample preparation may have produced organic species contributing to a positive thymol test. Once the task of cleaning fibre to such low levels of sucrose had been achieved (successive hot and cold water washes followed by centrifuging to remove excess water) results for hygroscopic water became somewhat reproducible (see Table 3, dry fibre column).

One point not investigated in previous hygroscopic water determinations of this nature was the effect of evaporation on the fibre/sucrose slurry, and consequently on the % hygroscopic water on fibre. In order to counteract any Brix increase eventuating from evaporation, a sucrose solution containing no fibre was used for each analysis (i.e. control), with any deviation in Brix observed for the control being corrected for in the sample concentrations during the analysis.

This proved successful as in some cases, depending on ambient conditions, Brix increases due to evaporation reached 0.01-0.02°Bx which, if uncorrected, would have led to an overestimation of hygroscopic water in the order of 1.9-3.7% on fibre.

In addition to using control sucrose solutions to correct for Brix increases due to evaporation losses, fibre/water mixtures were also analysed to correct for any sucrose still present after the washing stage.

Having developed a method for determining % hygroscopic water in fibre with some degree of reproducibility, the method was used throughout the entire project.

TABLE 3
Method Development
% Hygroscopic Water

Method of Analysis	
Wet Fibre Analysis	Dry Fibre Analysis
33.4	2.6
36.7	6.6
17.6	35.3
48.8	37.1
143.2	43.9
63.9	17.5
150.4	29.3
70.0	24.3
19.0	24.4
-23.3	24.5
-2.3	16.6
351.8	24.0

Determination of Equilibration Time - Fibre Contact Method

Referring to Figure 12, a duplicate analysis which monitored Brix over the first 60 minutes of the analysis revealed that 30 minutes after the fibre and sucrose solution had come into contact, no further increase in sucrose concentration was observed. Therefore for all following analyses, an equilibration time of one and a half hours was chosen, a time which was well past the point at which the system attains equilibrium.

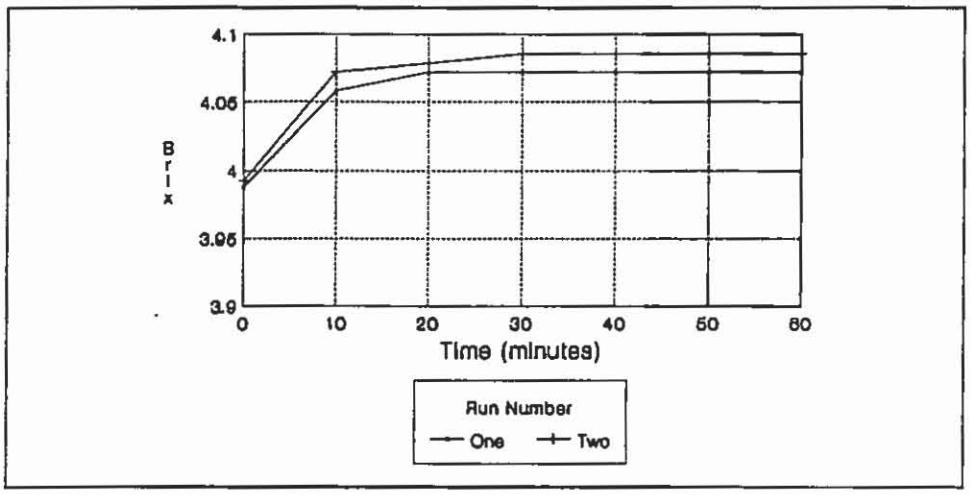


FIGURE 12: Monitoring Brix changes in a fibre/sucrose solution slurry as a function of time.

In addition to determining this equilibrium time, the following section of this report deals with extending the equilibration time to 24 hours in order to investigate the effect upon the final result for hygroscopic water.

Extended Equilibration Times - Dry Fibre Contact Method

A series of trials using Jeffco cutter-grinder fibre in conjunction with 10°Bx sucrose solutions (see Table 4) indicated that the 24 hour equilibration time gave higher results in the order of 1 to 4 units for % hygroscopic water on fibre.
Statistically (95% CI, t = 4.44), a significant difference existed between results obtained over the 1.5 and 24 hour equilibration periods, contradicting results reported in previous determinations (Harris, 1962), in which hygroscopic water results determined over 1 and 24 hour periods were reported as having no significant difference.

This result indicates that the longer equilibration time allows a greater number of water molecules to leave the bulk solution and locate vacant hydroxyl groups in the sorbing fibre. Hence, owing to the physical nature of Jeffco-cutter or similar material, the water sorbing capacity over a short equilibration time (i.e. 1.5 hours) would tend to be lower (1-4%) through the inhibiting effect of that fibre, in comparison to results obtained over longer equilibration periods.

TABLE 4
Extended Equilibrium Periods
% Hygroscopic Water

Equilibration Period	
1.5 Hours	24 Hours
23.6	24.3
23.7	24.4
28.9	32.8
28.4	30.8
20.9	23.4
20.2	23.4
22.5	22.9
22.7	23.4
Mean 23.9	25.7
StdDev 3.2	3.8

Comparison Between 4°Bx and 10°Bx Slurry Concentrations

Table 5 indicates the reproducibility obtained by using the previously examined method which employed 4-5°Bx solutions, in comparison to the current method incorporating a higher Brix solution for the analysis.
A possible cause for the smaller deviation in results using 10°Bx (% C.V. = 5.9) rather than 4°Bx solutions (% C.V. = 15.8) may have resulted from the fact that absolute errors in Brix measurement, sometimes in the order of 0.01 Brix, represent a smaller relative error for the 10°Bx solutions.

i.e. 0.01 Brix error in 10°Bx = 0.1% error
where
0.01 Brix error in 4°Bx = 0.25% error.

Hence, having reduced the error in reading solution Brix, the final result for % hygroscopic water would be closer to the true value.
For all hygroscopic water analyses, 10 Brix solutions were adopted in order to gain some advantage over previous determinations in which 4-5 Brix solutions had been utilised.

TABLE 5
Varying Sucrose Concentration
% Hygroscopic Water

Sucrose Concentration	
4°Bx	10°Bx
24.9	23.0
23.8	23.3
20.3	24.8
23.8	24.5
22.9	24.9
29.8	27.5
21.8	27.4
19.3	24.7
27.6	24.0
28.5	23.6
29.8	25.6
31.8	25.5
Mean 25.7	25.4
% C.V. 15.8	5.9

Varying Cane Sample Preparation

On contacting fibres prepared as previously mentioned, results (see Table 6) reflected the similarity of fibre behaviour for both wet disintegrator and Jeffco cutter-grinder material, and that machine ground fibre indicated a greater ability to adsorb water in producing a higher result for hygroscopic water.

This increased adsorptivity of machine ground fibre may have occurred as a result of the ease with which water molecules locate vacant hydroxyl groups on the dry fibre surface. In the case of wet disintegrator and Jeffco cutter material, the mere nature of the fibre, having a proportion of vascular bundles still unbroken after processing, could have possibly hindered the migration of water molecules to the fibres internal surface over the equilibration time chosen for the analysis.

On inspecting the three fibre preparations, it appeared that machine ground, owing to its fine consistency, would yield the highest surface area per unit mass, suggesting it should yield higher results for hygroscopic water than the other preparations examined.

A statistical (95% CI) comparison of hygroscopic water results obtained for the three sample preparations revealed the following :-

1. W.D. vs Machine : t = 5.9, significant,
2. W.D. vs Jeffco : t = 4.5, significant,
3. Jeffco vs Machine : t = 1.6, insignificant.

In an attempt to explain the occurrence of the varied results, one section of this report investigated equilibration times of 1.5 hour and 24 hour periods using Jeffco cutter-grinder fibre.
Jeffco cutter-grinder material, being the coarsest of the three materials produced, was chosen in an attempt to highlight the effect of partially unbroken fibre cells\vascular bundles on hygroscopic water via inhibition. If any additional moisture was to be taken up by a fibre sample over a longer equilibration period, Jeffco cutter material would indicate that, owing to the proportion of unbroken cells contained in such a sample.

Referring to results derived from the "**Extended Equilibration Time**" section , it was found that a significant difference existed between results obtained over 1.5 and 24 hour equilibration times, indicating that additional moisture was adsorbed onto the fibre over a longer period of time. This result supported the concept that the physical nature of the fibre analysed will effect the result obtained for hygroscopic water, and that various fibre preparations analysed under similar conditions would yield different results for hygroscopic water depending on the availability of the water sorbing components in that fibre.

TABLE 6
Varying Sample Preparation
% Hygroscopic Water

Method of Sample Preparation		
Wet Disintegrator	Jeffco Cutter-Grinder	Machine Ground
31.3	26.1	37.4
30.4	26.2	36.9
29.2	23.8	37.8
26.5	22.7	37.3
24.7	27.6	28.2
23.6	26.9	28.1
22.8	26.4	24.6
22.9	24.0	25.8
24.2	-	28.5
23.9	-	28.7
22.4	-	23.4
21.8	-	24.7
Mean 25.3	25.5	30.1
StdDev 3.3	1.7	5.6

Varying Equilibration Temperature - Ambient vs 50°C

Having analysed two varieties of wet disintegrator ground material at both ambient and approximately 50°C (actual temperature range was 49.8 - 52.5°C), results (see Table 7) indicated that no major difference existed between hygroscopic water determined at varying equilibration temperatures.

Statistically (95% CI, t = 1.0), no significant difference existed between results obtained at the two equilibration temperatures, indicating that hygroscopic water values determined under ambient conditions were of similar magnitude to those present during wet disintegrator analyses.

In an investigation by Stamm and Harris (1953), desorption isotherms for Sitka spruce at varying temperatures (Figure 13) indicated that the hygroscopicity of such cellulose\lignin materials decreased at higher temperatures, a phenomena which was only observed for one of the two cane varieties analysed in this experiment (see Table 7, sample Q115).

