SECOND ANNUAL REPORT (1927) OF THE COMMITTEE ON STANDARDIZATION OF CHEMICAL CONTROL.

Official Methods of the S.A. Sugar Technologists' Association for Chemical Control.

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Chapter I.

DEFINITIONS AND CALCULATIONS.

For the purposes of Chemical Control, the following definitions apply:—

CANE:
The raw material to be crushed.

The weight of cane crushed is taken as recorded at the weighbridge. No deduction may be made for trash, tops, dirt, etc., or for cane which is crushed and subsequently found to be below the standard of rejection.

FIBRE:
The insoluble matter in the cane.

BRIX OR TOTAL SOLIDS:
The soluble solids in solution indicated by the Brix hydrometer.

REFRACTIVE SOLIDS:
The soluble solids in solution as indicated by the refractometer.

DRY SUBSTANCE:
The solids determined by drying.

APPARENT SUCROSE:
The sucrose content indicated by single polarization.

CLERGET SUCROSE OR TRUE SUCROSE:
The sucrose content as determined by double polarization method.

APPARENT PURITY:
The percentage proportion of apparent sucrose in the Brix or total solids.

CLERGET PURITY:
The percentage proportion of Clerget sucrose in the Brix or total solids.

DRY SUBSTANCE—CLERGET PURITY:
The percentage proportion of Clerget sucrose in the dry substance.

POLARIZATION:
The actual reading of a solution on the saccharimeter scale in degrees Ventzke.

REDUCING SUGARS:
The reducing substances in cane and its products calculated as invert sugar.

ASH:
The residue remaining after burning off the organic matter.

REDUCING SUGAR RATIO:
The ratio of reducing sugars to sucrose.

REDUCING SUGAR-ASH RATIO:

$$= \frac{\text{per cent reducing sugar}}{\text{per cent ash.}}$$

NORMAL JUICE:
The juice expressed from the cane. It is assumed to have the Brix of the first crusher juice and the purity of the mixed juice.

FIRST CRUSHER JUICE:
The juice expressed by the first two rollers of the crushing plant.

MIXED JUICE:
The total juices from the mills sent to the clarification department.

The weight of mixed juice extracted from the cane crushed is taken as recorded by the weighing machine, without making any deduction for matters in suspension, or for rejected cane.

MACERATION WATER:
The water applied to bagasse during crushing.

This may be either weighed or measured in bulk, making the necessary volume corrections where hot water is used. Water meters may not be used for measuring maceration water.

BAGASSE:
The residue left after expressing the juice from the cane.

The weight of bagasse is calculated and equals: Weight of cane + weight of maceration water – weight of mixed juice.

SUCROSE PER CENT MIXED JUICE:
The value indicated in the mixed juice by the Clerget method of double polarization.

WEIGHT OF SUCROSE IN MIXED JUICE:

$$= \frac{\text{Weight of mixed juice} \times \text{Clerget sucrose per cent. mixed juice}}{100}$$

WEIGHT OF SUCROSE IN BAGASSE:

$$= \frac{\text{Weight of bagasse} \times \text{sucrose per cent. bagasse}}{100}$$

SUCROSE IN CANE:

= Sucrose in mixed juice + sucrose in bagasse.

JAVA RATIO:

$$= \frac{\text{Sucrose in Cane} \times 100}{\text{Sucrose in first crusher juice.}}$$

LAST ROLLER JUICE:

The juice expressed by the top and bagasse rollers of the last crushing unit.

FIBRE PER CENT BAGASSE:

$$\text{Dry substance per cent. bagasse} - \frac{\text{Sucrose per cent. bagasse)} \times 100}{\text{Purity of last roller juice.}}$$

FIBRE PER CENT. CANE:

$$= \frac{\text{Weight of fibre in bagasse} \times 100.}{\text{Weight of Cane.}}$$

DILUTION WATER:

The portion of the maceration water which finds its way into the Normal Juice and reduces its Brix to that of mixed juice.

DILUTION PER CENT. NORMAL JUICE:

$$= \frac{\text{(Brix first crusher juice} - \text{Brix mixed juice)} \times 100}{\text{Brix of Mixed Juice.}}$$

WEIGHT OF NORMAL JUICE:

$$= \frac{\text{Weight of Mixed Juice} \times 100}{100 + \text{Dilution per cent. Normal Juice.}}$$

WEIGHT OF DILUTION WATER:

= Weight of mixed juice – Weight of Normal Juice.

MACERATION PER CENT. CANE:

$$= \frac{\text{Weight of Maceration Water} \times 100}{\text{Weight of Cane.}}$$

CLARIFIED JUICE:

The juice entering the evaporator.

SCUMS JUICE:

The clear juice from the second decantation of the mud.

FILTER PRESS JUICE:

The total juice running from the filter presses. Where double filtration of the mud is practised, the juices should be sampled, analysed and recorded separately.

FILTER PRESS CAKE:

The mud removed from process by filtration.

SYRUP:

The concentrated juice leaving the evaporator.

MASSECUITE:

The mixture of crystals and mother liquor as concentrated in, and discharged from, vacuum pans.

JELLY:

A string-proof boiling of molasses.

MOLASSES OR RUN-OFFS:

The liquid removed from massecuite by centrifugals and subsequently re-boiled.

FINAL MOLASSES:

The molasses from low-grade boiling from which no further sugar is to be removed, and which is discarded from the factory.

RAW SUGAR:

Sugar which is intended to undergo refining.

WHITE SUGAR:

High grade sugar produced for direct consumption.

LOW GRADE SUGAR:

Sugar obtained from low-grade massecuites and intended for sale on the open market.