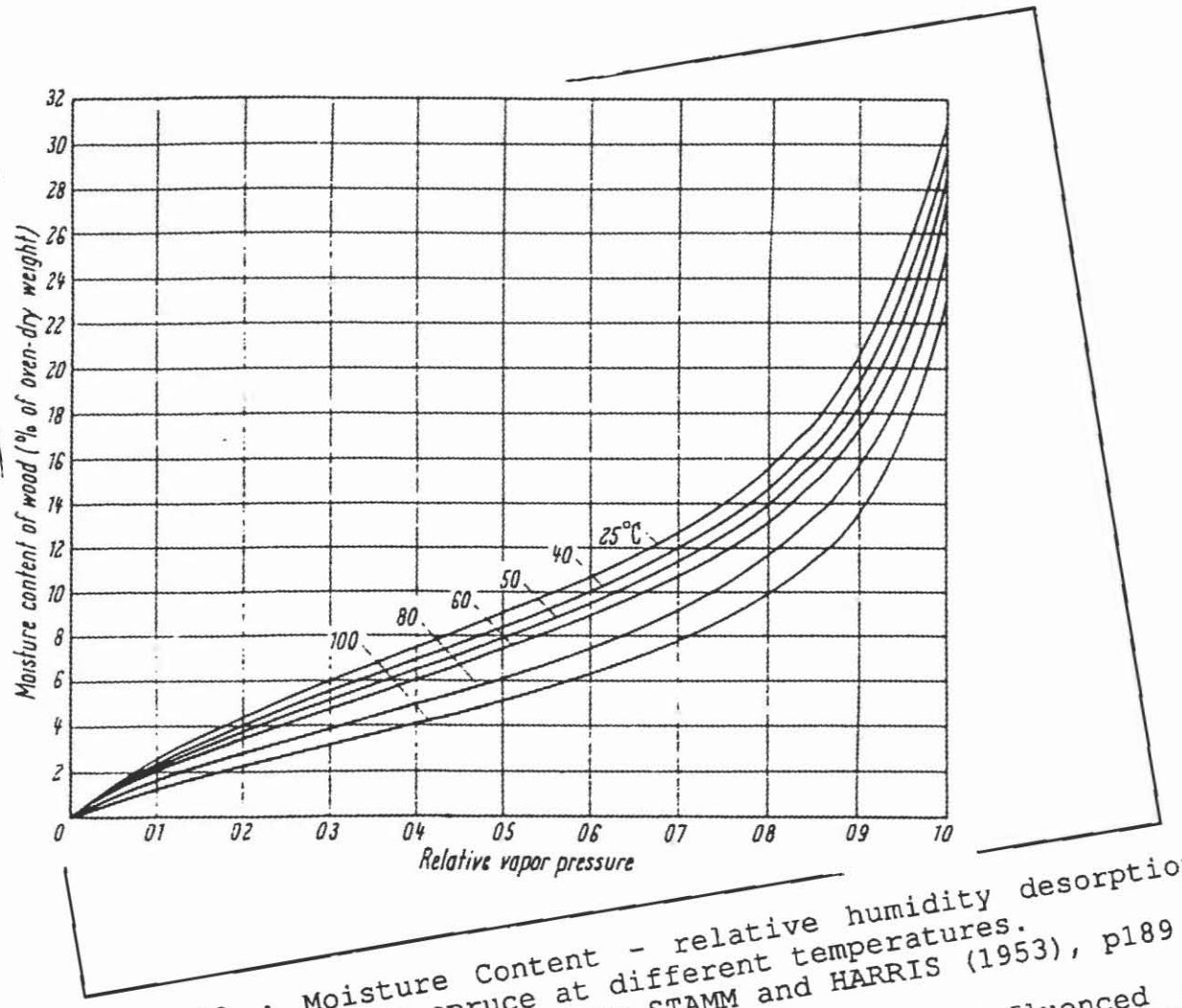


FIGURE 13 : Moisture Content - relative humidity desorption isotherms for Sitka spruce at different temperatures.
COTE and KOLLMANN (1968) from STAMM and HARRIS (1953), p189

However, the degree to which adsorption is influenced at elevated temperatures for cane fibre has not been fully investigated, and its significance or its magnitude may not have been measurable using the current methodology.

TABLE 7
Varying Equilibration Temperature
% Hygroscopic Water

Sample Type	Equilibration Temperature	
	Ambient	50°C
Q115	19.9 17.1 21.4 22.4	16.2 13.8 20.6 20.4
Mean	20.2	17.8
Triton	20.1 21.2 18.5 18.3 15.7 14.4	20.9 22.4 26.7 22.1 18.4 19.4
Mean	18.0	21.7

Variation of Hygroscopic Water within a Plant

Each of the varieties analysed exhibited similar results for hygroscopic water in relative portions of the cane plant. Results (see Table 8) indicated that for both varieties, tops yielded the highest results (22-24%) for hygroscopic water whilst root material yielded the lowest (5-8%).
In terms of stalk and trash material, no major differences were observed for hygroscopic water measurements, these being 4-5% lower than those obtained for tops material.

Results therefore signified the changing composition and varying affinity for hygroscopic water throughout the plant, which reflected to some extent, the varying proportion of water sorbing cellulose and lignin materials in the growing plant.
Deerr (1921) reported cellulose, pentosan and ligneous body levels in selected portions of sugar cane (see Table 9), which were converted to a % of total cane basis. These values indicated that leaf and root material should basically retain similar quantities of hygroscopic water, and that stalk material should retain less than either root or leaf material, owing to the relative amounts of sorbing material in each.

TABLE 8
Variation Within a Cane Plant
% Hygroscopic Water

Cane Portion	Cane Variety	
	Q115	Triton
Tops	23.5	24.1
1/3 Top	19.2	22.9
1/3 Middle	18.9	16.9
1/3 Bottom	20.3	19.6
Roots	6.7	4.5
Trash	19.7	20.9

In terms of the cellulose and pentosan levels reported by Deerr (1921), the observed difference between stalk and leaf hygroscopic water results verified the varied compisition of each portion. However, root material yielded much lower results than would be expected if Deerr's information is taken into consideration.

TABLE 9
Composition Of Fibre in Different parts of Cane

Constituent in Fibre	% Constituent in Portion		
	Stalk fibre	Leaf fibre	Root fibre
Cellulose	48.4	48.8	45.9
Pentosans	30.3	29.2	33.7
Ligneous Material	21.3	22.0	20.4

From DEERR (1921), p12.

Two possible causes for this are given below :-

a. The relative strength of root material would be greater than that of leaf or stalk material. Hence during mechanical processing as in wet disintegrations, root material would be ground to a lesser degree (as was observed), the product having a lower surface area per

unit mass and expose less internal surface than that of leaf or stalk material.
Thus, the reduced availability of water sorbing constituents (i.e. lignin, cellulose) in processed root material would produce lower results for hygroscopic water, as results have indicated.

b. As quantities of soil were present in the root material after processing, its presence will tend to lower hygroscopic water results, as results in this report indicate that soil retains much less hygroscopic/bound water (4-5%) than any other material present in the cane plant (18-24%).

Hygroscopic water content of Pith and Fibre in Cane

Of the pith and long fibre samples analysed for hygroscopic water, results (Table 10) indicated pith to have a greater affinity for water than the long fibre material by yielding higher values for hygroscopic water.

Reasoning for this result came from the apparent nature of each component separated from the original wet disintegrator samples, with pith material being a much finer material and would tend to have a greater surface area per unit mass than the long fibre component. The increased surface area would thus tend to produce higher results for hygroscopic water, as was observed in the experiment.

The fact that pith and long fibre portions of sugar cane adsorb different amounts of moisture per unit mass, gave some explaination as to why hygroscopic water results for mixed pith/long fibre samples varied considerably in the order of 4-6 units for hygroscopic water, even though sampling techniques employed were considered adequate for the analysis.

Meade and Chen (1977) gave some indication of the cellulose and lignin fractions in cane fibre, expressing that pith and long fibre contain approximately equal portions of these water sorbing compounds (see Table 11).
Hence, it appeared that pith and long fibre should yield similar results for hygroscopic water owing to their relative cellulose and lignin levels, however, the adsorbtivity of each portion was again affected by its physical structure, resulting in lower hygroscopic water results for the fibre material.

Variation of Hygroscopic Water with Cane Variety

Cane varieties (80% there of) grown in the Herbert region yielded hygroscopic water results in the 17-20% region (see Table 13), with the majority of results closer to the higher end of this range.
Variability in results for cane samples fluctuated by as much as 10 units (min to max), with this variation seen to be arising from several factors including :-

1. Possible variations in pith:fibre ratios in the individual samples analysed,

2. Errors involved with the methodology, from weighing errors to Brix measurement errors.

Overall, standard deviations obtained for each variety were of the order of 2-3 units in % hygroscopic water, an improvement on previous determinations of this nature.

TABLE 10
Pith and Fibre in Cane
% Hygroscopic Water

Cane Variety	Pith	Fibre
Q117	12.8	11.0
	15.6	10.3
	17.3	11.1
	-	9.8
	16.9	9.9
	19.9	10.1
	17.1	13.5
	17.5	11.0
Mean	16.7	10.8
Q96	23.3	19.3
	26.4	19.4
	24.4	18.4
	22.1	19.7
	23.2	19.5
	22.6	20.1
	23.5	18.2
	20.2	17.6
Mean	23.2	19.0

TABLE 11
Cane Fibre Characteristics

Constituent in Plant	Plant Component	
	Pith	Fibre
Lignin (%)	18-22	20-23
Cellulose (%)	26-36	38-43
Length (mm)	0.25-0.40	1.0-2.0

Data from MEADE and CHEN (1977), p105.

TABLE 12
Sample Identification Data

Sample Type	Farm #	Mill #	Ratoon #	Farm Owner
Herbert Region - Victoria Mill				
Q115	F215	422B	3RG	Bosworth
Q119	F663	450A	3RG	Pennisi
Q117	F162	588B	2RG	Board
Cassius	F533	412B	10RG	Gollogly
Triton	F53	418B	10RG	Balanzategui
Q96	F162	450B	3RG	Board
Burdekin Region				
Kalamia Mill				
Q96	F69B	n/a	3RG	n/a
Q117	F195	n/a	Plant	n/a
Pioneer Mill				
Q96	n/a	n/a	n/a	n/a
Q117	n/a	n/a	n/a	n/a

TABLE 13
Variation of % Hygroscopic Water
With Cane Variety

Mill	Variety	Mean	n	Standard Deviation
Herbert Region Victoria Mill	Q115	17.0	21	2.6
	Triton	20.3	20	2.9
	Cassius	18.9	19	2.3
	Q117	17.8	20	1.6
	Q96	19.8	20	1.8
	Q119	19.8	19	2.5
Burdekin Region Pioneer Mill	Q96	19.5	20	1.2
	Q117	18.8	20	1.3
Burdekin Region Kalamia Mill	Q96	17.6	20	1.2
	Q117	19.7	16	1.5

The following table represents statistical data for varieties Q96 and Q117 grown in 3 locations.

TABLE 14
Statistical Data for
Varieties grown in different regions

Variety & Mill Area	n	Student t Randomly paired data
Q96		
Pioneer v Kalamia	20	6.84*
Pioneer v Victoria	20	0.26
Kalamia v Victoria	20	4.44*
Q117		
Pioneer v Kalamia	16	1.76
Pioneer v Victoria	20	2.14*
Kalamia v Victoria	16	0.97

* - Statistically significant at the 95% CI.

Variation of Hygroscopic water with Soil

From farms producing the six varieties of cane fibre analysed in this report, soil samples were collected in order to assess their hygroscopic water capacity (see Table 15 for sample details).

Of the soils analysed, results showed that soil from farm 162, on which cane variety Q96 was grown, yielded hygroscopic water results twice as great as all other soils tested.

A test gauging particle size distribution for each soil revealed that soil collected from farm 162 contained a greater proportion of smaller sized (colloidal) particles than all other soils tested (see Table 16). Hence, owing to the physical size of particles present in the analysed sample, it would be expected that smaller particle size leads to increased surface area, thus increasing the water sorbing capability of that soil.

TABLE 15
Hygroscopic Water Results
Herbert Region Soils

Run N°	Variety Grown on Soil				
	Cassius	Q119	Triton	Q115	Q96
Farm	533	663	63	215	162
1	5.7	4.5	4.1	4.9	8.3
	5.2	4.4	3.9	4.5	8.4
2	6.6	4.9	3.3	4.6	9.6
	5.7	5.4	3.3	4.4	9.6
3	5.2	5.7	3.4	4.4	9.2
	4.9	4.9	4.8	4.0	9.0
4	3.9	3.0	1.1	2.1	6.8
	4.1	2.0	1.3	2.2	6.4
5	4.1	2.6	2.6	3.1	7.6
	4.2	3.6	2.8	3.1	7.0
Mean	5.0	4.2	3.1	3.7	8.2
Std Dev	0.9	1.4	1.2	1.0	1.2

Table 16
Statistical Data - Soil particle size analysis

Statistical Data	Q119	Q115	Q96	Triton	Cassius
% < 1.6μ	5.6	5.1	7.5	4.9	5.7
n	15	14	10	15	14
Std Dev	0.4	0.6	0.4	0.4	0.6

The following table describes the moist soil samples for which analytical results have been previously mentioned.

TABLE 17
Characteristics of moist soil samples

Farm Number	Observed colour	Consistency, Texture	Munsell Colour rating and description
533	Dark reddish brown	Medium coarse, sandy clay	2/3* - dusky red
63	Reddish brown	Moderately fine, sandy clay	4/3 - weak red
663	As for 63	As for 63	4/3 - weak red
215	As for 533	As for 533	2/3 - dusky red
162	Dark brown to blackish, finely mottled with dark reddish brown portions	Clayey, fine textured	2/1 - reddish black

* - Munsell Value/Chroma rating - Soil Survey Staff (1951)

Result Comparison - Differential vs Dipping refractometer

A comparison between the two instruments used in the investigation was performed in order to determine whether the application of differential refractometry made any improvement on previous determinations of this nature.

Two areas investigated reveal the greater accuracy obtained for differential refractometer results.

(i) Individual hygroscopic water results for 72 determinations indicated that differential refractometry provides a more accurate reading than those obtained via dipping refractometry, see Table 18. Statistical data (95% CI, t = 18.0) indicates that a significant difference exists between hygroscopic water results obtained for the two instruments, with Dipping refractometer results tending to be 3-5 units higher than Differential refractometer results.

(ii) Refractive indices obtained from the instruments at 4 and 10 Brix (see Table 19). In the measurement of 4 Brix solutions, the Dipping refractometer (% C.V. = 0.84)

yielded a larger spread of results in comparison to the Differential refractometer (% C.V. = 0.77). Similarly at 10 Brix, the Differential refractometer (% C.V. = 0.07) performed better (%C.V. = 0.16).

Consequently, all hygroscopic water and statistical data were generated using Differential refractometer results unless otherwise stated.

TABLE 18
Refractometer statistical data
% Hygroscopic Water results

Instrument	Mean	Std Dev
Differential Refractometer (a)	18.9	1.6
Dipping Refractometer (b)	19.7	3.6
Difference (a-b)	-0.7	4.0

TABLE 19
Refractometer Statistical Data
for Brix readings

Statistical Data	Brix			
	4°Bx		10°Bx	
	Refractometer Type		Refractometer Type	
	Dipping	Differential	Dipping	Differential
Mean	4.02	4.03	10.00	10.01
n	42	42	39	39
Std Dev	0.03	0.03	0.02	0.01
% C.V.	0.84	0.77	0.16	0.07

Polarimeter - comparison to Differential refractometer

As the polarimeter is one of the main instruments used for sucrose measurements accessible to staff at a mill or

refinery, polarimeter readings were used to calculate hygroscopic water results in comparison to differential refractometer results.
In order to investigate the reproducability of the analysis using another method of sucrose measurement, a polarimeter was used to measure sucrose concentration for the hygroscopic water analysis. A Schmidt & Haensch automatic Polatronic-D was used for the evaluation, capable of measuring Pol accurately to ± 0.01°S.

Table 20 reveals results obtained from both polarimeter and differential refractometer readings. Statistically (95% CI, t=1.5), differences between the paired data over various hygroscopic water readings were not significant, indicating the suitability of polarimeter derived results for hygroscopic water determinations. Results also indicate that polarimeter and differential refractometer results tend to follow each other, regardless of the range of hygroscopic water measured, evidence that the method is somewhat reproducible when monitoring sucrose changes in hygroscopic water analyses via polarimetry.

TABLE 20
Refractometer vs Polarimeter Data
% Hygroscopic Water

Instrument Type		
Differential Refractometer (a)	Polarimeter (b)	Difference (a - b)
23.5	19.8	3.7
19.6	17.0	2.6
21.8	26.4	-4.6
19.2	21.8	-2.6
13.9	13.3	0.6
24.2	24.2	0
24.7	22.6	2.1
23.8	15.0	8.8
23.9	14.5	9.4
25.3	20.1	5.2
25.2	21.0	4.2
21.1	21.4	-0.3
21.2	20.0	1.2
Mean 22.1	19.8	1.7
StdDev 3.1	3.7	3.5
% C.V. 13.9	18.8	-

E. Discussion\Conclusion

In order to assess whether the results obtained in this project were comparable to the currently used value of 25% for hygroscopic water, points such as the analysis and chemistry of hygroscopic water in cane fibre were examined.
Theories supporting the possible occurrence of lower than expected hygroscopic water results came from several sources. In an attempt to explain hysteresis loops for hygroscopic isotherms relating to eucalypt cellulose, Urquhart et al (1924, 1929) stated that

"...hydroxyl groups of cellulose and lignin in drying wood are drawn closely together and may satisfy each other instead of binding water molecules. When water is adsorbed some of the hydroxyl groups continue to satisfy each other and therefore are not available for taking up water."

The tendency for hydroxyl groups to satisfy one another may also explain Hermans' (1949) observations (Figure 14), in that dried cellulose is less capable of sorbing moisture than fresh or undried cellulose.

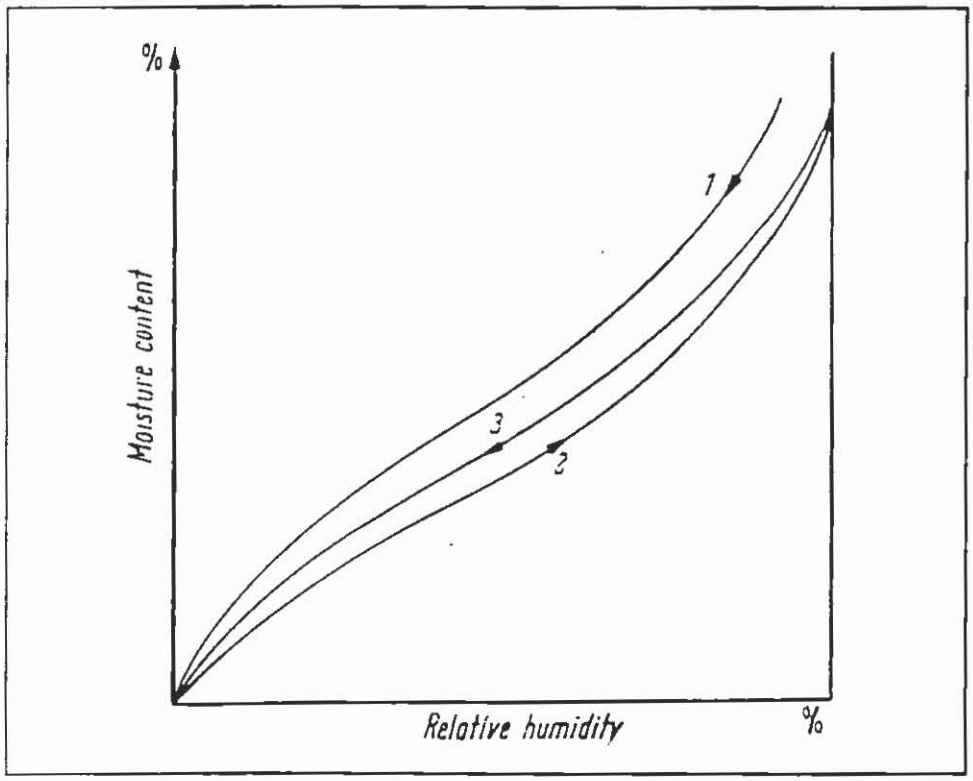


FIGURE 14 : Sorption isotherms of Cellulose objects.
1. Fresh fibre (desorption); 2. Adsorption and desorption after first drying.
COTE and KOLLMANN (1968) from HERMANS (1949), p193