EXTRACTION:

$$= \frac{\text{Weight of Sucrose in Mixed Juice} \times 100}{\text{Weight of Sucrose in Cane.}}$$

TONNAGE RATIO:

An arbitrary figure for comparing the crushing rate of mills of different sizes obtained by dividing the tons of cane crushed per hour by a factor calculated as follows:-

$$\frac{(\text{L'gth. of Roller in ft.})^2 \times \text{No. of Units (mills) in train}}{6}$$

When rollers are of different lengths, that of the majority is used. A two-roller crusher counts as one unit.

For value of factors see Table I.

LELY RATIO:

$$= \frac{\text{Sucrose \% bagasse} \times 10{,}000.}{\text{Sucrose \% Normal Juice} \times \text{Fibre \% bagasse.}}$$

EXTRACTION RATIO:

The ratio of the unextracted sucrose to the fibre in cane. The formula is:

$$\frac{100 - \text{Extraction}}{\text{Fibre per cent. cane}}$$

BOILING HOUSE RECOVERY:

$$= \frac{\text{Weight of sucrose recovered} \times 100}{\text{Weight of sucrose in Mixed Juice}}$$

AVAILABLE SUCROSE % SUCROSE IN JUICE:

$$\frac{\text{Purity Sugar (Purity Mixed Juice} - 45) \times 100}{\text{Purity Mixed Juice (Purity Sugar} - 45)}$$

N.B.—Gravity purity of sugar and Clerget purity of mixed juice should be used.

BOILING HOUSE EFFICIENCY:

$$= \frac{\text{Sucrose recovered} \times 100}{\text{Available Sucrose}}$$

OVERALL RECOVERY:

$$= \frac{\text{Sucrose recovered} \times 100}{\text{Sucrose in cane}}$$

CRUSHING TIME ANALYSIS:

Cane Shortage: This only includes actual loss of time for want of cane, *e.g.*, a factory making a practice of crushing only 18 hours per day does not record the other six hours as cane shortage.

Mechanical Stoppages: These include all stops of mills caused in the manufacturing process, including chokes of milling units. Slow crushing for any reason is not reckoned as a mechanical stoppage.

Total Available Time: = Hours lost in cane shortage + Hours lost in mechanical stoppages + Hours actual crushing.

NOTE: For mills using the Petree Process, it will be necessary to use local modifications of some of the above definitions and calculations.

Chapter II.

STANDARDIZATION OF APPARATUS.

POLARISCOPES (SACCHARIMETERS):

All polariscopes or saccharimeters should be standardised at 20°C. and have a normal weight of 26.00 gms. as recommended by the "Committee of the International Congress of Applied Chemistry." Each laboratory should be equipped with at least two Quartz Control Plates, one between 94° and 98°V. and the other between 50° and 60°V. The Experiment Station undertakes the work of further checking polariscopes by means of Quartz Control Plates and graduated telescopic control tubes.

BRIX HYDROMETERS:

These should not be less than 54 cm. in total length, and should have a range of not more than 10° Brix, graduated in one-tenth of a degree. There should not be less than 22 mm. between each unit on the scale. Brix Hydrometers should be standardised at 20°/4°C. The Experiment Station undertakes the work of standardization of all hydrometers used in Sugar Factories. Any correction required is to be applied to every reading.

HYDROMETER JARS:

These should be of glass without a lip and have a clearance of not less than one-half inch between the bulb of the hydrometer and the side of the jar.

VOLUMETRIC GLASSWARE:

The true or metric cubic centimetre, that is, the volume occupied by one gram of water weighed in vacuo at 4°C., is adopted as the standard for all volumetric apparatus and adjusted at 20°C.

PIPETTES:

For the discharge of pipettes, the method laid down by the United States Bureau of Standards is adopted: "After filling, remove the excess of liquid adhering to the tip. In emptying, hold in a vertical position, with the outflow unrestricted, until the surface of the liquid reaches the upper end of the tube below the bulb, then touch the tip to the side of the receiving vessel, keep in contact until the liquid has ceased to run freely and immediately withdraw."

BAGASSE APPARATUS:

This consists of a cylindrical vessel 8 inches in diameter and 10 inches high. The whole to be a brass casting or in copper, the object being strength with a minimum of weight. It should be provided with a ⅛ inch machined flange to form an air-tight joint with the cover, the flange to carry hinged bolts with winged nuts for securing the cover. The cover to be of metal, ⅛ in. thickness, and having in the centre a hole 1½ ins. in diameter, encircled by a collar, ½ in. in height and very slightly tapered to hold a rubber stopper. This hole to be provided with a rubber stopper through which passes the lower end of a suitable condenser. The vessel should be fitted with one cock, with a straight outlet, one inch from bottom.

The apparatus may be fitted with a basket of perforated metal plate, 8 inches high, and of a diameter such that it conveniently fits into the vessel. This basket should be provided with vertical handles for convenient withdrawal from the vessel while hot.

All types of apparatus should be fitted with a suitable appliance which ensures that the whole of the bagasse is in contact with the water throughout the boiling. A plain circular disc of metal, fitting loosely into the vessel or basket is found to be effective.

Chapter III.

REAGENTS.

BASIC ACETATE OF LEAD—STOCK SOLUTION:

Heat nearly to boiling for about half an hour, 860 gms. of neutral lead acetate, 260 gms. of litharge and 500 ccs. of water. Add water to compensate for the loss by evaporation. Cool, settle and decant the clear solution.

This solution may be prepared without heat, provided the mixture is set aside several hours with frequent shaking.

BASIC ACETATE OF LEAD—DILUTE SOLUTION:

Dilute the Stock Solution to 54° Brix with cold recently boiled water. In all cases where a solution of basic acetate of lead is mentioned in the methods of analysis, this dilute solution should always be used.