The effect of sample pretreatment on fibres ability to adsorb moisture was also demonstrated by Kollmann and Schneider (1963) in Figure 15. Their work highlighted the effects of heat-treated wood in comparison to untreated wood, indicating that heat-treated samples were less capable of adsorbing moisture. As the above observations indicated the importance of sample treatment prior to analysis, vacuum dried samples (80°C, 625mm Hg) were used for the analysis over the conventional drying method (105-110°C), in an attempt to minimise heat damage of the fibre samples.

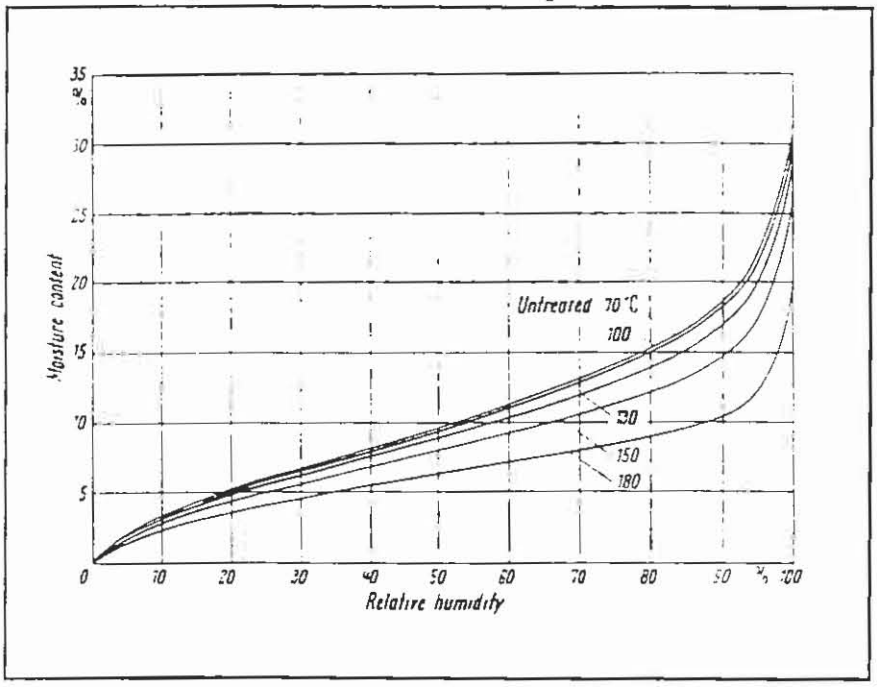


FIGURE 15 : Average adsorption-desorption isotherms for untreated and heat treated wood at 20°C.
COTE and KOLLMANN (1968) from KOLLMANN & SCHNEIDER (1963), p195

Results of the 6 varieties examined showed % hygroscopic water to range between 17 and 20 %, and although falling short of the previously determined figure of 25 %, are considered to be nearer the true value.
As the inclusion of other than cane stalk material can be expected at the shredding stage of the milling process, the contribution to hygroscopic water of foreign material was also considered. Firstly, the inclusion of soil or root material will tend to reduce the total hygroscopic water component of the fibre sample analysed, if present in sufficient

quantities, leading to an overcorrection in hygroscopic water (i.e. an underestimation of the Brix in cane).
Secondly, the presence of tops in the processed sample, displaying a greater affinity for hygroscopic water (23-25%) than stalk material, will yield higher than expected results for the direct cane analysis, overestimating the Brix in cane.

It therefore seemed possible, depending on the composition of the harvested cane, to produce samples of varying hygroscopic water content, leading to erroneous readings for Brix in cane over a period of time if a constant value for hygroscopic water were used.

In terms of cane variety it appeared that for all varieties, regardless of crop location, hygroscopic water was found to be present in similar quantities ranging from 17-20% on fibre. These results were considered to be comparable to the currently used value of 25%, owing to the possibility of reduced adsorptivity for oven dried fibres as discussed above.

Although the use of modern equipment (e.g. Differential refractometer, analytical balances etc..) had reduced the uncertainty of readings taken during the analysis, errors associated with the methods of analysis still made some contribution to the variation in hygroscopic water results obtained.

Wet Disintegrator

From the derivation of the unbound water formula (see equation 4), the following equation was used to calculate the error associated with the system used.
For one additional dilution -

$$Q = \frac{(W_2 - W_1)\,C_2}{C_1 - C_2} - W_1$$

where C_1, C_2 = initial and first dilution slurry concentrations respectively.

W_1, W_2 = inital and first dilution cane:water ratios.

Typically,

C_1 = 5.510 ± 0.001 g/100g

C_2 = 4.843 ± 0.001 g/100g

W_1, W_2 = 300 and 350 respectively

therefore, Q = 63.01 ± 1.16

Using values for total moisture (w), fibre (f) and Bx of cane as 66, 14 and 20 respectively, % hygroscopic water was calculated via equation (8) as 21.4 ± 9.0.

Reducing the error in solution concentration readings to ± 0.0001 g/100g, Q = 63.01 ± 0.12 and % hygroscopic water = 21.4 ± 1.6. As experimental data did not reflect this degree of accuracy, it was concluded that other errors in experimental procedure led to the variation in results.

Dry Fibre contact method

From equation (9) we are given -

$$\%\ Hygroscopic\ Water = W\left(1 - \frac{p^0}{p^1}\right)$$

where

$$W = \frac{weight\ sucrose\ solution}{weight\ of\ fibre} \times 100$$

p^0 = Brix of sucrose solution prior mixing with fibre

p^1 = Brix of sucrose solution after mixing with fibre

At 4°Bx, p^0 = 4.00 ± 0.01°Bx, p^1 = 4.05 ± 0.01°Bx, W = 1875 ± 1 and % hygroscopic water = 23.2 ± 11.2.
At 10°Bx, p^0 = 10.000 ± 0.001°Bx, p^1 = 10.130 ± 0.001°Bx, W = 1875 ± 1 and % hygroscopic water = 24.1 ± 2.3. Hence, having reduced the error in reading solution concentration, % hygroscopic water readings should have become more accurate.

As the fibre contact data did not provide this degree of accuracy, it was concluded that other errors associated with the analysis contributed to the variation in results.

Soxhlet extraction

The comparison of Soxhlet extraction to wet disintegration results for % Brix on cane demonstrated that homogeneity of the analysed sample was of primary importance in order to obtain repeatable, reproducible results.
The fact that both analyses were investigated using smaller than desired sample size (i.e. 30 to 100 g instead of 1 to 2 kg) may have contributed to the variation in results obtained (see Table 21).

TABLE 21
Brix % Cane results
Soxhlet Extractor vs Wet disintegrator

Sample Type	Soxhlet Extraction	Wet Disintegration
Q113	14.2 15.6 14.7 13.2 14.2 14.1 15.8 14.4 14.8	15.4 14.6 15.4 13.3 15.5 13.7 - 13.5 14.6
Mean	14.6	14.6
Standard Deviation	0.7	0.9
Q124	15.6 14.9 14.1 14.8 15.8 14.4 14.6 15.4 14.8 14.9	15.6 15.7 15.7 16.5 16.9 16.5 16.6 16.9 16.8 16.6
Mean	14.9	16.4
Standard Deviation	0.5	0.5

Results of sample Q113 indicated that the soxhlet extraction technique may be used for the determination of solubles in cane, however sample Q124 results did not confirm this due to the higher than expected wet disintegrator results obtained. Previous full scale wet disintegrator analyses were reported as having a maximum accuracy of ± 0.04 Brix % cane for a single determination (RICHARDSON, 1970).
Statistically (95% CI), no significant difference existed between soxhlet extraction and wet disintegrator results for Q113 ($t = -0.6$), while for Q124 ($t = -6.8$), a significant difference existed possibly due to dehydration of the sample during transport. In addition, correlation between extractor and disintegrator results yielded a higher coefficient for Q113 results ($R^2 = 0.83$) than for Q124 results ($R^2 = 0.76$) see Figures 16 and 17. In order to improve the repeatability of the analysis, it is recommended that -

a) larger sample size, representative of the gross sample be analysed (e.g. Kg amounts)

b) prompt analysis of the extract solution in order to maintain solution integrity.

c) increased ratio of cane to solvent in order to obtain a more concentrated extract for final analysis. For results generated in this report, final extract Brix ranged from 1 to 2 °Bx depending on initial weight of cane, stage of extraction.

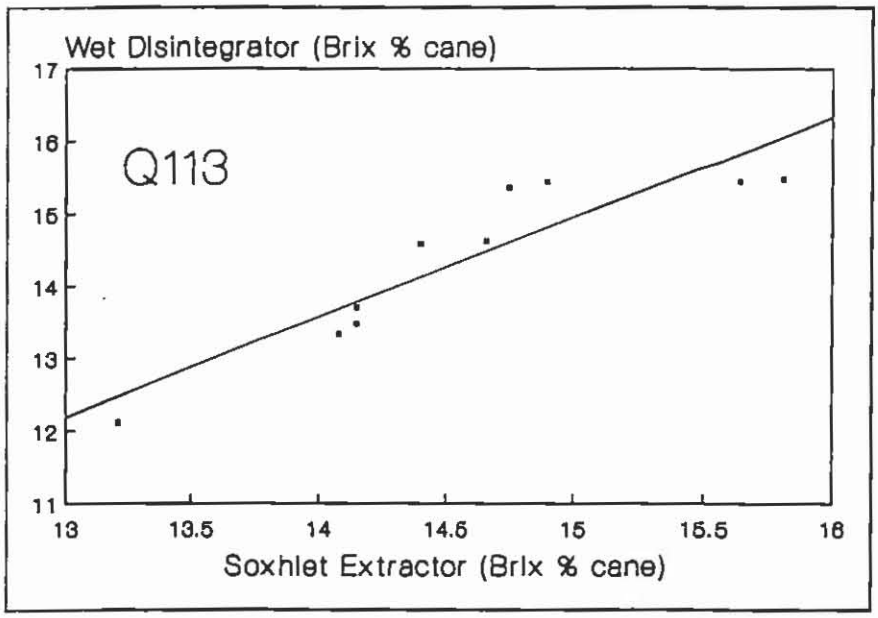


FIGURE 16 : Soxhlet extraction vs wet disintegrator results - Brix % cane for sample Q113

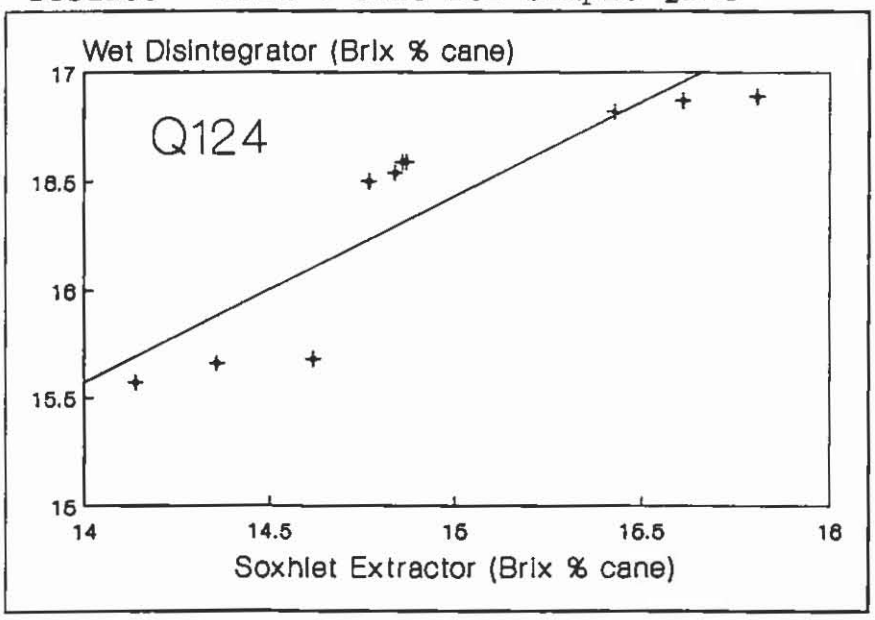


FIGURE 17 : Soxhlet extraction vs wet disintegrator results - Brix % cane for sample Q124

In terms of the quantity of cane present in the extraction thimble during analysis (i.e. density of processed cane), result trends indicated Brix % cane to be proportional to the mass of cane used, signifying that complete extraction was not

achieved over the 4 hour extraction period chosen for the analysis (see Figure 18). As thymol tests were conducted in order to assess the time period required for complete extraction, solubles not accessible via the extraction method must have been retained by the fibre.

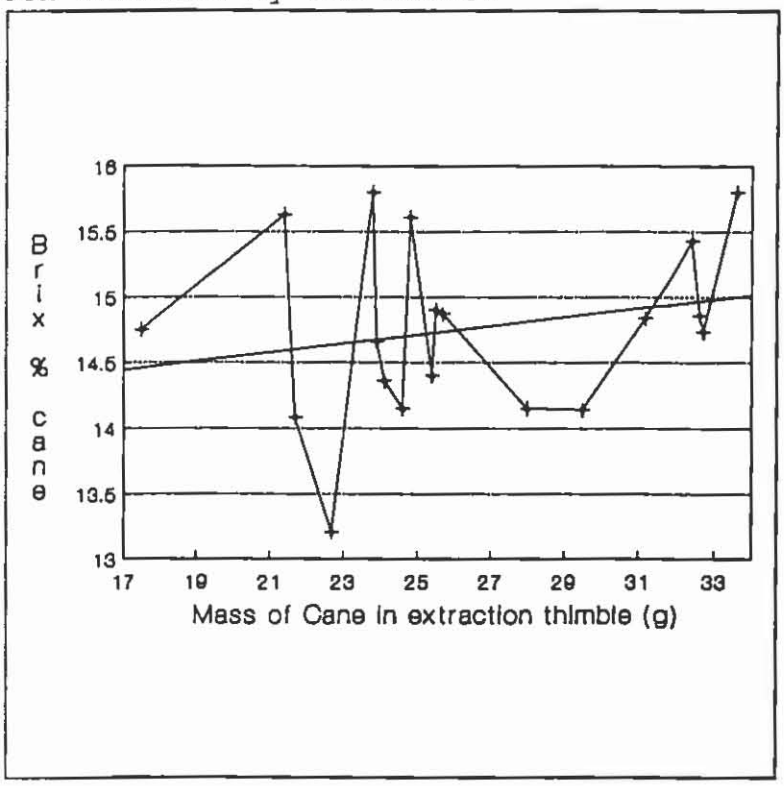


FIGURE 18 : Brix % cane as a function of Soxhlet extractor cane packing density

Possible solutions to this problem may be -

a) simply increase the extraction period from 4 hours (although initial tests indicated no significant, measurable improvement using increased extraction times).

b) obtain a finer preparation of cane to be analysed

c) devise a method of moving the extraction bed through the solvent (i.e. counter current contact)

d) mix the extraction bed during the analysis to expose a greater proportion of the cane to the solvent

e) reduce the solvent surface tension (e.g. by way of surfactants), in order to allow the solvent to permeate through the extraction bed more intimately.

F. Acknowledgments

Firstly, I wish to express thanks to members of staff at Hambledon, Kalamia, Pioneer and Victoria mills for their assistance in the collection of material required for the project.

Also, the help of Darryl Gibson, who assisted with the majority of analyses during the final stages of the project, is very much appreciated.

Finally, I would like to thank Dr N. Berding from the Bureau of Sugar Experiment Stations Research Centre, Meringa, for his co-operation in preparing suitable samples for the Soxhlet extraction work carried out.

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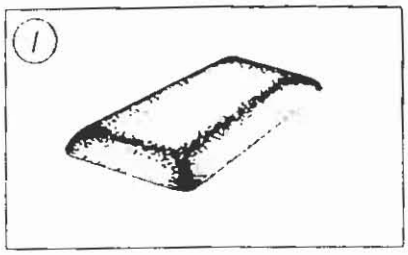
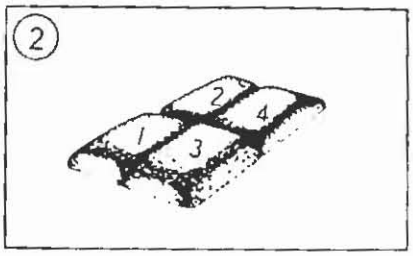
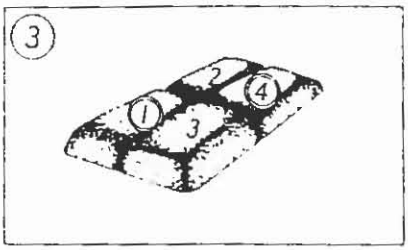
Appendix A - Soil Sampling Technique (Japanese Industrial Standard, 1965)



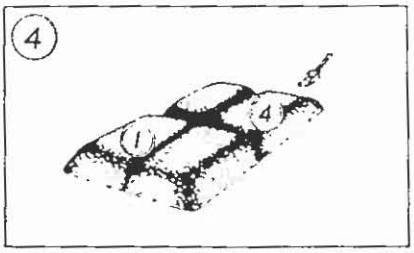
Spread crushed increment to form square of thickness as specified in Table 3 of the body of standard.



Divide the square, into 4 equal parts of cell, e.g., mark 2 x 2 matrix.