NEUTRAL LEAD ACETATE SOLUTION:

Dissolve a quantity of neutral lead acetate in about twice its weight of water, and dilute the solution to 54° Brix.

ALUMINA CREAM:

To a saturated solution of common alum in water, add ammonia in slight excess. The sulphate may be removed, if desired, by washing by decantation until the odour of ammonia is not apparent.

FEHLING'S SOLUTION:

This is prepared according to Soxhlet's method, in two parts:—

(A) 34.639 grams of copper sulphate are dissolved in water, and accurately diluted to 500 ccs. in a graduated flask.

(B) 173 grams of tartrate of soda and potash (Rochelle Salt) are dissolved in water, mixed with 100 ccs. of a solution containing 51.6 gms. of sodium hydroxide (caustic soda) and the volume completed to 500 ccs.
Chemically pure salts should be used.

In every case where Fehling's Solution is mentioned in these methods, a volume containing equal volumes of solutions A and B, prepared as above, is to be used.

POTASSIUM OXALATE SOLUTION (for de-leading):-
10% solution in water.

METHYLENE BLUE SOLUTION:
1% solution in water.

SODIUM CARBONATE SOLUTION:
5% solution of commercial washing soda in water is recommended for use in bagasse analysis.

PHENOLPHTHALEIN SOLUTION:
1% solution in 60% pure methylated spirits.

THYMOL SOLUTION:
A 5% solution in absolute alcohol.

STARCH INDICATOR:

One part of starch is first mixed with cold water to form a smooth emulsion, then gradually poured into about 150 or 200 times its weight of boiling water, and the boiling continued for a few minutes. Best results are obtained by using freshly prepared starch indicator, and by using the clear solution obtained on allowing the mixture, prepared as above, to stand and settle thoroughly. Potato starch or arrowroot starch gives very good results.

INDICATORS FOR THE DETERMINATION OF HYDROGEN ION CONCENTRATION:

These are prepared according to the Method of Clark & Lubs. For the preparation of stock solutions, 0.1 gram of the dry indicator is ground in an agate mortar with the following quantities of N/32 sodium hydroxide:—

Methyl red	11.6 ccs.
Bromcresol purple	5.8 ccs.
Bromthymol blue	5.3 ccs.
Phenol red	9.1 ccs.

When solution is complete, dilute with water so that the concentration of the indicators is as follows:-

Methyl red	0.02%
Bromcresol purple	0.04%
Bromthymol blue	0.04%
Phenol red	0.02%

STANDARD SOLUTIONS:

Oxalic acid is used as the basis for Acidimetry and Alkalimetry.

N/3.2 STANDARD OXALIC ACID SOLUTION:

19.695 grams of the purest obtainable crystals of Oxalic Acid ($H_2C_2O_4\ 2H_2O$) are accurately weighed out, and carefully transferred to a 1,000 cc. graduated flask. It is dissolved in water and the volume completed to the mark with the solution at a temperature of 20°.

The oxalic acid crystals should be preserved in a well stoppered glass bottle, sealed with paraffin wax. Crystals showing signs of efflorescence should be rejected.

N/3.2 CAUSTIC SODA SOLUTION—
(STOCK SOLUTION):

Rapidly weigh off about 14.0 grams of pure sodium hydroxide (caustic soda) for every litre of Stock Solution required. Immediately dissolve in freshly boiled distilled water and make up to approximately the required volume.
Titrate this solution against 20 ccs. of standard N/3.2 Oxalic Acid, using phenolphthalein indicator. Calculate and add the quantity of water necessary to bring to N/3.2. Shake well and repeat the titration and addition of water until the standard Oxalic acid solution (N/3.2) is exactly neutralized by an equal volume of caustic soda solution.

It should always be remembered that the titration value of caustic soda solutions, using phenolphthalein as indicator, changes on exposure to air, due to absorption of carbon dioxide. The normality of the stock solution should therefore be checked from time to time.

N/32 CAUSTIC SODA SOLUTION:

A volume of the stock solution (N/3.2) is accurately diluted with freshly boiled distilled water to 10 times the original volume.

(1 cc. of this solution=1 mg. sulphur dioxide).

It is important that the water used for this dilution should be free from carbon dioxide (*i.e.* freshly boiled). Owing to the dilute nature of this solution, the comparative effect of carbon dioxide is more marked. Only small quantities of this dilute solution should be prepared as required.

N/2.8 HYDROCHLORIC ACID—STOCK SOLUTION:

37.5 ccs. of concentrated hydrochloric acid diluted to one litre gives a solution of approximately the required strength. The method of standardisation is similar to that described above for N/3.2 Caustic Soda solution, using freshly standardised N/3.2 Caustic Soda for the titration.

28.0 ccs. of N/2.8 hydrochloric acid should exactly neutralize 32 ccs. of N/3.2 caustic soda solution.

N/28 HYDROCHLORIC ACID:

A volume of the stock solution (N/2.8) is accurately diluted to 10 times the original volume.

(1 cc. of this solution=1 mg. CaO).

N/32 IODINE SOLUTION:

Dissolve 4 grams of iodine (3.968 for accurate work) in a solution of 12 grams of Potassium Iodide (free from Iodate) in a little water, and the whole made up in a graduated flask to exactly one litre.

(1 cc. of this solution = 1 mg. SO_2).

Chapter IV.

SAMPLING.

The reliability of the results of Chemical Control depends to a very large extent on the methods of sampling. Due care should therefore be taken that all samples are truly representative.

CANE:

It is pointed out that the nature of cane makes it impossible to obtain representative samples, so that hand sampling of cane should only be resorted to in cases of necessity. Small samples selected in the fields or at the mills are of little value in judging the general character of cane. The best the chemist can hope to accomplish, under favourable conditions, is to obtain a sample that will, in a very general way indicate the condition of the cane. When no other method of analysis is possible, the following methods of sampling should be employed.