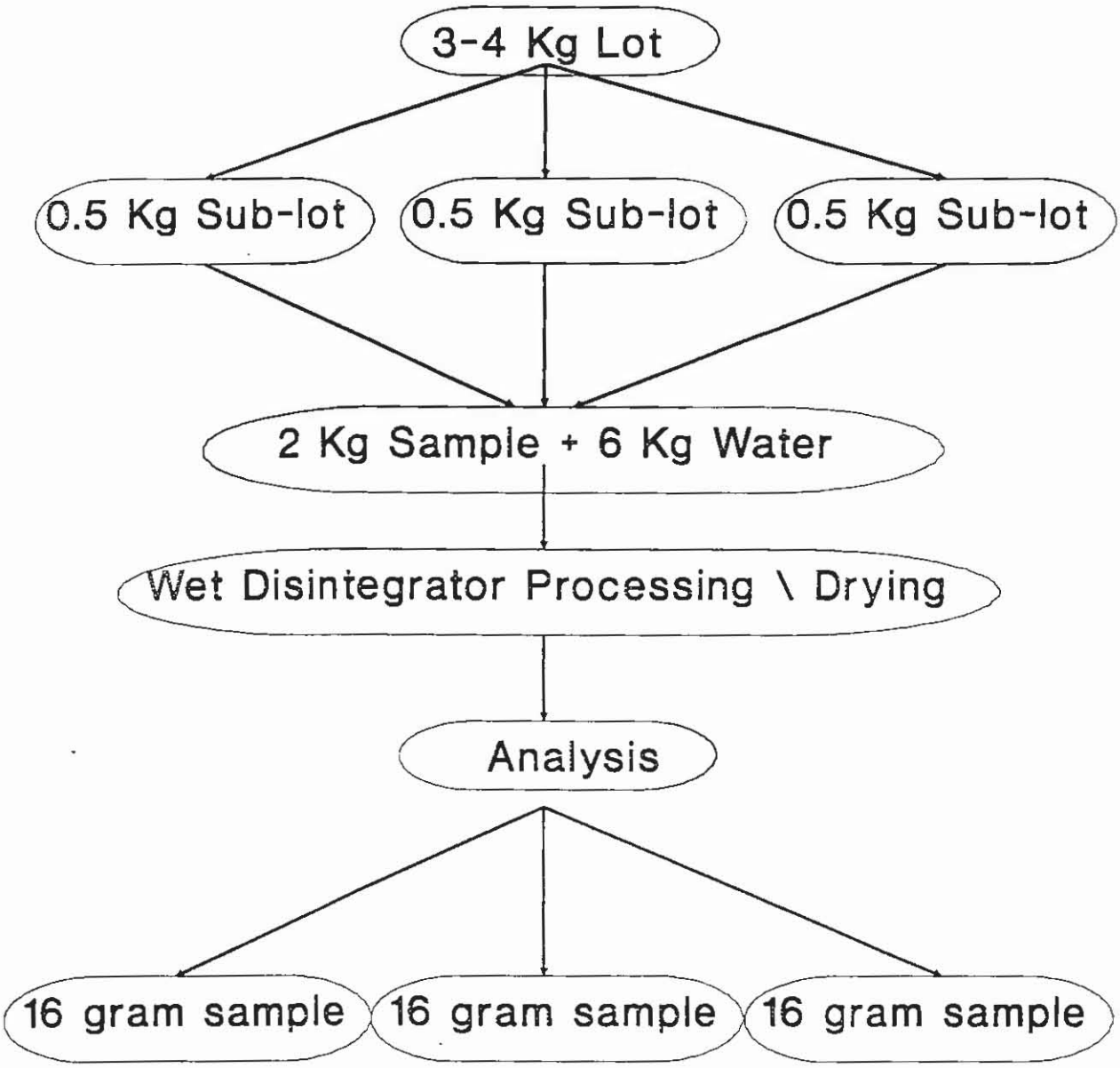
In the case when the number of increments as taken from a lot is 20 and up to 50, select at random two cells, e.g., (1) and (4), in accordance with 4.5.2 (2) (c).



Take at random each of one scoopful with a scoop from the cells (1) and (4) as selected at random, by inserting the scoop to the bottom, and obtain sample by combining these scoopfuls.

Appendix B

Sampling technique - cane fibre samples



Appendix C
Standard Mill Wet Disintegrator Analysis

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. Percent 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 rpm) 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 x 32 mm wide x 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 preferably 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 20L drum should be provided in case of blade failure.

3.3 **Water dispenser** 9 kg $\pm$ 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; or 400 mm long for visual polarimeters.

3.7 **Balance** to weigh 900 $\pm$ 10 g, plus container.

4.0 REAGENTS

4.1 **Basic lead acetate powder (dry lead) or basic lead acetate solution (wet lead).** Refer to the method for "Basic Lead Acetate - Specification and Preparation of Reagents and Methods of Analysis" for specifications and safety precautions.

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 $\pm$ 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) to ensure a reasonable fibre to water ratio.

6.2 Add 9 kg $\pm$ 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. If the wet disintegrator has a water jacket, 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°S (average of 4 readings).

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

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°S (average of 4 readings).

7.0 CALCULATIONS

7.1 **Record** the percent water in bagasse obtained by the method, "Water - Determination in Bagasse by Drying in a Spencer Oven".

7.2 **Correct** the pol reading of the liquor -

Corrected pol reading = pol reading (6.8) - water reading (6.7)

7.3 **Percent pol in bagasse** is obtained from Table 16.

Table 16 gives the pol in bagasse according to the corrected polarimeter reading, (in a 200 mm pol tube) and the percent water in the bagasse sample.

NOTE : (i) If the pol is read in a 400 mm tube, then divide the corrected pol reading (7.4) by 2, before obtaining % pol in bagasse from Table 16.

(ii) If the sample to water ratio is not 1 in 10 (see Note 6.1), use the relationship given in Table 16 to calculated % 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

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

Appendix D
Conversion of RI to Brix - Chebyshev Series (@ 589nm)*

P_{air} as a function of n_{air} at temperature of 20°C

i	a_i	
	546 nm	**589 nm**
0	90.886541	**92.616348**
1	44.346558	**44.197959**
2	-3.735553	**-3.705839**
3	0.465150	**0.462087**
4	-0.045687	**-0.045247**
5	0.004356	**0.004160**
6	-0.000854	**-0.000856**

$$P_{air}(n_{air}) = 0.5\ a_0 + a_1T_1(x) + \ldots\ a_6T_6(x)$$

where

$$x = \frac{(n_{air} - 1.42)}{0.09}$$

and

$$T_i\ (x) = \cos(i \times \cos^{-1}x)$$

* Rosenbruch R. J. , (1982)

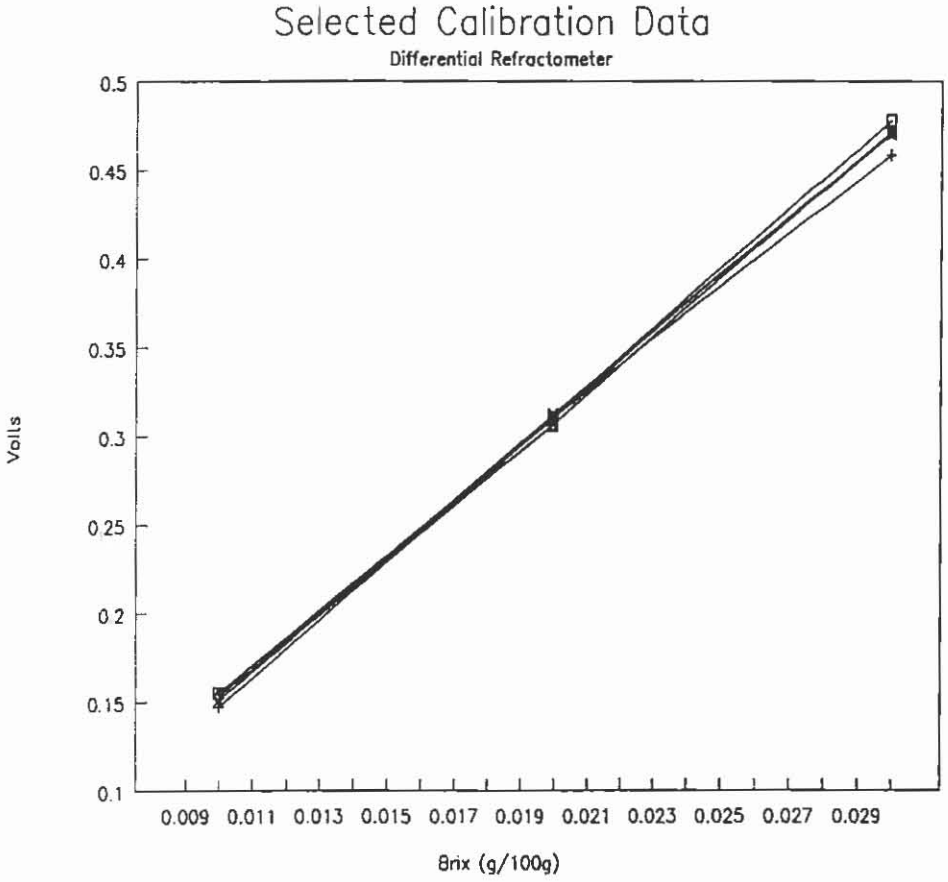
Appendix E
Standard Graph Data

Collected Data – Differential Refractometer Calibration

Date Analysed	Conc (Brix)	Reading (Volts)	Temp (Deg C)	Intercpt (Brix)	Slope	Correlation
27/9/89	0.010	0.155	23.5	0.0052	16.19	0.999
	0.020	0.310				
	0.030	0.478				
28/9/89	0.010	0.147	22.3	0.0062	15.59	0.999
	0.020	0.310				
	0.030	0.458				
29/9/89	0.010	0.153	20+/–0.1	0.0049	15.9	1.000
	0.020	0.306				
	0.030	0.471				
11/10/89	0.010	0.155	20+/–0.1	0.0004	15.05	1.000
	0.020	0.306				
	0.030	0.471				
17/10/89	0.011	0.151	20+/–0.1	0.0060	16.02	0.999
	0.021	0.312				
	0.030	0.470				
26/10/89	0.010	0.151	20+/–0.1	0.0005	15.84	1.000
	0.020	0.310				
	0.030	0.471				
2/11/89	0.010	0.150	20+/–0.1	–0.0100	15.69	1.000
	0.020	0.310				
	0.031	0.470				
6/12/89	0.010	0.149	20+/–0.1	–0.0031	15.3	1.000
	0.020	0.300				
	0.030	0.455				
11/12/89	0.010	0.159	20+/–0.1	0.0160	14.48	0.999
	0.020	0.315				
	0.030	0.450				
18/12/89	0.010	0.153	20+/–0.1	0.0005	15.25	1.000
	0.020	0.301				
	0.030	0.458				
20/12/89	0.010	0.154	20+/–0.1	0.0085	14.63	1.000
	0.020	0.304				
	0.030	0.448				
3/1/90	0.010	0.145	20+/–0.1	–0.0012	14.65	1.000
	0.020	0.288				
	0.030	0.438				

5/1/90	0.010	0.148	20+/−0.1	−0.0068	15.3	0.999
	0.020	0.291				
	0.030	0.454				
9/1/90	0.010	0.149	20+/−0.1	−0.0038	15.35	1.000
	0.020	0.300				
	0.030	0.456				
11/1/90	0.010	0.150	20+/−0.1	−0.0081	15.5	0.999
	0.020	0.291				
	0.030	0.460				
15/1/90	0.011	0.146	20+/−0.1	−0.0339	16.8	0.998
	0.020	0.313				
	0.030	0.467				
17/1/90	0.010	0.175	20+/−0.1	0.0197	15.22	0.999
	0.020	0.318				
	0.030	0.478				
22/1/90	0.010	0.148	20+/−0.1	−0.0091	15.75	1.000
	0.020	0.302				
	0.030	0.463				
24/1/90	0.010	0.151	20+/−0.1	−0.0001	15.2	1.000
	0.020	0.301				
	0.030	0.455				
30/1/90	0.010	0.148	20+/−0.1	−0.0042	15.18	1.000
	0.020	0.297				
	0.030	0.450				
5/2/90	0.010	0.149	20+/−0.1	−0.0005	15	1.000
	0.020	0.299				
	0.030	0.449				
7/2/90	0.010	0.151	20+/−0.1	0.0071	14.45	0.999
	0.020	0.293				
	0.030	0.440				
9/2/90	0.010	0.153	20+/−0.1	−0.0088	15.4	1.000
	0.020	0.295				
	0.030	0.453				
12/2/90	0.010	0.151	20+/−0.1	−0.0002	14.9	0.999
	0.020	0.289				
	0.030	0.449				
15/2/90	0.010	0.151	20+/−0.1	0.0071	14.6	1.000
	0.020	0.299				
	0.030	0.443				
19/2/90	0.010	0.151	20+/−0.1	0.0037	15	1.000
	0.020	0.303				
	0.030	0.451				
21/2/90	0.010	0.149	20+/−0.1	−0.0017	15.02	1.000
	0.020	0.298				
	0.030	0.448				
26/2/90	0.010	0.150	20+/−0.1	0.0022	14.95	1.000
	0.020	0.300				
	0.030	0.449				

1/3/90	0.010	0.151	20+/−0.1	0.0037	14.88	0.999
	0.020	0.301				
	0.030	0.450				
5/3/90	0.010	0.151	20+/−0.1	0.0028	15	1.000
	0.020	0.302				
	0.030	0.451				
8/3/90	0.010	0.150	20+/−0.1	−0.0025	15.25	1.000
	0.020	0.298				
	0.030	0.455				
12/3/90	0.010	0.150	20+/−0.1	−0.0013	15.08	1.000
	0.020	0.303				
	0.030	0.450				



Appendix F - Statistical Formulae

1.0 From "Principles and Procedures of Statistics", STEEL and TORRIE (1982).

1.1 Section 5.7, "Comparison of Sample Means; Meaningfully Paired Observations", pp 102.

$$S_D^2 = \frac{\sum (D^2) - \frac{(\sum D)^2}{n}}{(n-1)}$$

$$S_{\bar{D}} = \sqrt{\frac{S_D^2}{n}}$$

$$t_{\bar{D}} = \frac{\bar{D}}{S_{\bar{D}}}$$

Where :-

Sum D^2 = sum of the squared differences between paired data points

$(\text{Sum } D)^2$ = sum of the differences between paired data, squared

n = number of paired data

$\bar{D}$ = mean of the differences for the paired data

S_D = standard deviation for the difference between the paired data

$S_{\bar{D}}$ = mean standard deviation

2.0 NWA - Statpak, (1985), Appendices 4, 5

2.1 Arithmetic Mean ($\bar{X}$)

$$\bar{X} = \frac{\sum X_i}{n}$$

for i = 1, 2, 3 .....n

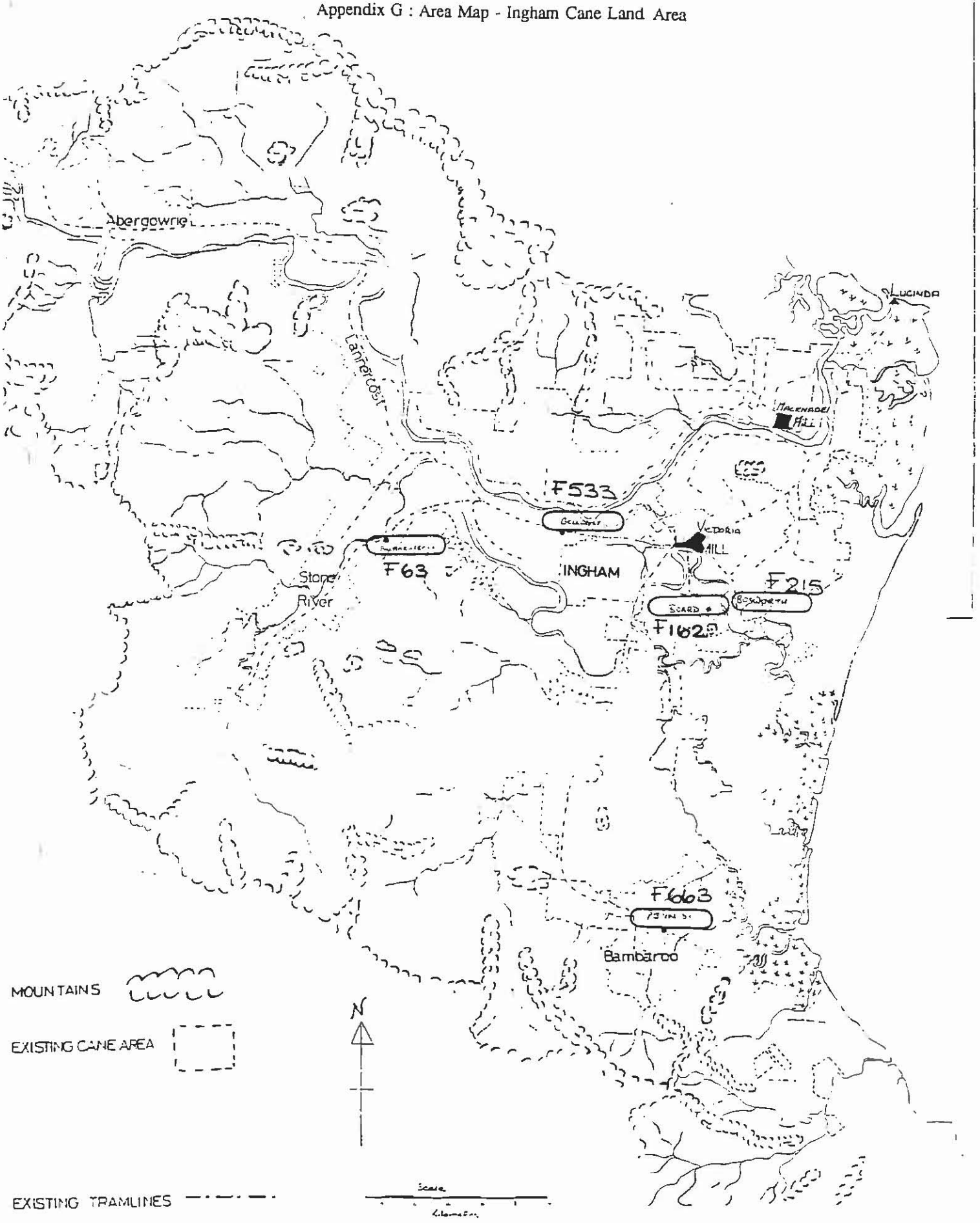
2.2 Standard Deviation - Sample

$$S = \sqrt{\frac{\sum (X_i - \bar{X})^2}{(n-1)}}$$

2.3 Coefficient Of Variation

$$C.V. \% = \frac{S}{\bar{X}} \times 100$$

Appendix G : Area Map - Ingham Cane Land Area



GEND	PLAN OF ASSIGNED LAND	DESCRIPTION OF ASSIGNED LANDS	ASSIGNMENT HOLDER
	SCALE 1 4000	PARISH	FARM No
			HILL AREA

Appendix H : Mill Manufacture Data*

CSR Limited. Mills Group S E A S O N S U M M A R Y Season 1989

2 PRODUCTIVITY, HARVESTING & TRANSPORT Page 7

Variety	% Harv Tonnes	Tonnes p Ha Plant	Tonnes p Ha Ratoon	CCS Plant	CCS Ratoon	CCS p Ha Plant	CCS p Ha Ratoon	% Area Planted 1989	% Area Planted 1990
			2.1 Productivity						
2.1.6 Variety Performance : Victoria									
Q115	33.7	104.9	89.6	14.65	13.82	15.3	12.3	35.3	
Q119	15.6	113.4	88.2	13.63	12.92	15.4	11.4	13.5	
Q117	10.2	114.1	96.4	14.43	14.08	16.4	13.5	11.0	
Triton	9.3	98.9	70.8	13.80	13.24	13.6	9.3	0.2	
Cassius	6.9	114.8	80.8	14.04	13.72	16.1	11.0	2.7	
Q124	4.7	116.8	96.8	14.26	13.52	16.6	13.0	16.6	
Q96	4.6	119.6	87.9	14.58	13.48	17.4	11.8	1.9	
2.1.6 Variety Performance : Pioneer									
Q96	73.2	143.7	110.3	14.60	13.90	20.9	15.3	62.7	51.0
Q117	18.6	153.5	118.6	15.20	15.04	23.3	17.8	32.9	46.6
Q63	4.8	80.8	90.1	14.89	13.77	12.0	12.4	0.2	
Q133	2.1	121.8	102.8	14.33	13.60	17.4	13.9	2.3	0.2
2.1.6 Variety Performance : Kalamia									
Q96	55.8	130.0	105.8	14.45	14.12	18.7	14.9	30.1	34.9
Q117	27.3	137.9	117.8	15.40	14.96	21.2	17.6	59.6	57.4
Q63	8.8	106.4	90.6	13.41	14.25	14.2	12.9	1.4	
Q133	6.9	114.5	98.7	13.30	13.13	15.2	12.9	7.9	6.6

* - Data taken from "CSR Sugar Mills, Mill Manufacture Report, season 1989"

Appendix I - Soil particle size - Method

1.0 Weigh approximately 2 grams of soil into a pre-weighed beaker. Add volumetrically 100 mL of distilled water and place the covered solution on a magnetic stirrer for 5 minutes.