FACTORY SAMPLING:

25 lbs. of the cane should be sampled by each of two individuals acting separately, and the whole sub-sampled to a 25 lb. sub-sample.

FIELD SAMPLING:

All the canes from a representative stool are taken for a sample, each stool to be taken not less than 15 yards from any boundary or break. The number of stools to be taken for a sample from a number of fields of cane of the same age should be as follows:—

Number of Stools.	Area in acres.
1	10
2	25
3	50
4	100
5	200

Each cane should be trashed and topped one node below where the lowest green leaf joins the stem.

BAGASSE:

A sample to be taken once every hour during a period of five minutes over the entire width of the carrier. Care should be taken that this sample is not collected during a choke. The sample should be conveyed to the laboratory in a closed receptacle and analysed immediately for sucrose and moisture.

As an alternative method, a composite sample is made up for sucrose determination every hour, with a catch sample every two hours for moisture.

JUICE SAMPLES:

These should be collected in covered containers of a suitable size. All juice sampling devices and containers should be kept scrupulously clean. It is advisable to keep duplicate sets and to use the sets alternately. Enamelled buckets are particularly suitable for factory samples. Sub-samples in the laboratory should be collected in wide mouthed clear glass bottles with ground glass stoppers, of about 2 litres capacity. (Bottles such as are used for sweet containers are suitable for this purpose). All sub-samples should be preserved with 2 ccs. of a 40 per cent solution of formaldehyde per litre of juice. Special analyses are to be made from time to time to ascertain if the preservation of samples is effective. Where any discrepancy is found, a saturated solution of mercuric chloride is to be used at the rate of 0.5 cc. per litre of juice.

It is impressed that care should be taken that all samples are homogeneous before testing.

FIRST CRUSHER JUICE:

Wherever possible this sample should be automatic. A good arrangement is a half inch hole drilled in the juice chute from the first crusher, in a suitable position with a bucket immediately underneath it. A large hole or a number of small holes, will be necessary for samples for the determination of approximate sucrose in cane. Should a continuous sample be impracticable, representative samples should be drawn by hand at frequent intervals from the flow of juice from the first crusher.

The routine samples are brought to the laboratory hourly, and sub-sampled into collective sample bottles.

LAST ROLLER JUICE:

This product should be sampled over the same period as the bagasse. It should be drawn by hand at frequent intervals from different points along the roller and conveyed to the laboratory and sub-sampled.

MIXED JUICE:

As the entire chemical control is based on the weight and analysis of the mixed juice, the greatest care must be taken to obtain a representative sample.

Wherever practicable, this sample must be automatic. Various suitable methods of carrying this out may be devised. Wherever it is impracticable to employ mechanical methods, samples may be drawn from each tank as weighed, and mixed to form a general sample. The sample is taken to the laboratory every hour and sub-sampled.

A catch sample of mixed juice for acidity and sulphur dioxide determination is taken from the juice leaving the sulphur apparatus.

CLARIFIED JUICE, SCUMS JUICE AND FILTER PRESS JUICE:

These samples should be automatic wherever possible from the juice flow, or may be taken by hand at frequent intervals. Samples are brought to the laboratory and sub-sampled every hour.

Where the scums are double pressed the juice from the two processes should be sampled separately.

FILTER PRESS CAKE:
This sample should be taken from the trolleys or from each press as emptied by means of a metal tube of suitable size. Samples are to be composited in a closed receptacle and analysed every four hours.

SYRUP:
Representative samples may be taken from the syrup storage tanks, or the sampling may be automatic if desired. Samples are taken to the laboratory and the composite sample analysed every four hours.

MASSECUITES:
These may be sampled while being discharged from the pan, or from the crystallizer as desired. In the former case, not less than three separate portions are to be taken at different periods during the discharge, and thoroughly mixed.

MOLASSES FOR RE-BOILING OR RUN-OFFS:
These may be sampled by hand or automatically from the molasses storage tanks after blowing up. Samples should represent the run-off from each individual massecuite struck.

FINAL MOLASSES:
Samples are to be taken from the discharge side of the molasses pump—continuously if possible. A sample may be taken from a storage tank by hand every four hours, and composited for analysis. Representative portions of this are put aside and mixed for complete weekly analysis:

SUGAR:
For factory control, representative samples are to be made as bags are filled.

For each type produced an average weekly sample is composited from weighed proportionate amounts of sugar from each lot bagged. For this purpose, a number of grams of the sample from a particular lot, equal to the number of bags in that lot is weighed out, and set aside for the weekly composite sample. At the end of the week, these proportionate amounts are thoroughly mixed for analysis.

If one gram of sugar per bag makes the composite sample too bulky, a smaller weight may be taken provided that it bears a definite constant ratio to the number of bags.

Chapter V.

GENERAL METHODS OF ANALYSIS

GENERAL PRECAUTIONS:
All apparatus should be used at room temperature so as to maintain a uniform temperature in the solution under test. Where the filtration for any reason is slow or delayed, adequate precautions are to be taken against evaporation by setting the funnel directly on to the receiving vessel, and covering with a watch glass. For ordinary rapid working, this precaution is not regarded as necessary for dilute solutions, such as mill juices or products diluted to the same density, or the dilute solutions from bagasse or press cake. In testing sugar, however, this precaution must always be taken. The first runnings from the filter are in every case to be discarded and used for rinsing the vessel receiving the filtrate.

BRIX:
Before filling hydrometer jars, samples should be well mixed, strained, and to be as near room temperature as possible. Samples should be allowed to stand for at least 20 minutes in the jars, or, in the case of abnormally muddy cane, until all foreign matter shall have subsided. The jar should be filled to overflowing, and it is with the juice in this condition that the Brix hydrometer is inserted, care being taken that it floats freely. A sighting reading should be taken and the stem of the spindle dried down to a little above that point. The spindle is carefully re-inserted to keep the stem dry above the liquid. All readings must be taken with the juice overflowing from the cylinder. The degree indicated at the point where the general level of the liquid would cut the stem if produced, indicates the reading, not at the top of the liquid in contact with the stem. The temperature is taken at the time of reading, and the temperature correction, as shown on Table II applied to the first decimal place only. The standard correction of the hydrometer must also be applied to every reading. "Uncorrected" Brix=Actual reading + standard correction. "Corrected" Brix = Actual reading + standard correction + temperature correction.

Where composite samples are made up, the Brix as measured in these samples is taken for the control.

If the same sample is to be used for polarising, it must be thoroughly mixed after reading the Brix as described.

DIRECT POLARIZATION:
Fill a 100-110 cc. flask to the lower mark with the sample in which the Brix is determined. Clarify with a minimum of basic lead acetate solution and complete the volume with water to 110 ccs. In samples where glucose has to be determined, a minimum of neutral lead acetate solution should be used for clarifying. Shake thoroughly, filter and polarise in a 200 mm. tube.

APPARENT SUCROSE:
The apparent sucrose content will be found on reference to Table III using the uncorrected Brix and direct polarization.

APPARENT PURITY:
The corrected Brix and apparent sucrose as determined in the composite sample, are used for the calculation of the Apparent Purity.

INVERT POLARIZATION:
With the aid of a pipette, place exactly 50 ccs. of the clarified solution used for direct polarization in a 100 cc. graduated flask, add about 25 ccs. of water

and heat in a water bath to 69°C. Remove from the bath and immediately add 10 ccs. of a mixture of equal volumes of concentrated hydrochloric acid and water. Allow to stand for thirty minutes (longer standing does not affect results). Bring to room temperature, make up to 100 ccs., shake, filter if necessary and polarize in a water-jacketed 400 mm. tube. When the solutions are dark coloured after making up to the mark add a small quantity of carbon, or zinc dust, or a few crystals of sulphite of soda. The temperature of the invert solution at the time of polarization should be determined to the nearest 0.1°C. and this temperature used in the calculation of Clerget polarization The direct and invert polarization should be made at the same temperature, a maximum difference of 1°C. being permissible in routine work. The most practicable way is to work at exactly room temperature, using water jacketed tubes for both direct and invert polarizations, filling the jackets with water at room temperature.

CLERGET POLARIZATION:

This is a calculated figure and is obtained by the use of the following general formula:—

$$\frac{100\ (D—I)}{F - 0.5\ T.}$$

Where D and I are the direct and invert polarizations obtained as above, D being the direct polarization in a 200 mm. tube and I the invert polarization in a 400 mm. tube.

F a factor.

T the temperature of the invert solution.

(It should be remembered that I is already a negative quantity).

The Herzfeld factor 142.66, though slightly in error, is retained until the correct factor is more definitely established. The factor changes slightly with differences in concentration. 142.66 is for 13 gms. of sucrose in 100 ccs. The exact factors recommended for specific concentrations are given below:-

HEZFELD'S FACTORS FOR DIFFERENT SUCROSE CONCENTRATIONS:

Gms. Sucrose in 100 ccs.	Factor.	Gms. Sucrose in 100 ccs.	Factor.
1	141.85	8	142.32
2	141.91	9	142.39
3	141.98	10	142.46
4	142.05	11	142.52
5	142.12	12	142.59
6	142.18	13	142.66
7	142.25		

It is urged that each factory determine its own factors at different concentrations, but the above may be retained until this is carried out.

Modifications of the above quantities may be used to give higher polariscope readings where desirable.

CLERGET SUCROSE:

This may be found by reference to Table III, using Clerget polarization and uncorrected Brix.

REDUCING SUGARS:

The volumetric method to be employed, using methylene blue as internal indicator. 10 ccs. of the solution as used for direct polarization, which has been clarified with neutral lead acetate solution is transferred, with the aid of a pipette to a 50 cc. graduated flask. It is diluted with about 20 ccs. with water and ½ cc. of a 10% solution of potassium oxalate added. The volume is completed to 50 ccs., shaken well and filtered, using a little diatomaceous earth, if necessary, as a filter aid. The filtrate is used for titration against Fehling's solution.

By means of a pipette. 5 ccs. of Fehling's solution is placed in a 300 cc. flask and is diluted with a little water so that the volume at the end of the titration will be about 50 ccs. Nearly as much juice as is estimated will reduce the copper is added in one charge in the cold. It is boiled for two minutes, 4 drops of methylene blue solution added, and the titration completed in one minute at boiling temperature. A suitable sand-glass will be found very convenient for timing purposes.

The percentage of reducing sugars in the solution taken for analysis will be found on reference to Table IV.

ACIDITY AND ALKALINITY:

Acidity: 20 ccs. of the solution are titrated against N/32 caustic soda solution, using a few drops of phenolphthalein solution as indicator.

Number of ccs. × 50 = acidity expressed as milligrams of SO_3 per litre.

Alkalinity: 20 ccs. of the solution are titrated against N/28 hydrochloric acid solution, using a few drops of phenolphthalein solution as indicator.

Number of ccs. × 50 = Alkalinity expressed as milligrams of CaO per litre.

SULPHUR DIOXIDE (SO_2):

5 ccs. of the solution are diluted with water and titrated against N/32 iodine solution, using a few drops of starch indicator to determine the end point. The end point may be made sharper by adding a few drops of concentrated hydrochloric acid when near the end point of the titration.

Number of ccs. × 200 = milligrams of SO_2 per litre of sample.