2.0 Once mixing is complete, filter the solution through the following steps.

2.1 Filter initial solution through a preweighed Whatman No91 filter (retains particles > 10 µm), collecting filtrate for step 2.2 .

2.2 Filter filtrate from 2.1 through Whatman GF/A glass filter (retains particles approximately equal to 1.6 µm), collecting filtrate for step 2.3 .

2.3 The filtrate collected in 2.2 is then evaporated to dryness in order to ascertain the quantity of soil particles smaller than 1.6 µm.

3.0 Having completed filtration, dry retentate + paper from steps 2.1 and 2.2 for 3 hours ± 5 minutes (105-110°C) until constant weight is obtained.

4.0 Calculate % soil retained at each stage of filtration as follows :-

$$\% \, Soil = \frac{(S) - (W)}{S_T} \times 100$$

where :-

W = mass of dry weighing dish + filter

S_T = initial mass of soil weighed (total)

S = mass of filter + soil

Appendix J - Derivation for the Calculation of Unbound Water

The formula for calculating Brix of cane from wet disintegrator analysis was shown by Deicke to be :-

$$Bx\ of\ cane = \frac{Bx\ extract\ (\phi + w)}{(\theta - Bx\ extract)} \quad \ldots\ldots\ldots (1)$$

where :

$$\phi = 100 \frac{(\frac{x + y}{y}) - (1 + \frac{z}{100})}{(1 + \frac{z}{100})}$$

$$\theta = \frac{100}{(1 + \frac{z}{100})}$$

y = weight of cane
x = weight of water
z = hygroscopic water
w = moisture % cane

By performing the wet disintegrator analysis at two different dilutions, it is possible to determine the Brix of cane without knowing the hygroscopic water in fibre or determining the water in cane (w).

This may be seen mathematically as follows :

Starting with the Deicke formula above :-

Let

$$\beta = 1 + \frac{z}{100}$$

y = 1 and x = ratio water/cane

then

$$\phi = 100\frac{(x + 1 - \beta)}{\beta}$$

$$\theta = \frac{100}{\beta}$$

$$Brix\ of\ cane = \frac{BE(\frac{100}{\beta}(x + 1 - \beta) + w)}{\frac{100}{\beta} - BE} \quad \ldots\ldots\ldots (2)$$

Let BC = Brix of cane
BE = Brix of extract

$$BC(\frac{100}{\beta} - BE) = BE(\frac{100}{\beta}(x + 1 - \beta) + w) \quad \ldots\ldots\ldots (3)$$

expand and collect terms :

$$BE(\frac{100}{\beta}(x + 1 - \beta) + w + BC) = BC(\frac{100}{\beta}) \quad \ldots\ldots\ldots (4)$$

$$(BC)\beta - \frac{100}{BE}BC - (100 - w)\beta + 100(x + 1) = 0 \quad \ldots\ldots\ldots (5)$$

if the analysis is done at 2 dilutions, we have 2 equations like this with x_1 and x_2 and BE_1 and BE_2 in each respectively. Subtracting one from the other :

$$-(\frac{100}{BE_1} - \frac{100}{BE_2})BC + 100(x_1 - x_2) = 0 \quad \ldots\ldots\ldots (6)$$

Thus :

$$BC = \frac{100(x_1 - x_2)}{\frac{100}{BE_1} - \frac{100}{BE_2}} \quad \ldots\ldots\ldots (7)$$

Rearranging terms in (5)

$$\beta = \frac{(\frac{100}{BE})BC - 100(x + 1)}{BC - (100 - w)} \quad \ldots\ldots\ldots (8)$$

Since BC can be obtained in terms of BE_1 and BE_2 using two dilutions x_1 and x_2, then ß can be calculated in terms of these quantities with the additional knowledge of water in cane (w).

Explicitly :

$$\beta = \frac{(\frac{100}{BE_1} - \frac{100}{BE_2}) + (\frac{100}{BE_1}x_2 - \frac{100}{BE_2}x_1)}{(\frac{100}{BE_1} - \frac{100}{BE_2})(1 - \frac{w}{100}) + (x_2 - x_1)} \quad \ldots\ldots\ldots (9)$$

An alternate treatment can be seen as follows :

If we take 100 part cane and disintegrate in W added water we can measure the concentration of the extract in units of g/100 solvent :-

$$C_1 = \frac{BC}{W_1 + Q} \times 100 \ldots\ldots\ldots (10)$$

where : Q is unbound water in 100 cane
BC is Brix % cane

similarly -

$$C_2 = \frac{BC}{W_2 + Q} \times 100 \ldots\ldots\ldots (11)$$

equations (10) and (11) can be solved for Q and BC :-

$$Q = \frac{(W_2 - W_1)C_2}{\Delta C} - W_1 \ldots\ldots\ldots (12)$$

Rearranging equation 11 we get :-

$$BC = \frac{C_2 W_2}{100} + \frac{C_2 Q}{100} \ldots\ldots\ldots (13)$$

Substituting equation 12 for Q and simplifying :-

$$BC = \frac{C_2^2 (W_2 - W_1)}{100 \Delta C} + \frac{C_2 (W_2 - W_1)}{100} \ldots\ldots\ldots (14)$$

If the total water in cane is "w" then :-

$$Hygroscopic\ water = w - Q$$

or expressed as a percentage on fibre :-

$$Z = \frac{w - Q}{F} \times 100 \ldots\ldots\ldots (15)$$

If we do not wish to measure the concentration of the extracts in g/100 solvent we can measure them as Brix and convert to g/100 solvent or vice versa as follows :-

If corresponding to C, we have BE, Brix of extract.

Then :

$$BE_1 = \frac{C_1}{100 + C_1} \times 100$$

$$= \frac{C_1}{1 + \frac{C_1}{100}}$$

and

$$C_1 = \frac{BE}{1 - \frac{BE}{100}}$$

One can also determine Q and BC graphically by using the inverted form of equation 11.

$$\frac{1}{C_2} = \frac{W_2}{100\ BC} + \frac{Q}{100\ BC}$$

Plotting $1/C_2$ against values of W_2, data will lie on a straight line giving a slope of 1/100BC and a y axis intercept of Q/100BC.
Q is calculated by dividing the intercept by the slope which is equal to 1/100BC. The physical interpretation of the intercept Q/100BC is the concentration in g/100 solvent of absolute juice.

If a sample is withdrawn from the disintegrator vessel, small errors are incurred unless corrections are applied to compensate. The following equations have been derived so that Q can be calculated from 2, 3 or more measurements on samples withdrawn after each solvent dilution.

Where S grams of sample is withdrawn after the first water addition :

$$Q = \frac{1}{C_1 - C_2}\left(C_2W_2 - C_1W_1 + V_1\left(\frac{C_1}{1 + \frac{C_1}{100}}\right) - \frac{C_2V_1}{100}\left(100 - \frac{C_1}{1 + \frac{C_1}{100}}\right)\right) \ldots (16)$$

$$V_1 = \frac{S}{M} \times 100\ g\ sample/100\ cane$$

$$S = mass\ of\ sample\ withdrawn\ (grams)$$

$$M = mass\ of\ cane\ used\ (grams)$$

where a second sample is withdrawn after second water addition, the equation expands to the form as appears in the main report.

Appendix K
Pilot Wet disintegrator method

Principle of Method

A weighed sample of processed cane is mixed with a weighed quantity of distilled water and disintegrated via a high speed homogeniser. The slurry obtained from the disintegration is then filtered, cooled and analysed for % solids (Brix). Brix of cane is then calculated using suitable formulae.

Apparatus

Ordinary laboratory apparatus and glassware and

High Speed homogeniser

Essentially a high speed blender, the pilot wet disintegrator method consisted of a suitably sized disintegrating vessel (10 L capacity) and an Ultra Turrax homogeniser (type : T45, 10000 rpm, 300 W).

Sample Pretreatment

Billets of cane were initially sliced via guillotine followed by preparation using a domestic food processor. Once processed samples had a coarse consistency, being frozen in readiness for analysis.

Procedure

1. Weigh out 100.0 ± 0.1 g processed cane into the 10 litre disintegrating vessel.

2. Add 1000.0 ± 0.1 g distilled water to the vessel, sealing the vessel with an appropriately holed out lid (i.e to accommodate the homogeniser head).

3. Run the homogeniser for four 1 minute intervals, allowing the system to rest for 1 minute in between each disintegration.

4. Having completed the disintegration, filter 15 mL of the slurry through Whatman 8 µm membrane, in order to obtain a clear, readable solution.

5. Once the filtered solution has cooled, determine the Brix of the solution via Dipping refractometer (see section C, "Experimental and Instrumentation" in main report.

Calculations

Brix of cane is calculated from wet disintegration via the Deicke equation -

$$Bx\ Cane = \frac{Bx\ Extract \times (\theta + w)}{(\phi - Bx\ Extract)} \quad \ldots\ldots\ldots\ldots (1)$$

where :

$$\theta = \frac{\frac{(X+Y)}{Y} - (1 + \frac{z}{100})}{1 + \frac{z}{100}} \quad \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

and

$$\phi = \frac{100}{(1 + \frac{z}{100})} \quad \ldots\ldots\ldots\ldots\ldots\ldots (3)$$

y = weight cane (g), x = weight water (g), z = % hygroscopic water, w = moisture % cane.

Note : 1. moisture % cane is determined via oven drying of hammer milled cane samples at 105-110 °C/5 hours.

2. a value of 25% (i.e. z = 25) is used for % hygroscopic water on fibre.