HYDROGEN ION CONCENTRATION (pH)

The comparator tube method is to be used wherever possible, failing which, the spot plate method is specified.

5 ccs. of juice are diluted to 10 ccs. with neutral distilled water and 0.5 cc. of a suitable indicator solution used for the test. In the case of syrup, 2 ccs. are to be diluted as above. It is recommended that the Experiment Station make trials of other methods of determining pH, including indicator and electrometric methods.

THYMOL TEST:

In testing for traces of sugar in condenser water and the like, 2 ccs. of the liquid are to be used with three drops of thymol solution and 2 ccs. of pure

concentrated sulphuric acid. This test may be made very sensitive as a "ring" test, by preserving the acid and aqueous solutions in separate layers, but for general purposes it is used as a colour test by mixing the reagents.

Chapter VI.

ROUTINE ANALYSES

CANE – APPROXIMATE SUCROSE

TEST MILL ANALYSES:

A sample of cane is crushed in a test mill weighing the cane and the resulting bagasse.

Sucrose in bagasse: The bagasse is chopped or disintegrated and its sucrose content determined as described on this page under "Bagasse." This must be done as rapidly as possible as the bagasse loses moisture.

$$\text{Weight of sucrose in bagasse} = \frac{\text{Weight of bagasse} \times \text{sucrose per cent. bagasse}}{100}$$

Sucrose in Juice: The method of determination is identical with that described below under Factory Samples for first crusher juice.

Weight of juice = Weight of cane – Weight of bagasse.

$$\text{Weight of sucrose in Juice} = \frac{\text{Weight of Juice} \times \text{Apparent sucrose in Juice}}{100}$$

Approximate Sucrose in Cane: Weight of sucrose in cane = Weight of sucrose in bagasse + weight of sucrose in juice.

$$\text{Sucrose \% cane} = \frac{\text{Weight of sucrose in cane} \times 100.}{\text{Weight of Cane.}}$$

FACTORY SAMPLES:

(First crusher juice for approximate sucrose in cane).

An average sample of the first crusher juice from the cane under examination is tested as follows:—

Brix: Special care should be taken to ensure that the juice is well sieved and settled, and all occluded air removed before determining the Brix as detailed under "Prix" on page 9.

Polarization: For the polarization a minimum of basic lead acetate solution is to be used for clarifying, the actual procedure being as described under "Direct Polarization," page 9.

Apparent Sucrose: This is found by reference to Table III using the polarization and the uncorrected Brix.

Approximate sucrose in cane: This is determined by applying the Java Ratio of the previous week's crushing.

$$\text{Approximate sucrose in cane} = \frac{\text{Sucrose in Crusher Juice} \times \text{Java Ratio}}{100}$$

MILL CONTROL ANALYSES:

BAGASSE:

Sucrose: Sucrose is determined in the hourly sample. 520 grams of bagasse are weighed and loosely packed in the basket of the bagasse apparatus. The basket is inserted in the apparatus and 3740 grams of hot water containing 20 ccs. of a 5% solution of sodium carbonate added. The plate is placed on the bagasse, and the cover and condenser attached, care being taken that the cover forms an air tight joint with the flange. The water is brought to the boil, and allowed to simmer for 30 minutes. Care must be taken that the condenser is efficient and that no vapour is lost. Cool with condenser still attached to about 70°C. By means of the cock withdraw sufficient liquid for the completion of the test, rejecting the first portion, and cool to room temperature. Clarify with a minimum of powdered neutral lead acetate; filter and polarize in a 400 mm. tube. The polarization shows direct the percentage of sucrose in bagasse. Modifications of the above quantities may be used to give a direct reading in a 600 mm. tube.

If it is so desired, the basket portion of the apparatus may be dispensed with, and the weighed sample of bagasse placed directly in the apparatus.

Moisture: Every two hours the moisture in bagasse is determined in a catch sample taken directly from the mill. Not less than 100 grams of bagasse are weighed into a tray 8 inches by 8 inches by ¾ inch deep, made of suitable gauze. It should be dried to constant weight at a temperature not exceeding 120°C.

FIRST CRUSHER JUICE:

The sample at the time of sub-sampling is tested for Brix. A composite sample is made up every four hours and tested as follows:

Brix: As described under "Brix" page 9.

Polarization: This is determined as described under "Direct Polarization," page 9, using a minimum of basic lead acetate solution for clarifying.

Apparent Sucrose: The uncorrected Brix and polarization are used for finding apparent sucrose by reference to Table III.

Apparent Purity: The corrected Brix and apparent sucrose, as determined in the composite sample, are used for the calculation of apparent purity.

Hydrogen Ion Concentration (pH): This is determined in the four-hour composite sample as detailed under "Hydrogen Ion Concentration," page 10.

LAST ROLLER JUICE:

The hourly sample at the time of sub-sampling is tested for Brix. A composite sample is made up every four hours and tested as follows:—

Brix: As under "Brix," page 9.

Polarization: A minimum of basic lead acetate solution is used for clarifying, the procedure being as detailed under "Direct Polarization," page 9.

Apparent Sucrose: This is found by reference to Table III using the polarization and uncorrected Brix.

Apparent Purity: This is calculated from the corrected Brix and the apparent sucrose, as determined in the composite samples.

MIXED JUICE:

The hourly mill sample is tested for Brix at the time of sub-sampling. A more complete analysis is carried out on a composite sample which is made up every 4 hours.

Brix: As described under "Brix," page 9.

Direct Polarization: This is determined as detailed under "Direct Polarization," page 9 using a minimum of neutral lead acetate solution for clarifying.

Invert Polarization: This is determined as detailed under "Invert Polarization," page 9 using a minimum of neutral lead acetate solution for clarifying.

Clerget Polarization: This is calculated as described on page 10.

Clerget Sucrose: The Clerget polarization and uncorrected Brix are used for finding Clerget sucrose by reference to Table III.

Clerget Purity: Clerget sucrose and the corrected Brix are used for the calculation of Clerget purity.

Reducing Sugars: Part of the filtrate used for direct polarization as above is used for the determination of reducing sugars, proceeding as detailed under "Reducing Sugars" page 10.

The hourly catch sample of the sulphited mixed juice is analysed as follows:—

Acidity: As detailed under "Acidity," page 10. This determination is only of value when sulphitation is practised before liming.

Sulphur Dioxide: This is determined as described under "Sulphur Dioxide," page 10.

CLARIFIED JUICE:

A composite sample is tested every 4 hours as follows:—

Brix: As under "Brix" page 9.

Polarization: As detailed under "Direct Polarization" page 9 using a minimum of neutral lead acetate for clarifying.

Apparent Sucrose: As shown on Table III using polarization and the uncorrected Brix.

Apparent Purity: Calculated, using the corrected Brix and the apparent sucrose.

Reducing Sugars: Use a portion of the filtrate from the direct polarization and proceed as described under "Reducing Sugars," page 10.

Acidity or Alkalinity: A portion of the composite sample is titrated with N/32 caustic soda solution, or N/28 hydrochloric acid solution, using phenolphthalein as indicator as detailed under "Acidity and Alkalinity," page 10.

pH: This is determined in the composite sample by the method described under "Hydrogen Ion Concentration," page 10.

Where clarified juice is weighed for the sucrose balance, Invert Polarization, Clerget polarization, and Clerget sucrose should be determined by the methods detailed under those headings on pages 9 and 10.

SCUMS JUICE AND FILTER PRESS JUICE:

A composite sample is made up every four hours. Where the scums are double pressed, the juice from the two processes should be analysed and recorded separately.

Brix: As detailed under "Brix," page 9.

Polarization: A minimum of basic lead acetate solution is used for clarifying, the procedure being as detailed under "Direct Polarization," page 9.

Apparent Sucrose: Refer to Table III using the uncorrected Brix and polarization.

Apparent Purity: This is calculated, using the corrected Brix and the apparent sucrose.

Hydrogen Ion Concentration (pH): As detailed under "Hydrogen Ion Concentration," page 10.

Acidity or Alkalinity: As described under the same heading, page 10.

FILTER PRESS CAKE:

A composite sample is analysed every four hours.

Sucrose: 25 grams of the sample are triturated with water in a mortar and washed with water into a 200 cc. graduated flask. 5 ccs. of basic lead acetate solution are added, and the volume of liquid made up to the 200 c.c. mark. The contents of the flask are thoroughly mixed by shaking, filtered, and polarized in a 400 mm. tube. The reading gives direct the sucrose per cent press cake.

Moisture: For moisture determination, 20 grams of the sample are dried in a flat bottomed dish at a temperature not exceeding 105°C.

SYRUP:

The composite sample is tested every 4 hours. A quantity of the sample is accurately diluted with twice its weight of water. This diluted sample is used for all determinations except pH.

Brix: As detailed under Brix, page 9. The corrected Brix multiplied by 3 represents Brix of the original sample.

Polarization: As described under "Direct Polarization" page 9, using a minimum of neutral lead acetate for clarifying.

Apparent Sucrose: This is determined in the diluted sample by reference to Table III using the uncorrected Brix and polarization. This value multiplied by 3 represents the apparent sucrose of the original sample.

Apparent Purity: The corrected Brix and the apparent sucrose, as determined in the composite sample, are used for this calculation.

Reducing Sugars: A portion of the filtrate used for polarization is used, the procedure being as detailed under "Reducing Sugars," page 10. The figure shown on Table IV multiplied by three represents the reducing sugars in the original sample.

Hydrogen Ion Concentration (pH): This is determined on the original composite sample of syrup, as described under "Hydrogen Ion Concentration," page 10.

Where syrup is weighed for additional sucrose balance, Invert Polarization, Clerget Polarization and Clerget Sucrose are determined as described under those headings on pages 9 and 10.

MASSECUITES:

From every strike, a sample is accurately diluted with four times its weight of water. Care should be taken that all crystals of sugar are dissolved. If hot water has been used for this purpose, the solution should be brought to about room temperature before proceeding with the analysis.

Brix: As described under "Brix," page 9. Multiply the corrected Brix by 5 to represent the original sample.

Polarization: The solution should be clarified with a minimum of basic lead acetate solution, proceeding as detailed under "Direct Polarization," page 9.

Apparent Sucrose: This is obtained by reference to Table III using the uncorrected Brix and polarization.

Apparent Purity: The apparent purity is calculated using the corrected Brix and the apparent sucrose.

MOLASSES OR RUN-OFFS:

Samples should represent, as near as possible, the run-off from individual massecuites.

Apparent Purity: The apparent purity may be rapidly determined by diluting a quantity of the sample with water to about 16° Brix. A 100-110 cc. flask is filled with the solution to the lower mark. The necessary minimum amount of basic lead acetate solution is added, and the volume completed to the higher mark with water. The contents of the flask are well shaken, filtered and polarized. If the liquid is too dark to be read in a 200 mm. tube, use a 100 mm. tube, multiplying the reading by 2. Its apparent sucrose content is found by reference to Table III using the uncorrected Brix and polarization. Apparent purity is calculated using the corrected Brix and apparent sucrose of the diluted sample.

FINAL MOLASSES:

Test analyses may be carried out as for ordinary molasses as desired.

For sucrose balance a weekly composite sample is made up. After thoroughly mixing, a quantity of this sample is accurately diluted with four times its weight of water. This diluted solution is used for the following determinations.

Brix: This is measured as described under "Brix," page 9. The corrected reading multiplied by 5 represents the Brix of the original sample.

Direct Polarization: A minimum of basic lead acetate solution is used for clarifying, proceeding as described under "Direct Polarization," page 9. If the filtrate prepared in the ordinary way, and clarified with 10 ccs. of basic lead acetate solution is found to be too dark to be read in a 200 mm. tube, 50 ccs. of the original diluted (1:5) solution can be similarly treated with a proportional quantity of clarifying agent as required, and the volume made up to 110 ccs. with water. If possible, the direct polarization should be carried out according to the standard method, to give a higher reading on the saccharimeter scale.

Invert Polarization: This is determined as detailed under "Invert Polarization," page 9. 1 cc. of the dilute hydrochloric acid should be added in all tests before heating. When solutions are dark coloured after making up to the 100 cc. mark, add a small quantity of zinc dust or carbon, or a few crystals of sodium sulphite. If carbon is used, the first 15 ccs. of the filtrate are to be rejected.

Clerget Polarization: This is calculated from the direct and invert polarizations as described under Clerget polarization, page 10.

Clerget Sucrose: The Clerget sucrose is found from Table III, using Clerget polarization and uncorrected Brix. Multiply this figure by 5, to represent the Clerget sucrose of the original sample.

Clerget Purity: The corrected Brix and Clerget sucrose as determined in the composite sample are used for the calculation of Clerget purity.

Reducing Sugars: 10 ccs. of the solution used for the determination of Brix (diluted 1 to 5) are diluted to about 100 ccs. with water, 1 cc. of potassium oxalate solution added, and the volume completed to 220 cc. The solution is filtered and titrated against 5 ccs. of Fehling's Solution as detailed under "Reducing Sugars," page 10. The reducing sugars in the diluted sample are determined by

reference to Table IV and when multiplied by 20, gives the amount of reducing sugars per 100 gms. of original molasses.

Ash: Ignite 2 grams in a platinum vessel to constant weight at not more than a faint red heat. The heating must be carefully controlled, especially in the earlier stages.

SUGAR:

Polarization and moisture are determined as often as desired. In the weekly composite sample, the following determinations are made.

Polarization or Sucrose: The normal weight (26.00 grams) is weighed out, transferred to an accurately calibrated 100 cc. flask and dissolved in about 70 cc. of water. If hot water has been used for this purpose, cool to room temperature, clarify and make up to the 100 cc. mark with water.

For white sugar, use 2 drops of basic lead acetate solution and ½ cc. of alumina cream for clarifying.

For raw sugar clarify with about 2 cc. of basic lead acetate solution, varying the quantity with the grade of sugar.

For low grade sugar, about 5 ccs. of basic lead acetate solution will be necessary.

The solution should be well shaken and filtered in a covered vessel to prevent evaporation. Polarize in a 200 mm. tube. The reading gives direct the polarization or sucrose content.

Moisture: 10 gms. of sugar are accurately weighed in a small dish or watch glass, and dried to constant weight in an oven at a temperature not exceeding 105°C.

Weight lost × 10 = Moisture per cent sugar.

Gravity Purity: This figure is required for the calculation of available sugar. A quantity of the composite sample is dissolved in exactly 5 times its weight of water.

Brix: This is determined as described under "Brix," page 9.

Apparent Sucrose: As detailed under "Direct Polarization" page 9.

Gravity Purity of Sugar: This is calculated from the Brix and apparent sucrose of the solution of the sugar prepared as above.

ACKNOWLEDGEMENTS:

It will be seen that there is relatively little that is original in the foregoing methods, which are largely compilations of such existing methods and specifications as appeared to be most suitable for our own conditions.

We are indebted to the following publications which have been more or less frequently drawn upon for information and assistance:—

Various circulars issued by the Bureau of Standards, Department of Commerce, Washington, D.C., U.S.A.

"Methods of Chemical Control for Cane Sugar Factories," by the Association of Hawaiian Sugar Technologists, 1923.

"General Instructions & Methods of Analysis and Chemical Control for Use in the Factories of the Cuban—American Sugar Co." by G. L. Spencer.

"A Handbook for Cane Sugar Manufacturers and their Chemists," by G. L. Spencer.

"Chemical Control in Cane Sugar Factories," by H. C. Prinsen Geerligs, Amsterdam.

"The Determination of Hydrogen Ions," by W. Mansfield Clark.

"Volumetric Analysis," by F. Sutton.

TABLES FROM I TO IV & LABORATORY REPORT FORM (OVERLEAF)

As the paper was such a lengthy one, it was decided that Mr. Jacobs should summarise the main points thereof for the benefit of the members.

Mr. Dodds then suggested that the discussion on the paper be postponed until members had had an opportunity of going through it. It was fully intended to circulate this before the meeting, and as Secretary of the Committee he was sorry it had not been done, circumstances having rendered it quite impossible. If the discussion could be postponed until tomorrow by which time members would have a chance of getting a copy and reading it, it would give a basis for discussion which did not exist at present.

The Chairman agreed with the suggestion. He did not call it a paper; it was more like a book. He certainly felt it would be difficult to read a paper like that and have it discussed at that meeting, in the way it should be. It was really a report which should be in every laboratory for everyday reference.

Mr. Jacobs seconded Mr. Dodds' proposal.

The Chairman remarked that so far as he could see it was going to be the same with the other papers.

It was then agreed that the discussion on the paper be postponed to the following day, whereupon the Chairman suggested that Mr. Dodds should read the summary of the previous year's work, which had been compiled by the Experiment Station. Mr. Dodds prefaced the summary by saying that it was the second annual summary of Chemical Laboratory Reports from Sugar Factories in South Africa and that the first one was presented at the Sugar Congress in April last year: