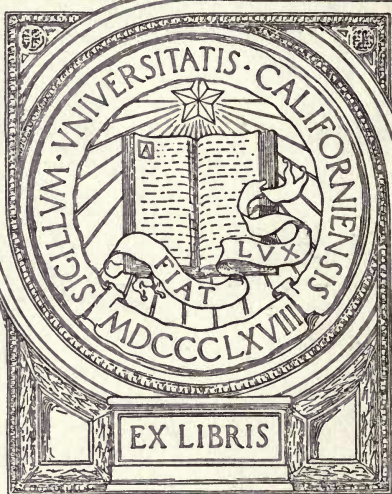


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SUGAR ANALYSIS

FOR CANE-SUGAR AND BEET-SUGAR
HOUSES, REFINERIES AND EX-
PERIMENTAL STATIONS

AND AS A

HANDBOOK OF INSTRUCTION IN SCHOOLS OF
CHEMICAL TECHNOLOGY

BY

FERDINAND G. WIECHMANN, PH.D.

THIRD EDITION, REWRITTEN

FIRST THOUSAND

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JOSEPH A. DEGHUÉE, Ph.D.

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PREFACE

IN preparing this, the third edition of his Sugar Analysis, the writer has endeavored to cast his material in a form in which it would prove most readily available in the several branches of the sugar industry.

With this aim in view the whole range of the subject—the methods and means used in the analysis of sugar and in the analysis of the materials used in sugar production, have first been fully discussed, and then the specific analytical control of cane-sugar manufacture, of beet-sugar manufacture, and of refining, has been taken up for detailed consideration.

In adopting this method of treatment a certain amount of repetition has been unavoidable. This however has been reduced to a minimum and, it is believed, that the greater convenience for practical use thus gained fully warrants the procedure. Although the International Commission for Uniform Methods of Sugar Analysis adopted—in 1897—26.00 grams for 100 metric cubic centimeters, at 20° C., as the normal sugar weight, yet the American sugar industry, to a large extent, still retains as normal weight 26.048 grams for 100 Mohr cubic centimeters.

For this reason both standards appear in this work; universal adoption of the Commission's standard is however strongly urged as an important step towards the unification of analytical methods in the industry. The

brief résumé of the Commission's work to date, will, it is hoped, prove of interest.

In the preparation of this book free use has been made of the publications of standard authorities and endeavor has been made to give due credit in every instance. The writer would however here once more specifically express his obligations to the work of Browne, Deerr, Herzfeld, Horne, Prinsen Geerligs, Spencer and von Lippmann.

F. G. W.

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SUGAR ANALYSIS

CHAPTER I

PROPERTIES OF SUCROSE

SUGAR, which is chemically known as sucrose or saccharose, is a compound of the elements carbon, hydrogen and oxygen. Its composition is expressed by the formula $C_{12}H_{22}O_{11}$, showing it to consist of 42.11% carbon, 6.43% hydrogen and 51.46% oxygen.

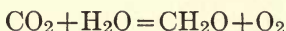
It is a product of the vegetable kingdom and occurs in many plants; it is found in leaves, stalks, fruits, seeds, grains etc. in widely varying amounts. The only plants however which produce it in sufficient quantity to serve as sources for its extraction on a commercial scale are the sugar cane, the sugar beet, the sugar maple and the palm; and to a very much smaller degree Indian corn (maize) and sorghum have also at times figured as sources of supply.

Placing the world's yearly production of sucrose at about 16 million long tons, at present approximately 53% of this is obtained from the sugar cane, and the greater part of the balance from the sugar beet.

Sugar is formed in plants from water taken from the soil and from carbonic acid gas (carbon dioxide) taken from the air, through the intervention of chlorophyll, the green coloring matter of plants. This chlorophyll transmutes

the energy of sunlight into chemical energy and this latter brings about the chemical combination of the carbon dioxide and the water to form sucrose. This reaction is termed one of assimilation; oxygen is set free in the process.

Baeyer in 1870 suggested that the first step in this process is the formation of formaldehyde as illustrated by the reaction,



in which the chemical symbols given represent respectively carbon dioxide, water, formaldehyde and oxygen.

It seems probable that the formaldehyde so formed suffers a condensation or polymerization to $\text{C}_6\text{H}_{12}\text{O}_6$, glucose, but this is a question upon which there is as yet a wide divergence of opinion, some holding that starch, others that sucrose is the first carbohydrate to be formed; however it seems likely that starch is an intermediary product in the formation of sucrose.

Sucrose is created in the leaves of plants and is thence distributed to other parts of the plant to be stored there as such, or transformed into other substances.

Pure sucrose crystallizes in monoclinic crystals; impurities however can greatly modify these forms, raffinose in particular, causes a pointed distortion of the normal tabular form. Sucrose crystals do not affect the plane of polarized light, whereas the power which aqueous sucrose solutions have of rotating polarized light has been made the basis of one of the most delicate and accurate methods for the determination of this substance.

Sucrose is soluble in water and in dilute alcohol, slightly soluble in boiling absolute alcohol, insoluble in ether.

At 20° C. 100 grams of water dissolve 203.9 grams of sucrose; at 25° C. 211.4 grams, at 30° C. 219.5 grams.*

* For a detailed statement of the solubility of sucrose in water see Herzfeld's table in Browne's Handbook of Sugar Analysis, p. 649.

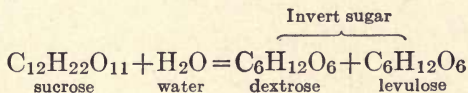
Whereas pure water, of about 20° C., will hold in solution only about 40 parts of sucrose, a beet molasses containing 20% of water will dissolve 50 parts, and a cane molasses of equal water content only 30 parts of sucrose. This is explained by Dubrunfaut, Von Lippmann, Prinsen Geerligs and others by the fact that in beet molasses the salts prevent the sucrose from crystallizing, whereas, in cane molasses, the organic salts enter into combination with the invert sugar, a constituent of cane molasses, but essentially absent from beet molasses, and that this combination of invert sugar and organic salts binds a considerable amount of water of hydration and thus lowers the amount of free water in which the sucrose would otherwise dissolve.

Sucrose is destroyed by heat. Its melting-point lies at about 170° C. If heated beyond this point it undergoes decomposition into a variety of bodies prominent among which is caramel, itself a mixture of several substances, notably of caramelane, caramelene and carameline.

Solutions of sucrose suffer changes even when heated only at 100° C. for some time, at higher temperatures the destruction of sucrose is rapid.

Acids, metallic salts and enzymes invert sucrose solutions, that is to say, by their influence the sucrose is caused to combine with water and is transformed into invert sugar, a substance which consists of equal parts of glucose and fructose, or dextrose and levulose, as they are frequently termed.

The inversion of sucrose by acids is chemically thus represented:



As stated, by this transformation the sucrose which has a pronounced dextrorotation is changed into invert sugar

which has a levorotation, that is to say which turns the plane of polarized light to the left instead of to the right.

Inversion of sucrose can be effected by minute amounts of acids; the rate at which the inversion progresses is always proportional to the concentration of the reacting substance.

Different acids at a given temperature have different inverting powers; hydrochloric, nitric and methyl sulphuric acids have the same inverting power, this is usually taken as the standard. Sulphuric acid has an inverting power about 54% of this standard, oxalic acid about 19%, and phosphoric acid only about 6%. A rise in temperature increases the inverting power of acids materially. Metallic salts show an analogous behavior; as a rule, the strongest inverting action is shown by the salts of the strongest acids.

Invertase is a white powder which readily dissolves in water. It is prepared from yeast, and has a very powerful inverting action on sucrose, acting in this respect purely as a catalyser, and causing the addition of a molecule of water to each molecule of sucrose.

Sucrose readily forms compounds with metallic bases, notably with the alkalis and with the alkaline earths. Such compounds are known as sucrates or saccharates. Technically the most important are the sucrates of lime of which three are known in which, respectively, one, two or three molecules of lime, CaO , are combined with one molecule of sucrose. The latter compound is made in the process of Steffens which is largely employed in the desucration of beet molasses.

Strontium monosucrate and bisaccharate are also well known, and in some countries the use of barium is also resorted to for reclaiming sucrose from molasses although in other countries its use for this purpose is forbidden on account of the poisonous nature of some of the barium salts.

CHAPTER II

INSTRUMENTS USED IN SUGAR LABORATORIES

Refractometers. Among the first to introduce the use of the refractometer in the sugar industry was Hugh Main, who in 1906 first called the attention of the members of the International Commission for Uniform Methods of Sugar Analysis to its possibilities in this connection.

The description of an Abbé refractometer with heatable prisms, which follows, has been abstracted from a circular of Carl Zeiss, Jena, a manufacturer of this form of refractometer.

The refractive index n_D of the solution under examination is read off directly from a graduated circle after a simple adjustment, for the method of measurement is based upon the observation of the position of the *border line of the total reflection* in relation to the faces of a prism of flint-glass, into which the light from the substance under investigation enters by the action of refraction.

The refractometer is essentially composed of the following parts:

1. The double Abbé prism, which contains the fluid and can be rotated on a horizontal axis by means of an alidade.

2. A telescope for observing the border line of the total reflection formed by the prism.

3. A sector, rigidly connected with the telescope, on which divisions—representing refractive indices, are engraved.

The double prism consists of two similar prisms of flint-glass, each cemented in a metal mount and having a refractive index $n_D=1.75$, the fluid to be investigated—a few drops, is deposited between the two adjoining inner faces of the prisms in the form of a thin stratum (about 0.15 m.m. thick). The former of the two prisms, that farther from the telescope (which can be folded up or be removed),

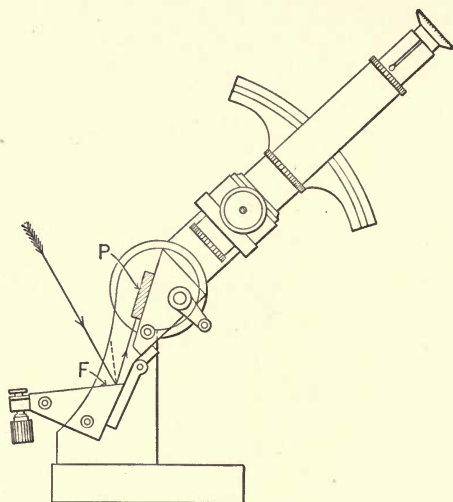


FIG. 1.

serves solely for the purpose of illumination, while the border line is formed by the second flint prism.

The *border line* is brought within the field of the telescope by rotating the double prism by means of the alidade in the following manner. Holding the sector, the alidade is moved from the initial position, at which the index points to $n_D=1.3$, in the ascending scale of the refractive indices until the originally entirely illuminated field of view is encroached upon, from the direction of its lower half, by a dark portion; the *line dividing* the bright and

the dark half of the field then is the "border line." When, daylight or lamp light being employed, the border line, owing to the total reflection and the refraction caused by the second prism, assumes at first the appearance of a *band of color*, which is quite unsuitable for any exact process of adjustment. The conversion of this band of color into a colorless line, sharply dividing the bright and dark portions of the field, is the work of the compensator.

The compensator, which has its place in the prolongation of the telescope tube beyond the objective, i.e., at a point between the objective and the double prism, consists of two similar Amici prisms, of direct vision for the *D*-line and rotated simultaneously, though in opposite directions, around the axis of the telescope by means of a screw-head. In this process of rotation the dispersion of the compensator passes through every value from zero (when the refracting edges of the two Amici prisms are parallel and on different sides of the optical axis) up to double the amount of the dispersion of a single Amici prism, the refracting edges of the two Amici prisms being parallel and on the same side of the axis. The above-mentioned dispersion of the border line, which appears in the telescope as a band of color, can thus be annulled by rotating the screw-head, thereby giving the compensator an equal, but opposite, dispersion. The opposite equal dispersions will then neutralize each other, with the result that *the border line appears colorless and sharply defined*.

The border line is now adjusted upon the point of intersection of the reticle by slightly inclining the double prism to the telescope by means of the alidade. The position of the pointer on the graduation of the sector is then read off by the aid of the magnifier attached to the alidade. *The reading supplies the refractive index n_D of the substance under investigation itself*, without any computation, and with a degree of exactness approaching to within about 2 units of the fourth decimal. Simultaneously the *reading* of the

scale on the drum of the *compensator* enables the *mean dispersion* $n_F - n_C$ being arrived at by the aid of a special table and a short process of computation. The accuracy of the measurement of dispersion is increased by taking the mean of two readings of the drum varying by 180 degrees.

As the refractive index of fluids varies with their temperature, it is of importance to know the temperature of the fluid contained in the double prism during the process of measurement.

If, therefore, it be desired to measure a fluid with the highest degree of accuracy attainable with the Abbé refractometer (to within 1 or 2 units of the fourth decimal of n_D), it is absolutely necessary to bring the fluid, or rather the double prism containing it, to a definite known temperature and to be able to control this temperature so as to keep it constant to within some tenths of a degree for a period of several hours; hence a refractometer principally required for the investigation of fluids must be provided with heatable prisms.

The double prism is opened out and closed up by means of a screw-head, which acts in the manner of a bayonet catch. In order to apply a small quantity of fluid to the prisms, without opening the casing, the screw is slackened and a few drops of fluid poured into the funnel-shaped mouth of a narrow passage. On again tightening the screw, the fluid is distributed by capillary action over the entire space between the two prisms. This arrangement facilitates the investigation of even rapidly evaporating fluids. Ordinarily, a drop of moderate size is applied with a glass rod to the dull prism surface, the double prism being opened out for the purpose. The prisms are then closed up again and before the measurement is proceeded with, the refractometer is left standing for a few minutes in order to compensate any cooling or heating of the prisms which may have occurred while they were separated.

The fitting for heating the prisms of the refractometer

is constructed in its essential parts on Dr. R. Wollny's plan by enclosing the prisms in a metal casing with double walls, through which water of a given temperature is circulated. The thermometer provided registers the temperature of the water circulating through the prism-casing at its point of exit. Attainment of a constant temperature in the current of water is necessary.

TESTING THE ADJUSTMENT OF THE REFRACTOMETER

Each refractometer is supplied with a testing plate made of glass of known refractive index, the value of which is marked on the upper ground surface of the plate. The plate has two plane polished surfaces which form a sharp edge. For the purpose of testing the refractometer the refractive index of the plate P is very accurately measured by applying it several times in succession, the prism surface being wiped clean after each measurement. The mean of the several measurements is compared with the value stated on the plate, and if agreement is found in the fourth decimal, the adjustment of the refractometer may be considered satisfactory.

Should the mean of several such measurements show a concordant deviation of several units in the fourth decimal, the refractometer should be adjusted in the following manner.

The testing plate is placed in position, and the index is set exactly at the value n_D engraved in the plate. The key is then slipped over the small square bar situate on the front side of the telescopic-tube and turned until the borderline exactly crosses the point of intersection of the hair-lines. Finally, the new adjustment thus secured is verified in the usual way by finding the borderline and taking the readings.

In using the refractometer for sugar solutions it must be borne in mind that its values really represent rather a

determination of the total substances in solution, that is to say, the total soluble solids, and that 100 minus these total solids is water. In other words, index of refraction values are essentially of the same order as specific gravity values, and are subject to the same limitations. The purer a sugar solution is, of course, the closer is the approach of the refractive index values to the true sucrose content of the solution.

The refractive index of sugar solutions of different concentrations, at the temperature of 17.5° C., was published by Stolle in 1901.*

In 1906 Tolman, and Smith worked out such a table for sugar solutions at 20° C.† This follows:

% Sucrose.	Index of Refraction 20° C.	% Sucrose.	Index of Refraction 20° C.	% Sucrose.	Index of Refraction 20° C.	% Sucrose.	Index of Refraction 20° C.
1	1.3343	24	1.3705	47	1.4137	70	1.4653
2	1.3357	25	1.3722	48	1.4158	71	1.4677
3	1.3372	26	1.3739	49	1.4179	72	1.4701
4	1.3387	27	1.3756	50	1.4200	73	1.4726
5	1.3402	28	1.3774	51	1.4221	74	1.4751
6	1.3417	29	1.3792	52	1.4242	75	1.4776
7	1.3432	30	1.3810	53	1.4263	76	1.4801
8	1.3447	31	1.3828	54	1.4284	77	1.4826
9	1.3462	32	1.3847	55	1.4306	78	1.4851
10	1.3477	33	1.3865	56	1.4328	79	1.4877
11	1.3492	34	1.3883	57	1.4351	80	1.4903
12	1.3508	35	1.3902	58	1.4373	81	1.4929
13	1.3524	36	1.3921	59	1.4396	82	1.4955
14	1.3539	37	1.3940	60	1.4419	83	1.4981
15	1.3555	38	1.3959	61	1.4442	84	1.5007
16	1.3572	39	1.3978	62	1.4465	85	1.5034
17	1.3588	40	1.3997	63	1.4488	86	1.5061
18	1.3604	41	1.4017	64	1.4511	87	1.5088
19	1.3621	42	1.4036	65	1.4534	88	1.5115
20	1.3637	43	1.4056	66	1.4557	89	1.5142
21	1.3654	44	1.4076	67	1.4581	90	1.5170
22	1.3671	45	1.4096	68	1.4605		
23	1.3688	46	1.4117	69	1.4629		

* Zeitschrift d. V.R.Z. Ind., Vol. LI, pp. 335 and 469.

† Journal Am. Chem. Soc., 1906, p. 1480.

By aid of the following table corrections may be made for observations taken at temperatures other than 20° C. The reading of the index of refraction is made at room-temperature and this reading is calculated to per cent sucrose, then the proper correction from the table is determined and applied.

% Sucrose.	Index at 20° C.	Index at 30° C.	Difference.	Correction for 10° C.	
				In % Sucrose.	In % Sucrose from Brix Table.
2.18	1.3358	1.3348	0.0010	0.66	0.64
7.43	1.3438	1.3428	0.0010	0.66	0.67
15.82	1.3569	1.3557	0.0012	0.70	0.70
51.71	1.4236	1.4219	0.0017	0.81	0.79
62.52	1.4477	1.4459	0.0018	0.78	0.76

A refractive-index table of sugar solutions at 28° C., was worked out by Prinsen Geerligs and Van West in 1907,* and this table gives values which are practically identical with those obtainable from Tolman and Smith's table by aid of the temperature-correction factors above given.

Hugh Main's table† is calculated for the temperature of 20° C. and is slightly at variance with the values given by Tolman and Smith.

Among recent experimental contributions on the refractive indices of sugar solutions are those made by Otto Schoenrock and by V. Stanek.

Schoenrock's values‡ show a close agreement with those of Main previously referred to; Schoenrock also furnishes a table of temperature corrections for temperatures varying from 10° to 30° C. and for water contents from 30% to 100%.

* Prinsen Geerligs, *De Fabricatie van Suiker*, etc., 1911, p. 11.

† *International Sugar Journal*, 1907, p. 481.

‡ *Zeitschrift der Deutschen Zucker Industrie*, 1911, pp. 421-425. *International Sugar Journal*, 1911, Vol. 13, p. 398.

Stanek's work* deals with the determination of water in raw sugars by the immersion refractometer and shows very promising results are to be obtained with this type of instrument.

Balances and Weights. For weighing out samples for polarization, a balance capable of weighing up to 300 grams and sensible to 1 milligram will answer. For water and ash determinations an analytical balance should be used; this should be sensible to 0.1 of a milligram, and be capable of bearing a charge up to 200 grams.

A good balance should give the same result in successive weighings of the same body; the two halves of the beam should be of equal length; the balance should be sensible to a small load, and should work quickly.

It is an easy matter to determine whether a balance possesses these properties. Repeated weighings of the same load will quickly establish whether the balance is consistent with itself; this depends principally on the truthness of its knife-edges.

To determine whether both halves of the beam are of the same length, the two pans should be loaded with equal weights. If the arms are of unequal length, the pan attached to the longer arm will descend.

To test the sensibility, load both pans with the maximum weight which they are intended to bear, and then add to one of the pans the weight to the extent of which the balance is supposed to be sensible. The addition of this slight extra weight should cause the pan on which it has been placed to descend.

The weights used, both the regular weights for analytical purposes, and the so-called sugar-weights, normal and half normal, should be verified from time to time, as they will, in daily use, unavoidably suffer some wear and tear.

* International Sugar Journal, 1911, Vol. 13, p. 90.

	For use with metric c.c. flasks.	For use with Mohr c.c. flasks.
Normal sugar-weight	=26.000 grams.	26.048 grams.
Half-normal sugar-weight	=13.000 grams.	13.024 grams.

Most of the weights are so made that the plug or stopper unscrews from the body of the weight, and slight deficiencies in weight can readily be corrected by inserting tin-foil or small shot into the cavity after removing the plug. Should the weights be too heavy, a little filing will readily remedy the evil.

Graduation and Calibration of Measuring Apparatus.

Detailed instruction for the calibration of volumetric apparatus is given in an article by Morse and Blaloch, Amer. Chem. Journ. Vol. XVI, p. 479, and in: "The Testing of Glass Volumetric Apparatus," by N. S. Osborne and B. H. Veazey, Reprint No. 92 from Bulletin of the Bureau of Standards, Vol. IV, No. 4, Washington, D. C., 1908, the following directions will however suffice for practical laboratory needs.

Graduation of Pipettes. The pipette to be tested for its accuracy is filled to the exact mark with water of the temperature at which the pipette has been graduated, the water is then run into a tared beaker, which of course must be perfectly clean and dry, the pipette allowed to drain in touch with the beaker and the beaker with its contents weighed at once.

Graduation of Burettes. The burette is placed in an upright position, filled with water of the temperature at which the burette was graduated and successive quantities, 2, 5 or 10 c.c. at a time are then run into a clean and perfectly dry beaker and quickly weighed.

Graduation of Flasks. Sugar flasks are graduated either in metric cubic centimeters, or in Mohr cubic centimeters.

A metric cubic centimeter is the space occupied by the mass of 1.000 gram of water weighed in vacuo at the temperature 4° C., the temperature of maximum density of water.

At 20° C. the metric (true) cubic centimeter is equiv-

alent to the volume occupied by 0.998234 gram of water weighed in vacuo, or 0.997174 gram of water weighed in air with brass weights.

The International Commission has officially adopted the metric cubic centimeter as the standard unit of volume for sugar flasks. A 100 metric c.c. flask at 20° C. must contain 99.7174 grams of distilled water weighed in air with brass weights.

A Mohr cubic centimeter is the space occupied by the mass of 1.000 gram of distilled water at the temperature of 17.5° C. weighed in air with brass weights.

Sugar flasks are graduated in Mohr c.c. by weighing into the dry flask, in air with brass weights, exactly 100 grams of distilled water having the temperature of 17.5° C.

To ascertain the weight of water which at t° C. shall be of the same volume, i.e. 100 c.c., as 100 grams of water at 17.5° C., use the formula:

$$100 \times \frac{\text{density of water at } t^{\circ} \text{ C.}}{\text{density of water at } 17.5^{\circ} \text{ C.}}$$

In order to be able to graduate or to verify graduated Mohr flasks at temperatures other than 17.5° C., the following convenient table of the weight of water of different

Temperature. Degrees C.	Weight of 50 c.c. after Mohr. 17.5° = 1.	Weight of 100 c.c. after Mohr. 17.5° = 1.
25.0	49.916	99.833
25.5	49.910	99.820
26.0	49.903	99.807
26.5	49.896	99.793
27.0	49.890	99.780
27.5	49.883	99.767
28.0	49.877	99.754
28.5	49.870	99.740
29.0	49.863	99.727
29.5	49.857	99.714
30.0	49.850	99.701

temperatures, also weighed in air with brass weights, is given by Prinsen Geerligs.*

Hydrometers. The hydrometers used in the analysis of saccharine solutions embrace specific-gravity hydrometers and instruments graduated according to an arbitrary scale. To the latter belong the Baumé hydrometers, and the Brix or Balling spindles. The degrees of a Brix hydrometer indicate percentage by weight of sugar when immersed in a solution of pure sugar.

The suggestion has been made to replace the Baumé scale by a scale graduated in so-called densimetric degrees. These values are found by taking the specific gravity corresponding to any given Baumé degree, ignoring the unit, and dividing the decimals by 100

Example.

Baumé Degrees.	Densities.	Densimetric Degrees.
0	1.0000	0.00
5	1.0356	3.56
10	1.0731	7.31
50	1.5161	51.61

This scale has, however, not been adopted in general practice, although it affords an advantage over the Baumé scale for purposes of calculation. Wherever it can be done in sugar work Brix hydrometers should be used in preference to Baumé instruments.

The range of scale in each and all of these hydrometers of course varies greatly, according to the ideas and preference of the makers, and of those who use the instruments. The following will be found to be convenient

* Methods of Chemical Control in Cane Sugar Factories. Altrincham, 1905.

graduations for the ordinary requirements of the house and laboratory:

Specific-gravity Scale. Range from 1.095 to 1.106. The scale bears twelve full divisions, and these are divided into halves.

The Brix Hydrometers. Range from 0° to 28° , spread over three instruments: the first from 0° to 8° , the second from 8° to 16° , the third from 16° to 28° . Each degree is divided into tenths.

The Baumé Hydrometers for Liquids heavier than Water. For general use in the refinery, a scale on a single instrument ranging from 0° to 50° , and divided into quarters or halves, will prove sufficient. For work at the "blow-ups" the range of scale is from 27° to 32° , and each degree is divided into tenths. For the syrup-boiler a scale from 32° or from 38° to 44° , also divided into tenths, is desirable. For laboratory work the range is from 0° to 45° , best carried over three or more instruments: for instance, from 0° to 20° , from 20° to 35° , and from 35° to 45° ; the subdivision to be in tenths of a degree.

It is a matter of great importance that the hydrometers used in analytical work be correct. Every instrument should be examined in at least three places, these being preferably chosen at points corresponding to the upper, the middle, and the lower part of the scale.

If a correct instrument is at hand (ascertained to be correct by careful examination), other hydrometers of the same scale are readily tested by comparison with the standard hydrometer. If a standard is not available, the testing must be done in comparison with very accurate specific-gravity determinations, made by a balance. If the instrument tested is a specific-gravity hydrometer, the balance determinations are of course directly compared with its readings; if it is a Brix or a Baumé spindle, the corresponding specific-gravity values can be ascertained from Table I.

Methods of Testing Hydrometers. Method I. The balance determinations are made by weighing first a specific-gravity flask or pycnometer,* perfectly clean and dry. The flask is then filled with distilled water at the temperature at which the hydrometer was graduated. This is usually 17.5° C. or 20.0° C.; the weight of the flask filled with water up to the mark is next taken. A solution is then prepared by dissolving pure sugar in water. The density of this solution is such that it corresponds approximately to one of the points marked on the scale of the hydrometer which is being tested. The temperature of the solution is made to correspond exactly with the temperature at which the specific-gravity flask was previously filled, and the weight of this flask, now filled with the sugar solution, is accurately determined.

Subtracting the weight of the flask from these two weighings gives respectively the weight of equal volumes of water and of sugar solution. Dividing the latter value by the former, gives the specific gravity of the sugar solution.

Example.

Weight of specific-gravity flask + water,	40.0403
“ “ “	15.0811
	<hr/>
Weight of water in flask,	24.9592
	<hr/>
Weight of specific-gravity flask + sugar solution,	42.5810
“ “ “	15.0811
	<hr/>
Weight of sugar solution in flask,	27.4999
	<hr/>

$$27.4999 \div 24.9592 = 1.1018$$

$$\text{Specific gravity of sugar solution} = 1.1018$$

* The neck where the mark is placed, should be narrow, and the flask should have a tightly-fitting stopper to prevent loss by evaporation.

Some of the sugar solution is poured into a glass cylinder, the temperature carefully brought to the temperature at which the hydrometer was graduated, and the hydrometer, perfectly clean and dry, inserted. It should be allowed to glide down slowly into the solution in order that no more of the stem shall be immersed than is necessary. Care must also be taken that the instrument floats free, that is, does not come in contact with the sides.

When the hydrometer has come to rest, a reading of the scale is made and compared with the specific gravity obtained by the balance.

The indications of specific-gravity hydrometers should of course agree exactly with the balance determinations; for Brix and for Baumé instruments the limit of agreement should be placed at $\pm 0.10^\circ$ and the necessary correction should be made when using them. The cheaper Baumé hydrometers, ranging from 0° to 50° , will, however, rarely agree closer than $\pm 0.20^\circ$, and this degree of accuracy will suffice for practical working purposes.

Method II. If the hydrometer is a specific-gravity hydrometer of limited range, it may be tested by immersion in solutions of chemically pure sugar; these solutions are prepared as follows:

Sp. Gravity.	Grams C. P. Sugar.	Grams Distilled Water at $17^\circ.5$ C.
1.095	22.6	77.4
1.097	23.0	77.0
1.100	23.7	76.3
1.103	24.3	75.7
1.106	25.0	75.0

Preparation of Pure Sucrose. The method recommended by the International Commission for Uniform Methods of Sugar Analysis, is as follows:

“Purest commercial sugar is to be further purified in the following manner. A hot saturated aqueous solu-

tion is prepared and the sugar precipitated with absolute ethyl alcohol; the sugar is carefully spun in a small centrifugal machine and washed in the latter with some alcohol. The sugar thus obtained is redissolved in water, again the saturated solution is precipitated with alcohol, and washed as above. The product of the second centrifuging is dried between blotting paper and preserved in glass vessels for use. The moisture still contained in the sugar is determined and taken into account when weighing the sugar which is to be used."

Method III. If a balance is not available, the testing of specific-gravity hydrometers may be accomplished by the aid of a polariscope. This method is also applicable to Brix and to Baumé hydrometers if their degrees are translated into the corresponding specific-gravity values.

A solution of pure sugar is made, its temperature is taken, and the hydrometer inserted in it with all the care and precautions previously referred to. After the reading of the hydrometer has been noted, the solution is polarized, and the polarization is multiplied by the factor (Table IV) corresponding to the specific gravity of the solution, corrected, if necessary, for temperature (Table II). If the hydrometer is correct (of course a correct polariscope is premised), the result of the multiplication of the polarization by the factor must be 100.

Example.

Specific gravity of solution corrected for	
temperature,	1.096
Factor,	1.042
Polarization,	96.0
	$96.0 \times 1.042 = 100.0.$

Determination of the Density of Solutions. *By the Specific-gravity Flask.* The most accurate way to determine the density (specific gravity) of a solution is by means of a specific-gravity flask (pycnometer) and a delicate

balance. The weight of the flask, empty and dry, having been ascertained, and the weight of distilled water which it will hold at 4° C., or at the temperature at which it was graduated, being known once for all, it is only necessary to fill the clean and dry flask exactly up to the mark with the solution whose specific gravity is to be determined. If the solution has not been brought to the temperature at which the flask was graduated, before the flask is filled with it, this must be done before the weighing is made, in order that the weight of equal volumes of the water and of the solution may be obtained.

The flask filled with the solution is weighed, the weight of the flask subtracted from this figure, and the remainder divided by the weight of the corresponding volume of water. The result is the specific gravity of the solution.

The Brühl flask especially intended for making specific-gravity determinations of viscous liquids is provided with a glass tube inserted at right angles to the neck of the pycnometer, somewhat above the mark. This tube, as well as the regular mouth of the flask, is closed by a glass stopper. The diameter of the neck should not be less than 2 c.m.

To fill the pycnometer the liquid is drawn into a pipette and the latter inserted in a rubber stopper placed in the neck of the flask. An aspirator is attached to the side-tube and the contents of the pipette are drawn into the flask by suction.

An instrument due to Scheibler used for the same purpose consists of the body of a pipette both ends of which are provided with glass stop-cocks. To each of these ends a well ground detachable glass tube is fitted. To carry out a specific-gravity determination with this both tubes are attached, the stop-cocks are opened and the lower tube is inserted in the liquid the density of which is to be determined. The body of the pipette and a small part of the upper tube are filled by suction with the liquid. The lower stop-cock is then closed, the tube attached to it

removed and the body of the pipette with its contents is immersed in water of the standard temperature until the desired temperature has been attained. The upper stop-cock is then closed, the remaining tube is detached and the apparatus dried and weighed. The weight of an equal volume of water of the standard temperature having been determined once for all, the weight of pipette plus liquid, divided by the weight of pipette plus water, gives at once the specific gravity of the liquid.

By Pipette and Beaker. An adaptation of the methods just described, and which is convenient for rapid working is the following:

A pipette capable of holding a certain volume, say 10 or 20 c.c., is placed in a glass beaker; both pipette and beaker of course must be perfectly clean and dry. The combined weight of the two is taken and noted.

The pipette is then filled with distilled water at the temperature selected as the normal temperature. The pipette is replaced in the beaker, and the combined weight of the pipette, beaker, and water is determined. The vessels having been again cleaned and dried, the solution whose specific gravity is to be determined, is brought to the standard temperature, and the pipette filled with it up to the mark. The weight of pipette, beaker, and solution is then determined. The calculation to be made is exactly as before explained, the combined weight of beaker and pipette taking the place of the weight of the pycnometer in the previous method.

By Hydrometers. The hydrometer selected for making the determination may be a specific-gravity hydrometer or an instrument graduated according to an arbitrary scale (Brix, Baumé).

Whenever a solution is to be tested, care must be taken to have it as free from air-bubbles as possible. If the solution whose density is to be determined is a thick syrup or a molasses, it had best be poured into a vessel provided

at the bottom with a stop-cock. This vessel, advantageously enclosed in a water-jacket, may be heated and the molasses thus readily warmed; this will greatly hasten and facilitate the rising of the air bubbles. When these have all risen to the top, the liquid is drawn off from below, without disturbing the frothy layer on the surface.

The liquid is then placed into a glass cylinder, which must stand perfectly level, and the hydrometer is carefully and slowly inserted. It must float free in the liquid, that is, it must not be permitted to touch the sides of the cylinder. When the hydrometer has come to rest, the point up to which it is immersed in the solution is read and recorded. The temperature of the solution is determined, and if not of the standard temperature, a correction therefore must be made. (See Table II or III.)

The readings of the specific gravity, the Brix, and the Baumé hydrometers can each readily be translated into the terms of the others by Table I.

By Gravimeter. A convenient instrument devised by W. K. Gird permits the convenient determination of the specific gravity of saccharine solutions and at the same time measures out the exact amount by weight of such solutions for analysis.

To quote Gird, this instrument consists of a brass tube standard about twelve inches high and one and one-half inches in diameter. About three inches from the top a small tube makes connection with the large one, extends parallel with and as high as the large tube, and then turning down again, forming a siphon. The idea of having the connection made below the top of the large tube is to allow clear fluid free from bubbles or froth to run out.

The large tube is filled with juice direct from the beet, until it runs out of the small tube, when it is allowed to settle, exactly level with the top of the siphon. The saccharometer, weighing 26.048 grams, is then inserted in the large tube, and settles down until it has displaced through

the siphon exactly its own weight—26.048 grams—of the juice. This is the exact weight of juice required and is ready for the filter and polariscope.

By Araeo-pycnometers. Araeometers of a somewhat modified form have been devised by Fritsch and Eichhorn. They offer the important advantage of requiring but a small amount of solution for specific-gravity determinations. These instruments are shaped like araeometers excepting that between the usual float and the mercury bulb another bulb is placed which is provided with a tight fitting glass stopper. This bulb is completely filled with the liquid to be tested, the stopper inserted and the instrument is then floated in a cylinder which contains water of the standard temperature. A reading of the scale in the stem of the araeo-pycnometer shows at once the specific gravity of the solution tested.

By Glass Spheres. For approximate density determination small glass bulbs of different weights are sometimes used; a number engraved or etched on each, designates the density of a liquid in which it will float.

Beginning with the heavier, these bulbs are successively thrown into the solution whose density is to be determined, until one is found which will float in the liquid tested. The number engraved on this bulb indicates the density of the solution. Of course regard must here also be had to the temperature of the liquid.

By Mohr's Hydrostatic Balance. From one end of the beam of this balance a glass bob, preferably one provided with an accurate thermometer, is suspended by a fine platinum wire*. The other end of the beam is provided with a counterpoise to the bob; this counterpoise terminates in a fine metal point, and serves as the tongue of the balance. It shows the beam to be in equilibrium when the same

*The Reimann thermometer-bob displaces exactly 5 c.c. of distilled water; its weight is less than 15.0 grams to which weight the total weight is readily brought by a metal sinker.

remains at rest in a horizontal position directly opposite to a fixed metal point.

The balance, when correctly adjusted, is in perfect equilibrium when the glass bob hangs freely suspended in air.

That part of the beam between the fulcrum and the end from which the bob is pendant, is provided with nine graduations, numbered from one to nine. Accompanying the balance are five weights or riders. The largest two are each equal to that weight of distilled water at a given temperature, which the glass bob displaces when it is immersed. The other three riders weigh respectively one tenth, one hundredth, and one thousandth as much as the large rider.

When the bob is immersed in water, one of the large riders must be placed at that end of the beam from which the bob is suspended. This will restore the equilibrium, and the balance then indicates the specific gravity 1.000.

If the bob is immersed in a liquid heavier than water, this liquid having been brought to the temperature for which the balance was graduated, some of the other riders also must be placed on the beam in order to restore the equilibrium. The position of these riders indicates the specific gravity of the solution, each rider according to its weight, representing respectively as many tenths, hundredths, or thousandths as is expressed by the numbered division on the beam where it is placed.

By Analytical Balance. Specific-gravity determinations of solutions can also be rapidly and easily effected in the following manner.

Take a piece of metal or glass of convenient form. Determine its weight in air and its weight in water. From these data calculate its specific gravity. The weight of the body in air divided by its specific gravity expresses the volume of the body and of course represents also the weight of the volume of water which this body displaces on immer-

sion, presuming the metric system of weights and measure is employed. These data having once been ascertained the body need only be immersed and weighed in the solution the specific gravity of which is sought and the result found by a simple calculation.

Example.

Weight of the metal in air	12.000	grs.
Specific gravity of the metal	2.4	
Weight of the metal in the sugar solution	6.495	grs.

$$\text{Volume of metal} = \frac{12.0}{2.4} = 5.0.$$

That is to say, this piece of metal when immersed in water will displace 5.0 grams of water. The metal when immersed in the sugar solution displaces:

$$\begin{array}{r} 12.000 \\ 6.495 \\ \hline 5.505 \end{array} \text{ grams of the sugar solution,}$$

hence the specific gravity of the sugar solution is equal to:

$$\frac{5.505}{5.000} = 1.101.$$

Calculation of Weight of Solids and Liquids from their Specific Gravity. One cubic foot of distilled water weighs 62.50 lbs. = 1000 ounces. The specific gravity of water is 1.000. If the decimal point of a specific-gravity value be moved three places to the right, the weight of a cubic foot in ounces will be obtained. This figure divided by 16 gives the weight of a cubic foot in pounds. From this the following rule is deduced;

To find the weight in pounds per cubic foot:

Determine the specific gravity, remove the decimal point three places to the right, and divide by 16.

Example. Specific gravity of a substance is 0.87904.

$$879.04 \div 16 = 54.94$$

Hence this substance weighs 54.94 lbs. per cubic foot.

As above stated, if the decimal point of a specific-gravity value is removed three places to the right, the weight of a cubic foot in ounces will be obtained, and this figure divided by 16 will give the weight of a cubic foot in pounds. But if the cubic foot be assumed equal to 7.5 gallons, $7.5 \times 16 = 120$. Therefore,

To find the weight of a gallon in pounds:

Determine the specific gravity, remove the decimal point three places to the right, and divide by 120.

Example. A syrup has a specific gravity of 1.413.

$$1413 \div 120 = 11.78,$$

hence the syrup weighs 11.78 lbs. per gallon.

Colorimeters. The color-tests made on sugars and on sugar solutions are generally only comparative, that is to say, the color of the sample examined is compared with that of some other sample which is taken as the standard.

In examining the color of sugar solutions, to learn, for instance, how effectively a certain sugar has been decolorized in passing through bone-black, two test-tubes, beakers, or cylinders made of white glass, are filled to an equal height with, respectively, the sample under examination and the "standard" solution with which the sample is to be compared, both solutions of course being of equal density.

Various forms of apparatus have been designed for effecting comparison of color.

The colorimeters of Duboscq, Stammer and C. H. Wolff are very similar in their construction. Duboscq's and Wolff's apparatus require standard solutions for com-

parison whereas in the colorimeter of Stammer, and also in the "tintometer" of Lovibond, the standard solution is replaced by colored glass discs of various tints, by the combination of which it is possible to produce almost any shade desired.

As the depth of color of a solution is proportional to the length of a column of such solution, there must be ascertained in instruments of the Stammer type the height of a column of the solution which will in color correspond to the tint of some "standard" colored glass disc or discs inserted in an adjoining tube. The scale is graduated in millimeters. If, for instance, a depth of one millimeter of the solution corresponds to the normal tint, the color is said to be 100. If two millimeters depth of solution are required to match the tint, the color is 50; if four millimeters, 25, and so on.

Thermometers. All thermometers should be, if possible, compared with some standard instrument. This applies especially to the thermometer which is to be used to determine the temperature while ascertaining the polarization of inverted sugar solutions. It will answer to verify, on Centigrade thermometers intended for ordinary use, the zero and the 100 mark; on a Fahrenheit instrument, the 32° and the 212° mark, and in both of course to see that the degrees are of equal size.

The zero mark on the Centigrade scale (32° Fahrenheit) is ascertained by placing the bulb and part of the stem in snow or pounded ice for about a quarter of an hour. The vessel in which the snow or ice is placed should be provided with a small opening at the bottom, through which the water is drained off as it is formed.

To obtain the 100° C. (212° F.) mark, the thermometer is suspended in the vapor of boiling water, care being taken that it does not dip into the water. The pressure of the atmosphere should be 760 m.m. at the time; if not, a correction for the variation must be made.

The reading of one scale can be translated into that of the other by the following formulæ:

$$\text{To change } ^\circ\text{C. into } ^\circ\text{F. } \frac{^\circ\text{C.} \times 9}{5} + 32 = ^\circ\text{F.}$$

$$\text{To change } ^\circ\text{F. into } ^\circ\text{C. } \frac{(^{\circ}\text{F.} - 32)5}{9} = ^\circ\text{C.}$$

See Table XIX for a comparison of different thermometric scales.

CHAPTER III

POLARISCOPES AND ACCESSORIES

Polarization. If a ray of light strikes a glass mirror and makes an angle of about 55° with the normal of the mirror, the ray is not only reflected, but is endowed with certain properties, and is said to be polarized.

In Fig. 2, ab is the incident ray, bc the polarized ray. A plane conceived as passed through abc is called the plane of polarization.

If a polarized ray is allowed to fall upon a second mirror, parallel to the first, it is again reflected at the angle above mentioned. If this second mirror is turned around bc , its inclination to the horizontal being preserved unchanged, the intensity of the reflected ray continuously diminishes until, when the rotation has been carried through 90° , the light is extinguished completely. If the rotation be carried beyond this point the mirror becomes again illumined; and when it has been turned through 180° , the reflection is again at its maximum of brightness. In other words, the intensity of the reflected light is greatest when the incident ray and the polarized ray, after reflection from the second mirror, are in the same plane, and least when these rays are in planes at right angles to each other.

Polarization of light can also be produced by other means: by repeated single refractions, or by double refraction in certain crystals—Iceland-spar, for instance.

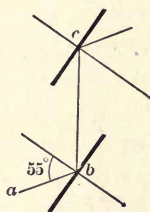


FIG. 2.

If a plate of quartz, cut at right angles to its principal axis, is inserted between two mirrors placed as above described, and traversed by a polarized ray, the image of the quartz will appear in color in the upper mirror. The color of the image changes with the turning of the mirror; the order in which the colors appear is the same as found in the solar spectrum: red, yellow, green, blue, and violet.

This phenomenon is termed circular polarization. It depends on the property possessed by quartz of rotating to a different degree the planes of polarization of the various colored rays which compose white light. One variety of quartz shows these colors in the order named when the mirror is turned to the right; a second variety of the mineral exhibits the colors in this sequence only when the rotation of the mirror is to the left. These varieties of quartz are respectively termed right-rotating and left-rotating, or dextrogyrate and levogyrate.

Among other bodies which share with quartz the property of circular polarization are the sugars when in solution. Some of the sugars are dextro-rotatory: for instance, sucrose, dextrose, and raffinose; others rotate the plane of polarized light to the left, as levulose and sorbinose.

The extent to which the plane of polarized light is turned by quartz, by a sugar solution or any other optically active substance, depends on the thickness of the layer which the polarized ray has to traverse. The thicker the plate or the longer the column of solution, the greater the rotation of the ray. Whereas in the case of a quartz plate the thickness of the plate is the only factor to be considered, in sugar solutions the concentration of the solution, i.e., the amount of sugar in the solution, must be taken into account.

Specific Rotation. The polarizing power of sucrose, termed its specific rotation or specific rotatory power, is the angular rotation given to the plane of polarization by a column 1 dm. long of a 100% solution of sucrose.

This angular rotation depends upon the wave length of the light used and it is therefore necessary to specify this. It is customary to denote the angular rotation by the letter a , and the specific rotation by the term $[\alpha]$.

Specific rotation of the mean yellow light ray is designated by the expression $[\alpha]_J$, that of the bright yellow line of sodium by $[\alpha]_D$. In addition it is customary to indicate also the temperature at which the value is determined, thus if this be 20° C. the symbol becomes $[\alpha] \frac{20}{D}$.

Specific rotation is calculated by the formula

$$[\alpha] = \frac{100 \times a}{c \times l},$$

in which: a = angular rotation of the solution,
 c = grams per 100 c.c. of solution,
 l = length in decimeters of the observation tube.

Or, it can be calculated by the formula:

$$[\alpha] = \frac{100a}{p \times d \times l},$$

in which: a = angular rotation of the solution,
 d = the specific gravity of the solution,
 p = parts by weight of the sucrose in 100 parts
 by weight of the solution,
 l = length in decimeters of the observation tube.

In this expression it will be seen that $p \times d$ corresponds to the c of the previous equation.

The specific rotation of sucrose is +66.6. This can be determined by obtaining the reading of a sucrose solution of known concentration in a polariscope, changing this value to angular degrees by the factor 1° Ventzke = 0.34657 angular degrees and applying the $[\alpha]_D$ formula.

If, for instance, a 13% by weight solution of sucrose be prepared and polarized at 20° C. in a 2 dm. tube, the reading found is multiplied by 0.34657 and by 100, and then divided by 13×2. Thus: 13 grams of sucrose dissolved at 20° C. to 100 metric cubic centimeters give, in a 2 dm. tube, a reading of 50° Ventzke on the polariscope.

$$[\alpha]_{D}^{20} = \frac{(50 \times .34657) 100}{13 \times 2} = +66.6.$$

*Conversion factors.**

Normal weight for Ventzke scale = 26.00 grams

French scale = 16.29 “

Wild scale = 10.00 “

1° Ventzke scale = 0.34657° angular rotation *D*.

1° French “ = 0.21666° “ “

1° Wild “ = 0.13284° “ “

1° Ventzke scale = 1.59960° French scale

1° “ “ = 2.60903° Wild scale

1° French scale = 0.62516° Ventzke scale

1° “ “ = 1.63098° Wild scale

1° Wild scale = 0.38329° Ventzke scale

1° “ “ = 0.61313° French scale.

Polariscopes—Saccharimeters. Basing on the property of circular polarization, instruments have been constructed by which the strength of solutions containing optically active substances can be determined. They are called polariscopes or polarimeters. Polariscopes intended for general scientific work are provided with a circular disc, graduated in such a manner that the angle of rotation can be conveniently read. Instruments intended for some special purpose, as

* C. A. Browne, Handbook of Sugar Analysis, 1912.

for instance, for sugar analysis, are generally provided with a scale which, if certain directions have been followed in the preparation of the solution, will at once indicate in percentage the amount of the optically active substance present. Polariscopes designed especially for sugar analysis are termed saccharimeters, although the former term is also often employed even for this type of instrument.

The principle on which these instruments are constructed is briefly this: A ray of light is polarized by passing through a prism, called the polarizer and generally made of Iceland-spar; the ray is then made to traverse a column of sugar solution of known length. Emerging from this, it passes through a second prism of Iceland-spar, the analyzer, which corresponds to the second mirror in the apparatus previously described. It now only remains to ascertain the extent to which the plane of polarized light has been rotated by the sugar solution. The arrangements by which this is effected differ in the various forms of saccharimeters, but in the more modern instruments it is generally accomplished by allowing the light on its emergence from the analyzer to pass through a layer of quartz, the thickness of which (capable of accurate measurement) can be so regulated as to compensate exactly the rotation produced by the sugar solution. It is assumed that the rotatory dispersion of sugar corresponds to that of quartz.

The field of vision of a saccharimeter is either one of color, or else exhibits, when correctly set at zero, a uniform faint tint; saccharimeters showing the latter are known as half-shade or half-shadow instruments, and can be used by color-blind persons, as well as by others.

The arrangement of the optical parts of saccharimeters is shown in the accompanying Figs. 2 and 3. Concerning the adjustment of the double-wedge compensating saccharimeter, the following directions, given by Schmidt and Haensch should be carefully followed.

Before using an instrument it is necessary to control

the zero point. After the red scale has been placed on zero the wedge corresponding to the black scale is moved

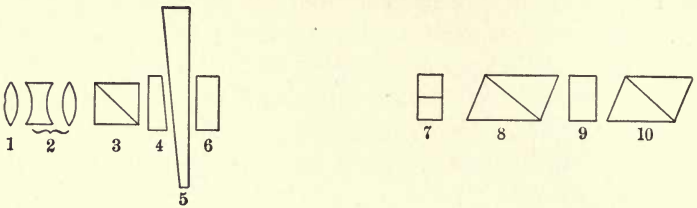


FIG. 3.—Soleil-Ventzke-Scheibler Saccharimeter.

- 1. Magnifying-glass for reading scale.
- 2. Telescope for observing field of vision.
- 3. Nicol prism, analyzer.
- 4. Quartz-wedge, fixed, bearing vernier.
- 5. Quartz-wedge, movable, bearing scale.
- 6. Quartz plate. { Dextro-rotatory if 4 and 5 are levo-rotatory. } Rotation Compensator.
- 7. Double quartz plate (dextro- and levo-rotatory).
- 8. Nicol prism, polarizer.
- 9. Quartz plate, dextro- or levo-rotatory. } Regulator.
- 10. Nicol prism.

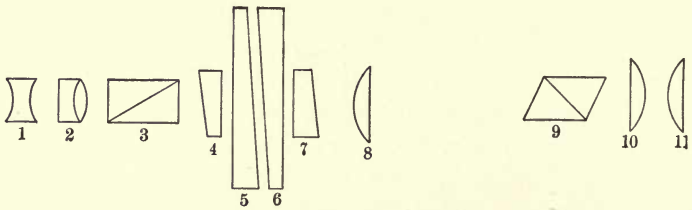


FIG. 4.—Double-wedge Compensator Saccharimeter, Schmidt and Haensch Construction.

- 1. Eye-piece.
- 2. Objective.
- 3. Nicol prism, analyzer.
- 4. Quartz-wedge. { Constituting the Double-wedge Compensator.
- 5. Quartz-wedge. {
- 6. Quartz-wedge. { Constituting the Double-wedge Compensator.
- 7. Quartz-wedge. {
- 8. Lens.
- 9. Nicol prism.
- 10. Lens.
- 11. Lens.

until the field exhibits the same tint throughout. The black nonius should then point exactly to zero. If this is

not the case the wedge is moved by means of the micrometer-screw on the left until absolute zero is obtained. The correctness of this zero is determined by repeated trials. If now, after both scales are placed at zero, one side of the field appears colored we must proceed as follows: The wedges are removed and the color of the field observed. The large wedges are removed by screwing them out as far as they will go and then simply pulling them out; then the two screws are removed and the small wedges taken out. If now the field does not show an even tint the analyzer is moved by the two screws placed at the side, loosening one and tightening the other correspondingly until the desired result is reached. The small wedges are now put back in the order in which they are marked, the large wedges placed in position, and the zero point corrected as before. The zero point of the instrument is now accurate, and if the divisions of the scale are correct the quartz plate should give the correct reading. If, however, an incorrect reading is obtained, the plate should be taken out and controlled by means of the red scale. If this gives the correct reading the black scale is wrong, otherwise both scales are inaccurate.

The U. S. Customs Regulations of 1907 prescribe the double quartz-wedge compensation saccharimeters and a polarizing system known as the Lippich half-shadow, and state:

“ Art. 58. All polariscopes shall be so adjusted that when a 200-millimeter tube filled with the standard sugar solution is polarized at 20° C. the instrument shall read 100° S (sugar degrees) $\pm 0.01^\circ$ S (sugar degree). All points on the scale shall indicate percentages of the standard solution. The standard sugar solution shall be prepared by dissolving 26 grams of pure sugar in pure water and making up the volume to 100 metric cubic centimeters, all weighings to be made in air with brass weights and the volume to be completed at 20° C. A length of 200 millimeters of the standard sugar solution shall be considered to give a

rotation of 40.728° for light of wave length 5,461 centimeters $\times 10^{-8}$."

Bates' Saccharimeter. This instrument a white light polariscope with an adjustable sensibility was designed by Frederick J. Bates of the Bureau of Standards, Washington, D. C.

It possesses some marked advantages over the several styles of half-shade saccharimeters in more general use.* It is an improved quartz compensating instrument the sensibility of which can be instantly adjusted so as to afford ample light for the observation of the darkest sugar solutions; its right and left rotating wedges cover a wide range from -20° sugar to $+120^\circ$ sugar. The scale is etched on ground glass, which permits of very sharp rulings, and, as this scale is illuminated by transmitted instead of by reflected light, the black dividing line between the vernier and the scale and which is oftentimes disturbing, is done away with, and scale readings to 0.01° sugar, are made possible.

As the temperature of the quartz-wedges plays so important a role in accurate polarimetric work, a thermometer with a horizontal scale, divided into one-fifth degrees, is mounted on the metal case containing the compensator and permits accurate ascertainment of the temperature of the wedges.

A further improvement of this instrument is the placing of the milled heads, which move the quartz-wedges, in a position at right angles to the position in which they are usually placed; furthermore these wedges can be instantly set and rigidly fastened at any position of the scale.

This saccharimeter is equally available for regular commercial usage and for the most refined research work and certainly marks an important advance in the evolution of this, the most important instrument used in the sugar industry.

* See American Sugar Industry, Chicago, Vol. XIII, No. 6, 1911, p. 254.

Saccharimeter Scales. The scales of saccharimeters are constructed by ascertaining the number of degrees, minutes, and seconds which a definite amount by weight of pure sugar dissolved in water and made up to 100 cubic centimeters will rotate the polarized ray. This point is marked as 100, and the scale is then divided into one hundred parts.

If the same weight of an impure sugar is brought into solution and polarized under the same conditions, the reading in the polariscope expresses percentage of the active substance present.

The scales of different saccharimeters have their 100 mark correspond to different weights of pure sugar. In the Laurent and in the Duboseq instrument it is 16.192 grams, in Wild's it is 10.000 grams, and in the Ventzke apparatus 26.048 grams dissolved to 100 Mohr c.c., or 26.000 grams dissolved to 100 metric c.c. These values are termed the "normal weights" of the respective instruments.*

The 100 mark in the Ventzke scale was originally determined † by preparing from pure sugar a solution having a specific gravity of 1.100. This solution placed in a tube 200 m.m. in length deviated the plane of polarized light to a certain extent and this point was marked 100° on the polariscope scale. Formerly, in order to test a sugar sample containing less than 100% of sugar, there was prepared of it a solution having the above specific gravity; this was placed in a 200 m.m. tube, and the reading on the scale of the polariscope indicated by this solution, was regarded as giving directly the percentage by weight of sugar in the dry substance.

This method of procedure was however soon abandoned because it was found to be inexact, as the salts contained

* Confer: Nasini and Villavecchia, *Sul Peso Normale Pei Saccharimetri*, Roma, 1891.

† Landolt: *Zeitschrift des Vereines für Rübenzucker-Industrie*, 1891, p. 515.

in the raw sugars also influence the specific gravity of the solution.

It was ascertained that 100 c.c. of Ventzke's normal solution contained 26.048 grams of pure sucrose—hence if this weight of a sugar sample is dissolved in water up to 100 c.c. a reading of such a solution in a 200 m.m. tube will indicate directly the percentage of sugar.

This method was introduced long before "Mohr's" c.c. were known. The 26.048 grams were therefore most likely originally intended to be dissolved up to 100 metric c.c. However gradually German manufacturers of saccharimeters came to place the 100 mark on their instruments secured by means of solutions containing 26.048 grams of pure sucrose in 100 Mohr c.c. and this practice was quite generally adopted and continued, until the International Commission for Uniform Methods of Sugar Analysis, in 1900, urged the adoption of the metric c.c. for sugar-work and prescribed 26.000 grams of pure sucrose as the normal weight to be dissolved up to 100 metric c.c. at 20° C., and observed in a tube 200 m.m. long at 20° C., in order to secure the 100° mark on saccharimeters.

In order to adjust a saccharimeter, first obtain by the telescope a sharply defined view of the field. Then turn the screw attached to the quartz-wedge until both halves of the field are, in color instruments, of the same tint; or if the saccharimeter is a half-shade apparatus, until both halves of the field are equally illumined.

When this has been done the position of the scale is carefully read through the magnifying-glass. The zero of the scale should be exactly in line with the zero mark on the vernier; if this is not the case, they must be brought into the required position by a slight turning of the screw-micrometer provided for the purpose. Care must be taken that the screw in connection with the analyzer be not mistaken for the other screw, or the whole apparatus will be thrown out of order.

If it is impossible to obtain a uniform shade or tint on both sides of the center line of the field, the polarizer and the analyzer must be brought into adjustment.

This is done by removing the movable and the stationary quartz-wedges, as well as the compensation quartz plate; the cover is then closed, and the key having been inserted in the screw-head connected with the analyzer (this screw-head is generally placed on the right-hand side of the polariscope), the key is turned until the tint in both halves of the field is uniform.

The wedges and the plate which had been removed are then replaced, and the zero point accurately adjusted.

When the instrument has been correctly set at zero a quartz plate of known value, preferably one approximating the average test of the sugar solutions to be examined, is inserted in the instrument, and the correctness of that part of the scale ascertained.

Adjustment and Examination of Saccharimeters. In all instruments the zero point should be determined before every observation; where press of work renders this impracticable, the observation should be insisted on at least twice daily—in the morning before a polarization is made, and again in the middle of the day. If the zero point is out a few tenths only, it is not necessary to adjust the scale each time but the needed correction should be made in noting the reading.

When a solution is introduced for reading, the telescope must first be properly focussed, as before stated, to insure a clear and sharply defined view of the field.

If the scale stood at zero before the tube filled with the solution was introduced, a glance through the glass will after its introduction show the halves of the field to be of different colors; or, if a half-shade saccharimeter is used, one half of the field will appear dark and the other light.

The screw attached to the quartz-wedge is then turned until equality in tint or shade shall have been restored to the whole field. The three different appearances presented by the field are shown in the following diagram.



FIG. 5.

It then only remains to read the scale. Most instruments have the degrees divided into tenths. First it must be determined how many whole degrees the zero of the scale is removed from the zero of the vernier. When this has been ascertained, attention must be given to the tenths of a degree indicated. The number of divisions marking tenths on the vernier are counted until one is found which coincides perfectly with a division on the movable scale, that is to say, which appears to form a continuation of that line. This division represents the number of tenths indicated. The accompanying figure, for instance, shows 30.7° .

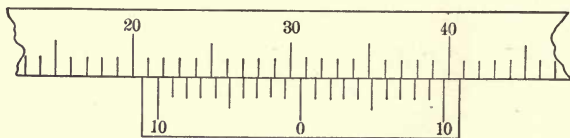


FIG. 6.

Most saccharimeters have scales possessing a range from 0° to 100° . Instruments have however been constructed, according to the suggestions of Stammer and Strohmmer, the scales of which have a range of only from 80° to 100° . A polarization tube 400 m.m. in length is usually employed

with these saccharimeters and readings to within $\pm 0.05^\circ$ can be obtained.

The sources of error in saccharimeters are numerous and therefore every instrument, before being placed in use, should be carefully examined.

To determine whether the scale is correct, adjust the zero point exactly. Make 100 c.c. of a sugar solution by dissolving the normal weight of chemically pure sugar in water, and polarize. This solution should read 100 degrees (per cent) on the scale if the instrument is correctly constructed. If it does not read 100, the instrument should be rejected.

The need of constantly controlling the adjustment of saccharimeters with quartz plates or sugar solutions of known value, has already been referred to.

In instruments of earlier make, provided with ivory scales, the scale itself was frequently a cause of introducing error, as it was liable to distortion by variation in temperature and of atmospheric moisture.

More modern saccharimeters are provided with nikelin scales—an alloy somewhat similar to German silver in appearance, or, best of all with glass scales; with the latter all difficulties of this kind are entirely obviated.

The scale may be right in some places, and wrong in others. This is the case when the surfaces of the quartz-wedges are not perfectly plane. In half-shade saccharimeters provided with double compensation wedges, this cannot occur, as any inequality would be noticed at once. In other saccharimeters, the scale may be examined by pure sugar solutions of different densities, by means of the "control tube" of Schmidt and Haensch, or by quartz plates.

The following figures, taken from a table calculated by Schmitz, show the number of grams of pure sugar which must be made up to 100 Mohr c.c. aqueous solution in order to show the corresponding degree on a saccharimeter

having 26.048 grams for its normal weight in 100 Mohr cubic centimeters.*

Polariscope Degrees.	Grams C. P. Sugar in 100 c.c. Solution.	Polariscope Degrees.	Grams C. P. Sugar in 100 c.c. Solution.	Polariscope Degrees.	Grams C. P. Sugar in 100 c.c. Solution.
1	0.260	35	9.097	69	17.954
2	0.519	36	9.357	70	18.216
3	0.779	37	9.618	71	18.476
4	1.039	38	9.878	72	18.738
5	1.298	39	10.138	73	18.998
6	1.558	40	10.398	74	19.259
7	1.817	41	10.659	75	19.519
8	2.078	42	10.919	76	19.781
9	2.337	43	11.180	77	20.042
10	2.597	44	11.440	78	20.302
11	2.857	45	11.701	79	20.564
12	3.117	46	11.961	80	20.824
13	3.376	47	12.222	81	21.085
14	3.637	48	12.482	82	21.346
15	3.896	49	12.743	83	21.608
16	4.156	50	13.003	84	21.868
17	4.416	51	13.264	85	22.130
18	4.676	52	13.524	86	22.391
19	4.936	53	13.784	87	22.652
20	5.196	54	14.044	88	22.912
21	5.456	55	14.305	89	23.174
22	5.716	56	14.566	90	23.435
23	5.976	57	14.826	91	23.696
24	6.236	58	15.087	92	23.957
25	6.496	59	15.347	93	24.219
26	6.756	60	15.608	94	24.480
27	7.016	61	15.868	95	24.742
28	7.276	62	16.130	96	25.002
29	7.536	63	16.390	97	25.265
30	7.796	64	16.651	98	25.525
31	8.056	65	16.912	99	25.787
32	8.316	66	17.173	100	26.048
33	8.577	67	17.433		
34	8.837	68	17.694		

* C. A. Browne, in his Handbook of Sugar Analysis, p. 118, gives a table calculated by him showing the effect of concentration of sucrose upon polariscope readings, based on the normal weight of 26.00 grams sucrose in 100 metric cubic centimeters at 20° C., which shows some variations from the table of Schmitz,

This method of testing requires a separate solution for each degree of the scale which is to be examined.

If the weights necessary to this mode of examination are not available, the tests can be made by dissolving the normal weight of chemically pure sugar in different volumes of water at the normal temperature. Thus, with a German saccharimeter 26.048 grams of such sugar will, when dissolved—

in 100 Mohr c.c. water	polarize	100.00°
“ 105	“ “ “	95.23°
“ 110	“ “ “	90.90°
“ 115	“ “ “	86.95°
“ 120	“ “ “	83.33°

If a control tube is used, but few solutions are needed, as this tube is so arranged that it can be lengthened or shortened at will. A funnel receives the superfluous solution when the tube is shortened, and a scale attached shows the length of the column in millimeters. A simple calculation gives the reading which will be shown by the polariscope if this is correct.

Example.

20.824 grams of pure sucrose made up to 100 Mohr c.c. indicate, when read in a 200 m.m. tube, 80° on a saccharimeter.

$$200 \div 80 = 2.5.$$

Therefore, shortening the control tube by 2.5 m.m. must decrease the reading on the saccharimeter by 1° provided that the scale is correct.

Thus, the above solution should read in a tube:

200 m.m.	80°
197.5 m.m.	79°
195.0 m.m.	78°

If these readings are not obtained, the instrument is incorrect at these points, and the necessary corrections must be made on all observations.

It is also necessary to verify that part of the scale which lies to the left of the zero point. This can be done by means of an inverted sucrose solution of known value. 26.048 grams of chemically pure sucrose are dissolved in water and made up to 100 Mohr c.c. If the polariscope scale to the right of the zero mark is correct, this solution will polarize 100° .

A part of this solution is inverted (see Clerget's Inversion Method), the inverted solution is polarized, and the usual calculation made. If this gives the value = 100.00 that part of the scale, to the left of the zero point, is correctly graduated.

Example: Polarization before inversion 100.00
Polarization after inversion at 20° C. - 32.66

$$\frac{100.00 - 32.66}{132.66} \text{ Sucrose} = \frac{100 \times 132.66}{142.66 - 10} = 100.00.$$

If this portion of the scale is not correctly graduated, but that part of the scale which lies between zero and 100° is correct, then the instrument may nevertheless be used for obtaining readings of inverted solutions.

All that is necessary is to insert and to read together with the inverted solution, a dextro-rotatory quartz plate of known value. The difference between the reading of this combination, and the reading of the quartz plate when read alone, represents the number of degrees which the inverted solution rotates to the left.

Example:

Quartz plate	+93.2
Quartz plate + inverted solution. (Calculated for full normal weight solution in 200 m.m. tube, at 20° C.)	+79.2
Inverted Solution	-14.0

Quartz Plates. If quartz plates are used to test the accuracy of different parts of the scale care must be taken that the surfaces of the plates are perfectly plane, that they are placed in the optical axis of the instrument and at right angles to it and that the plates are of the same temperature as the quartz-wedges of the saccharimeter. The quartz plates must of course have been carefully tested as to their accuracy before being put to use. Such testing is best done by means of pure sucrose solutions; another way of ascertaining their value, i.e., the amount by which they rotate a plane of polarized light, is to measure their thickness.

This measurement can be effected by means of a spherometer. This consists of a movable screw supported in the center of three arms, upon which the apparatus rests. The screw is provided at its lower end with a steel point; near its upper end there is fastened a circular plate of metal, the circumference of which is divided into several hundred equal divisions. Fastened to one of the supporting arms is a metal bar, also bearing a graduation; its graduated edge is placed at right angles to the circular disc.

Parallel to the latter, and attached to the bar, is a sliding-scale which can be set and fastened at any desired height. The graduation of the sliding-scale is so made, that nine of its divisions correspond to ten divisions on the disc.

When the thickness of a plate of quartz, for instance, is to be measured, the screw is first adjusted in such a manner that it shall just touch the perfectly level surface on which the apparatus has been placed.

The sliding-scale is next fastened on the bar exactly on a level with the circular disc.

Suppose the latter to bear five hundred equal divisions, and the graduated bar to be divided into halves of a millimeter. The threads of the screw are so cut that one complete revolution of the screw, indicated by the

graduated disc fastened to it, raises the screw through one half of a millimeter. To effect the measurement the screw is first raised sufficiently so as to allow the quartz plate to be slipped beneath it; when this has been done, the screw is carefully lowered until contact is secured between its point and the quartz plate. From the number of revolutions through which the screw has been turned, the thickness of the quartz plate is determined; with a spherometer graduated as here assumed, the measurement will be exact to the one ten-thousandth part of a millimeter.

The determination of the value of quartz plates is however so important a matter, that, if it is at all possible to secure them, only quartz plates which have been standardized by the Bureau of Standards or which have been tested against some plate so standardized should be used.

As quartz suffers modification of its optical value by stress or strain,* it is most essential that all quartz control-plates be mounted in such a way that no pressure whatever is exerted on the plate, yet they must not lie loose in their mounting.

Polariscope Tubes. The polariscope tubes must be of exactly the prescribed length, as the amount of deviation of the polarized ray produced by an optically active substance depends, of course, among other conditions, on the length of the column of the substance which it traverses. The length of tubes can readily be determined by measuring them with a metal rod made of the standard length. The ends of the polarization tubes must be ground perfectly plane-parallel.

Where a great number of polarizations must be made, Pellet's tube for continuous polarization can be advantageously employed.

* Wiechmann, School of Mines Quarterly, Vol. XX, 1899, The Optical Behavior of Quartz under Stress.

This tube is provided at each end with a metal tube of small diameter, placed at right angles to the main tube. One of the projecting tubules is connected with a funnel through which the solution which is to be polarized is introduced, the other tubule serves for the escape of the liquid when no longer needed. The tube is filled by displacement with the various solutions to be tested, about 20 to 40 c.c. of solution being requisite for the purpose. The readings are taken as soon as the field appears perfectly clear and uniform.

A convenient form of polariscope tube has its ends somewhat enlarged so that any air bubbles which may be introduced on filling the tube do not lie in the field of vision and interfere with the readings.

The cover glasses of the polarization tubes must be made of perfectly white glass, and must be free from scratches.

Another point to be borne in mind is the fact that the glass covers of the polarization tubes may be optically active, either by nature of the glass, by being screwed down too tight, or by not having both surfaces perfectly parallel.

To guard against the first of these sources of error the covers should be tested by placing distilled water in the tube and noting the absence of any optical activity. Non-parallelism of the surfaces can be readily recognized by taking a glass cover between two fingers and rotating it rapidly, at the same time looking through it at some fixed object. If the latter seems to be moving, the glass is not plane-parallel, and should be rejected.

The test can also be made with a simple apparatus devised for the purpose. A disc which can be rotated on its axis is placed between two black screens each one of which is provided with a small aperture.

A light is placed behind one of these screens and some of the light rays are permitted to fall upon the glass cover which has been placed on the movable disc.

The observer on looking through the aperture in the

second screen will see two images of the source of light, caused by reflection from the lower and the upper faces of the glass cover.

On rotating the disc these two images will retain their relative position unchanged only when the cover has been cut perfectly plane-parallel. If the images appear to change their relative position, the cover glass should be rejected.

To reduce the chance of accidental pressure on the cover glasses to a minimum it is recommended that the caps closing the ends of the polariscope tubes be provided with bayonet catches instead of being screwed into place as is customary.

Sources of Light. The light used for polariscopic work may be furnished either by gas, alcohol, oil, acetylene or electricity as all of these emit white light.

Of course each one of these light sources calls for its own style of lamp, burner, or bulb to yield the best results.

If illuminating gas be used, Argand triple-burners having flat, not circular, flames, may be employed. With the same illuminant, incandescent light mantles give good results. Where electric power is available, a 50 C.P. or a 100 C.P. spiral filament bulb gives satisfaction.

It is important to note that with the Ventzke saccharimeters a layer 1.5 cm. thick of a 6% solution of potassium dichromate must be used as a ray filter, as otherwise, colorless or very light-colored sugar solutions will make an even adjustment of both halves of the saccharimeter-field difficult, if not impossible. This phenomenon is due to a slight difference in the optical qualities of sugar and the quartz. If the light-filter is not used a normal sucrose solution will read 0.12° Ventzke too high. If an instrument of the Laurent type is used, a suitable monochromatic light must be provided. Various types of such lamps are in use, but it has been found to be a difficult matter to obtain an intensive illumination which shall prove constant for a considerable length of time.

A device due to H. W. Wiley has given satisfaction in this respect. Following is a description of this apparatus.*

It consists essentially of two wheels with platinum gauze perimeters and platinum wire spokes, driven by a clock-work, and mounted by supports. The sodium salt, chlorid or bromid, in saturated solution, is placed in porcelain crucibles, to such a depth that the rims of the platinum wheels dip beneath the surface as they revolve. The salt is volatilized by the lamp. By means of crossed bands the wheels are made to revolve in opposite directions. The solution of the salt, which is taken up by the platinum net work of the rim of the wheel, thus has time to become perfectly dry before it enters the flame, and the sputtering which a moist salt would produce is avoided. At every instant, by this arrangement, a minute fresh portion of salt is introduced into the flame with the result of making a perfectly uniform light, which can be used for hours without any perceptible variation. The mechanism of the apparatus is so simple that no further description is necessary. The polariscope should be so directed toward the flame as to bring into the field of vision its most luminous part. The platinum wheels are adjustable and should be so arranged as to produce between them an unbroken yellow flame. The wheels are eight cm. in diameter and driven at a rate to make one revolution in six to ten minutes.

F. Dupont has suggested sodium chloride and sodium tribasic phosphate, fused together in molecular proportions, as an excellent combination for the emission of monochromatic light.

The scale of a polariscope is illuminated either by transmitted light, by reflected light, or by means of a separate little electric light bulb which is switched on and off by the operator as required to make the readings.

Whatever be the source of light care must be taken that

* Journal American Chemical Society, Vol. XV, p. 121.

a clear bright illumination of the field is secured and that the source of light be so placed that any heating of the instrument is avoided. The source of light should be at least 200 m.m. from the end of the instrument and should be screened therefrom by a partition provided with an opening only sufficiently large to permit the passing of the light-rays to the saccharimeter.

CHAPTER IV

SUCROSE DETERMINATION BY OPTICAL ANALYSIS

Sampling. Before taking up the analytical determination of sucrose it is necessary to say a few words on the securing of correct samples, that is to say on the obtaining of samples representative of the cargo.

Samples of sugar are drawn with a long steel rod, pointed, and with a groove on one side. The package is pierced, the trier inserted, rotated completely and withdrawn. The sample filling the hollow in the trier is removed and put into a can.

Following are the U. S. Government directions (1907) for the sampling of sugars and molasses.

“Art. 3. All sampling shall be done at the time of weighing. In sampling imported raw sugars a general sample shall be taken; that is, each cargo shall be sampled without regard to marks, except as provided in Articles 12 and 13. In the event that a cargo is consigned to two or more consignees, any consignee's sugar shall be treated as a separate cargo, provided separate entry be made by such consignee. A separate general sample shall also be taken of (a) wet sugar, (b) damaged sugar not wet, (c) ship sweepings, (d) dock sweepings. In taking the general sample 100% of the packages shall be samples. In order to prevent any unnecessary labor and inconvenience in obtaining the sample, the inspector shall direct that the packages when discharged from the vessel upon the wharf shall be so placed that the sampler can readily obtain a 100% sample. All ship and dock sweepings shall be sampled before the sampler completes his half day's work.

“ Art. 4. In the treatment of sugars under these regulations great care will be taken by samplers and other appraising officers to prevent the drying out of the samples. All the sugar buckets in the possession of a sampler shall be either entirely filled or entirely empty, with the exception of one bucket for each general sample. The tag on each bucket must indicate the character of the sugar and the particular half day on which its contents were drawn. The covers of the buckets must be kept carefully closed, except when momentarily opened to receive the sample. The buckets shall be made of heavy galvanized iron and have a height of 31.5 centimeters and a diameter of 18.5 centimeters. * * *

“ Art. 5. The sugar triers used shall have the following dimensions:

	Short Trier. Centimeters.	Long Trier. Centimeters.	Barrel Trier. Centimeters.
Length overall.	40.6	152.4	104.0
Length of spoon.	22.9	132.1	91.4
Length of shank.	17.8	20.3	12.7
Length of handle.	26.7	38.1	30.5
Width of spoon.	2.7	2.5	2.5
Depth of spoon.	0.8	1.3	1.1
Diameter of handle.	3.8	3.8	3.8

“ Art. 6. Sugar in hogsheads and other wooden packages shall be sampled by putting the long trier diagonally through the package from chime to chime, one trierful to constitute a sample, except in small lots, when an equal number of trierfuls shall be taken from each package to furnish the required amount of sugar necessary to make a sufficient sample. In the sampling of baskets, bags, ceroons, and mats the short trier shall be used, care being exercised to have each sample represent the contents of the package. The greatest precaution shall be taken that the samples from each class of packages shall be kept separate and be

uniform in quantity. When the hard condition of the sugar renders the use of the short trier impracticable the sampler in charge shall notify the appraiser immediately and await his instructions. The keys of the sugar buckets shall be in possession of the examiner of the appraiser's office, who shall have sole custody of such keys. * * *

"Art. 13. When practicable all refined sugars shall be sampled with a trier specially provided for that purpose, and such percentage shall be sampled as in the judgment of the examiner or sampler in charge will afford a fair representation of the mark. When there is doubt whether a package of sugar is raw or refined, or whether a raw sugar is above or below No. 16 Dutch standard in color, such package shall be sampled in not less than two places to fairly represent the contents, and such samples shall aggregate not less than eleven ounces. The package shall be numbered by the weigher and weighed separately, and the weigher's return shall show the number and weight of such package.

"Art. 14. The utmost care must be taken to keep all the apparatus used in the process of sampling sugar clean and absolutely dry. * * *

"Art. 15. Of importations of molasses, as near as possible to 100% of the packages shall be sampled. If any package or packages invoiced as molasses shall, in the judgment of the sampling officer, have the appearance of sirup of cane juice, a separate sample from each such package, properly labeled as to mark and character, shall be taken for examination as hereinafter provided.

"Art. 16. In drawing samples of molasses care shall be taken to secure a fair representation and an equal amount of the contents from each package. Packages of the same size shall be sampled in groups of not more than 25; samples from all of the packages of each group being put into a bucket. * * * Molasses discharged from tank vessels shall be sampled as it is pumped from the tanks, a sample of uniform quantity being drawn at either regular intervals

of approximately fifteen minutes or for every 5,000 gallons discharged.

“ Art. 17. Inasmuch as the absorption of sea water or moisture reduces the polariscopic test of sugar, there shall be no allowance on account of increased weight of sugar importations due to unusual absorption of sea water or of moisture while on the voyage of importation, and the provisions of Article 1276 of the Customs Regulations of 1899 shall not apply to sugar importations. That portion of the cargo claimed by the importer to have absorbed sea water or moisture on the voyage of importation shall be sampled, tested, and classified separately. This claim must be made at the time of weighing. Special care must be taken that such sugars are sampled so as to fairly represent the contents of the packages.”*

When it is not feasible to make a separate analysis of every mark in a lot a representative sample must be prepared; this can be done in the following manner. Fix upon some definite quantity by weight as the unit weight. Weigh out this amount, proportionate to the number of packages in each mark, and place in a well-closed jar.

For example, suppose a lot of sugar contained four marks, A, B, C, and D, and that:

-	Mark A = 1000 packages
	“ B = 200 “
	“ C = 350 “
	“ D = 70 “

Then take from:

A = 100 grams
B = 20 “
C = 35 “
D = 7 “

* For a full account of sampling raw sugars in different countries see Wiechmann, *Int. Sugar Jour.* Vol. IX, pp. 18-28.

When the composite sample is completed mix it thoroughly and proceed with the analysis.

As some lots come in mixed packages, for instance partly in hogsheads, bags, tierces and barrels a certain relation between these is sometimes assumed.

1	hogshead	=	2	tierces
1	“	=	8	barrels
1	“	=	8	bags.

Besides the packages mentioned sugar is also shipped in baskets, mats and ceroons.

Dutch Standard. The Dutch Standard referred to in the U. S. Government Regulations consists of a number of samples of cane sugar which range from dark brown (No. 7) to almost white (No. 25) in color. They are prepared and put up in glass bottles in Holland under the direction of the Netherlands Trading Society. The samples are renewed each year and serve as standards in the assessment of duties.

A. Determination of Sucrose in the Absence of Other Optically Active Substances. *Method I. With use of balance.* Sugar samples for analysis should be kept in cans, boxes or glass vessels which must be practically airtight.

When such samples are brought into the laboratory from out-of-doors or from a cold store-room they should be kept in the laboratory for a sufficient time to allow their contents to acquire the laboratory temperature in order that no moisture may condense on the cold sugar.

The sample should then be quickly but thoroughly mixed; if the sugar contains lumps it must first be thoroughly crushed and only then mixed.

The normal weight of the sample, 26.000 grams, if the metric c.c. flask, 26.048 grams if the Mohr c.c. flask is used, is weighed out in the tared sugar-cup provided for the purpose, the sugar is then partially dissolved and washed into a 100 c.c. flask with distilled water or with clear water

which is optically inactive, and its solution is completed in the flask. Not more than 75 c.c. or 80 c.c. at most, should be used to bring this about.

If the sugar solution is sufficiently light in color but possibly somewhat opalescent, a few drops of alumina cream * are added, and the volume of the solution is made up with water to the 100 c.c. mark on the neck of the flask.

If the sugar solution requires decolorization, a clarifying reagent must be added before the volume of the solution is brought up to 100 c.c.

The reagent used for this purpose is known as basic acetate of lead, or subacetate of lead.† It is very important that sufficient of this reagent is added to effect the desired clarification, but equally important that the addition of an excess of the reagent be avoided, for the volume of the precipitate which is formed causes an error in polarization.

Experience must guide as to the proper amount to be used. If the amount added is sufficient, the precipitate formed will, as a rule, settle quickly; in any case, one additional drop of the reagent is put into the solution. If this disappears in the solution, the quantity of the subacetate of lead added has been insufficient and an additional amount must be given; if, on the other hand, the drop can

* Alumina cream is made by dissolving aluminum sulphate or aluminum chloride in a considerable amount of cold water and the alumina hydrate precipitated by the addition of ammoniac hydrate. The precipitate is then thoroughly washed until all acid has been removed, and the creamy alumina hydrate, suspended in water, is kept for use.

† Subacetate of lead is prepared as follows: Take 3 parts by weight of acetate of lead, 1 part by weight of oxide of lead and 10 parts by weight of water. Boil these together—the water being added gradually, until practically all has been dissolved to a turbid solution. This is then set aside to settle and is finally filtered. Subacetate of lead must show an alkaline reaction to litmus, but it must not redden phenolphthalein. Its specific gravity must be about 1.25. In place of the acetate of lead and the oxide of lead, solid subacetate of lead may be used for making this solution.

be traced through the solution as it flows along the glass of the flask, no more is required.

In some instances a few drops of alumina cream may have to be used in addition to the subacetate of lead in order to secure solutions entirely free from opalescence.

When the clarifying reagent or reagents have been added, the solution is made up to the 100 c.c. mark on the flask, taking care that the lowest point of the meniscus of the liquid just touches that mark. The contents of the flask are then well shaken and the entire solution poured at once on a perfectly dry filter.

The first 20 cubic centimeters of the filtrate should be entirely rejected and only a perfectly bright and clear filtrate used for polarization. During said filtration both funnel and beaker should be covered to avoid concentration of solution by evaporation.

A 200 m.m. polariscope tube is then filled with the solution and the readings made. Several readings must be made on each solution tested and the average of all but the first reading—which is discarded, is recorded.

Should the solution be too dark in color to admit of its being polarized in the 200 m.m. tube the reading should be made in a 100 m.m. tube and the polarization doubled.

The use of specially prepared bone-black dust * as a decolorant for very dark colored solutions is sometimes resorted to, but it should not be used unless it is absolutely impossible to do without it.

The saccharimeters now in universal use record the amount of sucrose in per cent, provided the normal weight of the sample has been used, and the reading has been effected in a 200 m.m. tube; if a 100 m.m. tube has been used, the reading must be doubled.

* Warm for several hours with hydrochloric acid to dissolve the phosphate and carbonate of lime; then wash with boiling water till all traces of chlorine are removed; dry at about 125° C., and keep in a well-closed jar.

If for any reason the normal weight has not been taken, a simple calculation will serve to figure the percentage of sucrose in the sample. Suppose, for instance, that 18.000 grams had been weighed for polarization and that these were dissolved up to 100 c.c. A polarization of this solution in a 200 m.m. tube = 62.00.

As a rotation of one degree represents 0.26048 gram sucrose (Mohr flask) there are contained in the sample $0.26048 \times 62 = 16.14976$ grams pure sucrose.

Hence $18.000 : 16.14976 :: 100 : x$. $x = 89.72$.

Therefore the sample contains 89.72% sucrose.

A more direct way of figuring this is by means of the formula:

$$\frac{P \times W'}{W} = \text{per cent sucrose.}$$

P = polarization of the solution;

W' = normal weight of the instrument used;

W = weight of substance taken for polarization.

Example. $\frac{62.0 \times 26.048}{18.0} = 89.72$.

Results so obtained can be verified by calculating the amount of sugar which would be necessary in order to indicate 100° on the polariscope. This is known as Scheibler's method of "One hundred polarization."

Example. In the case just discussed, a polarization of 89.72 required 26.048 grams of the sugar: how much will be required to produce a rotation of 100° on the instrument?

$$89.72 : 26.048 :: 100 : x. \quad x = 29.0325.$$

Therefore 29.0325 grams of this sample are polarized in the usual manner, and if they indicate 100% the result previously obtained, is correct,

Table VII, by Scheibler, obviates the necessity of this calculation, showing at once the amount that must be used.

Method II. Without use of Balance. The percentage of sucrose in a sample can also be obtained without making a weighing. A solution is made and the specific gravity of the solution is determined, either directly by a specific-gravity hydrometer or else by some other hydrometer (Brix, Baumé), the readings of which are translated into the corresponding specific gravity (Table I).

The polarization of the solution is then made, and the percentage of sucrose calculated by the formula:

$$S = \frac{P \times .2605}{D},$$

in which S = percentage of sucrose,
 P = polarization of the solution,
 D = specific gravity.

If the solution needs clarifying, it is placed into a graduated flask, the amount of basic acetate of lead solution that is added, is noted, and the reading increased in proportion.

Example. Specific gravity of solution, 1.0909;
 Polarization of solution = 35.0.

To 100 c.c. of solution added 5 c.c. basic acetate of lead solution; this corresponds to 5% of 35.0 = 1.75.

Hence corrected polarization = 36.75 per cent.

$$\frac{36.75 \times .2605}{1.0909} = 8.77 \text{ per cent sucrose.}$$

This calculation can be avoided by consulting Table VI. This table is used in the following manner:

Example. Corrected specific gravity = 1.0339;
 Polarization = 25.0.

In a line with the specific gravity 1.0339, and in the horizontal column marked 2, is found the number .504. This multiplied by 10 = 5.040.

In a line with the specific gravity 1.0339, and in the column marked 5, is found the number 1.260.

$$\begin{array}{r} \text{Adding these values,} \quad 5.040 \\ \qquad \qquad \qquad \qquad \qquad \quad 1.260 \\ \hline \text{Percentage of sucrose} = 6.300 \end{array}$$

The simple polarization of a sugar, syrup, liquor, magma, or sweet-water shows the percentage of sucrose in the sample as it is. Sometimes, however, it is necessary to know what this percentage would be if the water in the sample were removed; in other words, it may be desirable to ascertain the percentage of sucrose in the "dry substance."

Coefficient of Purity, or exponent, is the term which is applied to this value. There are several ways in which this can be determined. The most accurate method undoubtedly, but also the one which demands most time for its execution, is the following.

Method I. Determine polarization of the normal weight of the sample as previously described. Determine the percentage of water by drying to constant weight. Subtract the percentage of water from 100, and divide the remainder into the polarization multiplied by 100.

<i>Example.</i>	Polarization of syrup,	33.00;
	Water in syrup, per cent,	24.16.
	100.00	
	<u>24.16</u>	
	75.84	$3300 \div 75.84 = 43.5$

Polarization on dry substance = 43.5.

Method II. Determine polarization of the normal weight of the sample as previously described. Determine the degree Brix of the sample. Correct for temperature (Table III).

Calculate polarization on the dry substance by the formula:

$$\frac{\text{Pol.} \times 100}{\text{Degree Brix}}$$

Example. Polarization, 40.00;
 Density, 50° Brix at 24° C.;
 Correction for temperature, +0.49
 Degree Brix corrected for temperature,
 = 50.49.

100.00 ÷ 50.49 = 1.9806, factor;

40.00 × 1.9806 = 79.22, polarization on the dry substance,
 or coefficient of purity.

Method III. Ventzke's Method. Prepare a solution of the sugar which shall have the specific gravity 1.100 at 17.5° C. Take the reading of this solution in a 200 m.m. tube. This polariscope reading shows at once the percentage of *pure* sugar in the dry substance. This is the case, because a solution made by dissolving 26.048 grams of chemically pure sugar in water up to 100 Mohr c.c. has the specific gravity of 1.1000 at the temperature of 17.5° C., and a column of this solution 200 m.m. in length, indicates 100% in polariscopes of the German type.

The following table prepared by Gerlach shows the specific gravity of the above solution at the temperatures given:

Temperature. ° C.	Specific Gravity.	Temperature. ° C.	Specific Gravity.	Temperature. ° C.	Specific Gravity.
0	1.10324	16.5	1.10028	23	1.09834
5	1.10266	17	1.10014	24	1.09802
10	1.10192	17.5	1.10000	25	1.09770
11	1.10168	18	1.09986	26	1.09736
12	1.10144	18.5	1.09972	27	1.09702
13	1.10119	19	1.09957	28	1.09669
14	1.10095	19.5	1.09943	29	1.09635
15	1.10071	20	1.09929	30	1.09601
15.5	1.10057	21	1.09897		
16	1.10043	22	1.09865		

As the preparation of a solution which is to have a certain specific gravity at a certain temperature is apt to prove a tedious operation, the following modification of Ventzke's method will prove serviceable:

If the temperature at which the solution is prepared is not the normal temperature, a correction must be made (Table II).

This correction must be subtracted from the reading of the specific-gravity hydrometer if the temperature is lower than the normal, and added, if it is above the normal temperature.

The polarization obtained in the 200 m.m. tube must then be multiplied by the factor corresponding to the corrected specific gravity (Table IV).

Method IV. Casamajor's Method. Determine the specific gravity or the degree Brix of the solution. Correct for temperature if necessary (Table III). Determine the polarization of this solution and multiply the polarization by the factor corresponding to the degree Brix (Table V).

Example. Polarization of solution = 61.2;
 Brix, = 15.5° at 22° C.;
 Correction for temperature, +0.31
 Corrected degree Brix = 15.81;
 Factor corresponding to 15.8° Brix is 1.548

$61.2 \times 1.548 = 94.74$, which is the polarization on the dry substance, the coefficient of purity.

The coefficient of purity obtained by Method I (where the percentage of water is obtained by actual drying out), is called the "true" coefficient of purity; if hydrometers are resorted to, as in Methods II, III, and IV, the resulting coefficient is called the "apparent" coefficient of purity.

If a syrup or a molasses has been analyzed, the results of the analysis can easily be calculated into equivalents on the dry substance in the following manner:

The reciprocal of the degree Brix (that is, the quotient obtained by dividing 100 by the degree Brix), gives a factor by which the percentage of sugar, invert sugar, and ash must be multiplied in order to reduce them to the basis of dry substance.

Example. A syrup of 80.4° Brix shows on analysis:

Polarization, 31.2;
Invert sugar, 12.5;
Ash, 6.0.

$$100 \div 80.4 = 1.2437.$$

On Dry Substance.

Hence: Polarization,	$31.2 \times 1.2437 = 38.80$	per cent
Invert sugar,	$12.5 \times 1.2437 = 15.55$	“
Ash,	$6.0 \times 1.2437 = 7.46$	“
Non-ascertained (by difference)	$= 38.19$	“

100.00 per cent.

If sucrose has to be determined in a molasses, a syrup, or in sweet-water, the calculation of the result to dry substance can be avoided by aid of Table VIII.

This table has been calculated for use with the German polariscopes (normal weight 26.048 grams). It presupposes the addition of 10% by volume of basic acetate of lead to the sucrose solution examined, and in its preparation the variable specific rotatory power of sucrose has also been taken into account.

The use of the table is very simple.

Example. Density of a sugar solution, 22.0° Brix. Polarization (after using 10% by volume of basic acetate of lead solution for clarifying), 60.3.

In column headed 22.0° Brix, and opposite to the number 60 in the column headed “Polariscope degrees,” we find 15.72% sucrose. Then, turning on the same page to the division for tenths of a degree, in the section headed

“Per cent Brix from 11.5 to 22.5,” there is given opposite to 0.3 Brix the value 0.08% sucrose.

Hence $60.0^{\circ} = 15.72$ per cent.

$0.3^{\circ} = 0.08$ “

$60.3^{\circ} = 15.80$ per cent sucrose.

Sucrose in Fill Mass. Weigh out 250 grams of the massescuite, add 250 grams of water, carefully dissolve all crystals and insure a thorough mixing of the sample. Determine and note the degree Brix of this solution without temperature correction and then use a Spencer sucrose pipette. This is a pipette so graduated that if filled to the mark corresponding with the observed, i.e. the uncorrected degrees Brix, it will deliver twice the normal weight of the liquid. If the normal sugar-weight used is 26.048 grams, then 52.096 grams of solution will be delivered—if 26.000 grams is the normal sugar-weight employed, then 52.000 grams of solution must be delivered. The range of density for which these sucrose pipettes are graduated is usually from 5° to 25° Brix. Discharge the contents of this pipette into a 100 c.c. flask, clarify with basic lead acetate solution, using from 3 to 5 c.c. of this reagent, make the volume up to 100 c.c. with water, mix well, filter and polarize.

$$\text{Purity.} \quad \text{Real purity} = \frac{\text{Sucrose} \times 100}{\text{Dry Substance}}$$

$$\text{Apparent purity} = \frac{\text{Polarization} \times 100}{\text{Degrees Brix}}$$

Sucrose in Condensation, Boiler-feed and Waste Water.

The zero point of the polariscope must be accurately determined by means of the 400 m.m. tube filled with distilled, or at least with optically inactive water. The limit of accuracy of the average saccharimeter may be taken as $\pm 0.05^{\circ}$ Ventzke or possibly even as $\pm 0.10^{\circ}$ Ventzke, and

this limitation must be borne in mind in accepting the findings of such polarimetric determinations.

A sample of condensation-water, for instance, is examined as follows: Note the degree Brix of the solution. Measure out 100 c.c., if need be, clarify with basic lead acetate solution or with alumina cream, dilute up to 110 c.c., shake well, filter and polarize. Increase the reading by $\frac{1}{10}$, divide the corrected reading by 2, as the 400 m.m. tube has been used. Take the figure in the Schmitz table under the degrees Brix nearest that observed—the column headed “Per cent Sucrose” in the table then shows the value sought.

Example.

Degree Brix of solution	=0.54
Polariscope reading observed in 400 m.m. tube	=0.20
Corrected pol. reading ($+\frac{1}{10}$)	=0.22
Divide this by 2 as 400 m.m. tube was used	=0.11

Consult Schmitz's Table.

Degree Brix from 0.5 to 12.0.	
Tenths of Polariscope Degrees.	Per Cent Sucrose.
0.1	0.03
0.2	0.06
0.3	0.08
0.4	0.11
0.5	0.14
0.6	0.17
0.7	0.19
0.8	0.22
0.9	0.25

As will be seen, the polariscopic reading in the Schmitz table, nearest to 0.11 is 0.1, and this corresponds to 0.03% sucrose; this therefore is the percentage amount of sucrose in the condensation water examined.

If preferred, the sucrose in condensation and other waters can also be determined as invert sugar.

Possible Sources of Error in Polarization. In all determinations of sucrose by the saccharimeter, there are numerous sources of error which must be guarded against most carefully. These shall now be briefly referred to.

1. As already mentioned, the sample must be permitted to acquire the temperature of the room in its closed receptacle before it is taken out immediately before analysis. This is done to guard against its drying out, which would entail too high a polarization, or, on the other hand, to prevent a condensation of moisture on the sample if the latter be colder than the room in which it is to be analyzed. For the same reasons all mixing of the sample and its weighing, must be done as quickly as possible.

Concentration of the sugar solution during filtration must be avoided by keeping both funnel and receiving flask, or beaker, covered during the operation.

2. The polariscope must be set up in a place as free as possible from all jar and vibration of machinery. A thick plate of soft rubber placed beneath the instrument is helpful to this end.

The saccharimeter must be in a dark room, free from all interfering lights, yet the air must be allowed to circulate freely around it and the temperature must be kept as even as possible in the polariscope booth.

3. Polarizations must be made only on solutions which are perfectly clear and limpid and which show a perfectly circular field in the instrument. The scale-reading must be taken only when the halves of the circular field are of equal intensity to the eye of the observer.

4. An excess of subacetate of lead must be avoided in clarification. The 100° mark on a saccharimeter is determined by a length of 200 m.m. of a standard sugar solution obtained by dissolving the normal weight of pure sucrose in pure water and bringing the volume up to 100 cubic

centimeters at some given standard temperature, 20° C. is prescribed by the International Commission.

Any precipitate occupying a part of this space of 100 cubic centimeters which should be filled by the solution, naturally causes the solution to be too concentrated and therefore results in polarizations that are too high.*

If account is to be taken of this error and the same allowed for, use can be made of the double dilution method, due to Scheibler, and which is as follows:

The normal weight of sugar is dissolved in distilled water; a measured amount of subacetate of lead solution is added, the volume is made up to 100 c.c. and the solution filtered and polarized.

A second solution is prepared in the same manner, except that its volume is made double the volume of the former solution, that is to say to a volume of 200 c.c.; this solution is then also filtered and polarized.

Assuming that the volume of the lead precipitate formed is identical in both cases, it is evident that the polarization of the more dilute solution must be somewhat less than one half the polarization of the more concentrated solution.

The corrected polarization is then found as shown in the following example:

Polarization of 1st solution (100 c.c. in volume) = 96.80

“ 2d “ (200 c.c. in volume) = 48.25

$48.25 \times 2 = 96.50$

$96.80 - 96.50 = 0.30 \times 2 = 0.60$ and,

$96.80 - 0.60 = 96.20$ the corrected polarization.

Or, expressed more briefly,

corrected polarization = $(48.25 \times 4) - 96.80 = 96.20$.

To correct the errors of Scheibler's method Sachs washes the precipitate obtained in clarification with hot and cold

* Wiechmann, School of Mines Quarterly, Columbia University, Vol. XXV.

water until all sugar has been removed. This precipitate is then placed in a 100 c.c. flask, a half normal weight of sucrose is added and the volume, after complete solution has been effected, is made up to 100 c.c. After filtration, the polarization is made in a 400 m.m. tube.

Let P = true polarization of the sucrose used,

P' = polarization of sucrose plus the precipitate, then the volume of the precipitate is obtained by the formula

$$v = \frac{100(P' - P)}{P'}$$

and the correct polarization where the volume of solution = 100 c.c. is found by the formula:

$$P = \frac{100 \times P' - vP'}{100}$$

Example. Polarization of normal weight of a sugar, made up to 100 c.c. solution, = 95.0° Ventzke. Volume of precipitate formed $v = 0.5$ c.c.

the correct polarization of the sugar will be:

$$P = \frac{(100 \times 95.0) - (0.5 \times 95.0)}{100} = 94.52$$

Wiechmann * dries the washed precipitate and from its weight and specific gravity calculates its volume by the formula:

$$v = \frac{w}{\text{sp.gr.}}$$

and then determines the correct polarization by use of the formula above given.

* Proc. Fifth Int. Cong. Applied Chemistry, 1904.

A convenient and practical method of obviating the precipitate-volume error has been devised by Horne.* His description follows.

“The normal weight of sugar is dissolved in water in a 100 c.c. flask and made up to the mark without defecation. The concentration is thus at exactly the proper degree. It now remains to defecate the solution properly by precipitating the impurities in such a way as to produce the minimum change in the concentration of the solution of sucrose. This is accomplished by adding to the 100 c.c. of liquid small quantities of powdered anhydrous lead subacetate until the impurities are nearly all precipitated. This point is as easily determined as in the defecation by a solution of the same salt. The organic and mineral-acid radicals in the solution combine with and precipitate the lead and lead oxide of the dry salt, while the acetic-acid radical of the lead subacetate passes into solution to combine with the bases originally united to the other acid radicals.”

5. The quartz control plates must be mounted in such a manner that they shall be absolutely under no pressure or strain, as otherwise they incur marked changes in their optical value.†

6. The cover glasses of the polariscope tubes must be optically inactive and put under no strain by the cover-caps. Bayonet cover-caps are preferable for this purpose to the screw-caps in general use.

7. As changes in temperature affect both the optical rotation value of sugar solutions and of the polariscope, accurate readings can only be secured when:

1st. The polariscope is at the temperature at which it was graduated;

* J. Am. Chem. Soc., Vol. XXVI, p. 186.

† Wiechmann: School of Mines Quarterly, Columbia University, Vol. XX.

2d. The sugar solution of the normal weight of sugar, is made up to 100 c.c. at that same temperature;

3d. Is polarized at that same temperature.

The International Commission for Uniform Methods of Sugar Analysis accepted 20° C. as the standard temperature, and therefore, in conformity with the directions of that Commission, the saccharimeter must be graduated at 20° C., the sugar solution must be made up at 20° C. and must be polarized at 20° C.

The Commission however also authorized that, "for those countries, the temperature of which is generally higher, it is permissible that the saccharimeters (polariscopes) be adjusted at 30° C., or any other suitable temperature, under the conditions specified above, and providing that the analyses of sugar be made at that same temperature." *

7. The source of light must be carefully adjusted so that an absolutely uniform and sufficiently intensive illumination of the polariscopic field is insured. It should moreover be placed at a distance sufficiently far from the instrument to prevent any heating of the latter. White light such as emanates from a Welsbach burner or from an electric incandescent bulb, must never be used unless, before entering the polariscope, it is made to pass through a layer 1.5 cm. thick of a 6% aqueous solution of potassium dichromate.

B. Determination of Sucrose, in the Presence of Other Optically Active Substances. The determination of sucrose by the method now to be described is based on the fact that sucrose is changed by the action of acid from a dextro-rotatory substance into a levo-rotatory mixture of dextrose and levulose. The method is particularly applicable when

* In this connection see also: C. A. Browne, Seventh International Congress of Applied Chemistry, Vol. XIX.

Horne, Journal of Industrial and Engineering Chemistry, 1912, p. 41.

dextrose or starch sugar, considerable amounts of invert sugar, or when raffinose is present together with the sucrose.

This method is known as Clerget's method, but it has received some modifications by other investigators. According to Herzfeld's modification it is carried out as follows:

Clerget-Herzfeld Method.* Weigh out 26.00 grams of the sample, and determine the polarization as usual at 20° C. Of the solution, take 50 c.c. for inversion, or weigh out separately 13.00 grams of the sample. Dissolve with about 75 c.c. of water in a 100 c.c. flask; add, after complete solution has been effected, 5 c.c. hydrochloric acid (sp.gr. 1.188), containing 38 per cent HCl. Heat quickly, in two or three minutes, on a water-bath up to between 67° and 70° C. Then keep the temperature of the solution for five minutes as close to 69° C. as possible. Agitate constantly. Then cool quickly to 20° C., fill with distilled water up to the 100 c.c. mark, and polarize in a tube provided with an accurate thermometer. The temperature at which the reading is taken should be 20° C. and the temperature of the saccharimeter must also be 20° C.

The use of subacetate of lead for clarifying purposes is not permissible as this reagent affects the rotatory power of invert sugar. If a decolorant must be used, a little, specially prepared blood-carbon should be employed.

The result is calculated as follows:

Let R = sucrose,

S = sum of the two polarizations before and after inversion, the minus sign being ignored,

t = degrees Centigrade at which the polarization after inversion was observed,

* For use of invertase as hydrolyst see International Sugar Journal, 1911, p. 145. For use of hydrochloric acid and urea for determining sucrose in cane molasses, *ibid.*, p. 206.

then,

$$R = \frac{100 \times S}{142.66 - (0.5 \times t)} *$$

The two polarizations—the one before, the other after inversion, must always be made at one and the same temperature, because the optical rotation value of invert sugar is materially influenced by temperature changes. As the International Commission has accepted 20° C. as the standard temperature, 20° C. is used in the above formula

$$R = \frac{100 \times S}{142.66 - (0.5 \times 20)}$$

which, of course, is equivalent to:

$$R = \frac{100 \times S}{132.66}$$

As $100 \div 132.66 = 0.7538$, the formula may be given its simplest expression thus:

$$R = 0.7538 \times S$$

It is best to carry out both determinations at 20° C. if possible. If, however, the determinations are made at any other temperature between 10° C. and 30° C., Table X gives a series of factors by which it is necessary to multiply the sum of the indications, before and after inversion.

Example. Direct polarization, 86.0 at 22° C.

Polarization of half normal weight after
inversion—12.5 at 22° C.— $12.5 \times 2 = 25.0$
 $86.0 + 25.0 = 111.0$.

* The value 142.66 in this formula is based on the fact that a pure normal weight sugar solution which polarizes 100° Ventzke at 0° C. in a 200 m.m. tube, polarizes, after complete inversion—42.66° so that the entire decrease in rotation at 0° C. = 142.66. Account must be taken of the fact that the rotatory value of invert sugar decreases 0.5° for each 1° C. temperature elevation above 0° C.

Referring to Table X, opposite to 22° C. there will be found the factor 0.7595. Multiplying $111 \times 0.7595 = 84.3$; this is the desired result.

If any other weight than 13.00° grams is used for the determination, the value 142.66 in the formula $R = \frac{100S}{142.66 - \frac{1}{2}t}$ does not give correct results, because the specific rotatory power of an invert sugar solution varies with the degree of concentration of the solution.

The following table shows the value that under the circumstances, must be used in its stead.

Grams of Sucrose in 100 c.c.	Use Factor.	Grams of Sucrose in 100 c.c.	Use Factor.
1	141.85	11	142.52
2	141.91	12	142.59
3	141.98	13	142.66
4	142.05	14	142.73
5	142.12	15	142.79
6	142.18	16	142.86
7	142.25	17	142.93
8	142.32	18	143.00
9	142.39	19	143.07
10	142.46	20	143.13

If no other optically active body is present besides the sucrose, the Clerget polarization will of course return a value equivalent to the direct polarization value originally found.

Example. Polarization of normal weight before inversion, 87.5, at 20° C.

Polarization of half normal weight after inversion, -14.3 at 20° C.

$$\begin{array}{r}
 -14.3 \times 2 \\
 -28.6 \\
 \hline
 \end{array}
 \qquad
 \begin{array}{r}
 87.5 \\
 28.6 \\
 \hline
 116.1
 \end{array}
 \qquad
 R = \frac{100 \times 116.1}{142.66 - 10}$$

$$R = \frac{11610}{132.66} = 87.5$$

Sucrose and Invert sugar. The following case is a so-called invert sugar syrup, essentially a mixture of sucrose and invert sugar.

Direct polarization at 20° C. = $+15.0^{\circ}$

Polarization of half normal weight after inversion at 20° C = -13.0°

$$-13.0 \times 2 = -26.0, \text{ hence:}$$

$$S = 15.0 + 26.0 = 41.0$$

and

$$R = 0.7538 \times 41 = 30.90\%$$

That is to say this mixture contains 30.90% of sucrose.

Sucrose and Dextrose. In this instance some glucose syrup had been mixed with cane syrup.

Direct polarization, at 20° C. = $+72.0$

Polarization of half normal weight after inversion at 20° C. = $+9.0$

$$+9 \times 2 = 18.0$$

Hence $S = 72 - 18.0 = 54.0$

$$R = 0.7538 \times 54 = 40.70\%$$

Hence this mixture contains 40.70% sucrose.

Sucrose and Commercial Glucose mixtures can be analyzed optically by using the method of hot polarization due to Chandler and Ricketts.*

* Abstracted from Second Annual Report of the State Board of Health of New York, 1882.

This method depends upon the following well-known facts:

1. *Dextrose*, under the conditions of analysis, exerts a constant effect upon the plane of polarized light at all temperatures under 100° C.

2. *Levulose*. The action of levulose is not constant, the amount of rotation to the left being diminished as the temperature is increased.

3. *Invert sugar*, being a mixture of one half dextrose and one half levulose, does not affect the plane of polarized light at a certain temperature, somewhere near 87° C. (for it can easily be seen that the constant dextro-rotatory power of dextrose must be neutralized by the varying levo-rotatory power of levulose at some such temperature. The exact temperature is determined by experiment).

4. *Cane sugar*, when acted on by dilute acids, is converted into invert sugar, while dextrose remains practically unaltered.

Hence, if a "mixed sugar" is heated with dilute acids, the cane sugar present is converted into invert sugar, which, with that originally present due to the process of manufacture, is optically inactive at a certain temperature (near 87° C.); while the commercial glucose added, preserving its specific rotatory effect, will at this temperature show a deviation to the right in proportion to the amount present and according to its nature.

It is only necessary, therefore, to secure some means of heating the observation tube of the ordinary polariscope, so that readings may be taken at any temperature under 100° C. The middle section of a saccharimeter ordinarily intended for the observation tube alone, is so modified as to admit of the interposition of a metallic water-bath, provided at the ends with metal caps, which contain circular pieces of clear plate-glass. The tube for holding the sugar solution to be polarized, is provided with a tubule for the insertion of a thermometer in the sugar solution. The

metallic caps at the end of the tube rest on projecting shelves inside the water-bath, thus bringing the tube into the center of the bath, where it is completely surrounded by water. The cover of the water-bath is arranged for the insertion of a thermometer, so that the temperatures of the water-bath and of the sugar solution may both be ascertained. The water-bath is heated by spirit-lamps, gas-burners or some other heating device. By experiment it has been found that about 87° C. is the temperature at which the reading of a pure inverted sugar solution is *zero* on the sugar scale.

In working out this method it was next found necessary to determine the value of a degree of the scale in terms of the glucose known to be the variety used to adulterate cane sugar. It was found that the rotation to the right at 86° C. was 41°, when using a solution containing in 100 c.c. fifteen grams of a sample containing 85.476 per cent chemically pure glucose. Hence as fifteen grams was the amount taken, $15 \times \frac{85.476}{100} \div 41 \times 100 = 31.2717$ grams, which represents the amount of chemically pure glucose necessary to read one hundred divisions on the sugar scale of the instrument used; or, each division = 0.312717 grams chemically pure glucose. (A duplicate determination made, by using 26.048 grams, gave as a factor 0.312488.)

The success of the process depends greatly upon the care exercised in preparing the sugar solution for the polariscope. The inversion and subsequent clarification were accomplished as follows:

26.048 grams of the sugar to be examined were completely dissolved in about 75 c.c. of cold water, and were treated with 3 c.c. of dilute sulphuric acid (1 to 5 by volume) on a water-bath at a temperature of about 70° C. for thirty minutes. The solution thus inverted was then rapidly cooled, nearly neutralized with sodium carbonate solution (saturated), transferred to a 100 c.c. flask, and the gummy matters, etc., precipitated with 5 c.c. of a solution of basic

lead acetate. The flask was then filled to the mark, the solution transferred to a small beaker, mixed with enough bone-black to clarify completely, and then thrown on a fluted filter. The amount of bone-black necessary to effect decolorization depends on the grade of the sugar and on the color of the solution. It was not found necessary to use, even with sugars of the lowest grade, more than five grams.

The clarified inverted sugar solution was then placed in the polarization tube, the water-bath was filled with cold water, the thermometers were adjusted, and the temperature gradually raised to 86° C. This part of the operation should take about thirty minutes. If the sample is not adulterated, the polariscope reading will be zero at 86° C., while if glucose is present the amount of deviation to the right, in degrees and fractions, multiplied by the proper factor and divided by the amount taken, will give the percentage of glucose added as an adulterant.

The specific rotation of commercial glucose varies from about +100 to +125 for the liquid product. The value $[\alpha]_D + 108$ is however generally employed.

In using this method it is however very important not to overlook the fact that the liquid which is made, say at 20° C. and polarized at say 87° C., expands considerably and that therefore a correction must be made to allow for the dilution due to such expansion. 100 c.c. of water at 20° C. expand to 103.15 c.c. at 87° C. and therefore the factor 1.0315 must be used for multiplying the polarization observed at 87° C. to effect this needed correction.*

An additional difficulty in using this method lies in the fact that it is not possible to get as clear and accurate readings on solutions at high temperatures as at 20° C., owing to the fact, as determined by Browne, that striations are very apt to occur in the solution under such observation,

* C. A. Browne, Bureau of Chemistry, Bulletin No. 110, Washington, 1908.

unless the same is of an absolutely uniform temperature throughout.

Sucrose and Levulose solutions can also be analysed in a similar manner. Wiley * has shown that 1 gram of levulose in 100 c.c. of solution shows a variation of 0.0357° V. for each degree Centigrade.

To express this in a formula:

Let

F = grams of levulose in 100 c.c. of solution

P' = degrees Ventzke at high temperature t'

P = " " low temperature t

$$F = \frac{P' - P}{0.0357(t' - t)}$$

which means, that the difference between the direct polarization of a sucrose-levulose solution at 87° C. and at 20° C., for instance, divided by $0.0357 (87^{\circ} - 20^{\circ})$ gives the grams of levulose in 100 c.c. of a solution so examined.

Sucrose and Raffinose. The analytical details of the Clerget method to be followed, when dealing with mixtures of sucrose and raffinose, are precisely as previously given, however a different formula must be used in calculating the results, because the dextro-rotation of the raffinose is also affected by the inversion brought about by the use of the acid.

The specific rotation of raffinose decreases during the inversion from $+104.5$ to $+53.5$

Using metric cubic centimeters, the normal weight for the raffinose hydrate is 16.545 grams, and 14.037 grams for the anhydride.

These amounts of raffinose show before inversion a polarization of $+100^{\circ}$ V., after hydrolysis, a decrease of 48.76° Ventzke, i.e. after hydrolysis they show a polarization of $+51.24^{\circ}$ V. at 20° C.

* Principles and Practice of Agricultural Analysis, 1897, Vol. III, p. 267.

Herzfeld's modification of Creydt's formulæ, which were based on the old Clerget process, are at present generally used.

Let the normal weight (26.00 grams) of a sample consisting of $S\%$ sucrose and $R\%$ raffinose anhydride be dissolved to 100 metric c.c. at 20° C. and polarized at 20° C.

The polarization of the sucrose will be represented by S , that of the raffinose by:

$$\frac{26.000}{14.037} = 1.852 R$$

in other words, this 1.852 is the ratio of the normal weight of raffinose anhydride to that of sucrose. Let the sum of the direct polarizations of the sucrose and raffinose = P

Then:

$$P = S + 1.852 R$$

$$R = \frac{P - S}{1.852}$$

$$S = P - 1.852R.$$

Inversion having been effected, let P' represent the sum of the sucrose and raffinose invert polarizations, then

$$P' = -0.3266S + 0.9490R.$$

$$R = \frac{0.3266P + P'}{1.554}$$

$$S = \frac{0.5124P - P'}{0.839}$$

Example. Direct Pol. = 51.0

Pol. after inversion = -12.5

$$S = \frac{0.5124 \times 51 - (-12.5)}{0.839} = 46.04\%$$

$$R = \frac{0.3266 \times 51 + (-12.5)}{1.554} = 2.67\%$$

This sample therefore contains

$$\text{Sucrose} = 46.04\%$$

$$\text{Raffinose} = 2.67\%$$

If the observation of the inverted raffinose solution has not been made at 20° C. a correction of 0.0038° for each degree Centigrade above or below 20° C. must be introduced. This correction is effected by the formula.*

$$\left. \begin{array}{l} \text{Polarization} \\ \text{after inversion} \\ \text{at } 20^{\circ} \text{ C.} \end{array} \right\} = \left\{ \begin{array}{l} \text{Polarization} \\ \text{after inversion} \\ \text{at } t^{\circ} \text{ C.} \end{array} \right\} + 0.0038S(20-t),$$

in which S represents the sum of the polarizations before and after inversion.

Example. Suppose a solution of sucrose and raffinose polarized:

Before inversion, 105.0°;

After inversion, -22.0° at a temperature of 18.2° C.

Then the polarization after inversion at 20° C. will be equal to:

$$\begin{aligned} & -22.0 + 0.0038(105.0 + 22.0)(20.0 - 18.2) \\ & -22.0 + 0.0038(+127.0)(+1.8) \\ & -22.0 + 0.86868 \\ & = -21.13 \end{aligned}$$

The following table by Hammerschmidt † permits the ready determination of the value $0.0038S(20-t)$ occurring in above formula, and proves of great convenience where many calculations of this kind are called for.

* Zeitschrift des Vereines für Rübenzucker-Industrie, Vol. XL, p. 201.

† Correspondenz-blatt des Vereines Akademischgebildeter Zucker techniker, Vol. I, No. 3.

$S =$	134	132	130	128	126	124	122	120
$20-t=1$	0.51	0.50	0.49	0.49	0.48	0.47	0.46	0.46
2	1.02	1.00	0.99	0.97	0.96	0.94	0.93	0.91
3	1.53	1.50	1.48	1.46	1.44	1.41	1.39	1.37
4	2.04	2.01	1.98	1.95	1.92	1.88	1.85	1.82
5	2.55	2.51	2.47	2.43	2.39	2.36	2.32	2.28
6	3.06	3.01	2.96	2.92	2.87	2.83	2.78	2.74
7	3.56	3.51	3.46	3.40	3.35	3.30	3.25	3.19
8	4.07	4.01	3.95	3.89	3.83	3.77	3.71	3.65
9	4.58	4.51	4.45	4.38	4.31	4.24	4.17	4.10

$S =$	118	116	114	112	110	105	100	95
$20-t=1$	0.45	0.44	0.43	0.43	0.42	0.40	0.38	0.36
2	0.90	0.88	0.87	0.85	0.84	0.80	0.76	0.72
3	1.35	1.32	1.30	1.28	1.25	1.20	1.14	1.08
4	1.79	1.76	1.73	1.70	1.67	1.60	1.52	1.44
5	2.24	2.20	2.17	2.13	2.09	2.00	1.90	1.81
6	2.69	2.64	2.60	2.55	2.51	2.39	2.28	2.17
7	3.14	3.09	3.03	2.98	2.93	2.79	2.66	2.53
8	3.59	3.53	3.47	3.40	3.34	3.19	3.04	2.89
9	4.04	3.97	3.90	3.83	3.76	3.59	3.42	3.25

$S =$	90	85	80	75	70	65	60	55	50
$20-t=1$	0.34	0.32	0.30	0.29	0.27	0.25	0.23	0.21	0.19
2	0.68	0.65	0.61	0.57	0.53	0.49	0.46	0.42	0.38
3	1.03	0.97	0.91	0.86	0.80	0.74	0.68	0.63	0.57
4	1.37	1.29	1.22	1.14	1.06	0.99	0.91	0.84	0.76
5	1.71	1.62	1.52	1.43	1.33	1.24	1.14	1.05	0.95
6	2.05	1.94	1.82	1.71	1.60	1.48	1.37	1.25	1.14
7	2.39	2.26	2.13	2.00	1.86	1.73	1.60	1.46	1.33
8	2.74	2.58	2.43	2.28	2.13	1.98	1.82	1.67	1.52
9	3.08	2.91	2.74	2.57	2.39	2.22	2.05	1.88	1.71

$S =$	45	40	35	30	25	20	15	10	5
$20-t=1$	0.17	0.15	0.13	0.11	0.10	0.08	0.06	0.04	0.02
2	0.34	0.30	0.27	0.23	0.19	0.15	0.11	0.08	0.04
3	0.51	0.46	0.40	0.34	0.29	0.23	0.17	0.11	0.06
4	0.68	0.61	0.53	0.46	0.38	0.30	0.23	0.15	0.08
5	0.86	0.76	0.67	0.57	0.48	0.38	0.29	0.19	0.10
6	1.03	0.91	0.80	0.68	0.57	0.46	0.34	0.23	0.11
7	1.20	1.06	0.93	0.80	0.67	0.53	0.40	0.27	0.13
8	1.37	1.22	1.06	0.91	0.76	0.61	0.46	0.30	0.15
9	1.54	1.37	1.20	1.03	0.86	0.68	0.51	0.34	0.17

CHAPTER V

SUCROSE DETERMINATION BY CHEMICAL ANALYSIS

Qualitative Test for Sucrose. A delicate test for the presence of sucrose is afforded by the use of α -naphthol.

Place 2 c.c. of the solution to be examined in a test-tube. Add 2 drops of a 10% solution of α -naphthol in alcohol. Then allow about 5 c.c. of concentrated sulphuric acid to flow down the side of the test-tube and settle at the bottom.

If any appreciable quantity of sucrose is present a violet color zone will appear at the line of contact of the two liquids. This color is caused by reaction products between the decomposition products of the sugar and of the phenol derivatives.

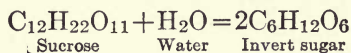
As nitric acid interferes with the reaction care must be used that this is not present. Ammonia, lime salts and most organic impurities found in water do not disturb the test. Solutions of invert sugar however also give the violet color reaction and this must be borne in mind in interpreting the test.

As there is no absolutely distinctive chemical test for sucrose, it is best to transform the sucrose into a saccharate—strontium bisaccharate for instance—to set the sucrose free from the strontium by precipitating the same as carbonate and then to identify the sucrose optically or otherwise.

Quantitative Determination of Sucrose as Invert Sugar. This is effected by taking advantage of the fact that sucrose

by inversion with acids or invertase, is changed into invert sugar which reduces alkaline copper solutions.

According to the reaction:



95 parts of sucrose yield 100 parts of invert sugar and therefore, the amount of invert sugar yielded by an inverted sucrose solution multiplied by 0.95 represents the amount of sucrose originally present.

The solution generally employed for the purpose is a strongly alkaline solution of sulphate of copper and Rochelle salts. This process was first suggested by Trommer in 1841 and later has received much attention at the hands of others, among them Barreswil, 1844, Fehling, 1848, Soxhlet, 1878, and Herzfeld in more recent years.

The invert sugar takes away a part of the oxygen from the cupric oxide and reduces the same to cuprous oxide which latter separates as a bright scarlet colored precipitate, insoluble in water. The invert sugar itself is broken up into several compounds among which oxalic acid, formic acid, etc., may be identified and all of which remain in solution.

It has been ascertained that the degree of reducing power of invert sugar is dependent upon the relative excess of the copper salt present in the solution and therefore it is necessary in making such determinations to adhere strictly to prescribed condition of analysis.

It is a matter of choice whether one carries on such determinations by gravimetric or by volumetric analysis—in the former, the weight of cuprous oxide produced, or of this oxide reduced to metallic copper, is weighed and the amount of sucrose corresponding to this amount is determined; in volumetric analysis the volume of a sugar solution of known strength which is required to complete the reaction, is measured.

Fehling's Solution. The Fehling solution now generally used in these determinations is prepared as follows:

Solution I. Dissolve 34.639 grams of sulphate of copper, recrystallized, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in water, making the solution up to 500 c.c.

Solution II. Dissolve 173 grams of pure, crystallized Rochelle salts in distilled water. To this solution add 100 c.c. of a solution of purest sodium hydrate containing 516 grams NaOH per liter, and make the volume of this mixed solution up to 500 c.c.

Solutions I and II must be kept in separate flasks; immediately before use mix solutions I and II in equal volumes.

Inversion of Sucrose. The first step to be taken in order to determine sucrose by means of Fehling's solution is of course the transformation of sucrose into invert sugar. This is accomplished as follows according to Herzfeld's directions.*

Weigh out 13.0 grams of the sugar sample and dissolve with 75 c.c. distilled water in a 100 c.c. (metric) flask. When solution has been completely effected, add 5 c.c. of hydrochloric acid, having a specific gravity of 1.188, mix thoroughly, insert a thermometer in the flask and then place the same into a water-bath which has a temperature of 72° to 73° C.

The flask with its contents is kept for 5 minutes at a temperature of 69° C. the flask being frequently agitated during this time. As it may take from two to five minutes to raise the temperature of the flask's contents up to 69° C. the whole operation may take from seven to ten minutes time—but it must never exceed ten minutes.

When the inversion has been achieved the flask is immediately immersed in water having a temperature of 20° C.,

* Originally given in the *Zeitschrift der Deutschen Zucker Industrie*, 1888, p. 699, but modified to meet the present standards of the International Commission.

the thermometer is cautiously removed from the solution, any solution adhering to the thermometer is carefully washed back into the flask, its contents neutralized with sodium hydrate solution, made up to exactly 100 c.c., and filtered.

The inverted sugar solution thus prepared is now ready for determination by either volumetric or gravimetric analysis.

Volumetric Analysis. Before using Fehling's solution it is necessary that its reducing value be exactly determined. This can be accomplished as follows:

9.5 grams of perfectly dry chemically pure sucrose or best grade of refined sugar are inverted exactly as above described. As 9.5 parts of sucrose yield on inversion 10 parts of invert sugar, the inverted solution will contain 10.0 grams of invert sugar.

50 c.c. of this solution, containing 5.0 grams of invert sugar, are measured into a 1000 c.c. flask, sodium carbonate is added until a bit of red litmus paper thrown into it turns blue, and then the contents of the flask are made up to the 1000 c.c. mark with distilled water and well mixed.

Each c.c. of this solution now contains 0.005 gram invert sugar.

With an accurate pipette 25 c.c. of Fehling's solution I, and 25 c.c. of Fehling's solution II are measured into a deep porcelain dish or casserole; to this 50 c.c. of distilled water are added and the mixture is rapidly raised to the boiling point.

The inverted sugar solution is then run into the Fehling solution from a burette, graduated in $\frac{1}{10}$ c.c., under constant stirring and renewed boiling, until all of the copper in the solution is precipitated as cuprous oxide. The operator is warned of the approach of the end of the reaction by the gradual change in the color of the solution; the blue color disappears and the solution becomes colorless.

The end-point of the reaction is however determined by

filtering a few drops of the solution through a very small paper filter into a very dilute solution of potassium ferrocyanide (20 grams in 1000 c.c. water) with a little acetic acid (10% strength) added.

If a brownish-red color appears, owing to the formation of cupric ferrocyanide two-tenths c.c. more of the inverted sugar solution are added to the Fehling solution, the same is again boiled and the test repeated. This operation is repeated until a few drops of the inverted sugar solution added to the ferrocyanide solution no longer produce the red coloration.

It is always well to confirm the finding by a duplicate test.

As previously stated, 1 c.c. of the inverted sugar solution used contains 0.005 gram invert sugar.

Suppose that 49 c.c. of the inverted sugar solution have been used in the test, then—to effect the complete reduction of the 50 c.c. of the Fehling solution used, there were required: $49.0 \times 0.005 = 0.245$ gram invert sugar. This gives the strength of the Fehling solution.

The determination of sucrose in a given sample is conducted in precisely the same manner as the standardization above described.

Thus, suppose we were dealing with a pure sugar solution. Weigh out 13.0 grams of the sample; invert, as described—under exact adherence to the directions given. Of the 100 c.c. of the inverted solution thus obtained, place 50 c.c. in a liter flask and make up to 1000 c.c. with distilled water.

As 100 c.c. of the original inverted solution contain 13.0 grams of the sugar solution to be analyzed, 50 c.c. will contain 6.5 gram of the sugar solution and as these have been made up to 1000 c.c. each c.c. of this last named solution will contain 0.0065 gram of the sugar solution to be analyzed.

Suppose now that 67 c.c. of this solution have been required to decolorize completely the 50 c.c. of Fehling

solution used in the test, then those 67 c.c. of the inverted sugar solution must also contain 0.245 gram of invert sugar. But these 67 c.c. correspond to $67 \times 0.0065 = 0.4355$ gram of the sugar solution used for analysis.

Hence, $0.4355 : 0.245 :: 100 : x$

$x = 56.25\%$ invert sugar in the sugar solution after its inversion.

As 1.0 part of invert sugar corresponds to 0.95 part of sucrose, $56.25 \times 0.95 = 53.44\%$ sucrose in the solution analyzed.

Gravimetric Analysis. Inversion of the sucrose to be determined is conducted precisely as previously directed and the Fehling solution is to be prepared as there described. The procedure is as follows:

Weigh out 13.024 grams of the sample. Dissolve with about 75 c.c. of water in a 100 Mohr c.c. flask. Add 5 c.c. hydrochloric acid (sp.gr. 1.188). Heat quickly, in two or three minutes, on a water-bath up to 69° C. Then keep at this temperature, as close to 69° C. as possible, for five minutes, with constant agitation. Cool quickly; make up to 100 c.c. Remove 50 c.c. by a pipette, place in a liter flask, and fill up to 1000 c.c. Of this solution take 25 c.c., corresponding to 0.1628 gram of sample, neutralize all free acid present by about 25 c.c. of a solution of sodium carbonate prepared by dissolving 1.7 grams crystallized sodium carbonate in 1000 c.c. of water. Then add 50 c.c. of Fehling's solution and heat to boiling in the following manner. Over the wire-gauze above the flame lay a sheet of asbestos provided with a circular opening of about 6.5 cm. diameter; on this place the flask, and arrange the burner in such a manner, that about four minutes are consumed in heating the solution to the boiling-point. From the time that the solution starts to boil—the moment when bubbles arise not only from the center, but also from the sides of the vessel—continue to boil for exactly three minutes

with a small flame. Then remove the flask from the flame immediately, and add 100 c.c. of cold distilled water, from which the air has previously been removed by boiling.*

Then filter through an asbestos filter, wash, and reduce to metallic copper †

This operation is carried out in the following manner: Clean a small straight calcium-chloride tube, or other tube of similar pattern thoroughly. Introduce asbestos fibers ‡ so as to fill about half of the bulb. Draw air through while drying, cool, and weigh. Connect with an aspirator, filter the precipitated Cu_2O , wash with hot water, and then, having changed the receiving flask, wash twice with absolute alcohol and twice with ether. Having removed the greater part of the ether by an air-current, connect the upper part of the filter tube by means of a cork and glass tubing with a hydrogen apparatus, and, while the hydrogen gas is flowing through, cautiously heat the precipitate with a small flame whose tip is about 5 cm. below the bulb containing the Cu_2O . The reduction should be completed in from two to three minutes.

After the tube has been cooled in the current of hydrogen, air is once more drawn through and the tube is then weighed.

After an analysis is completed, the asbestos is readily freed from the adhering copper by washing with dilute nitric acid.

* The water is added to prevent subsequent precipitation of cuprous oxide.

† This last step is sometimes omitted, the cuprous oxide being weighed after washing and drying, and the corresponding amount of copper calculated by multiplying by 0.8. This should be done however only when the amount of cuprous oxide formed is not large.

‡ The asbestos must first be prepared by washing successively with a solution of caustic soda (not too concentrated), boiling water, nitric acid, and again with boiling water. When filled into the glass tube the asbestos is made to rest on a perforated platinum cone.

The use of the electric current has also been advocated for reducing the precipitate to metallic copper.

The cuprous oxide is dissolved with 20 c.c. nitric acid (sp.gr. 1.2), the solution is placed into a weighed platinum dish, made up to between 150 and 180 c.c. with distilled water, and the copper precipitated by the electric current.

Calculation. In Table XI seek the number of milligrams of copper which agree most closely with the amount of copper found. The corresponding number in the column to the left, shows at once the number of milligrams of sucrose.

Example. 25 c.c. of the inverted solution = 0.1628 gram of sample, yielded 0.1628 gram copper. This corresponds to 0.082 gram sucrose; hence there are present in the sample 50.4 per cent sucrose.

If a number of such determinations are to be carried out it will prove convenient to construct a table which will indicate directly the percentage of sucrose corresponding to the milligrams of copper found. Thus 79 mgr. copper correspond to 40 mgr. of sucrose. As 0.1628 gram of the sample has been taken for analysis,

$$0.1628 : 0.0400 :: 100 : x$$

$$x = 24.6\% \text{ sucrose.}$$

Sucrose in Condensation, Boiler-Feed and Waste Waters. If preferred to an optical determination the sucrose in condensation, waste waters, etc., may be transformed into invert sugar and determined as such. To do this concentrate the condensation water to 5% of its original volume and invert by means of hydrochloric acid, using 1 part of conc. HCl acid for 10 parts of the concentrated water. After inversion neutralize by sodium hydrate and determine the invert sugar formed, volumetrically or gravimetrically as preferred. Multiply the amount of

invert sugar thus found by 0.95 as 95 parts of sucrose on inversion yield 100 parts of invert sugar. The result is the amount of sucrose in the sample of water examined.

Sucrose and Invert Sugar. If the sample analyzed contains invert sugar as well as sucrose, two determinations with Fehling's solution must be made—one without inversion, the other after inversion. The difference between these invert sugar values multiplied by 0.95 represents the sucrose in the sample.

Sucrose, Invert Sugar and Levulose or Dextrose. Fehling's solution can also be used—employing the gravimetric method—to determine sucrose, invert sugar and levulose or dextrose.

The necessary determinations to be made are those of the sucrose, the total reducing sugars and of the dextrose after destruction of the levulose by Sieben's process.

Take 100 c.c. of a solution made to contain 2.5 grams on the dry substance of invert sugar, or of invert sugar and levulose, place in a flask, add 60 c.c. of a hydrochloric-acid solution which is six times the strength of a normal solution, and heat the flask for three hours while it is suspended in boiling water. After this has been done, cool immediately, neutralize with a sodium-hydrate solution which is six times the strength of a normal solution, make up to a volume of 250 c.c., and filter. Of the filtrate use 25 c.c. for the determination of the dextrose; this is obtained as follows:

Take 30 c.c. copper-sulphate solution; *
 [30 c.c. Rochelle-salt solution; †
 60 c.c. water.

* Prepared by dissolving 69.278 grams C.P. sulphate of copper in distilled water, and making the solution up to 1 liter.

† Prepared by dissolving 173 grams Rochelle salt, cryst. and 125 grams potassium hydrate in distilled water, and making the volume up to 500 c.c.

Heat to boiling. Add 25 c.c. of the solution, prepared as above, and keep boiling for *two* minutes. Then proceed as with a gravimetric determination of invert sugar. Table XV shows the amount of dextrose corresponding to the weight of copper found.

Unfortunately, however, the destruction of the levulose by hydrochloric acid (Sieben's process), on which this whole scheme of analysis is based, is not always accomplished with certainty, and the results obtained by this method must therefore be received with some caution and reserve.

The method of calculating the results consists of two steps:

Step I. is always the same, and merely establishes whether the dextrose and the levulose are present in the proportion of 1 to 1, or whether either is in excess.

Step II. determines the amount of this excess, be it of dextrose or of levulose.

Values determined:

No. 1.	Copper reduced by total sucrose + total reducing sugars.
No. 2.	“ “ total reducing sugars.
No. 3.	“ “ dextrose (after Sieben's treatment).

CALCULATION.

Step I.

No. 1 = Cu reduced by inverted sucrose and total reducing sugars.

Less No. 2 = Cu reduced by total reducing sugars.

Difference = Cu reduced by inverted sucrose. Report the corresponding value as sucrose,

This difference $\div 2 = \text{Cu}$ reduced by the dextrose of the inverted sucrose. Call this value x .

No. 3 = Cu reduced by the total dextrose (after Sieben's treatment).

Less $x = \text{Cu}$ reduced by the dextrose of the inverted sucrose.

Difference = Cu reduced by the dextrose of the total reducing sugars. Call this value y . Then,
 $y \times 2 = 2y$ Cu reduced by invert sugar + free dextrose, if any is present.

Compare this value, $2y$, with No. 2:

If $2y = \text{No. 2}$, invert sugar *only* is present. If so, report as invert sugar.

If $2y > \text{No. 2}$, free dextrose is present.

If $2y < \text{No. 2}$, free levulose is present.

Step II.

When $2y > \text{No. 2}$, free dextrose is present.

No. 2 = Cu reduced by the total reducing sugars.

Less $y = \text{Cu}$ reduced by the dextrose from the total reducing sugars

Difference = Cu reduced by the levulose of the total reducing sugars. Call this value p .

$p \times 2 = 2p$ Cu reduced by invert sugar. Report as invert sugar.

No. 2 = Cu reduced by the total reducing sugars.

Less $2p = \text{Cu}$ reduced by invert sugar.

Difference = Cu reduced by the free dextrose.

Step II.

When $2y < \text{No. 2}$, free levulose is present.

No. 2 = Cu reduced by the total reducing sugars.

Less $2y$ = Cu reduced by the invert sugar. Report as
invert sugar.

Difference = Cu reduced by the free levulose.

In these calculations no attention has been paid to the fact that the reducing power of invert sugar, dextrose, and levulose for copper solutions is not identical, but allowance for this can readily be made.

CHAPTER VI

SUCROSE DETERMINATION BY OPTICAL AND CHEMICAL ANALYSIS

Sucrose in Presence of Invert Sugar. Method I. Determine the direct polarization as usual and the amount of invert sugar by means of Fehling's solution. Express the latter value in terms of percentage, multiply this value by 0.34 and add the product to the direct polarization.

Example.

Direct polarization	84.00
Invert sugar	6.00
$6.0 \times 0.34 =$	2.04
Direct Pol. =	84.00
	<hr/>
Sucrose =	86.04

Results so obtained are however only approximately accurate.

Method II. MEISSL-HERZFELD. Weigh out 26.048 grams of the sample. Place into a 100 c.c. flask, clarify with basic acetate of lead, make up to 100 c.c., filter, and polarize. Take an aliquot part of the filtrate, add sodium sulphate to remove any lead present, make up to a definite volume, and filter. It is best to arrange the dilution so, that the 50 c.c. of this filtrate, which are to be used for the determination of the invert sugar, will precipitate between 200 and 300 milligrams of copper.

To 50 c.c. of the sugar solution prepared as above, add 50 c.c. Fehling's solution (25 c.c. copper sulphate and 25

c.c. of Rochelle-salt-soda solution), and proceed as previously directed.

After having determined the amount of copper reduced, the method of calculating the amount of invert sugar, corresponding to the weight of copper found, can best be illustrated by an example. Suppose that 25 c.c. of the 26.048 grams of sugar dissolved in 100 c.c., had been removed, clarified with sodium sulphate, made up to 100 c.c., and filtered: 50 c.c. of this filtrate would correspond to 3.256 grams of substance.

Let this weight be designated by the letter p .

The *approximate* amount of invert sugar may be assumed to be

$$= \frac{\text{Cu}}{2}.$$

The *approximate percentage* of invert sugar will be

$$= \frac{\text{Cu}}{2} \times \frac{100}{p}.$$

Representing the former value by Z , the latter by y , we have

$$Z = \frac{\text{Cu}}{2},$$

and

$$y = \frac{\text{Cu}}{2} \times \frac{100}{p}.$$

The ratio between the invert sugar and the sucrose is determined by the following formulæ, designating sucrose by the letter S , and invert sugar by I .

$$S = \frac{100 \times \text{Polarization}}{\text{Polarization} + y}.$$

$$I = 100 - S.$$

Example. Polarization of 26.048 grams = 86.4. $p = 3.256$ grams.

Suppose these 3.256 grams have precipitated, on boiling with Fehling's solution, 0.290 gram of copper. Then,

$$1. \frac{\text{Cu}}{2} = \frac{0.290}{2} = 0.145 = Z.$$

$$2. \frac{\text{Cu}}{2} \times \frac{100}{p} = 0.145 \times \frac{100}{3.256} = 4.45 = y.$$

$$3. \frac{100 \times \text{Pol.}}{\text{Pol.} + y} = \frac{8640}{86.4 + 4.45} = 95.1 = S.$$

$$100 - S = I,$$

$$100 - 95.1 = 4.9,$$

$$4.9 = I,$$

and therefore the ratio of $S : I$ is expressed by 95.1 : 4.9.

In order to find the factor F we must hunt up the correct vertical and horizontal columns in Table XIII. The value $Z = 145$ is most closely approximated by the column headed 150; the ratio $S : I = 95.1 : 4.9$ is most closely approximated by the horizontal column 95 : 5. At the line of intersection of these two columns there will be found the factor 51.2, by aid of which the final calculation is effected.

$$4. \frac{\text{Cu}}{p} \times F = \frac{0.290}{3.256} \times 51.2 = 4.56\% \text{ invert sugar.}$$

The analysis would hence show:

Polarization,	86.40
Invert sugar,	4.56

If duplicate or comparative determinations of invert sugar are to be made by this method, the *same* weight of substance should always be taken. Otherwise, the value

of Z varying, will necessitate the employing of different factors, and in consequence discrepancies will ensue.

Example.

Weight used,	2.500 grams.
Polarization,	95.00
Cu reduced,	0.140

Invert sugar = 2.587 per cent.

Weight used,	5.000 grams.
Polarization,	95.00
Cu reduced,	0.278

Invert sugar = 2.768 per cent.

These methods of determining invert sugar are based on the assumption that there are no other substances present besides invert sugar which will precipitate the copper from its solution.

Sucrose in Presence of Dextrose and Levulose.* This method involves but three simple analytical operations: two gravimetric determinations, and one optical examination with the polariscope, is therefore easy and rapid of execution, and, with careful manipulation, yields accurate results

THE METHOD.

Preparation of Solution. If not already in solution, make of the sample to be examined a solution of arbitrary density. Of course, it goes without saying, that this method is to be applied only in cases where no other optically active substances are present besides sucrose, dextrose and levulose, and that care must be exercised that no preliminary treatment of the solution shall influence its original power of rotation.

* Wiechmann, School of Mines Quarterly, Vol. XIII, No. 3. International Sugar Journal, 1907, Vol. IX, p. 68.

The specific gravity of the solution above referred to is accurately determined by balance, and from this value there are calculated, in the following manner, the number of grams of solution which contain 10.000 grams of dry substance:*

Ascertain the degree Brix corresponding to the specific gravity found. Divide 100 by the degree Brix; the quotient represents the number of grams of solution which contain 1.00 gram of dry substance. This value is multiplied by 10, and the product represents the number of grams of solution, equivalent to 10.000 grams of dry substance.

This amount is weighed out, placed in an accurately graduated 100 c.c. flask, and the solution in the flask is made up to 100 c.c. with distilled water.

The determinations to be made, are as follows:

Optical Examination. Some of the solution is placed in a water-jacketed polarization tube, a thermometer is inserted in the solution, and a reading on this solution is taken in the polariscope, at the temperature of 20° C.

The reading thus obtained must be reduced to the basis of a reading made in a 100 m.m. tube.

Furthermore, if a sugar polariscope has been used for the observation, the reading obtained must be transformed into circular degrees. With a polariscope using 26.048 grams as the normal weight, the factor 0.346 is used, for the sodium ray.

Gravimetric Determination before Inversion. Of the 10 per cent solution, weigh out an amount equivalent to 1.0 gram of dry substance. Make this up to 150 c.c., and of this solution take 24.4 c.c., equivalent to 0.1628 gram dry substance. Take 50 c.c. of Fehling's solution, heat to boiling; while boiling add the 24.4 c.c. of sugar solution, and boil for three minutes.

* This concentration is chosen because the specific rotatory powers of the sugars, values needed in the calculation of results, vary with the concentration.

Then remove from flame, add cold distilled water, previously boiled, in order to cool the solution and prevent a further deposition of cuprous oxide.

Filter through a weighed asbestos filter, wash first with boiling water, then with absolute ethyl alcohol, and finally with ether. Dry perfectly, cool and weigh.

Calculate the cuprous oxide to its equivalent of metallic copper, and from the copper thus found, ascertain the corresponding amount of sucrose.* From this figure the total reducing sugars by adding $\frac{1}{20}$ to the sucrose value indicated.

Gravimetric Determination after Inversion. Of the original 10 per cent solution, weigh off an amount equal to 5.000 grams dry substance; invert with 4 c.c. of concentrated HCl (specific gravity 1.20) by heating on boiling water-bath up to a temperature of 69° C. and maintaining the solution at that temperature for five minutes. Then remove the flask, cool it and its contents to the temperature at which the flask was graduated, and then make the solution up to 100 c.c.

Of the solution thus obtained take 20 c.c. and neutralize with sodium carbonate; then make up to a volume of 150 c.c. with distilled water; of this solution take 24.4 c.c., equivalent to 0.1628 gram of dry substance, and proceed precisely as previously directed. Determine the value found, as before, from the table; the result obtained represents the total sugars present, *expressed as sucrose*. From this amount subtract the *sucrose* value found by prior determination,† and the difference represents the amount of sucrose actually present.

The results thus obtained represent: the polarization, in a 10 per cent solution, of the three sugars combined;

* Table published by the German Government, Table XI.

† That is, the amount as actually found by table, prior to the addition of $\frac{1}{20}$.

the total reducing sugars present; the amount of sucrose present.

In cases where the reducing sugars, i.e., the dextrose and the levulose together, exceed in amount the sucrose present, the gravimetric determinations before and after inversion should preferably be made according to E. Meissl's method for the determination of invert sugar.*

CALCULATION OF THE RESULTS OF ANALYSIS.

This can be accomplished by algebra, or by allegation.

BY ALGEBRA.

Let:

a = amount of sucrose present.

b = amount of total reducing sugars present.

x = amount of dextrose present.

y = amount of levulose present.

s = the specific rotatory power of sucrose, divided by 100.

d = the specific rotatory power of dextrose, divided by 100.

l = the specific rotatory power of levulose, divided by 100.

p = polarization observed, expressed in circular degrees.

Then

$$(as + xd) - yl = p.$$

$$(as + xd) = p + yl.$$

$$xd = p + yl - as.$$

$$x = \frac{p + yl - as}{d}.$$

Substituting this value of x in the equation

$$x + y = b$$

* *Zeitschrift des Vereines für Rübenzucker-industrie*, Vol. XXIX, p. 1034, and E. Wein: *Tabellen zur Quantitativen Bestimmung der Zuckerarten*.

there results:

$$\frac{p+yl-as}{d} + y = b.$$

$$p+yl-as+yd=bd.$$

$$yl+yd=bd-p+as.$$

$$y(l+d)=bd-p+as.$$

$$y = \frac{bd-p+as}{l+d}.$$

As y represents the amount of levulose, y deducted from b will give at once the amount of dextrose. Or, if preferred, the value of x can also easily be calculated independently.

Example.

Sucrose = $a = 8.50$.

Total reducing sugars = $b = 1.50$.

Polarization, expressed in circular degrees = $p = 5.6426$.

Specific rotatory power of sucrose $\div 100 = s = +0.665$.

Specific rotatory power of dextrose $\div 100 = d = +0.535$.

Specific rotatory power of levulose $\div 100 = l = -0.819$.

Dextrose present = x .

Levulose present = y .

$$(8.50 \times .665 + x \times .535) - (y \times .819) = 5.6426.$$

$$(8.50 \times .665 + x \times .535) = 5.6426 + (y \times .819).$$

$$.535x = 5.6426 + .819y - 5.6525.$$

$$(1). \quad x = \frac{-0.0099 + .819y}{.535}.$$

$$(2). \quad x + y = 1.50.$$

Substituting the value of x found in equation (1), in equation (2), there results:

$$\frac{-0.0099 + .819y}{.535} + y = 1.50$$

$$-0.0099 + .819y = 0.8025.$$

$$1.354y = 0.8124.$$

$$y = 0.600.$$

Total Reducing sugars present, . . . =	1.50
Levulose present, =	0.60
Dextrose present, =	<u>0.90</u>

BY ALLEGATION.

Example.

Determinations made on a 10 per cent solution.

	Per cent.
Sucrose present, =	66.0
Total Reducing sugars present, . . . =	34.0
Polarization observed, =	2.8199
Polarization due to the sucrose = $6.6 \times$	
0.665, =	<u>4.3890</u>
Difference due to the 3.40 per cent	
Reducing sugars, =	<u>1.5691</u>

$$34 : 100 :: 1.5691 : x$$

$$156.91 \div 34 = 4.615.$$

+53.5	}	46.15	35.75
-81.9			<u>99.65</u>
			<u>135.40</u>

$135.4 : 35.75 :: 34 : x = 8.98$ per cent of dextrose.

$135.4 : 99.65 :: 34 : y = 25.02$ per cent of levulose.

Or else, having found the percentage of dextrose as above,

	Per cent.
Total Reducing sugars, =	34.00
Dextrose present, =	<u>8.98</u>
Levulose present, =	<u>25.02</u>

As to reporting the nature of the reducing sugars, the analysis of course only warrants the stating of the actual amounts of sucrose, of dextrose, and of levulose as found.

Should it, however be so desired, equal quantities of dextrose and levulose might be considered as present in the form of invert sugar.

The dextrose or the levulose, of whichever one least is found, might be regarded as combined with an equal amount of the other, forming invert sugar, and any excess over the amount so disposed of, could be reported as free, or uncombined dextrose, or levulose, as the case might be.

Sucrose in Presence of Invert Sugar and Raffinose. The following method is due to J. Baumann and is based on the following considerations.

The reading of the normal weight of pure sucrose after inversion is $= -32.66^\circ \text{ V.}$ at 20° C. If $x\%$ of sucrose are present a reading of $-0.3266x^\circ \text{ V.}$ will be obtained; $y\%$ of anhydrous raffinose will after inversion produce a rotation of 0.9491° V.

Let a mixture of sucrose, invert sugar and raffinose, contain, for instance, $x\%$ sucrose+invert sugar (figured as sucrose) and $y\%$ raffinose then, if P' denotes the polarization after inversion,

$$1. \quad P' = -0.3266x + 0.9491y.$$

Assuming that 0.1628 gram of the mixture be inverted and tested with Fehling's solution the $x\%$ of sucrose will reduce

$$\frac{0.1628 \times F_1}{100} x \text{ grams of copper}$$

F_1 being the reducing factor of inverted sucrose. In a similar manner $y\%$ of raffinose will yield

$$\frac{0.1628 \times F_2}{100} y \text{ grams of copper,}$$

F_2 being the reducing factor of the inverted raffinose.

Having present $x\%$ of sucrose+invert sugar (figured as sucrose) and $y\%$ of raffinose, then, representing the total amount of copper precipitated by Cu,

$$2. \quad \text{Cu} = \frac{0.1628 \times F_1}{100} x + \frac{0.1628 F_2}{100} y$$

From these two equations there is deduced:

$$x = \frac{\frac{94.91}{0.1628} \text{Cu} - P' F_2}{0.9491 F_1 + 0.3266 F_2}$$

$$y = \frac{P' + 0.3266 x}{0.9491}$$

From these expressions the following formulæ are derived.

Let, S = Total sucrose, i.e., sucrose+invert sugar figured as sucrose,

R = Raffinose (anhydrous),

then,

$$S = \frac{582.98 \text{Cu} - P' F_2}{0.9491 F_1 + 0.3266 F_2}$$

$$R = 1.054 P' + 0.344 S.$$

The factors for F_1 are obtained from Table XI by finding therein the amount of copper obtained and dividing this value by the corresponding amount of sucrose.

The factors for F_2 are given in a table published in the *Zeitschrift des Vereins für Rübenzucker-Industrie*, 1888, Vol. XXXVIII, p. 741.

It is not necessary however to have these constants for every instance; it will answer to calculate these for every 10 milligrams of copper. The following table gives these

values calculated for copper ranging from 150 to 200 milligrams.

Cu=	S=
0.150	$248.1 \times \text{Cu} - 0.605 \times P'$
0.160	$248.4 \times \text{Cu} - 0.604 \times P'$
0.170	$248.7 \times \text{Cu} - 0.604 \times P'$
0.180	$249.2 \times \text{Cu} - 0.604 \times P'$
0.190	$249.7 \times \text{Cu} - 0.604 \times P'$
0.200	$250.0 \times \text{Cu} - 0.604 \times P'$

$$R = 1.054P' + 0.344S$$

If the customary amount of substance, 0.1628 gram has not been used for the determination of the inverted sugars, but, say w gram, the copper value found must be multiplied by $\frac{0.1628}{w}$ and the resultant value used in the above given formulæ in place of the other Cu value. For the calculation of the reduction factors however the actual weight of the precipitated copper must be employed.

In order to determine the sucrose and the invert sugar separately, in addition to the raffinose, an invert sugar determination must be made as usual, however instead of using the direct polarization for calculating the factor P' the total sucrose value obtained by the formulæ previously given must be used.

One-twentieth must be deducted from the invert sugar formed, to express it in terms of sucrose, and this remainder, subtracted from the total sucrose will represent the sucrose present as such.

Example. Analysis of a syrup yielded the following data:

Polarization after inversion, $P' = -8.5^\circ$
 0.1628 gram of sample gave after inversion $\text{Cu} = 0.184$ gram
 2.000 grams of sample gave before inversion $\text{Cu} = 0.250$ gram

The figure $Cu=0.184$ is, in the table previously given, approximated most closely by the figure $Cu=0.180$ and for this value

$$S = 249.2Cu - 0.604P'$$

Hence, $S = 249.2 \times 0.184 - 0.604(-8.5) = 50.98$

$$R = 1.054(-8.5) + 0.344(50.98) = 8.58$$

To determine the ratio of the sucrose, S , to the invert sugar, I ,

$$y = \frac{\frac{Cu}{2} \times 100}{p} = 6.25$$

hence, $S + I : I :: 50.98 : 6.25 :: 100 : 12$

$$S : I :: 88 : 12$$

$$I = \frac{0.250 \times 52.6}{2} = 6.58$$

$-\frac{6.58}{2} = \frac{0.33}{6.25}$ and this value must be subtracted from the total sucrose, i.e., $50.98 - 6.25 = 44.73$.

The syrup therefore contains:

Sucrose	= 44.73%
Invert sugar	= 6.58%
Raffinose	= 8.58%

CHAPTER VII

CONSTITUENTS OF SUGAR OTHER THAN SUCROSE

Reducing Sugars in General. A valuable reagent for the detection of reducing sugars, for instance, of dextrose and levulose, is phenyl hydrazine $\text{H}_2\text{N}-\text{NHC}_6\text{H}_5$. Emil Fischer, in 1884, first directed attention to this reagent for the purpose.

All sugars which contain a free carbonyl group respond to this test, giving compounds which are termed hydrazones.

Hydrazones are prepared by adding to the sugar solution, while cold, a solution consisting of equal volumes of phenyl hydrazine and a 50% acetic acid solution and three volumes of water. The hydrazones separate as crystalline compounds, some of them almost at once, others after a longer time, that of glucose possibly only after a day or two. Purification is effected by recrystallization from water or from alcohol, the purity of the product being proven by its exhibiting a constant melting-point. The sugars are separated from their hydrazones either by the use of concentrated hydrochloric acid or by means of formaldehyde; in the former case phenyl hydrazine chloride and free sugar are formed. The former substance is removed by filtration, and the filtrate after neutralization with carbonate of lead and filtration is evaporated to a syrup. This is then shaken up with alcohol, 95% strength, refiltered and the filtrate allowed to crystallize by evaporation.

If a reducing sugar be heated with an excess of phenylhydrazine a dihydrazone, usually termed an osazone, is formed. Osazones are but slightly soluble in water they crystallize well and have definite melting-points.

Dissolve in 20 parts of water, one part of the sugar to be tested, two parts of pure phenyl hydrazine hydrochloride and three parts of cryst: sodium acetate.* Place test-tube and contents, loosely corked, into boiling water the solution being occasionally stirred to facilitate crystallization.

All of the monosaccharides form precipitates within 20 minutes. Maltose and lactose yield precipitates only on cooling the solution. Sucrose will yield a precipitate only after it has been changed to dextrose and levulose—this is effected only after heating for about 30 minutes in the water-bath.

One to two hours' heating in the bath and then cooling the solution produces the largest yield of osazones. The osazones usually have a yellow color, and can be purified by recrystallization from 50% boiling alcoholic solution. The osazones of the monosaccharides crystallize from hot solutions, those of the disaccharides only after cooling.

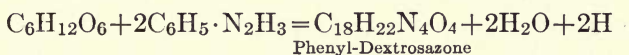
The osazones yielded by some of the more important sugars are:

Sugar.	Osazone.	Melting-point (when reached in 3 or 4 minutes).
Dextrose	Glucosazone	204°-205° C.
Levulose	“	
Mannose	“	
Galactose	Galactosazone	193° C.
Maltose	Maltosazone	206° C.
Lactose	Lactosazone	200° C.
Xylose	Xylosazone	160° C.
Arabinose	Arabinosazone	160° C.

* H. C. Sherman, School of Mines Quarterly, 1905, p. 148.

Careful tests made by R. H. Williams showed that solutions of pure dextrose yielded the osazone in 5 minutes' time when 0.10 gram was present, whereas 17 to 19 minutes were required when only 0.01 gram was present. Sucrose had no appreciable influence on the result even when present up to ten times the amount of dextrose present:

The reaction by which an osazone is formed is probably indicated by the empirical equation:



The hydrogen does not escape but is taken up by any excess of phenyl hydrazine which breaks up into ammonia and aniline.

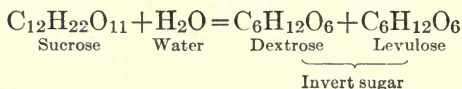
All phenyl hydrazones are changed to osazones on being heated with an excess of phenyl hydrazine.

Maquenne pointed out that the weight of osazones produced under identical conditions was a characteristic of value. The following weights of osazones were obtained by heating for one hour at 100° C. 1.0 gram of sugar with 100 grams of water and 5 c.c. of a solution containing 40 grams of phenyl hydrazine and 40 grams of acetic acid in 100 c.c.

After cooling the liquid, the osazones were placed upon a weighed filter, washed with 100 c.c. of water, and dried at 110°.

Sugar.	Weight of Osazone.
Sorbine crystallized	0.82
Levulose "	0.70
Xylose	0.40
Glucose, anhydrous	0.32
Arabinose, crystallized	0.27
Galactose "	0.23
Rhamnose "	0.15
Lactose "	0.11
Maltose "	0.11

Invert Sugar is composed of equal parts of dextrose and levulose. Its formation from sucrose by inversion is expressed by the reaction:



Qualitative Test. A qualitative test for the presence of invert sugar is easily effected by the use of methyl blue. To prepare this reagent dissolve 1.0 gram of methyl blue in 1 liter of water. To test for the presence of invert sugar, dissolve 20 grams of the sugar in water, add basic acetate of lead solution, make up to 100 cubic centimeters, and filter. Make the filtrate slightly alkaline with a 10 per cent solution of sodium carbonate, and filter again. Of this filtrate take 50 cubic centimeters, representing about 10 grams of the sugar tested, place in a porcelain casserole, and add 2 drops of the methyl-blue solution. Then place the casserole over a naked flame, and note accurately when the solution begins to boil.

If the solution is decolorized by boiling inside of one half minute, there is sufficient invert sugar present to permit of a quantitative determination. If it requires from one half to three minutes boiling to effect disappearance of the blue color, traces of invert sugar are to be reported; and if decolorization does not take place within three minutes, "no invert sugar" is recorded.

If the normal weight has been dissolved up to 100 c.c., 20 c.c. of the solution, clarified by basic acetate of lead, are made up to 50 c.c. The lead is removed by adding five drops at a time of the sodium-carbonate solution, and the addition of this reagent, in the same quantity, is continued, until no more precipitation can be detected.

To 25 c.c. of the filtrate one drop of the methyl-blue solution is added; about 10 c.c. of this solution are kept actively boiling over a naked flame for one minute.

If, after thus boiling for one minute, the solution is completely decolorized, it must have contained at least 0.01 per cent of invert sugar. If it is not decolorized, it contained no invert sugar, or certainly less than 0.01 per cent.

Quantitative Determination. The quantitative determination of invert sugar is effected by means of alkaline copper solutions either volumetric or gravimetric methods being employed.

Full details of these methods have been given in Chapters V and VI. As there directed keep the sulphate of copper solution in one flask, and the Rochelle-salt-soda solution in another. Mix the two immediately before use. It will be found very convenient to have the solutions in flasks or jars provided with a siphon-arrangement, and to have the delivery-tube so graduated that the required amount may be rapidly, yet accurately measured out. The accompanying figure shows an arrangement answering this purpose.

The standardizing of Fehling's solution has previously been fully discussed yet it may be of interest to add the following.*

“For the standardization of a solution for the determination of invert sugar in sugar-house products, dissolve 2.5 grams of pure sucrose in 100 c.c. of water, add 10 c.c. of hydrochloric acid (specific gravity 1.188), and invert according to the method given for double polarization. Neutralize the acid with sodium carbonate and dilute to 1 liter. The 2.5 grams of sucrose become 2.6316 grams of invert sugar.

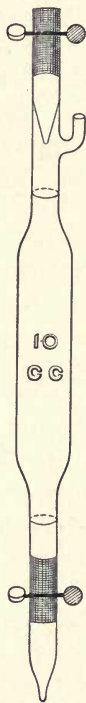


FIG. 7.

* Bulletin No. 38, U. S. Dept. of Agriculture, Division of Chemistry.

The weight of invert sugar equivalent to 10 c.c. of the copper reagent is calculated as follows:

$$\frac{2.6316 \times \text{by number of c.c. of the standard sugar solution used}}{1000} = X,$$

the weight of invert sugar required to completely precipitate the copper in 10 c.c. of the reagent under the conditions used for the test titration. For the calculation of the result of the titration of an unknown solution:

Let X = the factor obtained as above;

V = the number of c.c. of unknown sugar solution required to precipitate the copper from 10 c.c. of copper solution;

W = the weight of the material under examination in 1 c.c. of the solution.

Then $\frac{100X}{VW}$ = per cent of invert sugar in the sample.

“The calculation can be much simplified by so standardizing the copper reagent that 50 milligrams of invert sugar will be required to reduce the copper from 10 c.c. of the copper reagent. The various tables given in works on sugar analysis then become applicable. These tables are arranged for a ‘glucose normal solution’ containing 5 grams of the material to be examined in 100 c.c. When the weight per 100 c.c. is more or less than 5 grams the number found in the table is increased or diminished accordingly.”

a. By volumetric analysis. The details of procedure have been so fully given in Chapter V in describing the determination of sucrose (after inversion) that it will suffice to give here in briefest outline the course to be followed.

Five, ten or more grams of sugar are weighed out, dissolved in a flask, and the solution made up to 100 c.c. The weight of sugar varies, of course, with the nature of the sample examined, that is to say, with the amount of invert sugar it contains. It is advantageous to have the solution of such a strength that 20 c.c. to 50 c.c. will completely precipitate the copper in 10 c.c. of the Fehling solution.

Ten c.c. of the Fehling solution are measured out (using 5 c.c. each of the copper sulphate and of the Rochelle-salt-soda solution), placed in a porcelain dish, and quickly brought to the boiling point. The sugar solution is then run in from a burette (graduated in tenths of a cubic centimeter) until all of the copper in the solution is precipitated as cuprous oxide. The operator is warned of the approach of the end of the reaction by the change in the color of his solution. The blue color disappears and the solution becomes colorless, or, if the sugar solution is colored, assumes a yellow tinge.

The end-point, however, is determined by filtering a few drops of the solution through paper or linen-cloth into a very dilute solution of potassic ferrocyanide and acetic acid.

If a brownish-red color shows, owing to the formation of cupric ferrocyanide, two tenths c.c. more of the sugar solution are added to the copper liquor, the solution is again boiled, and the test repeated. This is continued until the addition of a few drops of the solution to the ferrocyanide no longer produces the red color.

As 10 c.c. of the copper solution are assumed to correspond to 0.05 gram of invert sugar, the calculation is an easy one. If 5 grams of sugar have been dissolved up to 100 c.c., the reciprocal of the number of cubic centimeters required of this solution to precipitate all of the copper in 10 c.c. of the copper liquor, multiplied by 100,

is the direct percentage of invert sugar sought. (See Table XII.)

Example. Dissolved 5 grams of sugar in 100 c.c. Of this solution used 22 c.c. to precipitate all of the copper in the Fehling solution. Referring to Table XII, 22 c.c. will be found to correspond to 4.55 per cent of invert sugar; hence there is this amount of invert sugar present in the sample.

b. By gravimetric analysis. This method also has been previously discussed. Where very small amounts of invert sugar are to be determined—in beet sugars for instance, containing 1% or less of invert sugar, the following method is applicable. The solution of the material to be examined is so prepared as to contain 20 grams in 100 c.c., and must be freed from suspended impurities by filtration through paper. In a beaker of 250 c.c. capacity place 50 c.c. of the mixed copper reagent and 50 c.c. of the sugar solution. Heat this mixture at such a rate that approximately three minutes are required to bring it to the boiling point and then continue to boil for exactly two minutes more. Add 100 c.c. of cold, recently boiled, distilled water. Filter immediately under pressure through a weighed filtering tube of hard glass. The asbestos film in the filtering tube is supported by a perforated disc or cone of platinum and should be moistened previous to the filtration. The precipitate is all transferred to the filter and thoroughly washed with hot water, following the water by alcohol and ether successively. After being dried the tube is connected with an apparatus for supplying a continuous current of dry hydrogen, gently heated until the cuprous oxide is completely reduced to the metallic state, cooled in the current of hydrogen, and weighed. The increase in weight represents the weight of copper reduced by 10 grams of the sample. The corresponding percentage of invert sugar is found by use of the following table:

HERZFELD'S TABLE FOR THE DETERMINATION OF
 INVERT SUGAR IN MATERIALS CONTAINING 1 PER
 CENT OR LESS OF INVERT SUGAR

Copper Reduced by 10 grams of Material. Milligrams.	Invert Sugar. Per Cent.	Copper Reduced by 10 Grams of Material. Milligrams.	Invert Sugar. Per Cent.	Copper Reduced by 10 Grams of Material. Milligrams.	Invert Sugar. Per Cent.
50	0.05	120	0.40	190	0.79
55	0.07	125	0.43	195	0.82
60	0.09	130	0.45	200	0.85
65	0.11	135	0.48	205	0.88
70	0.14	140	0.51	210	0.90
75	0.16	145	0.53	215	0.93
80	0.19	150	0.56	220	0.96
85	0.21	155	0.59	225	0.99
90	0.24	160	0.62	230	1.02
95	0.27	165	0.65	235	1.05
100	0.30	170	0.68	240	1.07
105	0.32	175	0.71	245	1.10
110	0.35	180	0.74		
115	0.38	185	0.76		

Such quantitative determination should be made on all beet sugars which react acid to the phenol phthaleïn test or which darken materially in the vacuum oven.

Water. Weigh out 2 to 10 grams of the sample. If the determination is to be made on a rather moist sugar or on a syrup, a corresponding amount of perfectly dry powdered glass or of sand must be intimately mixed with the sample.

Place in an air-bath, the heating of which should be commenced only after the introduction of the assay. The heat should be gradually carried up to 100° C.,* and continued at that temperature until the sample has attained to practically constant weight.†

* German and Belgium chemists recommend a temperature of not less than 105° C.

† Until the loss of weight in one hour is not greater than 0.20 per cent.

The loss in weight sustained, represents the water.

<i>Example.</i> Weight of dish, sand, and sample,	23.0000
" " and sand,	18.0000
	<hr/>
Sample taken,	5.0000
	<hr/>
Original weight of dish, sand, and sample,	23.0000
Final weight (after drying to constant weight), . .	21.1546
	<hr/>
	Water = <u>1.8454</u>

$$5.000 : 1.8454 :: 100 : x.$$

$x = 36.91$ per cent water.

Various drying ovens have been devised in which water determinations can be quickly made.

Instead of drying in an air-bath, the drying can be done in a current of any inert gas, or it can be still more rapidly accomplished by drying in a vacuum. A tube provided with a number of small branch-tubes, each of which can be closed independently by means of a stop-cock, is put into connection with a vacuum-pump. The samples of sugar in which the moisture is to be determined, are weighed into metal dishes provided with a cover and of known weight, and these dishes, after being placed on a steaming water-bath, are connected with the branch-tubes and the air exhausted.

Any convenient form of vacuum apparatus may be made to serve equally as well, but it is essential that the temperature of the liquid surrounding the containing vessel, be kept at 100° C. or above throughout the drying.

Another method for determining water in saccharine products, is the method of Josse.

About 2.0 grams of the substance are placed in a small flat-bottomed dish, 6 to 10 c.c. of water are added and the substance is dissolved, by aid of a gentle heat, if necessary. The liquid is absorbed by a coil of paper about 20 inches

long and 2 inches wide and this is dried for 2 hours at approximately 105° C.

After the first weighing the coil should be dried for an additional hour and the operation repeated until practically constant weight has been attained. The temperature of 105° C. can be conveniently obtained and held by drying in a jacketed air-bath using for the filling a salt solution containing about 30 parts of salt in 70 parts of water.

The following excellent method for the determination of water in saccharine products has been devised by Paul Poetschke.

Place 10 grams of pure quartz sand and a short stirring rod into a 100 c.c. beaker. Dry thoroughly at 100° C., cool in a desiccator and weigh. Introduce about five grams of the sample, mix with the sand and add gradually, with stirring, 10 c.c. of alcohol (absolute alcohol is preferable). Evaporate to dryness on a water-bath (do not place directly over steam, since the material is liable to bump, but stand the beaker on the metal surface of the bath). Again add 10 c.c. of alcohol and repeat the evaporation to dryness as before. During the evaporation the beaker should be protected against dust. After the second portion of alcohol has completely evaporated, place the beaker in an air-bath and dry for 2½ hours at 100° C. Cool in a desiccator and weigh. Repeat the heating at 100° C. for one hour and again weigh to note whether drying is complete. Usually 2½ hours are sufficient for all saccharine products. The method is applicable to syrups, honey, molasses, maple syrup, confectionery, etc. Quartz sand is prepared in the usual manner by digesting with strong hydrochloric acid, washing, drying and igniting. The alcohol used should leave only an inappreciable residue on evaporation.

A method for *approximately* determining the amount of water in a sample of syrup, liquor, or sweet-water, is to take the Brix hydrometer reading of the solution, and to

subtract this from 100. The difference is accepted as representing the water.

Example. Density of syrup in degrees Brix, 75.0°.

$$\begin{array}{r} 100 \\ \text{Less } 75 \\ \hline 25 \text{ per cent of water.} \end{array}$$

Ash. *Scheibler's Method.* Weigh out 2.5 to 5 grams of sample into a platinum ash-dish. Moisten with eight to ten drops of chemically pure conc. sulphuric acid, or better, with sixteen to twenty drops of dilute sulphuric acid (1 : 1). Pour a little ether over the contents of the dish and ignite. This treatment yields a porous carbonized mass, and avoids in a great measure the danger of loss by the assay mounting and creeping over the sides of the dish. When all gases have burned off, place in a platinum muffle, or in a Russia sheet-iron muffle (the metal should be about $\frac{1}{8}$ inch in thickness), and keep the muffle at a dull-red heat until the sample has been turned completely to ash; cool and weigh.

As the addition of sulphuric acid has converted a number of the salts present in the sugar into sulphates, 10 per cent is deducted from the weight of the ash found in order to make the results obtained by this method harmonize with those obtained by the method of carbonization.

Example. Used 2.5 grams of sugar.

Weight of dish + ash, . .	13.9030
“ “ . .	13.8490
	<hr/>
Ash,	0.0540
Subtract 10 per cent, . .	0.0054
	<hr/>
Total ash, =	0.0486
	<hr/>
Total ash, =	1.944 per cent.

This subtraction of one tenth of the weight of the ash is generally assumed to answer for beet-sugars, but is entirely misleading where cane-products are analyzed, because the ash of the latter possess a composition entirely different from the ash of the former.* At present, however, the subtraction of one tenth is still the general practice.

That unreliable results are obtained by this method of incineration with sulphuric acid and the subsequent subtraction of one tenth from the weight of the sulphated ash, even when beet-sugars are analyzed, is now generally admitted.

Von Lippmann advocates taking the dried-out sample, on which the water determination has been made, saturating it with vaselin-oil (having a boiling point of about 400°), and igniting the mixture. The carbonized mass is then to be burned to ash in a mixed current of air and oxygen.

Carbonization Method. Weigh out 2.5 to 5.0 grams of the sample. Carbonize at a low heat. Extract the soluble salts from the carbonaceous mass with boiling water; ignite the residue. Add the ash obtained to the aqueous extract and evaporate to dryness. Moisten with ammonium carbonate, drive off all ammonia, cool, weigh, and report as carbonate ash.

A number of other methods for determining ash in sugar have been advocated; the use of oxalic acid, zinc oxide, quartz sand and of benzoic acid has been recommended for the purpose.

In the method of Alberti and Haempel, employing pure quartz sand, 5 grams of sugar are placed in a platinum dish and are intimately mixed with about 6 to 7 grams of coarsely ground pure quartz sand. The mixture is ignited in a platinum muffle at a moderate red heat and the incineration completed in from one half to an hour's time, according to the nature of the sample.

* Wiechmann, "Ash Determinations in Raw Sugars," School of Mines Quarterly, Vol. XI.

This method depends on the fact that when a mixture of sugar and quartz (silica), is caused to undergo combustion, this combustion is complete and no carbon is left unconsumed. Furthermore the organic-acid salts are transformed into silicates and not into carbonates, and the sulphates and alkaline chlorides originally present in the sugars, are not decomposed by the silicic acid at the temperature at which the operation is carried out. This method has been extensively tested abroad, and the results are reported as satisfactory, *provided that pure quartz sand be employed.*

The benzoic acid method, devised by Boyer is described as follows: * “The benzoic acid is dissolved in alcohol of 90 per cent, 25 grams of the acid to 100 c.c. of alcohol; 5 grams of the sugar are weighed in a capsule and moistened with 1 c.c. of water. The capsule is heated slowly in order to caramelize the sugar without carbonizing it; 2 c.c. of the benzoic-acid solution are next added, and the capsule warmed until all the alcohol is evaporated; the temperature is then raised until the sugar is converted into carbon. The decomposing benzoic acid produces abundant vapors which render the mass extremely porous, especially if a circular motion is imparted to the capsule. The slow heating is continued until all the benzoic acid is volatilized.

“The carbon obtained is voluminous and of a brilliant black color. The incineration is accomplished in a muffle at a low red heat. The capsule should be weighed quickly when taken from the desiccator, in order to avoid the absorption of water by the alkaline carbonates. Ammonium benzoate may be employed instead of benzoic acid, and the analyst should previously assure himself that neither the acid nor the ammonia salt leaves a residue on incineration. In addition to giving the mineral matters directly, this method permits the determination of their composition also, a matter of no small importance.”

* U. S. Dept. of Agriculture, Division of Chemistry, Bul. No. 38, 1893.

Analysis of Sugar-Ash. Dissolve 10 grams of the sugar in hot water and filter; * wash the residue thoroughly with boiling water and evaporate the filtrate and the washings to dryness. Carefully carbonize the mass, and then extract the same with boiling water until nitrate of silver no longer gives the reaction for chlorine. Evaporate the solution to small bulk. The residue must be dried, ignited, and weighed. This weight is noted as insoluble ash. The solution and the ash obtained are then combined, hydrochloric acid is added, and the solution evaporated to dryness. All the chlorine is then driven off, the residue is taken up with water and a little hydrochloric acid, and filtered. The insoluble residue on the filter is thoroughly washed, and the washings are added to the filtrate. This residue is silica. To the filtrate ammoniac hydrate is added, and the solution is boiled and filtered; the residue, iron and alumina, must be thoroughly washed, and the washings added to the filtrate.

To this ammonium oxalate is added, and the whole is evaporated to dryness. The ammonia is burned off, and the oxalates are changed to carbonates by adding a little ammonium carbonate, and again driving off the ammonia.

The mass is then taken up with water, filtered, washed, and the washings added to the filtrate. The residue consists of the carbonates of calcium and magnesium. The filtrate is evaporated to small bulk, ammonium carbonate is added, and the evaporation is then continued to dryness, the ammonia is cautiously driven off, and the residue weighed. This gives the alkalies in the form of carbonates, and this weight added to the weight of the insoluble ash previously determined, represents the total carbonate ash.

* This should be done in every case so as to have all the analyses made under the same conditions; in most instances it will be imperative, for the inorganic suspended impurities (sand, clay, etc.) in a sample of cane-sugar often weigh more than the total sugar-ash.

Suspended Impurities. It is often necessary to determine the share of work done in filtration respectively by the bag-filters and the bone-black.

The former, of course, remove only the mechanically suspended impurities, or at least the greater part of them, and leave to the bone-black the rest of the work to be accomplished.

The suspended impurities are both mineral and organic; their determination is effected in the following manner:

Dissolve from 2.5 to 10 grams of the sample in hot water. Pour on a filter paper which has previously been dried and weighed between watch glasses, and wash with boiling water until all of the sugar has been removed. This is most conveniently done by the aid of a vacuum-pump. Then dry filter and contents to constant weight, and weigh as before between watch glasses. The increase in weight over the previous weight, represents the total suspended impurities. Ignite the filter and contents in a platinum crucible, and record the weight of the ash as mineral or inorganic suspended impurities; the difference between the total suspended impurities and this figure gives the organic suspended impurities.

An ash determination made as previously described represents the mineral matter contained in the sugar, in the form of salts, etc., as well as the mineral matter mechanically suspended, and which latter, the bag-filters are supposed to remove.

The inorganic suspended impurities when subtracted from the total ash show the "soluble" ash, the more or less complete removal of which is expected of the bone-black.

Example. Used 2.5 grams of raw sugar.

Weight of watch glasses+filter+total suspended impurities,	22.5071
Weight of watch glasses+filter,	22.5000
Total suspended impurities,	<u>0.0071</u>

Weight of crucible+ash of filter+inorganic sus- pended impurities,	13.20020
Weight of crucible,	<u>13.20000</u>
Ash of filter+inorganic susp. impurities,	0.00020
Ash of filter,	<u>0.00008</u>
Inorganic susp. impurities,	<u>0.00012</u>
Total suspended impurities,	0.00710=0.2840 per cent.
Inorganic " "	<u>0.00012=0.0048</u> "
Organic " "	<u>0.00698=0.2792</u> "
Total ash (previously determined),	0.5040 per cent.
Inorganic suspended impurities,	<u>0.0048</u> "
Soluble ash,	<u>0.4992</u> "

Woody Fiber. About 20 to 25 grams of the sample, in as finely divided a state as possible, are placed in a flask or beaker, into which cold water is poured. The water, after having been in contact with the chips or shavings from 20 to 30 minutes, is decanted carefully, in order to avoid any loss of the weighed sample. This treatment with cold water is repeated two or three times, and is then followed by a similar treatment with hot water; finally, the sample is boiled several times, fresh water being taken for each treatment, and the treatment continued until all the soluble material has been washed out. Sometimes this is followed by washings with alcohol and ether.

The sample is then transferred to a weighed filter, preferably made of asbestos, and gradually dried to constant weight. If dried in the air-bath, a temperature of 110° C. should not be exceeded. If the sample can be dried in vacuo, and subsequently weighed in a covered dish or capsule, all danger of oxidation and absorption of moisture are avoided.

The increase in weight which is noted of course represents the woody fiber.

Organic Non-Sugar. In regular technical analyses the organic matter not sugar, raffinose, or invert sugar is not determined. It is assumed to be represented by the difference between 100 and the constituents determined, viz., sucrose, raffinose, invert sugar, water, and ash. This difference is frequently recorded as "non-ascertained," or "undetermined matter."

Herzfeld has suggested that the "total non-sugar" could be determined by ascertaining the sucrose and the dry-substance, and subtracting the former from the latter.

If instead of doing this, the sucrose plus the ash be subtracted from the dry substance, the remainder may be regarded as "organic non-sugar." In that case, of course the invert sugar and the raffinose would also be embraced under the heading "organic non-sugar," unless these were separately determined.

There are several methods for the direct determination of this organic matter, but the results which they yield are of value chiefly for comparative purposes. The following method is perhaps the most satisfactory:

Dissolve 10 to 20 grams of raw sugar in warm water. Add basic acetate of lead solution in excess. Warm for a short time and filter. Wash the precipitate thoroughly; then suspend it in water and pass in sulphuretted hydrogen until all the lead is precipitated as sulphide. Filter out the sulphide of lead, wash thoroughly, and evaporate the filtrate and washings to dryness (constant weight), in a dish previously weighed. The temperature at which the drying is done, must not exceed 100° C.

Example. Used 10 grams of raw sugar.

Weight of dish and organic matter,	17.0973
" dish,	17.0482
	<hr/>
Organic matter,	0.0491
	<hr/>
Organic matter = 0.491 per cent.	

Organic Acids. Determination of some organic acids—non-volatile and volatile, can be approximately effected by the following scheme.*

APPROXIMATE DETERMINATION OF ORGANIC ACIDS, NON-VOLATILE AND VOLATILE

Non-volatile Acids.			Volatile Acids.
<p>A. Precipitation by neutral acetate of lead. Oxalic, citric, tartaric, and malic acids. Incompletely: pectic, parapectic, glucoic, melassinic, ulmic, and succinic acids.</p> <p>50 grams of the sample are dissolved in distilled water and made slightly acid with acetic acid. The solution is boiled to expel the carbonic acid, and neutralized with sodium hydrate (free from carbonic acid). A slight excess of neutral acetate of lead is added, and digested for one hour. The residue is placed on a dry and weighed filter, and is washed with boiled distilled water until the washings give no longer the reaction for lead. (For treatment of the filtrate, see B.)</p> <p>The precipitate contains the lead salts of the above named acids, and besides sulphate and phosphate of lead, if the sample examined contained sulphates and phosphates. The filter with its contents is dried at 110° C., and weighed. The precipitate is removed, the filter is burned in a weighed porcelain crucible, the precipitate is again added, and heated to dull redness.</p> <p>To facilitate the combustion of the carbon, small doses of ammonium nitrate are repeatedly added, great care being taken to prevent loss by spitting. After cooling, the crucible is weighed. The weight of the contents of the crucible subtracted from that of the precipitate dried at 110° C. represents the weight of the organic acids, because the sulphate and phosphate of lead are not altered by the ignition.</p>	<p>B. Precipitation by basic acetate of lead. Pectic, parapectic, glucoic, melassinic, ulmic, and succinic acids.</p> <p>Parapectin, incompletely: aspartic and metaplectic acids, and pectin.</p> <p>—</p> <p>To the filtrate from the lead salts precipitated by neutral acetate of lead, there is added a slight excess of basic acetate of lead, and the precipitate filtered out. (For filtrate, see C.)</p> <p>The precipitate is placed on a dried and weighed filter, then washed, dried at 110° C., and weighed. A part is incinerated as in A, and the weight of the organic acids determined by difference, as there described.</p>	<p>C. Precipitation by ammoniacal acetate of lead. Aspartic and metaplectic acids.</p> <p>—</p> <p>The filtrate obtained from the precipitation with basic acetate of lead is mixed with several cubic centimeters of an ammoniacal acetate of lead solution. Allow to stand for twelve hours. Filter, allow to drain off, and wash once with distilled water to which a little ammoniacal acetate of lead has been added. The precipitate, dried and weighed, is treated as described under A and B.</p> <p><i>Note.</i>—The ammoniacal acetate of lead must be added only gradually and in small amounts for without this precaution it is apt to precipitate sugar, and then even an approximate determination of the acids sought for, becomes very difficult.</p>	<p>D. Not precipitated by acetate of lead: formic, acetic, lactic, propionic, and butyric acids.</p> <p>—</p> <p>50 grams of the sample to be examined (in case of juices a larger amount must be taken; thick syrup must be diluted), are strongly acidified with dilute sulphuric acid. All the chlorine which has been previously determined volumetrically in a separate sample, is precipitated by a standardized sulphate of silver solution. The filtrate from the argentic chloride is distilled until acid fumes no longer pass over. This distillate is then mixed with a solution of barium hydrate, any excess of this reagent is precipitated by carbonic acid, and the solution filtered. The filtrate is evaporated to dryness at 110° C. in a weighed platinum capsule: the residue represents the weight of the organic acid salts of barium, which are determined as sulphates or carbonates.</p> <p>If nitrates were present in the sample analyzed, the residue contains also barium nitrate. In that case the nitric acid must be determined, the weight of the barium nitrate calculated from the result, and this value subtracted from the weight of the organic acid salts of barium previously found.</p>

* Translated by the author from the French, E. Langier-Bittman's arrangement.

NITROGENOUS SUBSTANCES. THE ABSOLUTE OR CUPRIC
OXID METHOD.*

[Applicable to all nitrogen determinations.]

"The apparatus and reagents needed are as follows:

APPARATUS.

Combustion tube of best hard Bohemian glass, about 66 cm. long and 12.7 m.m. internal diameter.

Azotometer of at least 100 c.c. capacity, accurately calibrated.

Sprengel mercury air pump.

Small paper scoop, easily made from stiff writing paper.

REAGENTS.

Coarse cupric oxid or wire form. To be ignited and cooled before using.

Fine cupric oxid. Prepared by pounding ordinary cupric oxid in a mortar.

Metallic copper. Granulated copper, or fine copper gauze, reduced and cooled in a current of hydrogen.

Sodium bicarbonate. Free from organic matter.

Caustic potash solution. Make a supersaturated solution of caustic potash in hot water. When absorption of carbonic acid ceases to be prompt, the solution must be discarded.

MANIPULATION.

Filling the tube. Of ordinary commercial fertilizers take 1 to 2 grams for analysis. In the case of highly nitrogenized substances the amount to be taken must be regulated by the amount of nitrogen estimated to be present.

* Reprinted from Bulletin No. 38, U. S. Dept. of Agriculture, Division of Chemistry.

Fill the tube as follows: (1) About 5 cm. of coarse cupric oxid. (2) Place on the small paper scoop enough of the fine cupric oxid to fill, after having been mixed with the substance to be analyzed, about 10 cm. of the tube; pour on this the substance, rinsing the watch glass with a little of the fine oxid, and mix thoroughly with a spatula; pour into the tube, rinsing the scoop with a little fine oxid. (3) About 30 cm. of coarse cupric oxid. (4) About 7 cm. of metallic copper. (5) About 6 cm. of coarse cupric oxid (anterior layer). (6) A small plug of asbestos. (7) 0.8 to 1 gram of sodium bicarbonate. (8) A large, loose plug of asbestos; place the tube in the furnace, leaving about 2.5 cm. of it projecting; connect with the pump by a rubber stopper smeared with glycerol, taking care to make the connection perfectly tight.

OPERATION.

Exhaust the air from the tube by means of the pump. When a vacuum has been obtained allow the flow of mercury to continue; light the gas under that part of the tube containing the metallic copper, the anterior layer of cupric oxid (see 5th above), and the sodium bicarbonate. As soon as the vacuum is destroyed and the apparatus filled with carbonic acid, shut off the flow of mercury and at once introduce the delivery tube of the pump into the receiving arm of the azotometer just below the surface of the mercury seal, so that the escaping bubbles will pass into the air and not into the tube, thus avoiding the useless saturation of the caustic-potash solution.

When the flow of carbonic acid has very nearly or completely ceased, pass the delivery tube down into the receiving arm, so that the bubbles will escape into the azotometer. Light the gas under the 30 cm. layer of oxid, heat gently for a few moments to drive out any moisture that may be present, and bring to red heat. Heat gradually

the mixture of substance and oxid, lighting one jet at a time. Avoid a too rapid evolution of bubbles which should be allowed to escape at the rate of about one per second or a little faster.

When the jets under the mixture have all been turned on, light the gas under the layer of oxid at the end of the tube. When the evolution of gas has ceased, turn out all the lights except those under the metallic copper and anterior layer of oxid, and allow to cool for a few moments. Exhaust with the pump and remove the azotometer before the flow of mercury is stopped. Break the connection of the tube with the pump, stop the flow of mercury, and extinguish the lights. Allow the azotometer to stand for at least an hour, or cool with a stream of water until a permanent volume and a temperature have been reached.

Adjust accurately the level of the KOH solution in bulb to that in the azotometer; note the volume of gas, temperature, and height of barometer; make calculation as usual, or read results from tables.

THE KJELDAHL METHOD

[*Not applicable in presence of nitrates.*]

REAGENTS.

(1) *Acid.* (a) Standard hydrochloric acid, the absolute strength of which has been determined by precipitating with silver nitrate and weighing the silver chlorid as follows:

To any convenient quantity of the acid to be standardized: Add solution of silver nitrate in slight excess, and 2 c.c. pure nitric acid, specific gravity, 1.2. Heat to boiling point, and keep at this temperature for some minutes without allowing violent ebullition, and with constant stirring, until the precipitate assumes the granular form. Allow to cool somewhat, and then pass the fluid through the asbestos. Wash the precipitate by decantation, with

200 c.c. of very hot water, to which has been added 8 c.c. nitric acid and 2 c.c. dilute solution of silver nitrate containing 1 gram of the salt in 100 c.c. of water. The washing by decantation is performed by adding the hot mixture in small quantities at a time, and beating up the precipitate well with a thin glass rod after each addition. The pump is kept in action all the time, but to keep out dust during the washing the cover is only removed from the crucible when the fluid is to be added.

Put the capsule and precipitate aside, return the washings once through the asbestos so as to obtain them quite clear, remove them from the filter, and set aside to recover excess of silver. Rinse the receiver and complete the washing of the precipitate with about 200 c.c. of cold water. Half of this is used to wash by decantation and the remainder to transfer the precipitate to the crucible with the aid of a trimmed feather. Finish washing in the crucible, the lumps of silver chlorid being broken down with the glass rod. Remove the second filtrate from the receiver and pass about 20 c.c. of 98 per cent alcohol through the precipitate. Dry at 140° to 150° . Exposure for half an hour is found more than sufficient, at this temperature, to dry the precipitate thoroughly.

Or (b) standard sulphuric acid the absolute strength of which has been determined by precipitation with barium chlorid and weighing the resulting barium sulphate.

For ordinary work, half normal acid is recommended; i.e., acid containing 18.2285 grams of hydrochloric acid or 24.5185 grams sulphuric acid to the liter; for work in determining very small amounts of nitrogen, one-tenth normal acid is recommended. In titrating mineral acids against ammonia solutions, use cochineal as indicator.

(2) *Standard ammonia*, the strength of which, relative to the acid, has been accurately determined. One-tenth normal ammonia solution, i.e., containing 1.7051 grams of ammonia to the liter, is recommended for accurate work.

(3) *Sulphuric acid*, specific gravity 1.84, free from nitrates and also from ammonium sulphate, which is sometimes added in the process of manufacture to destroy oxids of nitrogen.

(4) *Metallic mercury* or mercuric oxid, prepared in the wet way. That prepared from mercuric nitrate can not be safely used.

(5) *Potassium permanganate* finely pulverized.

(6) *Granulated zinc*, pumice stone, or 0.5 gram of zinc dust are to be added to the contents of the flasks in distillation, when found necessary, in order to prevent bumping.

(7) *Potassium sulphid*. A solution of 40 grams of commercial potassium sulphid in 1 liter of water.

(8) *Soda*. A saturated solution of sodium hydrate free from nitrates.

(9) *Indicator*. Solution of cochineal prepared as follows: Tincture of cochineal is prepared by digesting and frequently agitating 3 grams of pulverized cochineal in a mixture of 50 c.c. of strong alcohol with 200 c.c. of distilled water, at ordinary temperatures, for a day or two. The solution is decanted or filtered through Swedish paper.

APPARATUS.

(1) *Kjeldahl digestion flasks* of hard, moderately thick, well-annealed glass. These flasks are about 22 cm. long, with a round, pear-shaped bottom, having a maximum diameter of 6 cm., and tapering out gradually in a long neck, which is 2 cm. in diameter at the narrowest part, and flared a little at the edge. The total capacity is 225 to 250 c.c.

(2) *Distillation flasks* of ordinary shape, of 550 c.c. capacity, or preferably flasks of the same capacity, of pear-shaped bottom, of well-annealed glass, for both digestion and distillation, fitted with a rubber stopper and a bulb tube above to prevent the possibility of sodium hydrate

being carried over mechanically during distillation. The bulbs are about 3 cm. in diameter, the tubes being of the same diameter as the condenser and cut off obliquely at the lower end. This is adjusted to the tube of the condenser by a rubber tube.

MANIPULATION.

(1) *The digestion.* 0.7 to 0.8 gram of the substance to be analyzed, according to its proportion of nitrogen, is brought into a digestion flask with approximately 0.7 gram of mercuric oxid or its equivalent in metallic mercury and 22 c.c. of sulphuric acid. The flask is placed in an inclined position, and heated below the boiling point of the acid for from five to fifteen minutes or until frothing has ceased. If the mixture froths badly, a small piece of paraffin may be added to prevent it. The heat is then raised until the acid boils briskly. No further attention is required till the contents of the flask have become a clear liquid, which is colorless or at least has only a very pale straw color. The flask is then removed from the frame, held upright, and while still hot potassium permanganate is dropped in carefully and in small quantities at a time till, after shaking, the liquid remains of a green or purple color.

(2) *The distillation.* After cooling, the contents of the flask are transferred to the distilling flask with about 200 c.c. of water, with a few pieces of granulated zinc, pumice stone, or 0.5 gram of zinc dust when found necessary to keep the contents of the flask from bumping, and 25 c.c. of potassium sulphid solution are added, shaking the flask to mix its contents. Next add 50 c.c. of the soda solution, or sufficient to make the reaction strongly alkaline, pouring it down the side of the flask so that it does not mix at once with acid solution. Connect the flask with the condenser, mix the contents by shaking and distil until all ammonia has passed over into the standard acid. The first 150 c.c.

of the distillate will generally contain all the ammonia. This operation usually requires from forty minutes to one hour and a half. The distillate is then titrated with standard ammonia.

The use of mercuric oxid in this operation greatly shortens the time necessary for digestion, which is rarely over an hour and a half in case of substances most difficult to oxidize, and is more commonly less than an hour. In most cases the use of potassium permanganate is quite unnecessary, but it is believed that in exceptional cases it is required for complete oxidation, and in view of the uncertainty it is always used. The potassium sulphid removes all the mercury from the solution, and so prevents the formation of mercur-ammonium compounds which are not completely decomposed by soda solution. The addition of zinc gives rise to an evolution of hydrogen and prevents violent bumping. Previous to use the reagents should be tested by a blank experiment with sugar, which will partially reduce any nitrates that are present, which might otherwise escape notice."

The Gunning Kjeidahl method is frequently employed for the determination of nitrogenous substances in cane juices.

Use a flask made of strong, hard glass having a capacity of about 350 c.c.

25 c.c. of the sugar solution (cane juice) concentrated to a small volume, are introduced into the flask and the same amount of concentrated sulphuric acid is added. 12.0 grams of finely powdered potassium sulphate are then gradually added and flask with contents very gently heated until the frothing ceases. Then the temperature is raised and the solution boiled until it becomes colorless.

The flask is then cooled, its contents transferred to a liter flask, the solution made distinctly alkaline with sodium hydrate and all of the ammonia is distilled into a small flask containing 50 c.c. of a $\frac{n}{10}$ sulphuric acid solution.

Cochineal is preferably used as an indicator and any remaining acid which has not been neutralized by the ammonia distilled over, is determined by titration with $\frac{n}{10}$ ammonia hydrate solution. Of course the sulphuric acid used must be entirely free from nitrogen, if not, a determination of the same must be made and the amount found must be allowed for in all determinations made.

Amides and Albumenoid Nitrogen.*

Non-Nitrogenous Substances. The determination of non-nitrogenous organic substances is effected by aid of basic and neutral acetate of lead and alcohol (pectin and parapectin), by the successive use of water, alkalies, acids, alcohol, and ether (cellulose), by treatment with ether (fats, essential oils), by the aid of yeast fermentation, and alcohol (isolation of mannite).

Cellulose. To make this determination, place 10 grams of the sample, 30 to 40 grams of pure potassium hydrate, and about 30 to 40 c.c. of water into a glass retort. Close the retort by a glass stopper, place in an oil-bath, provided with a thermometer, and heat up gradually. At about 140° C. the solution will commence to boil and foam considerably. Increase the temperature to about 180° C., and continue heating for about one hour. When the contents of the retort cease foaming, become quiet, and begin to turn dry, the end of the reaction has been reached.

Remove the retort from the oil-bath, and after cooling to about 80° C., add hot water and rinse the contents of the retort carefully first with hot and then with cold water, into a beaker.

After cooling, acidify with dilute sulphuric acid; this acid will precipitate the particles of cellulose which have been kept in suspension in the strong alkaline solution.

* Confer: Bulletin No. 107, Bureau of Chemistry U. S. Dept. of Agriculture, or abstracts thereof in Noël Deer, Cane Sugar, Altrincham, 1911, pp. 481-482.

Then, with very dilute sodium hydrate, produce anew a faintly alkaline reaction, so that all of the precipitated substances, excepting the cellulose, may be again brought into solution.

The residue is then transferred to a weighed filtering tube provided with a finely perforated platinum cone and washed out thoroughly, first with hot water, and then with cold. Drying is effected on a water-bath, and the filter with its contents weighed.

The residue is then removed from the filter, ignited, and the weight of the ash found subtracted from the value previously obtained. The difference in weight represents pure cellulose.

Gums. The gummy substances occurring, for instance, in cane-juices are insoluble in alcohol and their determination is based on this property.

Concentrate 100 c.c. of juice to 20 c.c. Pour these into 100 c.c. of alcohol (90% strength), which has been acidified with 1 c.c. of hydrochloric acid.

Allow the precipitate to settle, wash with strong alcohol, first by decantation, and then on a weighed filter. Dry to constant weight and note weight of filter with its contents. Incinerate, and weigh the mineral matter (ash) remaining.

The first weight, after allowing for the tare of the filter paper, represents the weight of the gums plus the weight of the ash in the juice. The second weight, after allowing for the weight of the crucible, is the weight of the ash. This value subtracted from the combined weight of the gums and the ash, is the weight of the gums sought for.

Alkalinity of Sugars. Indicators. Indicators are chemical compounds which exhibit markedly different colors according to their being placed in acid or in alkaline solutions.

A large number of indicators have been suggested for, and tried in, the sugar industry, but, for various reasons,

most of them have been discarded and to-day phenol phthaleïn, litmus, and rosolic acid, (corallin) are about the only indicators that find practical application in sugar work.

These indicators exhibit the following colors:

	In Acid Solution.	In Alkaline Solution.
Phenol phthaleïn	Colorless	Red
Litmus	Red	Blue
Rosolic acid (Corallin)	Pale yellow	Red

These indicators are prepared as follows:

Phenol phthaleïn. 1.0 gram is dissolved in 30.0 grams of 90% alcohol. Two drops of this solution are added to 100 c.c. of the sugar solution to be tested.

Litmus. Powdered litmus is extracted with boiling 80% alcohol which is then discarded. The residue is boiled with distilled water and filtered. The filtrate is divided into two equal parts, one of these is neutralized with sulphuric acid and then intimately mixed with the other portion and this process is repeated until exact neutrality of the whole solution has been attained.

To make litmus paper, a certain volume of the above solution is divided into two equal portions. One of these portions is made faintly acid with sulphuric acid and strips of filter paper are dipped into the same and then dried. The other portion is made faintly alkaline with a weak sodium hydrate solution and also used for preparing filter paper strips in the same way.

The former, the red litmus paper serves as an indicator for alkaline solutions which turn it blue; the other, the blue litmus paper serves as an indicator for acid solutions, which turn it red. Care must be taken to preserve both kinds of filter paper in well stoppered bottles and undue exposure to strong light must be avoided.

Rosolic acid (Corallin). Dissolve 3.0 grams of rosolic acid in 150 c.c. of 90% alcohol and make the solution almost neutral. To 25 c.c. of a sugar solution to be tested, add 2 drops of the rosolic acid solution and 10 c.c. of neutral ether. Shake well, and then allow the fluids to separate—a yellow color indicates acidity, a red color, alkalinity.

Considering all things, phenol phthaleïn and litmus solution appear to be the most satisfactory indicators for general sugar work; the color change exhibited by phenol phthaleïn is well defined even in sugar solutions of a distinctly yellowish cast.

It must be borne in mind however whichever indicator is selected, that indicators vary considerably in their sensitiveness of reaction and therefore any given set of comparative determinations must be carried through with one and the same indicator.

Determination of Alkalinity. To get a qualitative indication as to whether a sugar is alkaline, acid or neutral, dissolve two grams of the sample in about eight or ten cubic centimeters of distilled water. On this solution pour carefully an alcoholic solution of phenol phthaleïn.

If the sample is alkaline, a red ring is formed at the contact zone of the two liquids.

If there is no color-reaction, a few drops of alcoholic phenol phthaleïn solution and one drop of a tenth-normal alkali solution are mixed with 100 c.c. of distilled water, and about 10 grams of the sample under examination are dissolved in this liquid.

If the latter becomes decolorized, the sample is acid, if the solution retains its red color, the sample must be pronounced neutral.

The alkalinity of different sugar products may be caused by potassium, by sodium, by lime, or even partially by free ammonia. It has, however, become customary to report the alkalinity in terms of calcium oxide (caustic lime).

Alkalinity is determined quantitatively by the addition of an acid of known strength to a known weight or volume of the product examined, until neutrality has been established.

The acid used may be either sulphuric or hydrochloric acid, the first of these is the one most commonly employed.

The acid used is generally of "tenth-normal" strength. To prepare this there are needed of

Sulphuric oxide	4.00	grams	SO ₃	in 1 liter of water.
Hydrochloric acid	3.637	"	HCl	" "
Nitric acid	6.289	"	HNO ₃	" "

The acid should be delivered from a burette divided into tenths of a cubic centimeter.

To effect an alkalinity determination, 10 to 20 grams of the product to be tested are weighed and dissolved, or, if a solution is to be examined, from 10 to 20 cubic centimeters are measured out and placed in a porcelain dish. A few drops of the indicator having been added, the acid is allowed to flow in from a burette until the change in color of the indicator shows the reaction to be finished.

1 cubic centimeter of $\frac{n}{10}$ (tenth normal) sulphuric acid corresponds to 0.0040 gram sulphuric oxide, 0.0028 gram calcium oxide, or 0.0047 gram potassium oxide.

The number of cubic centimeters of acid used, multiplied by 0.0028, show therefore the amount of calcium oxide present.

Example. 25 cubic centimeters of a sugar solution (specific gravity 1.198) required 2.4 cubic centimeters $\frac{n}{10}$ sulphuric acid to effect neutralization. This represents $0.0028 \times 2.4 = 0.00672$ gram calcium oxide

$$25.0 : 0.00672 :: 100 : x.$$

$x=0.02688$ per cent calcium oxide. This is *percentage by volume*. If *percentage by weight* is required, the above value must be divided by the specific gravity of the solution, or if a specific-gravity determination and this subsequent calculation are to be avoided, the solution to be tested must in the first place be weighed out, and not measured.

A method devised by Pellet permits determining separately the alkalinity due to lime and that due to the alkalies (potassium, sodium) liberated by the action of lime from their organic acid salts.

Total alkalinity: Titrate with sulphuric acid at the boiling point using litmus as indicator.

Soda and potassa alkalinity: In a certain volume of the sugar juice precipitate the lime as saccharate, by the addition of an equal volume of alcohol. Filter, and in the filtrate determine the alkalinity due to the soda and potassa. This value however is also generally expressed in terms of lime (CaO).

If no stress is laid upon knowing whether the alkalinity is due to lime or to the alkalies, put 50 c.c. of the solution to be tested into a white porcelain dish, use litmus as indicator and titrate with sulphuric acid solution of such strength that 1 c.c. is equivalent to 0.001 gram of lime (CaO).

Following is the standard method of determining alkalinity in raw sugars, as published in 1910 by the Directorate of the Verein der Deutschen Zucker Industrie, and here given by courtesy of Professor Dr. Alexander Herzfeld.

In order to make alkalinity determinations, the following reagents are necessary:

I. *The Concentrated Solution of Phenol Phthaleïn*. Dissolve one part of phenol phthaleïn in thirty parts of 90% alcohol. To carry out the test two drops of this solution are taken for every 100 c.c. of the solution to be tested.

Technical phenol phthaleïn frequently has a slightly acid reaction, but no attention whatever is paid to this,

in other words, the phenol phthaleïn solution is not made either neutral or faintly alkaline before use.

II. *Water of Solution.* For the preparation of the water used for dissolving, there is added, to a large quantity of freshly boiled distilled water, $1/2,000$ of its volume of phenol phthaleïn solution, thus, for instance, to ten liters of water 5 c.c. of the phenol phthaleïn solution are added, and then this solution is made strongly alkaline with a solution of caustic soda, until the fluid retains a pronounced red color. As this red coloration disappears again after one or two days, the amount of water used for dissolving should only be prepared in an amount sufficient for the time. It should, however, always be prepared a few hours before use.

III. *The Titration Acid.* This titration acid is prepared in such a way that 1 c.c. of the same corresponds to a lime alkalinity of 0.0001. Such a solution can be prepared with a sufficient degree of accuracy if one dilutes 36 c.c. of $1/1$ normal sulphuric acid with water up to a volume of ten liters.

IV. *Titration Alkali.* For this purpose one uses a solution of caustic soda which is so diluted that, as with the sulphuric acid, 1 c.c. corresponds to a lime alkalinity of 0.0001. For the present purpose it suffices to standardize such diluted caustic soda solution against the titration acid prepared as above directed.

Determination of Alkalinity. In order to test a raw sugar for its alkalinity, one weighs out ten grams of raw sugar, and measures out 100 c.c. of the faintly red water of solution, which has been prepared as above described, and neutralizes the same in a white porcelain dish as carefully as possible, until it is perfectly colorless, employing for this purpose the titration acid, the preparation of which has been previously described. Then one adds a sufficient amount of the titration alkali solution to it until the fluid again assumes a faintly red coloration.

This coloration must, however, be only sufficiently marked that it can be caused to disappear again, immediately before the addition of the raw sugar, by the addition of 1 c.c. of the standard titration acid.

Then, without delay, the ten grams of raw sugar which have been previously weighed, are dissolved in the fluid. If the indicator-red coloration of water remains on dissolving the sugar in it, or if it grows more intense, then the sugar is alkaline; if the red coloration disappears, the sugar is acid.

In case one is in doubt, one determines the true condition by titrating both with the standard acid as well as with the standard alkali, in order to decide in which way the color turns.

With dark sugars, as a rule, 100 c.c. of the water of solution are not sufficient, and then as much water must be used as is necessary in order to have the sugar solution sufficiently light in color that the titration may be carried out. This, however, is permissible only then when it is found impossible to get on with 100 c.c. In this connection it must be specifically remarked that in the application of this method neutral sugars are classed together with alkaline sugars.

Acidity of Sugars. To determine the acidity of a solution, syrup, molasses, etc., the same course is followed as above described, only, of course, the solution added to effect neutralization is one of sodium hydrate (caustic soda), potassium hydrate (caustic potash), or calcium hydrate (slaked lime), and the change of color of the indicator, if litmus, must be from red to blue, or if phenol phthalein or rosolic acid is employed, from colorless to a bright crimson. Of these solutions the calcium hydrate solution is least desirable, as the carbonic acid of the atmosphere readily precipitates in it calcium carbonate, and so changes the strength of the solution. A $\frac{n}{10}$ sodium-hydrate solution contains 3.996 grams NaOH in 1 liter.

To carry out an acidity determination on a sugar solution or juice, place 50 c.c. of it in a white porcelain dish. Add five drops of phenol phthalein solution as indicator and titrate with a $\frac{n}{10}$ solution of sodium or potassium hydrate. Appearance of a pink color indicates the end point of the reaction.

Sulphurous Oxide in Sugar. *Qualitative Test.* To test qualitatively for sulphurous oxide dissolve from 10 to 20 grams of the sugar in about 25 cubic centimeters of distilled water. Pour into a flask, and add about 5 grams of chemically pure zinc (free from sulphur), and 5 cubic centimeters of chemically pure hydrochloric acid. Suspend a paper moistened with acetate of lead solution in the neck of the flask. If sulphur dioxide is present, it will be liberated from its combinations as sulphuretted hydrogen, and this gas will turn the acetate of lead on the paper a brown or a black color, owing to the formation of sulphide of lead.

Sulphurous oxide can also be qualitatively tested for by impregnating filter paper with a mixture of starch and potassium iodide. This paste is prepared by dissolving 2.0 grams of starch in about 200 c.c. of boiling water and then adding 0.20 gram iodide of potassium in 5 c.c. of water. Paper thus prepared should be well dried and kept in closed bottles or jars. On being moistened and dipped into the solution to be examined, even small amounts of sulphurous acid will be indicated by the appearance of a blue color on the paper, caused by the iodine which is liberated and which reacts with the starch.

Quantitative Determination of sulphurous oxide can be made by either the gravimetric or the volumetric method.

a. By Gravimetric Method. 500 grams of the sugar to be tested are dissolved in a flask and made up to a volume of 500 c.c. with distilled water, 5 grams of chemically pure glacial phosphoric acid are added, the solution poured into a distilling flask, attached to a condensing worm and

a current of washed carbonic acid gas led into the flask insuring therein an atmosphere of carbonic acid gas. When this has been achieved, a flame is lighted under the flask and distillation commenced.

The distillate is received in 50 c.c. of chemically pure bromine water. When at least 50 c.c. have distilled over—the current of carbonic acid gas being constantly maintained in the flask in the meantime—the receiving flask is changed, and the operation is continued until another 50 c.c. of distillate have been secured.

Any sulphite which may have been present in the sample will be found in the distillate, changed to sulphuric acid. To this are now added 10 c.c. of a 20% barium chloride solution and the same thoroughly boiled. The white precipitate of barium sulphate is filtered out, well washed with water to remove any adhering barium chloride, dried, ignited, weighed, and the amount of sulphurous oxide originally present in the sugar calculated by the equation:

$$\text{BaSO}_4 : \text{SO}_2 :: \text{Weight of the barium sulphate found} : x$$

$$233.5 : 64 :: \text{—————} : x$$

If it is desired to express the finding in terms of sulphurous acid, the calculation to be made is as follows:

$$\text{BaSO}_4 : \text{H}_2\text{SO}_3 :: \text{Weight of the barium sulphate found} : x$$

$$233.5 : 82 :: \text{—————} : x$$

*b. By Volumetric method.** Distil 100 grams of the solution to be tested in a current of carbon dioxide after the addition of about 5 c.c. of a 20% solution of glacial phosphoric acid until 50 c.c. have passed over. Collect

* Bulletin No. 107 (Revised) Reprint, July 1, 1909, Bureau of Chemistry.

the distillate in a $\frac{n}{10}$ iodine solution in a flask closed with a stopper perforated with two holes, through one of which the end of the condenser passes and through the other a U-tube containing a portion of the standardized iodine solution. Twenty-five c.c. of $\frac{n}{10}$ iodine solution may be employed, diluted with water to give the desired volume. The method and apparatus may be simplified without material loss in accuracy by omitting the current of CO_2 , adding 10 c.c. of phosphoric acid instead of 5 c.c., and dropping into the distilling flask a piece of NaHCO_3 , weighing not more than a gram, immediately before attaching to the condenser. The CO_2 liberated is not sufficient to expel the air entirely from the apparatus, but will prevent oxidation to a large extent. The U-tube trap may also be omitted if the end of the condenser tube is made to extend below the surface of the iodine solution, and the distillation conducted with a steady flame. When the distillation is finished wash the contents of the U-tube into the flask and determine the excess of iodine with standard thiosulphate solution. On account of its lack of permanence the iodine solution employed should be titrated from time to time with a $\frac{n}{10}$ thiosulphate solution (containing 24.8 grams $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ per liter). 1 c.c. of $\frac{n}{10}$ iodine solution is equivalent to 0.0032 gram of SO_2 .

H. Pellet has suggested a simple and rapid method of estimating sulphurous acid, free and combined, in sugar house products.*

It is an adaptation of the Dujardin sulphurooenometric tube used for testing wine in France, and consists essentially of a tube which, for use, is set up in a vertical posi-

* Suererie Belge, 1910, Vol. XXXIX, p. 152. Intern. Sugar Jour., 1911, Vol. XIII, p. 48.

tion. Into this are poured successively prescribed amounts of the sugar solution to be tested, of potash solution, an acid and some starch as indicator. Titration is carried out with standard iodine solution until the blue color appears. From the graduation which is etched over the upper part of the tube, the sulphurous acid content of the solution examined can be read off directly in milligrams per liter.

Iron Oxide in Sugars. Place about 25 c.c. of the sugar solution to be examined in a white-glass test-tube. Add 5 drops of concentrated hydrochloric acid and 20 drops of ammonium- or potassium-sulpho-cyanide solution and mix well.

Add about 10 c.c. of ether, shake well, and allow the ether to separate. If iron is present the ether will show a red coloration.

CHAPTER VIII

MATERIALS USED IN THE SUGAR INDUSTRY

Bone-Black. *Water.* Heat 10.0 grams at 130° C.–140° C. to constant weight, making the first weighing after three hours.

To execute the following determinations the bone-black must be ground to a fine powder.

Carbon, Sand and Clay. To 5.0 grams of bone-black add some water and 25 c.c. concentrated hydrochloric acid. Boil for 15 minutes, transfer to a dried and tared filter and wash with hot water until disappearance of the acid reaction. Dry filter with contents at 110° C., to constant weight, and burn in weighed crucible. Weight of residue in crucible=sand and clay; this weight subtracted from weight of carbon, sand and clay=carbon.

Calcium Carbonate. Place from 3 to 5 grams of the bone-black in a Geissler, Schrötter or similar apparatus. The latter is weighed with its charge of bone-black, and hydrochloric or sulphuric acid, the acid, of course, being kept in its separate compartment for this weighing. The acid is then admitted to the bone-black, the latter is attacked by the acid and the carbonic acid gas generated is permitted to escape. Dry air, free from carbonic acid gas is then drawn through the apparatus and the latter reweighed. The loss in weight represents the carbonic acid gas liberated and is calculated to its equivalent of calcium carbonate by the formula:

$$44 : 100 :: \text{weight of CO}_2 \text{ found} : x$$

x = calcium carbonate, and this is, as usual, expressed in percentage on the bone-black used.

This determination is also frequently effected in a Scheibler apparatus which is based on a direct volumetric determination of the carbonic acid gas.

Free Lime. (CaO). Two carbonic acid gas determinations are required.

The first of these is made on the original bone-black sample, as above described; the second is carried out on a sample of the bone-black which has been moistened several times with a concentrated solution of ammonium carbonate and which has then been heated repeatedly and sufficiently to cause the volatilization of that reagent. Care must however be taken not to heat to redness. By such treatment the free lime is of course converted into calcium carbonate and from the difference in carbonic acid found in the two determinations the amount of free lime is readily calculated.

Calcium Sulphide. To 25 grams of bone-black add 0.5–1.0 gram potassium chlorate. Mix intimately, then moisten with hot water and add 100 c.c. pure hydrochloric acid. After evolution of the carbonic acid gas boil for 15 minutes. Filter into a 500 c.c. flask, wash residue on filter until tests with barium chloride solution show that all of the sulphates have passed into the flask; cool, fill up to the 500 c.c. mark, mix well and take 200 c.c. of filtrate (10.0 grams of bone-black). In this portion precipitate the total sulphates by barium chloride, filter and weigh. This weight of barium sulphate represents the calcium present in the bone-black both as calcium sulphate and as calcium sulphide. From this weight subtract the amount of calcium sulphate present as such, to be determined as directed below, and multiply the remainder by 0.3087. The value so found is to be recorded as calcium sulphide.

Calcium Sulphate. Proceed exactly as directed above except that the treatment with the potassium chlorate is

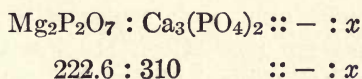
omitted. The weight of the barium sulphate found multiplied by 0.5828 represents the calcium sulphate in the bone-black.

Iron and Aluminum Oxides. 5.0 grams are dissolved in 25 c.c. conc. nitric acid (Sp.Gr. 1.2) and 12.5 c.c. hydrochloric acid (Sp. Gr. 1.12) and the solution is made up to a volume of 500 c.c. with water. This solution is filtered and 100 c.c. of the filtrate (=1.0 gram of bone-black) are placed in a 250 c.c. flask, 25 c.c. sulphuric acid (Sp. Gr. 1.84) are added and the mixture shaken several times during 5 minutes. Then 100 c.c. of a 95% alcohol are added, the mixture is cooled, made up to the 250 c.c. mark with alcohol, well shaken, again made up to the mark with alcohol as there is a decided contraction in volume, and once more well shaken.

After one half hour's standing, the solution is filtered. 100 c.c. are evaporated in a platinum dish until all the alcohol is removed, then poured into a beaker, about 50 c.c. of water are added and heated to boiling. The solution is next made alkaline with ammonium hydrate and the superfluous ammonia boiled off. After cooling the solution is filtered, the precipitate is well washed with hot water, dried, ignited and weighed as $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$.

Tri-calcic Phosphate. Take 5.0 grams of bone-black, add 50 c.c. nitric acid (Sp. Gr. 1.42) and 50 c.c. pure conc. sulphuric acid. Boil gently for one half hour. Cool, wash into a 500 c.c. flask, fill to mark, mix well and pour through a dry filter. In 50 c.c. of the filtrate (=0.5 gm. bone-black) determine phosphoric acid by the ammonium molybdate method to be discussed later.

From the weight of the magnesium pyrophosphate ($\text{Mg}_2\text{P}_2\text{O}_7$) found, the amount of tricalcic phosphate $\text{Ca}_3(\text{PO}_4)_2$ is obtained by the formula:



Sugar in Bone-black. Take 100.0 grams of bone-black, boil four or five times, for 10 minutes each time, with 150 c.c. water. To these washings add a few drops of sodium carbonate solution. Evaporate to small bulk, place in a 100 c.c. flask, neutralize carefully (using phenol phtalein solution as indicator), with acetic acid, add basic lead acetate, fill up to mark, and polarize in a 200 m.m. tube. The polarization found $\times 0.26 =$ percentage of sucrose in the moist bone-black. Determine the percentage of moisture separately and calculate result to dry substance.

Example. Water in bone-black examined = 25.0%, then dry substance = 75.0%. Sucrose found in moist bone-black = 0.70%. Then:

$$75.0 : 0.70 :: 100 : x$$

$$x = 0.93\% \text{ sucrose in dry bone-black.}$$

Organic Matter in Bone-black. Boil 100 grams of sample with 100 c.c. caustic soda solution, of 10° Brix density, for three minutes. The solution is poured into a test-tube and color examined. If colorless or of a very pale yellow, the revivification is satisfactory; if dark yellow or brown the bone-black has been underburned; if green in tint, overburned.

Decolorizing Power of Bone-Black. For this determination use some colorimeter, for instance Stammer's or Lovibond's tintometer.

Dry the bone-black, which is to be tested, at 140° C. Make up a solution of raw sugar or molasses by dissolving about 250 grams in water and make the volume of the solution up to 1000 c.c.

In a large porcelain dish place 100 grams of the bone-black and pour over it 400 c.c. of the sugar or molasses solution. Weigh dish and contents. Heat and keep boiling for five minutes. Cool, reweigh and replace the water lost in the boiling. Mix well, filter through a dry

filter and then determine the color of the unfiltered and of the filtered solution.

<i>Example.</i> Color of unfiltered solution.	=30.0
Color of filtered solution	= 5.0
	<hr/>
Decolorization effected by the bone-black	=25.0
	<hr/>

Expressed in percentage of the original color this is:

$$30.0 : 25 :: 100 : x$$

$$x = 83.3\% \text{ decolorization effected.}$$

Weight of Bone-Black. The impurities not removed by washing and revivification gradually accumulate in the black and cause an increase in its weight.

New bone-black ranges in weight from about 42 to 49 lbs. per cubic foot, depending to a considerable degree on the size of its grain. It is best to use a box of one cubic foot capacity for these weight determinations, taking care to have the same filled in the same manner and to the same extent every time. The weight can also be determined in smaller vessels, flasks, etc., but the results are not as reliable.

Phosphoric Acid Paste. From the fine bone-black dust which accumulates in process, an excellent defecating material can be prepared by the use of sulphuric or hydrochloric acid.

The materials are intimately mixed in vats by means of mechanical stirrers and according to the proportions of bone-black dust and of acid chosen, either free phosphoric acid (H_3PO_4) or monocalcic phosphate $\text{CaH}_4(\text{PO}_4)_2$ is produced. This paste should contain from 12–14% of P_2O_5 .

The total P_2O_5 and the soluble P_2O_5 are determined in the following manner.

Reagents Used. Molybdic Solution: Dissolve 100 grams molybdic acid in 400 grams, or 417 c.c. ammoniac hydrate

(sp.gr.=0.96), and pour the solution thus obtained into 1500 grams, or 1250 c.c. nitric acid (sp.gr.=1.20). Keep the mixture in a warm place for several days, or until a portion heated to 40° C. deposits no yellow precipitate of ammonium-phospho-molybdate. Decant the solution from any sediment, filter and preserve in a glass-stoppered vessel.

Magnesia mixture: Weigh out 110 grams of crystallized magnesium chloride ($MgCl_2+6H_2O$), also, 280 grams ammonium chloride. Dissolve this ammonium chloride in distilled water, and add 700 c.c. of ammonic hydrate (sp.gr. 0.96). Into this solution pour the magnesium chloride, previously dissolved in some distilled water, and make the total volume of the solution up to 2 liters.

Ammonic hydrate: (For washing). To 1 volume of ammonic hydrate (sp.gr. 0.96) add 3 volumes of distilled water. Ammonic nitrate solution: (For washing). Dissolve 200 grams ammonium nitrate in distilled water, and make the volume up to 2 liters.

Remarks: Nitric acid. To make nitric acid of sp.gr. 1.20 from nitric acid of sp.gr. 1.42 add 1 liter of distilled water to each liter of nitric acid of sp.gr. 1.42.

Ammonic hydrate. To make ammonic hydrate of sp.gr. 0.96 from ammonic hydrate of sp.gr. 0.90, add 1620 c.c. of distilled water to each liter of ammonic hydrate of sp.gr. 0.90.

Analysis: Water Soluble P_2O_5 . Weigh out by difference 2.0 grams of paste. Wash into a beaker with distilled water ($T=60^\circ C.$). Stir well so as to leave no lumps. Then wash on to a 9 c.m. S. & S. No. 589 filter and continue to wash with warm water ($T=60^\circ C.$) allowing all of the water to pass through each time before adding more, until the filtrate measures 250 c.c. Mix the washings and of these take 62.5 c.c.=0.5 gram and proceed as directed further on.

Total P_2O_5 . Weigh out 2.0 grams of paste. Wash into a beaker with as little water as possible, stir well so

as to leave no lumps. Add 30 c.c. concentrated nitric acid and 5 c.c. concentrated hydrochloric acid. Boil until all phosphates are dissolved and organic matter destroyed. Cool and dilute to 250 c.c. Mix well and pass through a dry filter. Of filtrate take 62.5 c.c. = 0.5 gram. Neutralize with ammonic hydrate and clear with a few drops of nitric acid and add about 10 grams of ammonium nitrate.

Having thus prepared both solutions proceed in each case as follows:

To the hot solution (about 65° C.) add 200 c.c. of warm molybdic solution (about 35° C.) Bring the temperature up to about 65° C. and digest for about 2 hours. Filter, and wash with ammonium nitrate solution (1:10). To filtrate add 50 c.c. more of molybdic solution, warm to 65° C. and set aside at a temperature of about 50° C. for 12 hours more to test whether all has been precipitated. Dissolve precipitate on filter with hot water and ammonic hydrate (sp.gr. 0.90) and wash into a beaker to a bulk not to exceed 100 c.c. Nearly neutralize with hydrochloric acid, cool, add 125 c.c. of magnesia mixture from a burette under constant stirring, one drop per second. After 15 minutes add 30 c.c. ammonic hydrate (sp.gr. 0.96). Allow to stand for 12-15 hours, filter, wash with dilute ammonic hydrate, dry, ignite intensely for 10 minutes and weigh the magnesium pyro-phosphate ($Mg_2P_2O_7$).

Retest the filtrate by adding 25 c.c. more of magnesia mixture and allow to stand for 6 hours more to see whether any further precipitation occurs. If a further precipitate is formed, filter it out, treat as before directed and add its weight to that of the precipitate previously obtained.

Calculate result as follows:



$$222.6 \quad : 142 \quad :: \text{---} : x.$$

or multiply by factor 0.6379.

Limestone furnishes the caustic lime and the carbonic acid gas both of which are extensively used in certain processes of sugar refining. In selecting a limestone for sugar purposes care must be had to secure a quality which is free from any considerable amounts of silica, gypsum, clay, magnesium salts and alkalies.

When limestone is subjected to a heat of between 700° and 1200° C. it is decomposed into calcium oxide and carbonic acid gas, the latter in fact begins to be set free even at a temperature but little above 400° C.

A very convenient method for determining both the calcium oxide and the carbonic acid gas is given by G. Lunge,* as follows:

Calcium oxide (CaO). 100 grams of the caustic lime are carefully slaked and run into a 500 c.c. flask. The volume is made up to 500 c.c. and, while agitating the mixture constantly, 100 c.c. are removed by a pipette. These 100 c.c. are run into another 500 c.c. flask, made up to volume, well mixed and from this solution 25 c.c. (=1 gram caustic lime) are taken for analysis.

To this solution a small amount of an alcoholic solution of phenol phthaleïn is added and then the solution is titrated with normal hydrochloric acid until the rose color has disappeared. This occurs when all free, uncombined calcium oxide is satisfied, but ere the calcium carbonate is attacked. Each c.c. of the normal hydrochloric acid = 0.028 gram CaO. This operation must be executed carefully and under constant shaking, in order to have it yield reliable results.

Carbonic Acid Gas (CO₂). The calcium oxide and the calcium carbonate are determined together by dissolving the sample in normal hydrochloric acid and then titrating back with normal caustic soda solution. From the value so found there is subtracted the amount of calcium oxide

* Chemisch-technische Untersuchungsmethoden, 1899, 4th Edition, Vol. I, p. 427.

determined as above described; the balance remaining represents the calcium carbonate.

Of course the calcium carbonate can also be found by determining the carbonic acid gas as described under Bone-black and calculating the calcium carbonate corresponding to the amount of carbonic acid gas so found.*

Coal. Properties. Coal is a hydrocarbon, the essential components of which are volatile combustible matter, carbon, hydrogen, ash and moisture.

The amount of heat required to do a certain amount of work is in fuels generally expressed in thermal units and the unit commonly used is known as the British Thermal Unit which represents the amount of heat required to raise 1 lb. of water from 39° to 40° F., i.e., 1° Fahrenheit in temperature. 2545 B.T.U. per hour equal 1 horsepower.

The heat yielded by the perfect combustion of 1 lb. of carbon to carbon dioxide is 14,500 B.T.U.; the heat yielded by the combustion of 1 lb. of carbon to carbon monoxide (incomplete combustion) is 4400 B.T.U.; the heat yielded by the perfect combustion of hydrogen to water is 62,100 B.T.U.

The evaporative value of a fuel is the amount of water which 1 lb. of the fuel will evaporate at normal boiling point; its calorific power divided by the latent heat of steam formation, viz.: 967. On an average 1 lb. of coal will evaporate 8 lbs. of water.

When coal is burned under a boiler the volatile gases are at once set free and these, combining with the necessary air are changed to carbon dioxide, either at once, or, possibly, they are first transformed into carbon monoxide and this, in turn, is changed into carbon dioxide by combining

* Also see HEYER, Chem. Ztg, Vol. XXXIV, p. 102, 1909, for a general method of determining, by means of a dilute solution of ammonium chloride, calcium as oxide, hydrate, and saccharate in the presence of calcium-carbonate, sulphate, etc.

with more oxygen of the air. Up to 700° C. (1292° F.) chiefly carbon dioxide is produced, above 700° C. in the presence of carbon, carbon monoxide results.

It must therefore be remembered that heated carbon dioxide is, by contact with heated carbon, again reduced to carbon monoxide which reaction involves the absorption, i.e., the loss of heat amounting to about 6700 B.T.U., and it is therefore a matter of great importance to have the combustion properly regulated and controlled, if the maximum efficiency is to be obtained from a fuel. If too little air is furnished the fuel for complete combustion, the carbon dioxide will react with more carbon and become reduced to carbon monoxide, as before stated; if too much air is admitted the furnace and gases are cooled—preheating the air for combustion means a higher temperature in the zone of combustion and makes for economy.

As air contains 21% of oxygen, an ideal combustion of pure carbon would produce 21% of carbon dioxide. The use of anthracite or coke actually does yield up to 19% of carbon dioxide, bituminous coal, at best, yields only about 16% of carbon dioxide.

Analysis. Great care must be taken to secure a correct average sample of the coal to be tested; this is finely ground and about 25 grams are reserved for the analysis.

Two samples of two grams each are taken. *Sample No. 1.* Dry in air-bath at 115° C. to constant weight. Loss in weight is water; the weight of the balance remaining represents the dry substance in the coal.

Expose this in a closed platinum crucible for three minutes to a strong Bunsen flame and then, without allowing it to cool, subject it for three minutes more to the intense heat of a blast lamp. The loss in weight thus found is recorded as volatile and combustible matter.

Sample No. 2. Ignite in a platinum crucible until all of the carbon is consumed and only the ash remains; determine the weight of this direct, by weighing.

State the percentage of water in the sample, but express the percentage of volatile and combustible matter, of fixed carbon and of ash, in terms of dry substance.

From the data of this analysis the calorific power of the coal can be *approximately* calculated by Lenoble's formula in which:

$$a = \% \text{ water,}$$

$$b = \% \text{ ash (in coal as is, i.e., not in the dry substance),}$$

$$k = a + b,$$

P = calorific power of coal in calories. To change this value into B.T.U. multiply by $9/5$. $P = 87.4(100 - k)$

Example.

$$\text{Let } a = 5.45$$

$$b = 17.90$$

$$k = 5.45 + 17.90 = 23.35$$

$$P = 87.4(100 - 23.35)$$

$$P = 6699 \text{ calories}$$

$$= \frac{6699 \times 9}{5} = 12021 \text{ B T.U.}$$

Carbon in Ash. To determine how thoroughly a coal has been burned, analyze the coal and the ash resulting from its burning exactly in the manner described, and then calculate the percentage of carbon lost (i.e., not burned) in the ash, by the following formula:

Percentage of lost carbon

$$= \frac{\% \text{ ash in coal (carbon + vol. matter of ash)} 100}{100 \text{ (carbon of coal + vol. matter)}}$$

Example.

	Coal.	Ash.
Carbon	76.63	9.61
Vol. matter	10.71	1.23
Ash	12.66	89.16

$$\frac{12.66(9.61 + 1.23)100}{(76.63 + 10.71)100} = \frac{13723.44}{8734} = 1.57\% \text{ carbon lost in ash.}$$

Flue-Gases. The constituents to be determined are carbonic acid, carbon monoxide, oxygen and nitrogen gases.

The apparatus generally used is that of Orsat, which consists of a burette, water-jacketed, and three absorption tubes. In these tubes are placed respectively the following solutions:

1. A solution of potassium hydrate, density approximately 60° Brix;

2. A solution of pyrogallic acid, 10 parts in 100 parts of hot water and 200 parts of potassium hydrate solution, about 50° Brix;

3. A solution of cuprous chloride. This is made as follows: 35 grams of cupric chloride are dissolved in a little water and stannous chloride is added in sufficient amount to change the color. The white precipitate is repeatedly washed by decantation with water, guarding the precipitate against exposure to the air. Transfer into a flask with 200 c.c. of concentrated hydrochloric acid and add about 120 c.c. of water. Place some copper wire into the flask and keep same carefully closed till used.

The sample of flue-gas having been drawn into the receiving bottle by displacement of water, pass the gas successively into the solutions contained in the tubes. It first passes into the potassium hydrate solution; this absorbs the carbonic acid gas and the volume of which is read off on the scale of the burette. The oxygen is absorbed by the pyrogallic acid solution and its volume is ascertained as before; the carbon monoxide is absorbed by the cuprous chloride solution and its volume is measured. The final, residual gas is essentially nitrogen, and its amount is found by noting the difference between 100 and the third burette reading.

Sulphurous acid gas is tested for by shaking up a sample of the gas with a starch solution containing iodine; sulphuretted hydrogen is evolved and detected by its action

on a test-paper moistened with lead acetate or basic lead acetate, sulphuretted hydrogen turning the same black.

Sulphur. Must be tested for its purity; the sample is finely powdered, and one-half a gram is placed in a glass-stoppered flask and well shaken with an excess of saturated bromine water, about 400 c.c. will answer. The bromine oxidizes the sulphur to sulphuric acid, the solution is boiled, expelling the excess of bromine (use a hood for this work), the residue is passed on a filter, washed well with hot water, dried and weighed, and the percentage of impurities thus determined.

Lubricating Oils. *Alkalinity and Acidity* are tested for by thoroughly shaking a sample of the oil with hot distilled water in a separatory funnel, allowing the water to separate and then testing the same with litmus or some other standard indicator.

Viscosity. This determination is of importance in judging of the quality of an oil. The test is conducted by allowing a measured volume of water at some standard temperature, say 20° C., to flow through an orifice or exit-tube and noting the time—in seconds—it requires to do so. An identical volume of the oil, at the same temperature, is then made to flow through the same opening and the time of its flow noted.

The specific viscosity of the oil is found by dividing the time of its flow by the time of flow of the water.

Congelation. Place 20 c.c. of the oil in a test-tube provided with a thermometer and insert in a freezing mixture. Stir well and note the temperature at which the oil stops flowing.

Saponification. This test is made to learn whether a mineral oil has been mixed with an animal or vegetable oil.

A solution of potassium hydrate is prepared by dissolving 100 grams of this reagent in 60 c.c. of distilled water. 4 c.c. of this solution are mingled with 4.0 grams of the oil

and the mixture is heated for an hour. After cooling, place in a separatory funnel, extract the mineral oil with ether, and, after evaporation of the ether, weigh the mineral oil which constitutes the residue. Any discrepancy between the weight originally taken—4.0 grams—and the weight of the residue obtained, is due to animal or vegetable oils admixed.

Hydrochloric Acid. *Density.* The density is determined by a specific gravity determination or by means of an hydrometer.

24.5	Baumé=	Sp.Gr.,	1.200	=	40.777%	HCl	at	15°	C.
20.0	“	=	“	1.157	=	31.805	“	“	
16.0	“	=	“	1.122	=	24.874	“	“	

The chlorine may also be determined by volumetric analysis, using a nitrate of silver solution of known strength, and potassium chromate as indicator.

Iron. The presence of iron is tested for by ammonium or potassium sulpho-cyanide, in a diluted sample of the acid, as previously described.

Arsenic. Mix 10 c.c. of the acid with 10 c.c. of water in a test-tube. Upon this solution pour 5 c.c. freshly prepared sulphuretted hydrogen water. A yellow ring developed at the zone of contact after an hour's standing indicates arsenic. A quantitative determination of arsenic is best made by the well known Marsh test.

Water. The quality of the water supply is of the greatest importance for sugar houses. It is necessary to determine in such a water certain constituents, and to control its composition from time to time.*

Total Solids. Evaporate 1 liter of the water in a porcelain or platinum dish on a water-bath and dry to constant weight at 130° C.

Organic and Volatile Matter. Evaporate 250 c.c. of the water to dryness in a weighed platinum dish. Dry at 130° C.

* Cairns-Waller: Manual of Quantitative Chemical Analysis.

cool and weigh. Heat the dish at a low red heat until all organic matter is destroyed. Add 25 c.c. of water saturated with carbonic acid gas, evaporate to dryness on a water-bath, repeat treatment, and finally dry at 130° C.; cool and weigh.

The *organic and volatile matter* is represented by the difference between the two weighings.

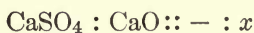
Silica, Lime, Magnesia, Oxides of Iron and Aluminum. Evaporate 5 liters of water nearly to dryness on a sand-bath; acidulate with hydrochloric acid and carry to dryness on a water-bath. Then dry at 110° C., until there is no more odor of hydrochloric acid.

Then take up with 20 c.c. of water and 1 c.c. of concentrated hydrochloric acid, heat to boiling, add 50 c.c. more water, filter and wash with hot water until a test with silver nitrate solution no longer gives any turbidity. Dry filter and contents at 110° C., ignite in a weighed platinum crucible, cool and weigh. This weight represents the *silica*.

Make the filtrate and washings obtained above, alkaline with ammoniac hydrate, boil out excess of ammonia, allow the precipitate to settle, decant on a filter, dissolve the precipitate in a little hydrochloric acid, reprecipitate by ammoniac hydrate and again boil. Filter, wash and dry the precipitate. The weight found is recorded as the *oxides of iron and aluminum*.

Concentrate the filtrate and washings to 100 c.c. Add 1 c.c. ammoniac hydrate, acidify with hydrochloric acid, boil for a minute and again make alkaline with ammoniac hydrate. The ammonium chloride thus produced prevents the precipitation of magnesium hydrate. Add 40 c.c. of ammonium oxalate solution (1 part of oxalate to 24 parts of water). Heat just to boiling and allow the precipitate to settle for some hours. Then decant through a filter, wash by decantation with about 25 c.c. of hot water and set filtrate aside as filtrate A.

Dissolve the precipitate in the beaker, this is calcium oxalate plus a little magnesium oxalate, with a very small amount of hot dilute hydrochloric acid. Make alkaline with ammoniac hydrate, add 5 c.c. ammonium oxalate solution, stir well and then allow the precipitate to settle. Filter, wash thoroughly with hot water. Dry at about 100° C. and brush the precipitate into a clock-glass. Burn the filter in a weighed platinum crucible until the ash is white. When the crucible is cool, add the precipitate from the clock-glass to the crucible contents, moisten the precipitate with concentrated sulphuric acid, place cover on crucible and heat very cautiously to expel the excess of the sulphuric acid. Then ignite strongly for a few minutes, cool in a desiccator and weigh. This precipitate is calcium sulphate and this is calculated to lime (CaO) by the equation:

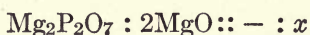


$$136 : 56 :: - : x$$

Enter the weight of the *lime*.

Filtrate A, previously set aside, contains the major portion of the magnesia, the second filtrate contains the balance. Acidify the latter and washings with hydrochloric acid, concentrate to a small volume and add it to the first. Make combined filtrates alkaline with ammoniac hydrate. Add 30 c.c. of a solution of hydrodisodic phosphate (1 part in 10 parts of water), stir well with a glass rod, avoid however touching the sides of the vessel with the glass rod. Allow to stand cold for 12-16 hours and then test a few drops of the clear fluid with a few drops more of hydro-disodic phosphate to see if enough of this reagent has been added. If not, add more, allow to precipitate as before, and then filter on a small filter. Wash with dilute ammoniac hydrate (1 part of strong ammoniac hydrate and 2 parts of water) until a few drops of the washings show no more turbidity with a solution of barium

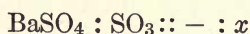
chloride acidulated with hydrochloric acid. Dry the precipitate on the filter, brush into a clock-glass and burn the filter in a weighed platinum crucible. Then add the precipitate from the clock-glass, ignite again at bright red heat, keeping the crucible covered in the meantime. Then remove the cover and heat to bright redness until the precipitate appears white. A few drops of nitric acid added will hasten the perfect incineration. Cool in a desiccator and weigh. The weight of the precipitate, which is magnesium pyro-phosphate, $Mg_2P_2O_7$ is calculated to magnesia, MgO , by the equation:



$$222 : 80 :: - : x.$$

Enter the weight of the *magnesia*.

Sulphuric Acid. To 4 liters of the water add hydrochloric acid until acidified, evaporate to about 100 c.c. filter and to filtrate add 20 c.c. of barium chloride solution (1 part in 10 parts of water). Boil thoroughly, test to see if sufficient barium chloride has been added, boil again, allow the precipitate to settle, wash by decantation with hot acidulated water several times, finally filter, wash with hot water until the test with nitrate of silver solution no longer reacts for chlorine. Dry the precipitate at about $100^\circ C$. brush the precipitate into a clock-glass, burn the filter in a weighed platinum crucible (moistened with a few drops of sulphuric acid), then add the precipitate to the crucible, ignite strongly, cool in a desiccator and weigh. The precipitate is barium sulphate which is calculated to sulphur trioxide (SO_3) by the equation:



$$232.8 : 80 :: - : x$$

Enter as *sulphur trioxide*.

Chlorine. Evaporate 4 liters of the water to about 100 c.c. Determine the chlorine volumetrically by means of a standardized solution of nitrate of silver, using potassium chromate solution as an indicator. Run the silver-nitrate solution in from a burette until the red chromate of silver is formed. Enter as *chlorine*.

Hardness. This term means the soap-destroying power of the water. One degree of hardness represents the effect produced on a soap solution by water which contains 1 grain of calcium carbonate per gallon.

Soap solution: Dissolve 10 grams of soda soap in 1 liter of 90% alcohol. Filter and keep in glass stoppered bottle. For use take of this soap solution 100 c.c. add to it 100 c.c. water and 33 c.c. of alcohol, adding the latter before the water, and shaking gently.

Calcium chloride solution: dissolve 1 gram of calcium carbonate in dilute hydrochloric acid. Evaporate to dryness, expel all free acid and dissolve the residue in 1 liter distilled water. 1 c.c. of this solution equals 1 mgr. of calcium carbonate. Dilute 10 c.c. of this solution with distilled water to 100 c.c. place in a narrow glass-stoppered bottle and introduce the soap solution from a burette, a little at a time, shaking after each addition of soap. Proceed thus until a permanent lather is formed which remains unbroken for 5 minutes. From the number of c.c. of soap solution needed for the formation of a permanent lather, the milligrams that 1 c.c. of the soap solution is equivalent to is readily determined.

To determine the hardness of the water place 100 c.c. of the water in a bottle as before directed and proceed exactly as described above. The number of c.c. of soap solution required multiplied by 10 = the number of mgrs. of calcium carbonate per liter that the hardness of the water is equivalent to. This value multiplied by 58,318 and divided by 1,000,000 = degrees of hardness of the water. Enter *degrees of hardness*.

Improvement of Waters. Waters must frequently be treated before they can be used in sugar houses.

Organic impurities are diminished by coagulation with alum followed by settling and filtration through sand or diatomaceous earth.

An excess of sulphate of lime is remedied by the addition of sodium carbonate. For improvement of waters rich in the bicarbonates, sulphates and chlorides of lime and magnesia, milk of lime and caustic soda are employed; for waters containing an excess of the bicarbonates of lime and magnesia, milk of lime in slight excess is used to advantage.

CHAPTER IX

ANALYTICAL CONTROL IN CANE-SUGAR MANUFACTURE

Determinations Required. Work in a cane-sugar house involves the analytical control of:

The Sugar Cane.

- (a) Cane.
- (b) Cane Juices: First Mill Juice, Mixed Juices, Last Mill Juice, Clarified Juice.
- (c) Syrup.

Bagasse.

Filter Press Work.

- (a) Juices.
- (b) Filter Press Cake.

Sugars.

- (a) Sugar.
- (b) Fill Mass.
- (c) Molasses: First Molasses, Exhaust Molasses.

Waters.

- (a) Condenser Water.
- (b) Waste Water.
- (c) Boiled-feed Water.

As an indication of the determinations which should be made, the following scheme is suggested; local conditions will of course determine the number of samples to be taken and submitted to analysis.

Sample	Determinations to be made	Number of Determinations in 24 Hours
SUGAR CANE		
<i>a. Cane fiber.</i>	Fiber	1
<i>b. Cane juices</i>		
First mill juice	Brix	24
	Sucrose	2
	Purity	2
	Alkalinity } Acidity }	2
Mixed juices	Brix	24
	Sucrose	2
	Purity	2
	Reducing sugar	2
	Ash	1
	Alkalinity } Acidity }	2
Last mill juice	Brix	24
	Sucrose	2
	Purity	2
	Alkalinity } Acidity }	2
Clarified juice	Brix	24
	Sucrose	2
	Purity	2
	Alkalinity	2
<i>c. Syrup</i>	Brix	2
	Sucrose	2
	Purity	2
	Reducing sugars	2
	Ash	2
	Acidity	2
BAGASSE	Sucrose	24
	Fiber	2
	Dry substance	12
FILTER-PRESS WORK		
<i>a. Juices</i>	Brix	24
	Sucrose	2
	Purity	2
	Alkalinity	2
<i>b. Filter-press cake</i>	Sucrose	2
	Water	2
SUGARS		
<i>a. Sugar</i>	Sucrose	2
	Reducing sugars	2
	Water	2
	Ash	2

Sample	Determinations to be made	Number of Determinations in 24 Hours
<i>b. Fill mass</i>	Brix	Each strike
	Sucrose	Each strike
	Purity	Each strike
<i>c. Molasses</i> First molasses	Brix	Each strike
	Sucrose	Each strike
	Purity	Each strike
Exhausted molasses	Clerget	Each strike
	Brix	Once in 10 days
	Sucrose	Once in 10 days
	Purity	Once in 10 days
	Clerget	Once in 10 days
WATERS.		
<i>a. Condenser water</i>	Alpha-naphthol	Frequently
	Sucrose by polariscope	As needed
<i>b. Waste water</i>	Alpha-naphthol	Frequently
	Sucrose by polariscope	As needed
<i>c. Boiler-feed water</i>	Alpha-naphthol	Frequently
	Sucrose by polariscope	As needed

The testing of these various substances and products must provide for a determination of the: Fiber, Brix, Alkalinity, Sucrose, Clerget, Purity, Reducing Sugars, Glucose Ratio, Ash, Water, Dry Substance, and application of the Alpha-naphthol reaction.

Sugar Cane. The choice of analytical methods to determine the sucrose in the cane lies, to a certain extent, between direct and indirect methods.

The former, that is to say, the direct method, is really available only in factories employing the diffusion process, or for the determination of sucrose in individual cane stalks. In ordinary cane-sugar house work however one or the other of the indirect methods must be employed.

Select at least two canes from each load delivered at the factory. Retain 10% of these canes at the end of each shift.

Sucrose in Cane: Direct Method. Although there are several such direct methods available, probably the method

by water extraction due to Zamaron, best answers the purpose. This method involves the use of:

1. A metal beaker with a discharge cock at the bottom.

2. A cylinder made of wire gauze, small enough to be placed on a tripod which is inserted in the first metal vessel spoken of, and permitting some space to exist between the bottoms of the two.

3. A perforated metallic disc attached to a rod, the disc to be of such dimensions as to fit well into the gauze cylinder.

Underneath vessel No. 1 there is placed a liter flask in such a position that the contents of this vessel No. 1 may be readily discharged into the flask; the flask itself is placed in a bath through which cold water circulates. Vessel No. 1 is placed over a Bunsen burner, or other heating device. A charge of 50 grams of the cane pulp is introduced into vessel No. 2 which is inserted in vessel No. 1. 1,000 c.c. of pure water containing enough lime to make it faintly alkaline, is heated, and then 200 c.c. of this water are poured on the pulp contained in the gauze cylinder. Vessel No. 1, in which vessel No. 2 is inserted, is now heated to boiling. This boiling is continued for a few moments and then the burner is removed. Next the contents of these vessels are run into the liter flask in which a few cubic centimeters of neutral lead acetate solution have been placed. When the fluid extract has passed from the metal vessels into the liter flask the pulp is strongly squeezed, and, when no more fluid flows out of the stop-cock, the same is closed, and the treatment with the hot alkaline water repeated. This time, however, only 150 c.c. are used. This treatment with 150 c.c. of the hot alkaline water is given five times in all. After the final treatment the pulp is very thoroughly squeezed in order to remove as much liquid from it as possible. Then 1 c.c. of basic lead acetate is added to the fluid in the liter flask, its contents are made up to the mark, well shaken, filtered, and polarized in a 400 m.m. tube. The readings multiplied

by 2.605 are taken as representing the percentage of sucrose in the cane.

While this method is not free from objections, it is possibly, for general purposes, the best direct method at present known for the determination of sucrose in the cane.

Sucrose in Cane: Indirect Methods: Indirect Method I. This is based on adding the sucrose extract in the juice and the sucrose retained by, i.e., lost in the bagasse.

The sucrose in the juice is determined in the usual way on a composite sample obtained from aliquot parts of each measuring tank. These samples are preserved during collection by the addition of one part of formaldehyde to 5,000 parts of juice. The result obtained expresses per cent of sucrose in 100 parts by weight of the juice.

The sucrose in the bagasse is determined according to the method of Zamaron previously described.

The sum of the sucrose found in the juice and that found in the bagasse represents the sucrose in the cane.

Indirect Method II. The sucrose found in the mill juice is multiplied by a factor.

The sucrose is determined in the following manner, on a sample which has not been preserved with subacetate of lead.

Place 100 c.c. of the juice into a 100–110 c.c. flask, clarify with the minimum amount of subacetate of lead solution, acidulate with acetic acid, make the volume up to 110 c.c. with water, mix thoroughly, filter and polarize the filtrate. The percentage of sucrose is found by aid of the Schmitz table. In accurate work the error caused by the volume of the lead precipitate must be allowed for. The sucrose in the cane is then calculated by means of the following formula:

Percentage of sucrose in cane

$$= \text{Percentage of sucrose in juice} \times \frac{(100 - \% \text{ fiber})}{100}.$$

At best, the figure thus obtained is for several reasons only an approximation. Various more or less empirical factors have been employed for the purpose in different countries at different times, thus, in Egypt at one time the factor 0.87 was in use, in Java 0.85, etc.

To obviate these difficulties, Noël Deerr conceived of an "absolute juice of the cane," which term he defines to mean all that is not left behind on extraction by water.

In this method Deerr uses this "absolute juice" as the basis for multiplication by a factor, said factor to be determined by each factory to meet its own conditions.

Cane Fiber is the term applied to that part of the sugar cane which remains after all water soluble matter has been extracted therefrom.

From 10 to 20 kilograms of cane are crushed in a laboratory mill, and about 75% of the juice is expressed. The resultant bagasse is weighed, cut into small pieces, and a part thereof is finely ground.

Of this pulp take 20 grams and extract first with cool water, not above 50° C., to insure extraction of such albuminoids as might be coagulated by hot water; then treat with water of about 70° C., and finally with water near the boiling temperature, until all soluble matter has been extracted. This entire operation should be completed in about 45 minutes. The exhausted bagasse, i.e., the fiber, is then removed and dried to constant weight.

The result is calculated as follows:

Cane used.....	20 kgs.
Bagasse	5 "
Extracted juice.....	15 "
Fiber in 20 grams of bagasse	9 grs. = 45.0%
Fiber in cane	$\frac{5 \times 45.0}{20} = 11.25\%$

This value can also be found in the following way:

$$\begin{aligned} \text{Bagasse} &= 25\% \text{ of cane} \\ \text{Fiber} &= 45\% \text{ of bagasse} \\ \text{Fiber in cane} &= \frac{45.0 \times 25.0}{100} = 11.25\% \end{aligned}$$

Available Sugar. This can be calculated by the formulae of Prinsen Geerligs or taken from his tables.*

Cane Juices. The samples should be taken from the mills preferably by some continuous sampling-device or automatic sampler, and formaldehyde 1:5000 added as a preservative.

First Mill Juice. After mixing thoroughly, strain the juice through a fine metallic gauze into a cylinder which has a discharge pipe a few inches above its bottom. This pipe is kept closed while the heavy suspended impurities settle; the froth will of course rise to the top. When the intermediate portion of the juice is clear it is drawn off for analysis.

Brix. Note the temperature of the juice and take the reading by an accurate Brix hydrometer, making this reading on the surface level of the juice. Correct the reading for temperature and record.

Alkalinity. Prepare a sulphuric acid solution for titration of such a strength that 1 c.c. corresponds to a lime alkalinity of 1 milligram. Place 50 c.c. of the juice to be tested in a white porcelain dish, use litmus paper as indicator, and make the titration. For *acidity* determinations titrate with alkali solution of known strength.

Sucrose. The sample on which this determination is made is obtained by taking aliquot samples of the first mill-juice samples taken throughout each factory shift. These samples are to be kept in wide mouthed glass jars, and are to be preserved by the addition of mercuric chloride, (corrosive sublimate), using strength 1:5000.

* International Sugar Journal, 1912, p. 274.

Place 100 c.c. of this sample in a 100/110 c.c. flask, add 3 to 5 c.c. subacetate of lead, complete the volume up to 110 c.c. with distilled water, filter and polarize in a 200 m.m. tube. Use Schmitz's table for determination of the sucrose percentage.

$$\text{Purity:} = \frac{\text{Sucrose} \times 100}{\text{Brix}}$$

This work can also be done advantageously by Horne's dry lead defecation method which obviates the use of the 110 c.c. flask.

Mixed Juices. Take continuous samples from each measuring tank, or samples equal and sufficient in amount, every ten minutes; of these make a composite sample each hour. Preserve this with mercuric chloride, strength 1:5000.

Brix. As previously directed.

Alkalinity, acidity. " "

Sucrose. " "

Purity. " "

Reducing Sugars. The sample on which this determination is made is obtained by reserving 25 c.c. of each sample of the mixed juices procured as previously described, but for this determination samples preserved with formaldehyde can not be used.

Determination of the reducing sugars is carried out by volumetric analysis, by means of Fehling's Solution.

Fehling's Solution is prepared as follows:

Solution No. 1. Dissolve 34.639 grams of re-crystallized sulphate of copper in water, making the solution up to 500 c.c.

Solution No. 2. Dissolve 173 grams of pure crystallized Rochelle salts in distilled water. Add 100 c.c. of a 50% solution of purest sodium hydroxide, and make the volume of the resultant solution up to 500 c.c.

Keep Solutions No. 1 and No. 2 in separate flasks.

Standardization of Fehling's solution: Take 9.5 grams

of a perfectly dry, chemically pure sucrose, or, in default of this, use for the purpose the stated amount of the highest grade of refined sugar, perfectly dry. Dissolve with 75 c.c. of distilled water in a 100 metric c.c. flask. When complete solution has been effected, add 5 c.c. of hydrochloric acid, having a specific gravity of 1.188, mix thoroughly, insert a thermometer in the flask and put the same on a water-bath having a temperature of 70° C. The flask and its contents are kept for five minutes at a temperature ranging between 67° C. and 70° C., the flask being frequently gently shaken during this time. As it may require from two to five minutes to raise the temperature of the flask's contents up to 69° C., the entire operation may take from seven to ten minutes; it must, however, never exceed ten minutes.

When the inversion has been thus achieved, the flask is immediately immersed in water having a temperature of 20° C. The thermometer is then cautiously removed from the solution, any solution adhering to the thermometer is washed back into the flask, its contents are made up to exactly 100 c.c., well mixed, and filtered. Clarification with basic lead acetate is not permissible; if necessary, decolorization must be effected with specially prepared bone-black.

As 9.5 parts of sucrose on inversion yield 10 parts of invert sugar, the solution prepared as above described will contain exactly 10 grams of invert sugar.

50 c.c. of this solution, containing 5.0 grams of invert sugar, are measured into a 1000 c.c. flask, sodium carbonate is added until a piece of red litmus paper put into the solution, begins to turn color. When this occurs the contents of the flask are made up to 1000 c.c. with distilled water, and thoroughly mixed. Each c.c. of this solution contains 0.005 gram of invert sugar.

25 c.c. of Fehling's Solution No. 1, and 25 c.c. of Fehling's Solution No. 2, are, with an accurate pipette, measured into a deep porcelain dish or casserole; to this 50 c.c. of

distilled water are added and the mixture is rapidly raised to the boiling point.

The inverted sugar solution is then run into the Fehling solution from a burette graduated in 1/10 c.c., under constant stirring and renewed boiling, until all of the copper in the copper sulphate solution has been precipitated as cuprous oxide. The operator is warned of the approach of the end of the reaction by the gradual change in the color of the solution; the blue color disappears and the solution turns colorless.

The end-point of this reaction is determined by filtering a few drops of the solution through a minute paper filter into a very dilute solution of potassium ferrocyanide (20 grams in 1000 c.c. water) and acetic acid of 10% strength.

If, owing to the formation of cupric ferrocyanide, a brownish-red color appears, two-tenths c.c. more of the inverted sugar solution are added to the Fehling solution, the same is again boiled, and the test is repeated. These operations are continued until the addition of a few drops of the inverted sugar solution to the ferrocyanide solution no longer produces the red coloration.

The finding should always be confirmed by a duplicate test.

As previously stated, 1 c.c. of the inverted sugar solution used contains 0.005 gram of invert sugar.

Suppose that 49 c.c. of the inverted sugar solution have been used in the test, then to effect the complete reduction of the 50 c.c. of the Fehling solution employed, there have been required: $49.0 \times 0.005 = 0.245$ gram of invert sugar, and this measures the strength of the Fehling solution.

The determination of the reducing sugars in the sample of mixed juices to be analyzed, is carried out precisely as above described for the standardization of the Fehling solution.

Take 20 c.c. of the composite juice sample, place in a 100 c.c. flask, make the volume up to 100 c.c., filter, and run the filtrate from a burette into the Fehling solution.

The amount of reducing sugars is obtained from Table XII.

Glucose Ratio. The reducing sugars found, multiplied by 100, and this product divided by the sucrose, is known as the glucose ratio.

Ash. This determination is made on 10 grams of the mixed juices. Evaporate carefully to dryness, moisten with 20 drops of dilute sulphuric acid (1:1), pour a little ether over the contents of the dish and ignite. This treatment yields a porous carbonized mass, and, in a great measure, avoids danger of loss. When all gases have burned off, place the dish in a muffle, keep the same at a dull-red heat until the sample has been completely ashed, then cool and weigh. Subtract 10% from the weight found and record the result as total ash.

Water. For rapid determinations, take 10 grams of the sample, mix intimately with perfectly dry ground glass or powdered quartz. Heat, preferably in a vacuum stove, at a temperature not to exceed 103° C., to constant weight. Should the last weighing made show a slight gain over the weighing immediately preceding, accept the latter as the true weight, for an increase in weight indicates incipient oxidation.

For very accurate work the following method advocated by Paul Poetschke is recommended. To carry this into effect place 10 grams of dry pure quartz sand and a short stirring rod into a 100 c.c. beaker. Dry thoroughly at 100° C., cool and weigh. Introduce about 5 grams of the sample, mix with the sand, and gradually add, while stirring, 10 c.c. of absolute alcohol. Evaporate to dryness on a water-bath, again add 10 c.c. of the alcohol, and repeat the evaporation to dryness as before. After the second portion of alcohol has been evaporated, place the beaker in an air-bath and dry for 2½ hours at 100° C. Cool in a desiccator and weigh. Repeat the heating at 100° C. for one hour, and again weigh, to learn whether the drying

is completed. Ordinarily 2½ hours will prove sufficient for the operation.

The quartz sand used is prepared in the well-known manner by digesting with strong hydrochloric acid, washing, drying and igniting. The alcohol used must of course be pure, and leave no appreciable residue on evaporation.

Last Mill Juice and Clarified Juices. The determinations to be made are the following:

<i>Brix.</i>	As previously directed.
<i>Sucrose.</i>	“ “
<i>Purity.</i>	“ “

Syrup, is the term assigned to the purified cane juice after concentration. Samples equal in amount are to be taken from every settling tank, and of these a composite sample is made, and analyzed once in each shift. The analytical determinations to be carried out are the following:

<i>Brix.</i>	As previously directed
<i>Alkalinity or acidity.</i>	“ “
<i>Sucrose.</i>	“ “
<i>Purity.</i>	“ “
<i>Reducing Sugars:</i>	“ “
<i>Ash.</i>	“ “

Bagasse, or Megass, is the refuse of the cane after crushing. Its essential constituents are cellulose-fiber and wood gum.

Saturation is the amount of water sprayed upon the bagasse in grinding. The water so used is measured, its temperature is taken, correction for temperature is made to reduce it to its corresponding volume at 4.0° C., at which temperature water attains its maximum density.

	Pounds.
1 cubic ft. of water at 4° C. weighs	62.427
1 “ “ 20° C. “	62.32
1 “ “ 30° C. “	62.16

Percentage Saturation of the cane weight

$$= \frac{62.427 \times \text{cu.ft. of water at } 4^{\circ} \text{ C.} \times 100}{\text{Weight of Canes in pounds}}$$

Small samples of bagasse should be frequently, if not continuously taken, placed in galvanized iron cans, sterilized with a few drops of formaldehyde, and analysis made every hour.

Sucrose. Cut or shred the bagasse as rapidly as possible, as it very quickly changes its moisture content on exposure to air. 50 grams of this comminuted bagasse are boiled with 500 c.c. of water, 8 c.c. of basic lead acetate solution and 10 c.c. of a 5% sodium carbonate solution in a metallic beaker provided with a reflux condenser. After boiling for one hour, cool and weigh beaker with its contents. Filter and polarize filtrate in a 400 m.m. tube.

The method of calculation is illustrated by the following example:

Fiber in bagasse (assumed)	= 45%	
Fiber in bagasse $\frac{50 \times 45}{100}$	= 22.5 grams	
Weight of beaker, bagasse and water	= 750	"
Weight of beaker	= 250.0	"
Weight of bagasse and water	= 500.0	"
Weight of fiber in bagasse	= 22.5	"
Weight of solution	= 477.5	"
Polarization of solution in 400 m.m. tube	= 5.0° Ventzke.	

$$\frac{5.0}{1.1} = 4.54$$

This, according to the Schmitz table = 1.27% sucrose.

This value, divided by two, in order to reduce to a reading in 200 m.m. tube = 0.635% sucrose in the solution.

The weight of this solution = 477.5 grams, hence, there are $\frac{477.5 \times 0.635}{100} = 3.03$ grams sucrose in the bagasse.

As 50 grams of bagasse were used for the analysis, this corresponds to 6.06% sucrose in the bagasse.

Fiber. Fiber is the cellular woody residue of the cane after this has been exhausted with water.

Take 50 or 100 grams of the material and make the determination as previously described.

If desired, a check on the analytical result found may be made by the formulæ:

Per cent Juice Solids in bagasse

$$= \frac{\text{Per cent sucrose in bagasse} \times 100}{\text{Purity coefficient of last mill juice}}$$

Per cent Fiber = 100 - (per cent juice solids in bagasse + per cent water in bagasse)

This calculation involves the assumption that the purity coefficient of the last mill juice and that of the bagasse juice are identical. Example:

Per cent sucrose in bagasse = 5%

Purity coefficient of last mill juice = 72

Per cent juice solids in bagasse = $\frac{5.0 \times 100}{72} = 6.94\%$

Per cent water in bagasse = 44.06%

Per cent fiber = 100 - (6.94 + 44.06) = 49.0%

Water. As previously directed.

Dry Substance in Bagasse. Chop up finely, and as quickly as possible, 50 grams of bagasse. Place in a tared vessel—shallow trays, or tubes made of gauze and provided with gauze net caps, and dry to constant weight at from 95° C. to 105° C. Cool in desiccator over sulphuric acid, and weigh quickly.

The loss in weight found represents the water in the bagasse. The difference between 100 and this value is the dry substance.

Filter-Press Work. *Juice.* The analytical determinations to be made are the following:

Brix. As previously directed.

Alkalinity. “ “

Sucrose. “ “

Purity. “ “

Cake. Samples must be taken from at least three press cakes, preferably five samples should be taken from each cake, one from the center of the cake, and one from each corner. The analytical determinations to be made are the following:

Sucrose. Weigh out 25 grams—to allow for the insoluble matter present. Work up with water into cream, wash into a 100 c.c. flask, add basic acetate of lead solution, from 2 to 4 c.c.—sufficient to clarify—make the solution up to 100 c.c. in volume, filter and polarize the filtrate in a 200 m.m. tube. The polarization found expresses the percentage of sucrose in the filter press cake.

Water. 5 grams are to be used for this determination which is to be carried out as previously directed.

Sugars. All samples must be systematically taken; a thorough mixing of such samples before analysis is of course imperative. The analytical determinations to be made are the following:

SUGAR: *Sucrose.* As previously directed.

Clerget. Weigh out 26.0 grams of the sample, and determine the polarization as usual. Of the solution take 50 c.c. for inversion, or, weigh out 13.0 grams of the sample separately. Dissolve with about 75 c.c. of water in a 100 c.c. flask; after complete solution has been effected add 5 c.c. hydrochloric acid (sp.gr. 1.188), containing 38 per cent HCl. Heat quickly, in two or three minutes, on a water-bath up to between 67° and 70° C. Then keep the temperature of the solution for five minutes as close to 69° C. as possible, while agitating constantly. Cool quickly to 20° C., fill with distilled water up to the 100 c.c. mark, and polarize in a tube provided with an accurate thermometer. The temperature at which the reading is taken should be 20° C., and that of the saccharimeter should also be at 20° C.

The use of subacetate of lead for clarifying purposes is not permissible as this reagent affects the rotatory power

of invert sugar. If a decolorant must be used, specially prepared bone-black should be employed.

The result is calculated as follows:

Let R = Sucrose.

S = Sum of the two polarizations before and after inversion, the minus sign being ignored.

t = Degrees Centigrade at which the polarizations, before and after inversion were observed,

then
$$R = \frac{100 \times S}{142.66 - (0.5 \times t)}$$

The two polarizations—the one before, the other after inversion, must always be made at one and the same temperature, because the optical rotation value of invert sugar is materially influenced by temperature-changes. As the International Commission has accepted 20° C. as the standard temperature, 20° C. is to be used in the above determinations and formula.

If no other optically active body besides the sucrose is present, the Clerget polarization will of course return a value equivalent to the direct polarization value originally found. Example:

Polarization of normal weight before inversion, at 20° C. = 87.5°

Polarization of half normal weight after inversion, at 20° C. = -14.3°

<u>-14.3</u> × 2	87.5	$R = \frac{100 \times 116.1}{142.66 - 10}$
<u>-28.6</u>	<u>28.6</u>	
	<u>116.1</u>	$R = \frac{11610}{132.66} = 87.5$

Reducing Sugars. As previously directed.

Water. " "

Ash. " "

FILL MASS. Fill mass, or massecuite, is the term given to the crystalline magma obtained by boiling sugar solutions in the vacuum pan. Distinctive names, such as first, second, mixed, etc. fill mass, are often assigned to fill masses boiled from specific products.

Thus, the designation first fill mass is often employed to denote the product boiled entirely, or at least in greater part, from syrup. Purged second sugars are often added to the first fill mass and so appear as centrifugal sugars.

By second fill mass there is generally understood the product obtained by boiling a small amount of syrup together with the molasses from a first fill mass.

During the discharge from the vacuum pan several samples of each fill mass should be taken, as strikes do not run uniformly. These various samples should be made up into a composite sample and the analysis made on this. Whenever samples are obtained from crystallizers or mixers, these should be taken only after the mass has undergone a thorough mixing.

The analytical determinations to be made on fill mass samples are the following:

Brix. As previously directed.

Sucrose. " " "

MOLASSES. The more or less impure sugar solutions spun off from the fill mass in the centrifugal machines, are known as molasses; the molasses lowest in purity is termed final or exhausted molasses.

The sampling of molasses, as well as the taking of its weight, must be conducted with great care, as molasses will readily imprison air, and this, of course, tends to reduce the weight of the molasses per unit volume to a considerable degree.

Brix. To 250 grams of the sample add 250 grams of water, and bring into perfect solution, however, without the aid of heat. Determine the degrees Brix of this solution, multiply by two, and then only correct for tempera-

ture, because the coefficient of expansion of the non-sugars in cane-sugar molasses varies from that of sucrose for which the tables were calculated, but which difference is allowed for by proceeding with the calculation as above directed.*

Sucrose. Weigh out the normal weight of the above solution, place in a 100 c.c. flask, clarify with basic lead acetate solution, using the smallest amount possible to effect the decolorization, bring the volume up to 100 c.c., filter and polarize. If the solution is sufficiently light in color use the 200 m.m. tube and multiply the reading found by two; if the 100 m.m. tube must be employed, the reading observed on the polariscope must be multiplied by four, and the resulting value recorded as sucrose.

Purity. Having determined the polarization of the normal weight, and the degree Brix of the sample corrected for temperature, use the formula:

$$\text{Purity} = \frac{\text{Polarization} \times 100}{\text{Degrees Brix}}$$

Example:

Polarization	=	40.0
Corrected Brix	=	51.0
Purity	=	$\frac{40.0 \times 100}{51.0} = 78.4$

Clerget. The direct polarization and the polarization after inversion should be carried out on portions of one and the same solution; for this reason two or three times the normal weight of the molasses should be dissolved in 500 c.c. of water. The determination is then carried out as previously directed.

Waters. Condenser Water. The term condenser water, or circulation water, is applied to the water which serves to condense the vapors coming from the vacuum concentration apparatus.

* Prinsen Geerligs, *Methods of Chemical Control in Cane Sugar Factories*, Altrincham, 1905, p. 15.

Samples of this water should be frequently and systematically, if possible automatically, taken from the tail pipes and condensers of all vacuum concentration apparatus.

The analytical determinations called for are the following:

Brix. As previously directed.

Sucrose. As the amount of sucrose which may normally be expected in condenser waters is very small, a delicate test for its presence must be employed.

Such a test is afforded by the alpha-naphthol reaction of Molisch, whose reagent consists of a 20% alcoholic solution of alpha-naphthol. 2 c.c. of the water which is to be tested for the presence of sucrose, are introduced into a test-tube, and five drops of the Molisch reagent are added. Then, 10 c.c. of chemically pure concentrated sulphuric acid are, by means of a pipette, allowed to flow to the bottom of the test-tube. This test, as has been said before, is a very delicate one. If any appreciable quantity of sucrose is present, a zone of color appears at the line of contact of the two fluids. If the contents of the test-tube are thoroughly mixed by shaking, the solution will show a pale violet coloration, even if only 0.001% of sucrose is present; 0.01% of sucrose gives a bright claret color, and, if as much as 0.1% of sucrose is present, the entire solution darkens and turns opaque.

Nitric acid interferes with this reaction, hence, both the sulphuric acid and the aqueous solution to be tested must be entirely free from nitric acid; ammonia, lime salts and most organic impurities to be found in water, do not interfere with this reaction.

If the alpha-naphthol reaction indicates any appreciable amount of sucrose in a condenser, or other waste water, the polarimetric determination of the sucrose must, in every instance, be made.

Such polarimetric observation is carried out in the following manner.

The zero point of the polariscope is determined with the utmost care; for this purpose it is best to employ the 400 m.m. tube filled with perfectly pure distilled water. When the zero point has thus been accurately determined and fixed, the water to be examined is put into the 400 m.m. tube, and a careful set of readings taken. In interpreting such readings the limit of sensibility of the polariscope employed must of course be carefully taken into account.

Purity. As previously directed.

Waste Waters. A continuous, preferably an automatic sampling of all waste waters is of the utmost importance in order to detect and to guard against excessive and often wholly unsuspected loss of sucrose through leaks and overflows. The analytical determinations to be made are:

Brix. As previously directed.

Sucrose. Alpha-naphthol test as previously directed; polarimetric test as directed.

Boiler-Feed Water. The danger of having sugar in the boiler-feed water, apart from the loss of sucrose itself, is so well understood, that it need not be here specifically referred to. As a safeguard, frequent sampling of the boiler-feed water is necessary. The analytical determinations to be carried out are as follows:

Alkalinity. A sudden drop in alkalinity indicates contamination by sugar.

Sucrose. Alpha-naphthol test as previously directed; polarimetric test as previously directed.

Calculations. While direct measurements and determinations are always preferable to values calculated indirectly from analytical or other data, the use of certain calculations is unavoidable and must be made as a matter of daily record.

To guard against misunderstanding a few explanatory words concerning the terms as commonly used and understood are given in connection with the formulæ by which

the values referred to are calculated. For the summation of these the writer is especially indebted to the work of Prinsen Geerligs.

Cane Formulæ. *Sucrose in 100 Cane.* As a correct sampling of cane is not possible, owing to its nature, the amount of sucrose in it is determined by aid of a factor by which the desired value is calculated from the amount of sucrose in the first mill juice. This factor varies for different fiber and different extraction, it ranges from about 0.84 to 0.86, but is usually taken as 0.85.

(1) Sucrose in 100 Cane = Sucrose in 100 parts of first mill juice \times factor. This formula, however, merely affords an approximation. The actual determination of the factor to be used can be effected by crushing a known weight of cane in the mills, analyzing the first mill juice, obtaining the weight of the bagasse, and determining the amount of sucrose it has retained.

Sucrose Extracted in 100 Cane. This corresponds to the sucrose in juice in 100 cane.

(2) Sucrose extracted in 100 cane = Sucrose in 100 parts of cane - sucrose in 100 parts bagasse. This value is of course also only an approximation value, as it embodies the approximate value obtained by Formula (1).

Weight of Available Sugar. This represents the estimated yield of sugar based on the analytical data.

(3) Weight of available sugar = Weight of sucrose entered in juice $\times \left(1.4 - \frac{40}{\text{Purity}}\right)^*$

(4) Available sugar in 100 cane = Sucrose extracted in 100 cane $\times \left(1.4 - \frac{40}{\text{Purity}}\right)$

* For derivation of this formula and for values of the factor $1.4 - \frac{40}{\text{purity}}$ for various degrees of purity between 77 and 93, confer Prinsen Geerligs, *Methods of Chemical Control in Cane-Sugar Factories*, Altrincham, 1905.

Calculated Weight of Cane Crushed. If all the cane delivered to the factory per day is crushed the same day, this cane delivery weight of course, represents also the weight of the cane crushed. Frequently, however, this is not the case, and then the value sought is found—approximately only, however,—by the formula:

$$(5) \text{ Weight of cane crushed} \\ = \frac{\text{Weight of sucrose entered in juice}}{\text{Sucrose extracted in 100 cane}} \times 100$$

Juice Formulæ. *Weight of Sucrose Entered in Juice.* This value is the fundamental factor of control in the working of a factory, and no care must be spared to get it as accurate as possible, whether the juice be measured or weighed.

$$(6) \text{ Weight of Sucrose entered in juice} = \text{Weight of mixed juice} \times \frac{\text{Sucrose in 100 parts mixed juice}}{100}$$

Sucrose in Normal Juice. Normal juice is the juice as it actually occurs in the cane and would be exemplified by juice extracted by all of the mills without any maceration. In practice this can not be obtained.

In some factories the juice from the first mill is considered as normal juice. This, however, is improper, for the juices from all the other mills are inferior in quality to first mill juice. It has therefore become customary to make certain assumptions, and to make the following calculations.

$$(7) \text{ Sucrose in normal juice} \\ = \frac{\text{Brix of first mill juice} \times \text{Purity of mixed juice}}{100}$$

Extraction. This represents the ratio between the sucrose extracted by the mills, and the total amount of sucrose in the cane.

$$(8) \text{ Extraction} = \frac{\text{Sucrose extracted in 100 cane}}{\text{Sucrose in 100 parts of cane}} \times 100$$

If, for instance, there are known the:

Percentage of sucrose in cane	=	13.00%
“ “ “ bagasse	=	4.30%
“ fiber “ cane	=	12.00%
“ “ “ bagasse	=	45.00%

$$\text{then the Bagasse per 100 cane} = \frac{12.0 \times 100}{45} = 24.45$$

$$\text{and the Sucrose in bagasse per 100 cane} = \frac{24.45 \times 45}{12 \times 100} = 0.92$$

$$\begin{array}{r} \text{Sucrose in juice per 100 cane} = 13.00 \\ - 0.92 \\ \hline 12.08 \end{array}$$

$$\text{Extraction} = \frac{12.08 \times 100}{13.0} = 92.92\%$$

J. H. Morse calls attention to the fact that the term extraction is used in a different sense in various localities. Thus, in Cuba, extraction indicates the yield of sugar; in Louisiana it signifies the per cent of juice by weight extracted from the cane; in the Hawaiian Islands this term is given to the sucrose in the juice compared with the sucrose in the cane.*

Maceration Dilution. Maceration, saturation, inhibition, signifies the water added to the bagasse on the mills; dilution means the amount of water which is added to 100 parts of normal juice. It is figured by the formula

(9) Maceration dilution

$$= 100 \times \left\{ \frac{\text{Brix of first mill juice}}{\text{Brix of mixed juice}} - 1 \right\}$$

* Calculations used in Cane-Sugar Factories, 1904.

Percentage of Normal Juice Extracted from 100 Cane. This value is only approximate; it is calculated by the formula:

$$(10) \text{ Percentage of normal juice extracted from 100 cane} \\ = \frac{\text{Sucrose extracted in 100 cane}}{\text{Sucrose in 100 parts of first mill juice}} \times 100$$

Weight of Sucrose Returned to Juice. When sugars are worked over in the factory it is necessary to redetermine their weight and analysis.

$$(11) \text{ Weight of returned sucrose} = \text{Weight of returned sugar} \times \frac{\text{Sucrose in 100 returned sugar}}{100}$$

Sucrose-loss Formulæ. *Sucrose Lost in Bagasse on 100 Cane.* Of approximate value only, as it is assumed that the juice is entirely free from fiber. Calculated by the formulæ:

$$(12) \text{ Sucrose lost in bagasse in 100 cane} = \text{Sucrose in 100 parts of bagasse} \left(\frac{\text{Fiber on 100 parts of cane}}{\text{Fiber in 100 parts of bagasse}} \right)$$

$$(13) \text{ Weight of sucrose lost in bagasse} = \text{Weight of cane crushed} \times \frac{\text{Sucrose lost in bagasse on 100 parts of cane}}{100}$$

$$(14) \text{ Weight of sucrose lost in filter-press cakes} = \text{Weight of press cakes} \times \frac{\text{Sucrose in 100 cakes}}{100}$$

CHAPTER X

ANALYTICAL CONTROL IN BEET-SUGAR MANUFACTURE

Determinations Required. The analytical determinations to be made in the systematic control of beet-sugar houses are as follows; the number of samples to be taken for analysis must of course be governed by local conditions.

Sample.	Determinations to be Made.	Number of Determinations in 24 Hours.
I. Beets	Polarization	1
<i>a. Fresh cosettes</i>	Polarization Invert sugar Dry substance	8
<i>b. Exhausted cosettes</i>	Polarization Dry substance	24
<i>c. Dry cosettes</i>	Polarization	4
II. Diffusion and press-juices	Brix Polarization Invert sugar Acidity Coagulation value	24
III. Diffusion waters	Brix Polarization Invert sugar Acidity Coagulation value	8
IV. Thin juices, non-saturated and saturated	Brix Polarization Color Alkalinity	Non-saturated juice 24 Saturated juice 24 Continuous saturation 48
V. Thick juices	Brix Polarization Invert sugar Dry substance Color Alkalinity	48

Sample.	Determinations to be Made.	Number of Determinations in 24 Hours.
VI. Press cakes	Polarization Alkalinity	3
VII. Fill mass <i>a. First Products</i>	Polarization Invert sugar Ash Lime Alkalinity Water (dry substance)	Each strike
<i>b. After products</i>	Brix Polarization Invert sugar Ash Lime Color Alkalinity Water (dry substance)	Each strike
VIII. Raw sugars <i>First products and after products</i>	Polarization Invert sugar Water Ash Alkalinity	Each lot
IX. Molasses	Brix Polarization Apparent purity Invert sugar Water Ash Water	Take 2 samples daily and make a composite sample for each week
X. Cattle food	Ash Crude protein Crude fiber Ether extract Sucrose	
XI. Waters <i>a. Condenser water</i>	Invert sugar α -naphthol test Polarization	4
<i>b. Waste water</i>	α -naphthol test Polarization	4
<i>c. Boiler feed water</i>	α -naphthol test Polarization	4

In the preparation of this chapter free use has been made of the directions issued by the Verein der Deutschen

Zuckerindustrie. For permission to do this the writer's cordial acknowledgements are due Professor Dr. Alexander Herzfeld, Berlin.

Sugar-Beets. It is extremely difficult to get a representative sample of the beets as delivered at a factory. If such is demanded, it is perhaps most convenient to take the beets on which the mud has been determined, select say every tenth beet of the lot, scour well, cut off the top and roots and then cut segments from the beet and grind to a pulp.

A far better average sample can however be obtained from fresh beet-cosettes. These are taken at short intervals of time, or, better still, continuously—placed in a closed vessel, and, after being thoroughly mixed, submitted to analysis at least every three hours.

The sample must be reduced to a very fine pulp before analysis and this is done by means of rasps, files, slicers, grinders, etc., of which a number of different kinds are available.

FRESH COSETTES. *Polarization.* Employ the method of hot aqueous digestion. Pass about 1 kilogram several times through slicing machine, weigh out 26.0 grams of this pulp on a small tin scoop, place latter with its contents into a dry metallic beaker provided with a cap, pour into this beaker 177 c.c. basic lead acetate solution (25 c.c. basic lead acetate per liter), close beaker, shake well, heat 30 minutes at 75°–80° C., cool, again shake thoroughly, filter and polarize.

Invert Sugar. 20 c.c. of the above solution are boiled for 2 minutes with $\frac{1}{2}$ c.c. Fehling's solution. If the filtrate remains blue, or if after deleading (cold) with a few drops of a bicarbonate of soda solution and subsequent filtration, the copper reaction with ferrocyanide of potassium and acetic acid is obtained, then abnormal amounts of invert sugar are not present.

Dry Substance. Weigh 20 to 25 grams of the pulp, well mixed, into a flat dish having a diameter of about

three inches. Spread well over the dish by means of a glass rod which has also been weighed in with the pulp. Dry for 2 hours at 70° C. Then again mix thoroughly, spread well over the dish and dry in vacuum at 105°–110° C. for approximately 8 hours, to constant weight.

EXHAUSTED COSETTES. *Polarization.* 60 grams of the pulp are weighed out, placed into a metallic beaker, digested with 177 c.c. basic lead acetate solution, as above directed for beet cosettes. Take the polarization reading in a 200 m.m. tube and read the percentage direct.

Dry Substance. This determination is effected as described above—the preliminary drying at 70° C. is however omitted.

DRY COSETTES. *Polarization.* Grind the dry cosettes to a fine powder. After a preliminary hot aqueous digestion take 12.6 grams of the dry powder and make up, with basic lead acetate water (100 c.c. basic lead acetate to 1 liter water) to 200 c.c., filter, polarize, and multiply reading observed by 4.

Diffusion and Press Juices. *Brix.* As customary. [In Germany the use of the table $\frac{20}{4}$ ° C. prepared by the Imperial Normal Standards Commission is prescribed.]

Polarization. Weigh out 26.0 grams into a 100 c.c. flask, clarify with basic lead acetate, make up to 100 c.c. filter and polarize. Or,

Place 100 c.c. of the juice into a 100–110 c.c. flask, add 10 c.c. basic lead acetate solution, shake well, allow to stand for 10 minutes, filter, polarize in a 200 m.m. tube and determine the percentage of sucrose from a Schmitz table.

Invert Sugar. 10 c.c. of the juice to be tested are boiled with 2 c.c. Fehling's solution for 2 minutes. If the solution still remains blue the juice contains less than 0.1% invert sugar.

If however the Fehling's solution is completely used up, then for every 10 c.c. juice, 2, 3, 4 etc., c.c. Fehling's solu-

tion are tried, in each instance boiling for 2 minutes, until the point is reached where copper is still present in the filtrate.

1 c.c. Fehling's solution = 0.005 gram invert sugar.

Acidity. 25 c.c. juice are diluted with sufficient phenol-phthaleïn-neutral water* so that the change in color is perfectly distinct, and are then titrated with 1/28 normal sodium hydrate solution. The CaO percentage is found by multiplying by 4.

Coagulation-Value. Place 25 c.c. of the raw-juice into a graduated cylinder about $\frac{3}{4}$ inch in diameter, add 3 drops of glacial acetic acid, heat for 3 minutes in a water-bath at 80°-85°C., remove, allow to stand at rest at room-temperature for 3 hours, read off the volume of the precipitate in cubic centimeters and multiply by 4.

Diffusion-Waters. Brix. Free from air and determine as usual by Brix hydrometer or by means of a pycnometer.

Polarization. Place 100 c.c. in a 100-110 c.c. flask, clarify with 5-8 c.c. basic lead acetate solution, fill up to 110 c.c., shake well, allow to stand for a few minutes, filter and polarize.

Invert Sugar. As previously directed.

Acidity. Allow the waters to settle by standing for about 5 minutes and then take the sample, 25 c.c., from the upper part of the vessel by means of a graduated pipette. Add sufficient phenol-phthaleïn-water until the change in color can be distinctly seen and then titrate with sodium hydrate solution of known strength.

Coagulation-Value. As previously directed.

* This is prepared by adding to a larger amount of freshly boiled distilled water $\frac{1}{2000}$ of its volume of a phenol-phthaleïn solution (1 part by weight of phenol phthaleïn dissolved in 30 parts by weight of 90% alcohol) and then making alkaline with sodium hydroxide solution until a distinct red coloration ensues. This solution is to be freshly prepared every few days, but must be some hours old before use.

Thin Juices: *non-saturated and saturated.*

Brix. As previously directed.

Polarization. As directed under diffusion juices. The juices of the first saturation are to be neutralized with acetic acid and phenolphthaleïn.

Color. Determine the color of the solution on which the Brix determination has been made, by Stammer's colorimeter.

Alkalinity. To 10 c.c. of the juice there is added sufficient phenol-phthaleïn-neutral water until the change in color is distinctly perceptible.

Thick Juices. *Brix.* As previously directed.

Polarization. Take 26.0 grams, neutralize with acetic acid and phenol phthaleïn, clarify with basic lead acetate make up to 100 c.c., filter and polarize.

Invert Sugar. If the sample has an acid reaction or an abnormal odor, dilute with water to about 10° Brix, add sodium hydroxide solution, shake well and boil a portion of the mixture. If a distinct brown coloration ensues, take 12.0 grams and determine in this the invert sugar quantitatively, as in raw sugars (which see).

Dry Substance. On 50 grams of pure sand (free from iron) weigh 3 grams of thick juice. Dry in vacuo at 105°-110° C. to constant weight.

Color. As directed under thin juices.

Alkalinity. To 10 grams of thick juice add sufficient phenol-phthaleïn-neutral water to make the color-change distinct, then titrate.

Press-Cakes. *Polarization.** Weigh out 53.0 grams of cold press-cake, place in a cold mortar and grind up with 177 c.c. ammonium nitrate solution containing approximately 10% neutral ammonium nitrate; this is added, gradually, from a burette. Do not add basic lead acetate. Filter. Polarize the filtrate in a 200 m.m. tube; the reading so found represents the sucrose percentage direct.

* Method of the German Sugar Institute.

Alkalinity 10 c.c. of the filtrate are titrated with acid (1 c.c. = 0.01 gram CaO) using rosolic acid as indicator. The number of c.c. of acid used, multiplied by 0.38 represents the percentage of CaO in the press-cake.

In case difficulties arise in the factory samples of the press-cakes from the second saturation are to be extracted with ether, the latter is to be evaporated and the residue examined for fats or oils; furthermore, after boiling with bicarbonate of soda the filtrate should be tested for oxalic acid. Magnesia may also have to be looked for. This is done by suspending a sample of the press-cake in water, oversaturating with carbonic acid gas, boiling the filtrate, dissolving the precipitate thus formed in hydrochloric acid, boiling with calcium carbonate and testing the filtrate with clear lime water for magnesia.

Fill Mass. **FIRST PRODUCTS.** *Polarization.* Weigh out 26.0 grams into a 100 c.c. flask. Cool to normal temperature, i.e., 20° C., add 3 to 4 c.c. basic lead acetate solution, make up to 100 c.c., shake well, filter, polarize.

Invert Sugar. As previously directed under invert sugar.

Ash. Weigh 3 grams into a platinum dish of about 60–80 c.c. capacity, carefully moisten with about 2 c.c. concentrated sulphuric acid, heat carefully at first, then char and finally incinerate in a muffle.

Lime. Test qualitatively for lime (CaO) with ammonium oxalate, or, if occasion arises, quantitatively with soap solution, or by one of the usual gravimetric methods.

Color. As previously directed.

Alkalinity. Dissolve 10 grams in phenol-phthaleïn-neutral water and titrate with acid of known strength.

Water (dry substance). Mix intimately from 2 to 3 grams of the fill mass with 50 grams of pure sand, free from iron. Dry at 70° C. for 15 or 20 minutes. Mix again most intimately and then dry for from 6 to 8 hours at 105°–110° C. in vacuo to constant weight. The latter point is assumed

to have been reached when the difference between two successive weighings, two hours apart, is less than 0.1%.

AFTER PRODUCTS. *Brix and Polarization.* Into a beaker in which there is a glass rod and both of which have been tared, weigh about 250 grams of the well mixed sample, and under constant stirring gradually add about 250 grams of warm water until all is dissolved. The solution is then cooled and made up to exactly 500 grams with water. The degrees Brix of this solution are then determined as usual and multiplied by 2. The polarization is found by weighing out twice the normal weight, i.e., 52 grams of the solution, adding 5 c.c. of basic lead acetate solution, making up to 100 c.c., filtering and polarizing.

Invert sugar, as previously directed.

Ash, “ “

Lime, “ “

Color, “ “

Alkalinity. 5. or 10. grams of the sample—according to the color of the sample—are dissolved in sufficient phenolphthaleïn-neutral water that the change in color is distinctly visible and then titrated with acid solution of known strength.

Water (dry substance). Weigh 2 to 3 grams of the sample in a dish containing 50 grams of sand, free from iron. Moisten with a few drops of warm water, mix well with the sand. Dry for 2 hours at 70° C., then, in vacuo, dry at 105°–110° C. to constant weight. This will require from 6 to 8 hours. The end-point is reached when there is only about 0.1% difference in the weight of the sample after a two hours' interval of drying.

Raw Sugar. *First Products and After Products.* *Preparation of the Sample.* The containers holding the samples must not be opened until they have acquired the temperature of the laboratory. The samples are then quickly, but thoroughly mixed and replaced in the containers and the analysis at once started.

Polarization. Weigh out 26.0 grams of the sample and place into a 100 c.c. flask, graduated in metric (true) cubic centimeters at 20° C., that is to say, these flasks must contain 99.71 grams (± 0.03 gram) of water at 20° C., weighed in air with brass weights. Basic lead acetate solution * is then carefully added until an additional drop of this reagent produces no further precipitation.

One or two c.c. of alumina cream are also added. The contents of the flask are then brought up to almost the 100 c.c. mark, any foam or bubbles are dispersed with a little ether vapor. The flask is next placed into a water-bath kept at 20° C. for about one half hour. Then it is removed and filled exactly to the 100 c.c. mark, any drops of water adhering to the neck being carefully removed with filter-paper. The contents are then thoroughly mixed and filtered through a dry filter into a receiving flask or beaker which is kept covered to guard against evaporation. The first few drops, which as a rule are turbid, are rejected and only the perfectly clear filtrate is used. The polarization is carried out in a 200 m.m. tube at 20° C. in a saccharimeter which has been graduated at 20° C. and which has been kept at 20° C. for at least 2 hours before the polarization readings are taken. Accuracy of the saccharimeter must be controlled by use of standard quartz plates mounted free from pressure.

* This solution is prepared as follows:

- 3 parts by weight of basic lead acetate,
- 1 part of litharge,
- 10 parts of water.

The two salts are intimately ground together and, after addition of one half part of water, are heated in a closed vessel on a water-bath until the mixture has turned white or a reddish white. Then the balance of the water is gradually added, and when the mass has been entirely or almost entirely dissolved to a turbid solution, the latter is set aside in a closed vessel to settle and is finally filtered.

Basic lead acetate solution must react alkaline to litmus, but must not redden phenol phthalein. Its specific gravity must be between 1.235 and 1.240.

The saccharimeter must be illumined by white light (petroleum, Welsbach or electric light) and the light rays filtered through a column 1.5 cm. long of a 6% potassium dichromate solution.

Invert Sugar. For the gravimetric determination of invert sugar use is made of Fehling's solution. This consists as previously described of two solutions kept in separate flasks and mixed only immediately before use.

Solution I. 34.639 grams pure recrystallized copper sulphate are dissolved in water and the solution is made up to 500 c.c.

Solution II. 173 grams pure crystallized Rochelle salts are dissolved in distilled water. To this solution are added 100 c.c. of a 50% purest sodium hydrate solution and the total volume of the solution is brought up to 500 c.c.

To make an invert sugar determination, 25 c.c. of Solution I and 25 c.c. of Solution II are mixed in a flask or in a porcelain dish of about 250 c.c. capacity. To this mixed solution are added 38.4 c.c. of the sugar solution used for the polariscopic determination (corresponding to 10 grams of raw sugar) and about 12 c.c. pure distilled water are added. The contents of the flask are well mingled by shaking and the flask or porcelain dish is then placed on some wire gauze on which has been placed a piece of asbestos sheeting having cut in it a circular opening $2\frac{1}{2}$ inches in diameter. A flame is placed under the flask or dish and its contents heated to boiling in about 3 minutes' time. As soon as active boiling has commenced the solution is kept boiling for 2 minutes; the flask or dish is then removed and 100 c.c. cold distilled water—from which the air has previously been removed by boiling, is poured into the same. The solution is then quickly filtered through a layer of asbestos-fiber contained in a glass tube, the precipitate of cuprous oxide is washed thoroughly with about 300 to 400 c.c. of hot water and finally with alcohol

and ether; it is then dried perfectly. The copper can be weighed as cuprous oxide, as cupric oxide into which it is transformed by heating while passing air through it, or, it can be reduced to metallic copper by means of a current of hydrogen gas and weighed as metallic copper.

The calculation of the invert sugar corresponding to the copper found is made by aid of Herzfeld's table.

Water. 5. grams of the sugar are dried at 110° C. in vacuo for from 2 to 3 hours until approximate constancy of weight has been obtained.

Ash. 3. grams of the sample are treated with concentrated sulphuric acid and ignited in a muffle as previously described.

Alkalinity. Weigh out 10.0 grams of the raw sugar. Measure out 100 c.c. of the phenol-phthaleïn-neutral water into a white porcelain dish and make the same as nearly colorless as possible by means of a solution of test acid, which is prepared by diluting 36 c.c. normal sulphuric acid with water to a volume of 10 liters. 1 c.c. of this test acid corresponds to a lime alkalinity of 0.0001.

Then add sufficient of the test sodium hydrate solution until the fluid has again turned a faint red. This test sodium hydrate solution is standardized against the test acid solution above referred to and hence 1 c.c. of the same also corresponds to a lime alkalinity of 0.0001.

The red coloration obtained must however be only strong enough that it will again completely disappear on the addition of one cubic centimeter of the test acid solution, immediately before the addition of the raw sugar solution.

The 10 grams of the raw sugar are then at once dissolved in this solution. If the red coloration of the water remains on dissolving the sugar in the same, or, if it increases in depth the sugar is alkaline, if the color disappears, the sugar is acid.

In case of doubt, one must determine by titration with

the test acid as well as with the test alkali solution in which direction the change of color occurs.

If the sugars tested are so dark that the use of 100 c.c. of water of solution are not sufficient to produce a sugar solution sufficiently light in color to permit of the titration, more water must be used, this however is permissible only when 100 c.c. will not suffice for the purpose.

Sugars which react neutral under this method of procedure are classed as alkaline.

Molasses. *Brix.* To 250 grams of the sample add 250 grams of water and effect perfect solution without aid of heat. Determine the degree Brix of this solution and then correct for temperature.

Polarization. Use 26.0 grams and proceed as usual.

Apparent Purity. Having determined the corrected degree Brix of the solution and its polarization calculate the purity by the formula:

$$\text{Apparent Purity} = \frac{\text{Polarization} \times 100}{\text{Degrees Brix}}$$

Invert Sugar. As previously described.

Water. Weigh out 10 grams of the sample, add 10 grams of absolute alcohol. Mix intimately with 50 grams of pure sand. Evaporate to dryness on a water-bath, add 10 grams more of absolute alcohol and repeat evaporation to dryness on a water-bath. Then place the sample in an air-bath or in a vacuum stove and dry for 2½ hours at 100° C. Cool in a desiccator and weigh. Repeat this heating for an additional hour at 100° C. and reweigh. Carry to constant weight.

Ash. Take 10 grams of the sample moisten with 20 drops of dilute sulphuric acid (1 : 1) pour a little ether over the contents of the dish and ignite *cautiously*. When mass is fully carbonized, place in a muffle and incinerate. Deduct 10% from the weight found.

Cattle Food. *Water.* Dry 5.0 grams at 100° C. for 12 hours. Cool, weigh. Continue drying and reweighing at intervals of one hour until practically constant weight has been attained.

Ash. Use 2.0 grams, moisten with sulphuric acid, heat carefully, then incinerate, cool and weigh.

Crude Protein. Make a Kjeldahl determination as directed in Chapter VII. The percentage found multiplied by the factor 6.25 represents the crude protein.

Crude Fiber. Use 2.0 grams for the determination. Extract with ether. Place the residue in a 500 c.c. flask and add 200 c.c. boiling sulphuric acid having a strength of 1.25 %. Boil for one half hour having the flask connected with a reflux condenser. Filter, wash residue with boiling water until free from acid.

Wash the material back into original flask with 200 c.c. boiling solution of sodium hydrate having a strength of 1.25%. Again boil for one half hour. Place residue in a tared Gooch crucible and wash to neutral reaction with boiling water. Dry residue at 110° C. Weigh, ignite and burn completely to ash. Then reweigh. The loss of weight found represents the crude fiber.

Ether Extract. Extract about 3.0 grams with anhydrous ether free from alcohol in a Soxhlet tube on a steam bath or on an electric plate. Run this extraction for 16 hours, remove the ether by evaporation and dry extract to constant weight at about 100° C. The residue, expressed in percentage on the original material used, is recorded as the ether-extract.

Sucrose. Invert and determine as invert sugar by Fehling's solution, as previously directed.

Invert Sugar. Determine by Fehling's solution, as previously directed.

Waters: Condenser, Waste and Boiler-Feed Waters.
 α -naphthol Test. Place 5 c.c. of the water to be examined into a test-tube, add 2 or 3 drops of a 10% solution of

α -naphthol. Then pour a few cubic centimeters of concentrated sulphuric acid—free from any trace of nitric acid—carefully along the sides of the tube so that it will settle at the bottom. If even very small amounts of sucrose are present a violet zone will appear at the line of contact of the two liquids. This test is a qualitative one, although some indication of the amount of sucrose present can be had from the intensity of the coloration. If any appreciable amount of sucrose is shown, a quantitative determination by the polariscope must be made.

Polarization. Evaporate about 4 liters of the sample in a dish, wash the residue into a 200 c.c. flask and add a few drops of phenol-phthaleïn solution. Decolorize with acetic acid, then clarify with basic lead acetate solution. When perfectly cold, shake well, filter and polarize in a 400 m.m. tube. Calculate the result on the original amount of water taken.

CHAPTER XI

ANALYTICAL CONTROL IN REFINERIES

Determinations Required. The following determinations are necessary for the current control of a sugar refinery; the number of samples taken for analysis are of course determined by local conditions.

Sample.	Determinations to be Made.	Number of Determinations in 24 Hours.
Raw sugar purchased	{ Polarization Invert sugar Water Ash }	Every lot
Raw sugar stacked	{ Polarization Invert sugar Water Ash }	Every lot
Raw sugar melted	{ Polarization Invert sugar Water Ash }	Every lot
Washed sugar	{ Polarization Invert sugar }	12
Remelt sugar	{ Polarization Invert sugar }	12
Car-sugar	{ Polarization Invert sugar Water Ash }	2
Refined sugar	{ Polarization Brix }	Every lot
Fill mass	{ Polarization }	Every strike

Sample.	Determinations to be Made.	Number of Determinations in 24 Hours.
Pan Specials Fill mass	Brix Polarization	Whenever taken
Blow-ups	Brix Polarization	12
Liquors, First, second, third, fourth	Brix Polarization Invert sugar	Hourly samples to be taken 12 composite samples of each grade
Washed sugar liquors	Brix Polarization Invert sugar	Hourly samples to be taken. 12 composite samples of each grade
Char-sweet water	Brix Polarization Invert sugar	Hourly samples to be taken. 8 composite samples.
Concentrated sweet-water	Brix Polarization Invert sugar	Hourly samples to be taken. 8 composite samples.
Bag filter washings	Brix Polarization Invert sugar	6
Pan syrups	Brix Polarization Purity	24
Cyclone syrups	Brix Polarization Purity	2
Barrel syrups	Brix Polarization Purity Clerget Invert sugar	Every lot
Molasses	Ash Brix Polarization Purity Clerget Invert sugar Ash	Every lot
Press-cakes	Polarization Water	3 composite samples
Press-water On:	Brix Polarization Purity	Hourly samples to be taken. 4 composite samples

Sample.	Determinations to be Made.	Number of Determinations in 24 Hours.
Press-water Off:	{ Brix Polarization Purity }	Hourly samples to be taken. 4 composite samples
Press-cloths water	{ Brix Polarization Purity }	3
Bag filter water	{ Brix Polarization Purity }	2
Bags Sheathes }	{ Brix Polarization Purity }	2
Condenser water	{ α naphthol Polarization when indicated }	Continuous automatic sample to be taken. 8
Waste water	{ α -naphthol Polarization when indicated }	Continuous automatic sample to be taken. 8
Boiler-feed water	{ Alkalinity α -naphthol Polarization when indicated }	Continuous automatic sample to be taken. 8
Phosphoric acid or phosphoric acid paste	Free { P_2O_5 hydrochloric or sulphuric acid }	3
Bone-black from kilns	Caustic soda test Density	Every kiln. 6
Hydrochloric acid	{ Iron Arsenic }	Every lot

The analytical determinations to be made as above indicated have been so fully considered in previous chapters that reference shall here be made only to such determinations as have not before received special attention.

Density Determination of Fill Mass. *Apparent Density.* Weigh out 250 grams of the fill mass, add 250 grams of water, carefully dissolve all crystals and mix the sample thoroughly. Determine the degree Brix of this solution, correct for temperature and multiply the corrected value by 2.

Noël Deerr * determines the apparent and the real specific gravity of fill masses in the following way.

* Cane Sugar, 1911, p. 483.

The fill mass to be tested is allowed to flow into a large wide-mouthed vessel of metal or glass the mouth of which slopes inward and which is provided with an accurately fitting stopper. This vessel is filled to about seven-eighths of its capacity. Vessel and contents are then cooled to the temperature at which the factory measurements of the fill mass are taken and the weight of the fill mass in the vessel ascertained.

If the apparent specific gravity of the fill mass is to be determined the vessel is next completely filled with water and the perforated stopper is inserted; any excess of water passes through the hole in the stopper and is removed.

Real Density. If the real specific gravity is desired, oil or any other fluid indifferent to sugar is used in place of the water, the specific gravity of such reagent has of course been previously determined.

Example.

	Grams.
Vessel, stopper, water	2163.40
Vessel, stopper—empty	416.35
	<hr/>
Water	1747.05
	<hr/>
Vessel, stopper, fill mass	2645.95
Vessel, stopper—empty	416.35
	<hr/>
Fill mass	2229.60
	<hr/>
Vessel, stopper, water, fill mass	2875.95
Vessel, stopper, fill mass	2645.95
	<hr/>
Water above fill mass	230.00
	<hr/>
Water	1747.05
Water above fill mass	230.00
	<hr/>
Water in space occupied by fill mass	1517.05
	<hr/>

$$\text{Apparent specific gravity of fill mass} = \frac{2229.60}{1517.05} = 1.469.$$

Whenever the real specific gravity is sought and an oil, for instance, is used, the calculation is effected as follows:

Example.

$$\frac{\text{Weight of fill mass}}{\text{Weight of an equal volume of oil}} = x.$$

Specific gravity of the oil referred to water = 0.862

True sp.gr. of fill mass = 0.862 x .

Composition of Fill Mass. A knowledge of the composition of the fill mass is a factor of prime importance in a sugar house.

The direct determination of the crystal content and of the syrup in a fill mass is analytically carried out by Sidersky * as follows:

Make an ash determination of the fill mass as usual by the sulphuric acid method. Then place some of the fill mass—if need be, after having slightly warmed it, upon a fine wire gauze and allow the syrup to drain from it. On this syrup make another ash determination, this, of course, represents the ash of the syrup originally adhering to the sucrose-crystals in the fill mass. As sucrose crystals are free from mineral matter all of the ash must be present in the syrup.

The amount of the adherent syrup is then calculated as follows:

Example. Ash found in fill mass = 3.14%
 " " syrup = 9.24%.

x = amount of syrup

$x : 100 :: 3.14 : 9.24$

$x = 33.98\%$ syrup,

and $100.00 - 33.98 = 66.02\%$ sucrose crystals in fill mass.

A number of methods have been devised for calculating the amount of sucrose crystals and of syrup constituting a

* Neue Zeitschrift für Rübenzucker-Industrie, Vol. 28, 1892, p. 161.

fill mass.* One of these methods is illustrated by the following example:

A fill mass has the composition:

Polarization	88.0
Water	3.5

Hence its Coefficient of Purity = $\frac{88.0 \times 100}{100 - 3.5} = 91.2$. The

sugar which is obtained from this fill mass has the composition:

Polarization	95.3
Water	2.0

$$\text{Coeff. of Purity} = \frac{95.3 \times 100}{100 - 2.0} = 97.2$$

The resultant syrup has the composition:

Polarization	66.0
Water	12.0

$$\text{Coeff. of Purity} = \frac{66 \times 100}{100 - 12.0} = 75.0$$

Represent the coefficient of purity of the

Fill mass by s

Sugar " r

Syrup " t

and, the amount of sugar by x , the amount of syrup by y .

Then:

$$1 = x + y$$

$$s = rx + ty$$

$$x = \frac{s - t}{r - t}$$

$$y = \frac{r - s}{r - t}$$

* For a detailed discussion of similar problems, see: Mittelstaedt-Bourbakis, Technical Calculations for Sugar Works, 1910,

Substituting in these formulæ the values of the present example,

$$x = \frac{91.2 - 75.0}{97.2 - 75.0} = 72.97 \text{ dry sucrose}$$

$$y = \frac{97.2 - 91.2}{97.2 - 75.0} = 27.03 \text{ dry syrup.}$$

$$\underline{\underline{100.00}} \text{ dry fill mass.}$$

Taking the water present into account,

$$\frac{100 \times 72.97}{98.0} = 74.46 \text{ sucrose,}$$

$$\frac{100 \times 27.03}{88.0} = 30.71 \text{ syrup.}$$

$$\underline{\underline{105.17}}$$

$$105.17 : 74.66 :: 100 : x = 70.8\% \text{ sucrose}$$

$$105.17 : 30.71 :: 100 : y = \underline{\underline{29.2\%}} \text{ syrup}$$

$$100.0$$

By analyzing the fill mass of each strike it is easy to keep track as to whether the boiling in the pans and the work at the centrifugals are being properly done. For this purpose a sample of the fill mass is secured, a part of it spun in a laboratory centrifugal and the purity of the resultant syrup determined.

Another portion of the fill mass is dissolved in water, the solution made up to about 15° Brix and its apparent purity ascertained.

Let x = per cent of sugar obtainable,

a = purity of fill mass,

b = purity of syrup resulting

then

$$x = \frac{(a - b) \text{ factor}}{a}$$

The table of factors, (Table XVIII) given by Irving H. Morse,* is especially adapted for use in refinery work.

To find a factor in this table consult the column headed by the polarization of the sugar obtained. The factor sought will be found in this column at the point of intersection of a line drawn straight across from the "Purity of Molasses" column.

Thus, if the polarization = 99.0 and the purity of molasses = 91.0 the factor sought = 12.35.

Yield or Rendement. The yield or rendement of a sugar is supposed to indicate how much crystallized sugar can be obtained from a given raw sugar. In other words it is supposed to represent the output in white, marketable product obtainable from a raw sugar—its refining value.

Different methods of calculating the yield have been and are in vogue in different countries. At best, all yield calculations are unreliable, and yet, as they afford a certain approximation to actual working possibilities, they continue to have a certain importance in the industry.

The method of yield calculation perhaps most commonly used is known as the ash-coefficient method; the ash is multiplied by 5 and the product subtracted from the polarization. This method is due to Monnier, 1863, and is based on certain relations which he observed between the ash of certain raw sugars and the amounts of sucrose therein which could not be brought to crystallization. From his observations he inferred that one part of the mineral impurities present (the ash) prevented five parts of sucrose from crystallizing and hence deducted five times the ash from the polarization to arrive at the refining value of the sugars.

As stated, this method of calculating the yield is perhaps the one most generally used. Frequently however the invert sugar contained in a raw sugar is also taken into account. Thus, in France, the formula, polarization less four times the ash plus twice the invert sugar, is much used;

* Calculations used in Cane-sugar factories, 1904.

from the figure thus obtained, an additional 1.5% is subtracted to allow for loss in refining operations.

In Germany, for a time, the total non-sugars were multiplied by 2.25 and this amount was deducted from the polarization. Of late however this method has been abandoned and the Monnier formula is now again used.

The yield in crystallizable sugar can be analytically determined by the Payen-Scheibler method.

This process is based on the treatment of the raw sugar, the rendement of which is to be ascertained, by solutions that will wash out the molasses-forming impurities, and leave behind the pure crystallizable sugar.

Five solutions are required:

No. 1 is a mixture in equal parts, by volume, of absolute alcohol and ether.

No. 2 is absolute alcohol.

No. 3 is alcohol of 96 per cent Tralles.*

No. 4 is alcohol of 92 per cent Tralles.

No. 5 is alcohol of 85 per cent to 86 per cent Tralles, to which 50 c.c. of acetic acid per liter have been added.

Solutions Nos. 3, 4, and 5 are all *saturated* with pure sugar, and, in order that they should remain saturated with sugar at all temperatures, they are kept in flasks which are half filled with best granulated sugar, previously washed with absolute alcohol.

These flasks are provided with a siphon arrangement; the air enters through chloride-of-calcium tubes, so as to be thoroughly dried; the solution is discharged through tubes filled with pure and dry sugar. Plugs of felt placed at the ends of these tubes prevent the carrying over of any sugar particles.

The washing operation is carried out as follows: The

* The alcoholometer of Tralles gives the percentage volume for the temperature of 60° F. = 15 $\frac{5}{8}$ ° C. Watt's Dictionary of Chemistry, Vol. I, p. 84.

accurately weighed sample, usually 13.024 grams, is placed into a 50 c.c. flask which has previously been dried.

A cork or a rubber stopper, through which two glass tubes are made to pass, serves to close the flask. One of these tubes reaches down almost to the bottom of the flask; it is provided with a felt-plug at its mouth; this serves as strainer. The shorter tube only reaches to just below the cork or stopper. The longer tube is connected, by means of a rubber tube with a large receiving bottle, from which the air is to a great extent exhausted by an aspirator or a vacuum pump. The rubber tube is provided with a pinch-cock, so that connection can be made or broken at will, between the receiving bottle and the small flask which holds the sample.

The apparatus being thus arranged, about 30 c.c. of solution No. 1 is allowed to flow into the flask containing the sugar. This solution is permitted to remain quietly in contact with the sample for from fifteen to twenty minutes, and is then drawn over into the receiving bottle. When it has all been drained over, 30 c.c. of solution No. 2 are introduced. After a contact of two minutes this solution is drawn off, and followed successively by about the same amounts of the other three solutions, in the order of their numbering.

The last of these, solution No. 5, is really the active reagent, the others principally serving to displace the moisture contained in the sugar.

This solution is allowed to remain on the sample for half an hour, being frequently and well shaken in the mean time to insure intimate contact.

It is then drawn off, and replaced by a fresh supply of the same solution. This in turn is drawn off, and the treatment is repeated with fresh amounts of solution No. 5, until the solution standing above the sugar, remains perfectly colorless. The time of contact is thirty minutes for each treatment.

The last traces of solution No. 5 are then removed by successive addition of solutions Nos. 4, 3, and 2, in the order named. These are added and drawn off at intervals of two minutes each. The last traces of alcohol are removed by drying on a water-bath, a current of dry air being continuously drawn through the flask in the mean time. When the sample is perfectly dry, the cork with its inserted tubes is carefully withdrawn, and any sugar clinging to the long tube or its felt plug, is carefully washed into the flask. The solution is then made up to 50 c.c. and polarized. The reading on the polariscope represents in percentage the yield in crystallizable sugar.

Other methods of determining the crystallizable sugar or, as it may be termed, the crystal content of sugars, obviating some of the difficulties inherent in the Payen-Scheibler method, have been devised by Koydl and by Herzfeld and Zimmermann. In the latter method the washing liquid employed is a saturated aqueous solution of sucrose; this method is to be recommended on account of its ease of manipulation.

If the crystal content of a raw sugar is known, it is a simple matter to determine the composition and purity of the molasses in the raw sugar by deducting the sucrose of the crystals from that of the raw sugar and then figuring the remainder as due to molasses.*

Hints on Reporting Sugar Analyses. In commercial analyses it is customary to report only—

Polarization,
Invert sugar,
Water,
Ash,
Non-ascertained,

* For a full account of these methods see C. A. Browne, *Handbook of Sugar Analysis*, 1912, p. 501 et seq.

the "non-ascertained" being the balance required to make the analysis figure up to 100.

When beet-sugars are examined, and a raffinose determination has been made, this substance, of course, makes another item in the report, which would then embrace:

Polarization,
Sucrose,
Raffinose,
Invert sugar,
Water,
Ash,
Non-ascertained.

The polarization in the first form of analysis given above, may either correspond to, be greater, or smaller than the amount of sucrose really present, for the presence of other optically active bodies influences the polariscope reading to a marked degree.

Invert sugar turns the plane of polarized light to the left. At 17.5° C. one part of invert sugar neutralizes the optical effect of about 0.34 part of sucrose. In order, therefore, to obtain the sucrose corrected for this disturbing influence, the amount of invert sugar found is multiplied by 0.34, and the result is added to the direct polarization. This sum is then regarded as approximately representing the sucrose.

Frequently a polarization after inversion is made, and compared with the direct polarization.

If there are no other optically active bodies present in the sample besides the sucrose, the result of the polarizations before and after inversion will be identical, or at least agree very closely. If the polarization after inversion is higher than the direct polarization, the presence

of levo-rotatory bodies is indicated; if it is lower, dextro-rotatory substances are present.

Investigation has, however, shown that this method of inversion and subsequent polarization (Clerget's test) is not always applicable to sugars rich in reducing sugars (so-called invert sugar), because the inverting acid (hydrochloric acid) changes the levo-rotation of the invert sugar, and because the reducing sugar sometimes consists of a mixture of levo- and of dextro-rotatory substances in varying proportions.

In dealing with samples of such description, as, for instance, with low sugars and molasses, sugar-cane products, etc., an exhaustive analysis is desirable, in order to gain all information possible with regard to the nature of the sample.

Such an analysis should record—

Reaction (acid, alkaline, or neutral),
Total sucrose,
Direct polarization,
Polarization after inversion,
Total reducing sugars,
Water,
Ash.

The correct interpretation of an analysis of this description is not always an easy matter.

If the polarization after inversion agrees with the direct polarization *plus* 0.34 times the total reducing sugar, this value *may* be regarded as the amount of sucrose present. As, however, all results obtained by the Clerget method on sugars rich in invert sugar are open to some doubt, it will be better, even in case the direct polarization *plus* 0.34 times the total reducing sugar is equal to the polarization after inversion, to resort to gravimetric determinations for verification.

In case of non-agreement of the direct polarization *plus* 0.34 times the total reducing sugar, and the Clerget test, gravimetric analysis must of course be employed.

Determine the total sucrose after inversion, by its reducing action on copper solution, and determine, also by gravimetric analysis, the total reducing sugar. Calculate the latter over to its equivalent of sucrose by subtracting one twentieth of the amount found; deduct this result from the total sucrose, and report the remainder as sucrose.

Example.

Polarization before inversion, . . .			52.70
Polarization after inversion, . . .			63.12
Total reducing sugar,			22.89
Total sucrose (gravimetric det.), . .			79.20
	22.89	Total sucrose,	79.20
Less $\frac{1}{20}$, 1.14	Less . . .		21.75
	<u>21.75</u>	Sucrose =	<u>57.45</u>

Sucrose Loss. By this term there is understood the difference between the amount of sucrose which enters into the process of manufacture and the amount of sucrose which is turned out by the process of manufacture in any and every form.

Presupposing correct weighing of all raw material received for refining purposes and of all products turned out; furthermore presupposing correct analytical work and correct clerical work in the making up of the technical statements, the sucrose losses sustained must be due to chemical causes, to mechanical causes, or to both.

When studying sucrose losses through chemical and mechanical causes, the following points should be especially kept in view.

Chemical Losses.

1. Formation of invert sugar through acidity, bacterial influence, overheating.
2. Formation of gums through bacterial influence.
3. Destruction of sucrose through caramelization, in vacuum pans through overheating.

Mechanical Losses.

4. In press cake.
5. In washing plant of bags, filter-cloths, etc.
6. By entrainment.
7. Through leaks and overflows.
8. By theft.

CHAPTER XII

RÉSUMÉ OF THE WORK OF THE INTERNATIONAL COMMISSION FOR UNIFORM METHODS OF SUGAR ANALYSIS

ALEXANDER HERZFELD, Chairman, F. G. WIECHMANN, American Secretary.

FIRST SESSION: Hamburg, Germany, June 12th, 1897.

(1.) *Kinds of quartz plates to be selected.*

At the start only quartz plates of high polarizing value shall be tested, later however, such also as will cover the entire scale-range of saccharimeters.

(2.) *Method of examination of quartz plates.*

Their examination is to be conducted in the same manner as has been done heretofore by the Commission of Tradeschemists, under guidance of the Society of the Beet Sugar Industry of the German Empire, with the participation of the Imperial Normal Testing Bureau and the Physical-Technical-Reichsanstalt.

(3.) *Temperature to be adopted as the normal-temperature for polarization.*

For the examination of quartz plates 20° Centigrade is to be chosen as the normal temperature, and the metric liter is to be adopted. The normal weight to be adopted is hence to be 26.00 grams, where 26.048 grams is the normal weight valid for Mohr's liter at the temperature 17½° Centigrade.

(4.) *Additional methods and means suggested in order to decrease differences in polarization work.*

In consideration of the well-known difficulties in sampling bagged sugar, sampling each bag does not offer sufficient advantages to justify a departure, in the interests of trade, from the customary method of sampling 20 bags in every 100 bags.

(5.) *Desirability of an endeavor to introduce uniformity of analytical methods for beet-sugar work in all countries concerned.*

Such an endeavor shall be made. For the computation of sugars, analyses shall be admissible only of such chemists as shall have pledged themselves to execute the analysis of sugar in accordance with the methods prescribed by the International Commission.

(6.) *The determination of invert sugar.*

The determination of invert sugar is to be made only in solutions which have been clarified with lead solution and from which the lead has then been removed. If volumetric determinations are made, the amount of reduction due to the chemically pure sucrose must be deducted.

SECOND SESSION: Vienna, Austria, July 31st, 1898.

(1.) *Results of the International examination of quartz plates.*

In general, such examinations proved satisfactory. Certain discrepancies were undoubtedly due to the fact that the examinations had not always been made at 20° C., as prescribed.

Reference was made to the observations of Herzfeld, Wiechmann, and Wiley, that pressures, due to varying temperatures affecting their mountings, exercise an influence on the rotation values of fixedly mounted quartz plates and quartz-wedges. Whereas the quartz plates heretofore used—owing to the fact of their being firmly held in their mountings—are apt to be strained when their temperature is raised, and whereas such strains cause irregularities in the polarizing values of these plates, it was resolved that the investigation above referred to should be repeated, making

use of other quartz plates not subject to the defect mentioned.

On the motion of Messrs. Dupont and Jobin it was resolved to employ plates the rotation values of which shall cover the entire scale of the saccharimeter, one levo-rotatory and four dextro-rotatory plates, the thickness of which is at the same time to be given, so that the plates shall remain serviceable when the normal weights shall be changed.

In determining the value of the plates there shall be employed not only the normal temperature of 20° C. but, on the motion of Messrs. Wiley and Wiechmann, also of 30° C., in order to take into due account the condition of warmer countries. In employing apparatus with quartz-wedge compensation the changes shall be studied which the saccharimeter itself suffers in consequence of variations of temperature. As source of light there shall be employed only yellow sodium light or light sufficiently purified by ray-filters.

Upon the motion of Dr. Hermann, of Hamburg, it was furthermore agreed to emphasize specifically in the protocol that the Commission had thus far made no such changes in the normal weight for polariscopes which could influence the results of polarization in the least. This had been mentioned already in the protocol of the Hamburg session of June 12th, 1897.

(2.) *Desirability of examining raw beet sugars for trade purposes according to the inversion method.*

Those present were unanimously of the opinion that the question should, in general, be decided in the negative. Exception should be made only in case of the products obtained in making sugar from molasses; with these it was recommended to make determinations of sugar and of raffinose.

(3.) *Discussion of applications of the Deutsche Zucker-Export-Vereine.*

Dr. Hermann suggested that uniformity in analytical methods should even now be striven for as much as possible, and the presiding officer was requested to prepare, with the assistance of the members of the Commission, a clear compilation of analytical methods which are in vogue in the different countries, and also to prepare a résumé of the directions which are to be followed in cases of differences in analysis. Basing on these documents, the attempt shall be made to secure International acceptance of a uniform method of procedure.

THIRD SESSION: Paris, France, July 24th, 1900.

(1.) *Normal sugar-weight to be adopted for saccharimeters of German make when the metric flask is used.*

The Imperial Physical-Technical Institute has, by its communication dated October 19th, 1898, called attention to the fact that an exact conversion of the normal weight 26.048 grams for Mohr's cubic centimeters at 17.5° C. corresponds to 26.01 grams (not 26.00) metric volume at 20° C., determined in air with brass weights.

The Commission decided that in consideration of the insignificance of the deviation the normal weight of 26.00 grams shall henceforth be adopted for 100 metric cubic centimeters, at 20° C., determined in air with brass weights.

(2.) *Examination and disposal of quartz plates.*

Prof. Herzfeld reported briefly on the results of the examination of quartz plates, and the Commission agreed that these quartz plates should be divided among the nations represented. For the United States, the plates were to be sent to the Department of Agriculture, at Washington; for France, to the Syndicate of Sugar Manufacturers; for Belgium, Holland, Austria-Hungary, and Russia, to the Associations of Sugar Manufacturers represented in the session by delegates.

(3.) *General principles governing the adjustment of saccharimeters.*

On motion of Messrs. Camuset and Saillard, the following was adopted:

“The Convention declares it to be necessary that the rotation of chemically pure sugar be accepted as the fundamental basis in saccharimetry.

“The chemically pure sugar which is to be employed for this purpose shall everywhere be prepared according to the same method which is as follows (method of the English chemists):

“Purest commercial sugar is to be further purified in the following manner: A hot saturated aqueous solution is prepared and the sugar precipitated with absolute ethyl alcohol; the sugar is carefully spun in a small centrifugal machine and washed in the latter with some alcohol. The sugar thus obtained is re-dissolved in water, again the saturated solution is precipitated with alcohol and washed as above. The product of the second centrifuging is dried between blotting paper and preserved in glass vessels for use. The moisture still contained in the sugar is determined and taken into account when weighing the sugar which is to be used.”

The Convention furthermore decided that central stations shall be designated in each country which are to be charged with the preparation and the distribution of chemically pure sugar. Wherever this arrangement is not feasible, quartz plates, the values of which have been determined by means of chemically pure sugar, shall serve for the control of saccharimeters.

Mention should be made of the fact that in the discussion on this topic, it was remarked, on the one hand, that the preparation of chemically pure sugar is not an easy task, and that in countries having hot climates, sugar is dried with difficulty and hence is not stable, and hardly available for transportation. Thereupon it was pointed out that the above control, by means of chemically pure sugar, should, as a rule, apply only to the central stations

which are to test the correctness of saccharimeters; for those who execute commercial analyses, the repeated control of the instruments is to be accomplished, now as before, by means of quartz plates.

Concerning the working temperature the following resolution of Mr. François Sachs was unanimously adopted:

“In general, all sugar tests shall be made at 20° C.

“The adjustment of the saccharimeter shall be made at 20° C. One dissolves (for instruments arranged for the German normal weight) 26.00 grams of pure sugar in a 100 metric cubic centimeters flask,* weighing to be made in air, with brass weights, and polarizes the solution in a room the temperature of which is also 20° C. Under these conditions the instrument must indicate exactly 100.00

“The temperature of all sugar solutions to be tested is always to be kept at 20° C., while they are being prepared and while they are being polarized.

“However, for those countries the temperature of which is generally higher, it is permissible that the saccharimeters be adjusted at 30° C. (or at any other suitable temperature), under the conditions specified above, and providing that the analysis of sugar be made at that same temperature.”

Objections were raised against the universal normal weight 20.00 grams by Mr. François Sachs as well as by Mr. Strohmer. In consequence, it was resolved not to undertake the introduction of the same, but to adopt the resolution:

“The general International introduction of a uniform normal weight is desirable.”

It was furthermore resolved, on the basis of the proposition of Mr. Strohmer, to observe the following rules in raw sugar analysis.

* Or during the period of transition, 26.048 grams in 100 Mohr's cubic centimeters.

I. POLARIZATION.

In effecting the polarization of substances containing sugar, half-shade instruments only are to be employed.

During the operation the apparatus must be in a fixed, unchangeable position, and so far removed from the source of light that the polarizing Nicol is not warmed by the same.

As source of light there are to be recommended lamps with intense flame (gas triple-burner with metallic cylinder, lens and reflector; gas lamp with Auer burner; electric lamp; petroleum duplex lamp; sodium light).

The chemist must satisfy himself, before and after the observation, of the correctness of the apparatus (by means of correct quartz plates) and in regard to the constancy of the light; he must also satisfy himself as to the correctness of the weights, of the polarization flasks, the observation tubes and the cover glasses. (Scratched cover glasses must not be used.)

Several readings are to be made and the mean thereof taken, but any one individual reading must not be selected.

II. SUGAR ANALYSIS.

1. *Sucrose*. To make a polarization, the whole normal weight for 100 cubic centimeters is to be used, or a multiple thereof for any corresponding volume.

As clarifying and decolorizing reagents there may be used: subacetate of lead, prepared according to the *German Pharmacopoeia* (three parts by weight of acetate of lead, one part by weight of oxide of lead, ten parts by weight of water), Scheibler's alumina cream, concentrated solution of alum. Bone-black and decolorizing powders are to be absolutely excluded.

After bringing the solution exactly to the mark and after wiping out the neck of the flask with filter paper, all of the well-shaken, clarified sugar solution is poured upon a dry,

rapidly filtering filter. The first portions of the filtrate are to be thrown away and the balance, which must be perfectly clear, is to be used for polarization.

2. *Water*. In normal beet sugars the water determination is to be made at 105° to 110° C.

For abnormal beet sugars, there is no commercial method for the determination of water.

3. *Ash*. To determine the ash-content in raw sugars the determination is to be made according to Scheibler's method, employing pure concentrated sulphuric acid. For an ash determination at least 3 grams of the sample are to be used. The incineration is to be carried out in platinum dishes, by means of platinum or clay muffles, at the lowest possible temperature (not above 750° C.).

From the weight of the sulphated ash thus obtained, 10 per cent is to be deducted and the ash-content, thus corrected, is to be recorded in the certificate.

4. *Alkalinity*. As, according to the most recent investigations, the alkalinity of raw sugars is not always a criterion of their durability, the Commission abstains from proposing definite directions for the execution of the investigations.

5. *Invert Sugar*. The quantitative determination of invert sugar in raw sugars is to be made according to the method of Dr. A. Herzfeld. (*Zeitschrift des Vereins für die Rübenzucker-Industrie des Deutschen Reiches*, 1886, pp. 6 & 7.)

Furthermore the following resolutions were adopted:

The Commission declares that only well closed glass vessels will ensure the stability of samples.

To obtain correct results it is desirable that the samples contain at least 200 grams of material.

All of the above resolutions were adopted unanimously by those present.

FOURTH SESSION. Berlin, Germany, June 4th, 1903.

(1.) *Professor Herzfeld outlined the previous work of the Commission.*

The sets of quartz plates which had been selected by

the *Physikalisch-Technische Reichsanstalt in Berlin*, and which had been tested in the laboratory of the *Verein der Deutschen Zuckerindustrie* as to their sugar value, have been distributed to proper central stations of the countries interested, and there kept at the disposal of chemists. These plates have been tested in almost all of the countries which have received the sets, and have been found correct. Some of these stations have thus far not made a report as to the result of this re-examination, and such a report is therefore requested.

Execution of the Paris agreement, according to which chemically pure sugar is to be used for the adjustment of polariscopes and for the testing of plates, has in some countries, met with difficulties because they could not succeed in preparing chemically pure sugar. The laboratory at Berlin, therefore, offers to furnish chemically pure sugar.

In the determination of invert sugar a difficulty has arisen, inasmuch as the English chemists have of late again declared against the clarification with basic lead acetate; the Commission will therefore have to seek means and methods to prevent, in this respect, loss of uniformity now secured in the methods of analysis.

The day's proceedings furthermore covered reports, concerning:

I. Practical experiences made with the uniform methods of analysis agreed upon in Paris.

II. The valuation of "Sand" and "Krystallzucker" in International trade.

III. Introduction of International uniform directions for sampling raw sugars.

IV. and V. Influence of temperature on the specific rotation of sucrose, and introduction of temperature-corrections when the temperature of observation differs from the temperature of 20° C., which has been accepted as the normal temperature.

VI. Determination of the sugar subject to duty or bounty contained in saccharine products and fruit preserves.

VII. Chemical control as an aid to the "Entrepôt" system, sanctioned by the Brussels Convention.

FIFTH SESSION: Bern, Switzerland, August 3d and 4th, 1906.

(1.) *The Chairman in a review of the achievements of the Commission designated the duties of the Commission to be purely analytical.*

The Commission has for its object the regulation of the methods of sugar analysis and endeavors to secure the working of chemists according to uniform and the best methods, but the Commission does not undertake to establish trade-customs. The Commission does not recognize resolutions carried by majority vote, it is in fact necessary that at least the representatives of the most important countries interested in sugar be in accord on a question before the same is presented for acceptance, as otherwise no reliance can be placed on the recognition of the resolutions by chemists.

(2.) *Determination of a method of preparing Fehling's solution as well as the manner of making invert sugar determinations.*

Messrs. Watt and Wiechmann communicated the results of their investigations. Mr. Watt preferred the volumetric method, Mr. Wiechmann the gravimetric method for commercial analyses. The latter moved that clarification with basic lead acetate shall be obligatory for the examination of syrups. This recommendation was endorsed by Messrs. Watt and Prinsen Geerligs and thereupon also by the entire Commission.

The Chairman reported on tests made for the comparison of Violette's and Fehling's solution, which had not yet been completed. He announced that Mr. Munson, the Chairman of the Association of American Agricultural Chemists had, through intervention of Mr. Wiechmann, sent him a resolution of the Association named, wherein

the same expressed the wish to work hand in hand with the Commission in the matter of securing a uniform alkaline copper solution.

Mr. Pellet also presented a paper on this subject which was published in the *Sucrerie Indigène* as well as in the *Deutsche Vereinszeitschrift*.

Mr. Strohmer promised a later report of his experiments bearing on this question which, at present, are not yet completed. He recommended retaining for the present the so-called Herzfeld method.

Mr. Watt declared himself against basic lead acetate clarification for solid sugars.

Mr. Sachs refrained from voting.

Mr. Saillard as well as Mr. Dupont expressed themselves in favor of retaining basic lead acetate clarification as long as the present method was used.

Mr. Pellet declared himself against the use of basic lead acetate as a clarifying reagent.

Mr. Schukow favored this clarification in commercial analysis.

Mr. Watt handed in the following declaration:

“The difference between the amount of the reducing substances in the clarified and the non-clarified solution of beet sugar lies so closely within the limits of the errors of observation, that a clarification is unnecessary; but in products which contain a large amount of glucose a clarification is of great importance.”

Mr. Herzfeld was of the opinion that he could not accept the first half of the declaration, the difference being indeed a small one but giving rise to considerable annoyance in trade.

Mr. von Buchka proposed to defer the question of the composition of Fehling's solution and to have the same studied further by a separate commission.

Mr. Geerligs expressed himself in favor of the basic lead acetate clarification for syrups.

Mr. Main spoke against the basic lead acetate clarification in raw sugars.

The Chairman proposed to request the chemists of Great Britain to discuss this question in a separate conference once more with delegates of the Commission in order to try, in this manner, to bring about an agreement.

The proposition of the Chairman was accepted by the Commission and a sub-commission was appointed, consisting of Messrs. Strohmer, Saillard, Sachs, Schukow, van Ekenstein, Watt, Main, von Buchka and Herzfeld, this sub-commission to take part in the conference with the chemists of Great Britain.

The Chairman agreed to ask for the intervention of the German Export Societies to the end that the conference might soon be called in London.

The question of Fehling's solution should be subjected to further study.

(3.) *Uniform International directions for sampling sugar products.*

(4.) *Resolution concerning a uniform form and manner of expression of certificates of analysis for the International sugar trade.*

Mr. Saillard presented the following resolutions:

1. As long as it is not settled that the degree of alkalinity is a sure criterion for the keeping qualities of sugars, the determination of alkalinity shall not be considered in International commerce.

2. The Commission shall determine upon a uniform method by which the trade yield (Rendement) for the sugar is to be calculated, in establishing scientific molasses coefficients for the impurities (ash and invert sugar) the relation of which is used in the certificates of analysis.

3. The state laboratories shall also take part in the endeavors to bring about uniformity, in order to cause a disappearance of the differences between trade ash determinations and Regie ash determinations. (France.)

These resolutions were accepted by the Commission, and the Commission also decided, for the present, not to endorse any specific form of certificate of analysis.

(5.) *Avoidance of the precipitate error in optical sugar analysis.*

(6.) *Suggestions for the preparation of unchangeable color standards in place of the raw sugar used for the Dutch standards.*

After a report by the Chairman on the substitution of samples of colored glasses for the Dutch standards, and a discussion of the question, the Commission unanimously expressed the wish that the valuation of sugar according to its color might soon be abandoned altogether, because this practice was to be condemned from the scientific as well as from the practical point of view.

(7.) *Concerning a method to be recommended for the determination of the sugar-content of beets.*

The Commission was of the opinion and unanimously adopted the resolution, that the aqueous digestion method for the determination of the sugar-content of the beets if it be executed with due regard to the precautions suggested by Pellet, Sachs and others, was to be recommended in preference to the alcohol method. The Commission charged Messrs. Pellet, Sachs and Herles with presenting detailed working directions of the method.

(8.) *A uniform International sugar-weight.*

After a review by the Chairman of the prior discussions on this topic, the introduction by Mr. Dupont of a proposition to accept 20.00 grams as the normal weight, and a thorough debate of the proposition, the Chairman put the question to those present concerning the desirability of retaining 26.00 grams as the normal sugar-weight for saccharimeters.

Mr. Sachs replied in the affirmative.

Mr. Saillard did not consider it necessary that all countries should have the same normal weight in order to have a uniform method.

On the question being put by the Chairman, the representatives present of America, Java, Great Britain, Russia, and Austria-Hungary declared themselves against the normal weight of 20.00 grams and for the normal weight of 26.00 grams.

Mr. Sachs declared himself in accord with Mr. Saillard to admit 20.00 grams, but not to prescribe it.

9. *Conference regarding measures to secure an Internationally valid uniform method of beet seed valuation.*

On the recommendation of the Chairman the proposition was accepted that the Commission should not occupy itself with the establishment of standards (Normen), but only with the establishment of methods of investigation.

Hereupon a sub-commission was chosen to work out uniform methods of examination; Mr. F. Strohmer was appointed chairman of the same.

The following were elected members of the sub-commission: Messrs. Strohmer, President; Saillard, Boussaud (Paris), Sachs, Schukow, Müller (Halle), Krüger (Bernburg), Herzfeld, Raatz, von Dippe, Heine, Briem, Neumann and Herles.

The Commission was authorized to increase its numbers by the election of further members.

SIXTH SESSION: London, England, May 31st., 1909.

(1.) *Report of the work of the International Commission since its last session.*

Mr. Herzfeld reported that the results of newer investigations speak against the views expressed by the members of the Commission in Bern, making obligatory the clarification with basic lead acetate for the determination of invert sugar in syrups. For this reason this matter was therefore that day given place on the program to permit of another resolution. All other resolutions taken in Bern have been put into practice.

Prof. Villavecchia in Rome has agreed, in a letter addressed to the Chairman, that the Commission shall

be called in consultation in case an International Commission of the Government works out directions for sugar analysis

Agreeable to the resolution taken in Bern, a compilation of the proceedings to date of the Commission has thus far been printed only in German. Copies of this pamphlet will be sent to the members of the Commission by the Chairman if they so desire. A compilation of the resolutions adopted thus far by the Commission, which had been prepared by Mr. Wiechmann of New York, was distributed. Messrs. François Sachs and Saillard promised to publish such a compilation in French.

Mr. Strohmer, Vienna, then reported on the doings of the Sub-Commission for Uniform Methods of Beet-Seed Analysis appointed in Bern, which had met under his direction in Vienna on May 24, 1907, but which had taken definite resolutions only with regard to uniform methods for the determination of water and the determination of impurities. A report concerning the session of the Sub-Commission has already been published in the technical journals.

At the suggestion of the referee, the International Commission decided that the introduction of uniform methods of beet-seed examination was to be postponed until it should be settled whether, and in what manner, the present standards (Normen) are to be changed.

(2.) *Unification of the tables for the calculation of the contents of sugar solutions from their density.*

Messrs. Saillard, Paris, and von Buchka, Berlin, reported on this topic. Their findings will be published in full in the technical journals.

On the motion of Mr. Sachs, seconded by Messrs. Saillard, Prinsen Geerligs, Strohmer, Neumann, and Pellet, the Commission voted unanimously to accept a single table as standard at the temperature of 20° C., which is to be based upon the official German table. From this, other tables may be calculated

at other temperatures, for instance, at 15° C., 17.5° C., 30° C., etc., as well as a table according to the Mohr system, 20° C.: 20° C.

(3.) *Propositions for the use of uniform clarifying reagents for the analysis of sugar products.*

Referees: Messrs. Prinsen Geerligs, Java, and François Sachs, Brussels. Their findings will also be published in the technical journals. In this connection Mr. Herles recommended basic lead nitrate as a clarifying re-agent.

After an extensive debate in which all delegates took part, the Commission unanimously decided that for the direct polarization of solutions of raw sugar products, basic lead acetate shall also in future be used for clarification, but not in excess. For fluid sugars (syrup and molasses), basic lead acetate may not be employed for the determination of invert sugar, but only neutral lead acetate as a clarifying reagent.

An agreement could not be reached concerning the clarifying of solutions of solid raw sugars for the purpose of the determination of invert sugar, as the English chemists remained firm in their former position to effect no clarification whatever for the determination of invert sugar.

(4.) *Agreement as to a uniform nomenclature for the products of sugar manufacture, especially in view of the food laws.*

Referee: Mr. Strohmer, Vienna, made a report on this topic which will be published in the technical journals. On the motion of the referee, and of Messrs. Saillard and Silz, a resolution in this matter was, for the present, postponed.

(5.) Dr. Wiley, of Washington, read two articles by Messrs. A. Hugh Byran and C. A. Browne, New York, concerning the conditions of basic lead acetate clarification and on temperature corrections in raw sugar polarizations, which are to be published at once. In consequence of the declaration of Dr. Wiley that in order to avoid temperature corrections, the American Government Laboratories for sugar analysis are soon to be provided with cooling arrange-

ments in order that the polarizations shall be made exclusively at the normal temperature of 20° C., the Commission avoided voting on the resolution of Mr. Browne, New York, in which the avoidance of any and every temperature correction in raw sugar analysis is demanded. (*See article by Mr. Browne in the technical journals.*) Prior to this, this resolution had been fully established by Mr. Horne, representing Mr. Browne.

An article by Mr. Horne, on the use of dry basic lead acetate clarification in sugar analysis, could not be read in this session of the Commission. This article has been published in the *Zeitschrift des Vereins der Deutschen Zucker-Industrie*.

SEVENTH SESSION: New York, September 10th, 1912.

(1.) *Report on the work done since the last session.*

Mr. Saillard reported that he and Mr. Sachs have conferred about the publication of a compilation of the proceedings of all sessions of the Commission in French, and expressed the belief that this will be achieved in time for the next session.

The German Imperial Bureau of Standards (*Normal-eichungsamt*) promised to prepare tables for the determination of the percentage composition of sugar solutions from their specific gravity at different temperatures, as was requested at the London conference.

The following resolution was unanimously adopted:

“Owing to the declaration of Dr. Wiley at the 6th Session of the International Commission for Uniform Methods of Sugar Analysis in London, 1909, that, in order to avoid temperature corrections the American Government Laboratories for sugar analysis are soon to be provided with cooling arrangements in order that polarizations shall be made exclusively at the normal temperature of 20° C., and to the fact that this declaration has not yet been carried into effect, this Commission again expresses the opinion that official polarizations of raw sugar products shall be made only at the constant standard temperature 20° C., the presence of invert sugar and other

impurities precluding the use of formulæ and tables, which have been elaborated for correcting the polarization of pure sucrose for changes of temperature."

(2.) *Uniform methods of sucrose determination in the raw materials of sugar manufacture.*

Mr. Saillard, at the behest of the Syndicate of Sugar Manufacturers, of France, explained that the trade in beets and the trade with molasses does not, for France, bear the same International character as the trade in sugar, and requested that the International Commission treat analytical methods for beets and molasses only from the viewpoint of analytical chemistry.

Upon the recommendation of Mr. Herzfeld, after a short debate, the Commission refrained from officially endorsing uniform methods of sucrose determination in beets and in sugar cane. It was agreed, by publication in the technical journals of the various countries, to bring the reports of Messrs. Strohmer, Sachs, Saillard, Wiechmann, Prinsen Geerligs, and Herles, which were read before the meeting, to the notice of those interested, and to call attention to the propositions of the referees.

(3.) *Uniform methods for the determination of the specific gravity of sugar solutions.*

Mr. Saillard and Mr. von Buchka presented reports on this topic; these reports are to be published; Messrs. Weinstein, Strohmer, and Saillard participated in the debate which followed.

It was agreed to publish the reports of the referees and the following resolutions were adopted. On motion of Mr. Strohmer that:

"The International Commission for Uniform Methods of Sugar Analysis expresses the wish that the various countries may prescribe a uniform temperature for the density determinations of sugar solutions.

"In trade analyses the use of temperature correction tables should be dispensed with, as far as possible."

On motion of Mr. von Buchka that:

"The normal temperature of +20° C. is to be retained for trade analyses.

In density determinations of aqueous sugar solutions, the density obtained at normal temperature shall be referred to the density of water at +4° C. and to vacuo.

In density determinations made by weighing, the results must be calculated to $\frac{20}{4}$ ° C. and to vacuo. To effect this it will be desirable to use tables prepared for the purpose."

Mr. Saillard agreed to the latter part of the resolution, provided that in France the normal temperature there customary, namely $\frac{15}{4}$ ° C., be retained in place of $\frac{20}{4}$ ° C.

(4.) *Resolution concerning a further examination of the inversion-constants of the double polarization (Clerget-Herzfeld) method.*

Mr. Herzfeld reported, that although the correctness of the constant 132.66 at 20° C. for a solution which contains the German half normal weight of pure sucrose has been confirmed by numerous chemists and, finally by an International Commission of Chemists, who worked in Berlin in 1910, yet, on the basis of more recent investigations, it seems to him desirable to re-test the accuracy of this constant in as far as its application in the analysis of beet-molasses is concerned. A committee consisting of Messrs. Bates, Bryan, Browne, Prinsen Geerligs, Saillard, Sachs, and Strohmer is appointed to re-test this constant.

The following reports were also made and the following resolutions adopted:

Mr. A. Hugh Bryan made a report on the necessity of using light-ray filters for polarizing high grade sugars, and on his motion the following resolution was adopted:

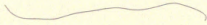
"Wherever white light is used in polarimetric determinations, the same must be filtered through a solution of potassium dichromate of such a concentration that the percentage content

of the solution multiplied by the length of the column of the solution in centimeters is equal to nine."

Furthermore, Mr. Bates reported on investigations conducted by the Bureau of Standards and Weights, which led to the conclusion that the one-hundred point of saccharimeters provided with Ventzke scales at present accepted, is not quite correct. On his motion the following resolution was adopted:

"Inasmuch as recent investigations tend to question the validity of the present 100° point of saccharimeters, and inasmuch as it is desirable that the Commission recognize and fix a transformation factor from absolute degrees to Ventzke degrees, the Chairman is hereby empowered to appoint a Committee of three to fully investigate this question and report at the next official meeting."

At the suggestion of the Chairman, Messrs. Bates, Schönrock, and Strohmeyer were elected as members of this sub-committee.



TABLES

I.

RELATION BETWEEN SPECIFIC GRAVITY, DEGREES BRIX AND DEGREES BAUMÉ, FOR PURE SUGAR SOLUTIONS FROM 0 TO 100 PER CENT.

MATEGCZEK AND SCHEIBLER.
(Temperature 17.5° C. = 63.5° F.)

FORMULÆ OF VON LORENZ. SPECIFIC GRAVITY AND DEGREES BRIX

Let d = specific gravity, s = degrees Brix.
For the range of:

0°–35° Brix.....	$d = \frac{29374 + 14s}{29375 - 100s}$
35°–70° Brix.....	$d = \frac{35036 + 43s}{35163 - 100s}$
70°–100° Brix.....	$d = \frac{42067 + 92s}{42908 - 100s}$

DEGREES BRIX AND SPECIFIC GRAVITY

For the range of:

1.00000–1.15411.....	$s = \frac{29375d - 29374}{100d + 14}$
1.15411–1.35088.....	$s = \frac{35163d - 35036}{100d + 43}$
1.35088–1.55785.....	$s = \frac{42908d - 42067}{100d + 92}$

SPECIFIC GRAVITY AND DEGREES BAUMÉ.

Let d = specific gravity, n = degrees Baumé.

$$d = \frac{146.78}{146.78 - n}$$

DEGREES BAUMÉ AND SPECIFIC GRAVITY

$$n = 146.78 \frac{d - 1}{d}$$

DEGREES BRIX AND DEGREES BAUMÉ.

Let s = degrees Brix, n = degrees Baumé.
For the range of:

0.00°–19.60° Baumé.....	$s = \frac{10 + 2097n}{1195 - n}$
19.60°–38.12° Baumé.....	$s = \frac{433 + 814.5n}{488 - n}$
38.12°–52.56° Baumé.....	$s = \frac{1342 + 457.2n}{306.3 - n}$

DEGREES BAUMÉ AND DEGREES BRIX.

For the range of:

0°–35° Brix.....	$n = \frac{1195s - 10}{s + 2097}$
35°–70° Brix.....	$n = \frac{488s - 433}{s + 814.5}$
70°–100° Brix.....	$n = \frac{306.3s - 1342}{s + 457.2}$

I.

Degrees Brix.	Specific Gravity.	Degrees Baumé.	Degrees Brix.	Specific Gravity.	Degrees Baumé.
0.0	1.00000	0.00	4.0	1.01570	2.27
0.1	1.00038	0.06	4.1	1.01610	2.33
0.2	1.00077	0.11	4.2	1.01650	2.38
0.3	1.00116	0.17	4.3	1.01690	2.44
0.4	1.00155	0.23	4.4	1.01730	2.50
0.5	1.00193	0.28	4.5	1.01770	2.55
0.6	1.00232	0.34	4.6	1.01810	2.61
0.7	1.00271	0.40	4.7	1.01850	2.67
0.8	1.00310	0.45	4.8	1.01890	2.72
0.9	1.00349	0.51	4.9	1.01930	2.78
1.0	1.00388	0.57	5.0	1.01970	2.84
1.1	1.00427	0.63	5.1	1.02010	2.89
1.2	1.00466	0.68	5.2	1.02051	2.95
1.3	1.00505	0.74	5.3	1.02091	3.01
1.4	1.00544	0.80	5.4	1.02131	3.06
1.5	1.00583	0.85	5.5	1.02171	3.12
1.6	1.00622	0.91	5.6	1.02211	3.18
1.7	1.00662	0.97	5.7	1.02252	3.23
1.8	1.00701	1.02	5.8	1.02292	3.29
1.9	1.00740	1.08	5.9	1.02333	3.35
2.0	1.00779	1.14	6.0	1.02373	3.40
2.1	1.00818	1.19	6.1	1.02413	3.46
2.2	1.00858	1.25	6.2	1.02454	3.52
2.3	1.00897	1.31	6.3	1.02494	3.57
2.4	1.00936	1.36	6.4	1.02535	3.63
2.5	1.00976	1.42	6.5	1.02575	3.69
2.6	1.01015	1.48	6.6	1.02616	3.74
2.7	1.01055	1.53	6.7	1.02657	3.80
2.8	1.01094	1.59	6.8	1.02697	3.86
2.9	1.01134	1.65	6.9	1.02738	3.91
3.0	1.01173	1.70	7.0	1.02779	3.97
3.1	1.01213	1.76	7.1	1.02819	4.03
3.2	1.01252	1.82	7.2	1.02860	4.08
3.3	1.01292	1.87	7.3	1.02901	4.14
3.4	1.01332	1.93	7.4	1.02942	4.20
3.5	1.01371	1.99	7.5	1.02983	4.25
3.6	1.01411	2.04	7.6	1.03024	4.31
3.7	1.01451	2.10	7.7	1.03064	4.37
3.8	1.01491	2.16	7.8	1.03105	4.42
3.9	1.01531	2.21	7.9	1.03146	4.48

Degrees Brix.	Specific Gravity.	Degrees Baumé.	Degrees Brix.	Specific Gravity.	Degrees Baumé.
8.0	1.03187	4.53	13.0	1.05276	7.36
8.1	1.03228	4.59	13.1	1.05318	7.41
8.2	1.03270	4.65	13.2	1.05361	7.47
8.3	1.03311	4.70	13.3	1.05404	7.53
8.4	1.03352	4.76	13.4	1.05446	7.58
8.5	1.03393	4.82	13.5	1.05489	7.64
8.6	1.03434	4.87	13.6	1.05532	7.69
8.7	1.03475	4.93	13.7	1.05574	7.75
8.8	1.03517	4.99	13.8	1.05617	7.81
8.9	1.03558	5.04	13.9	1.05660	7.86
9.0	1.03599	5.10	14.0	1.05703	7.92
9.1	1.03640	5.16	14.1	1.05746	7.98
9.2	1.03682	5.21	14.2	1.05789	8.03
9.3	1.03723	5.27	14.3	1.05831	8.09
9.4	1.03765	5.33	14.4	1.05874	8.14
9.5	1.03806	5.38	14.5	1.05917	8.20
9.6	1.03848	5.44	14.6	1.05960	8.26
9.7	1.03889	5.50	14.7	1.06003	8.31
9.8	1.03931	5.55	14.8	1.06047	8.37
9.9	1.03972	5.61	14.9	1.06090	8.43
10.0	1.04014	5.67	15.0	1.06133	8.48
10.1	1.04055	5.72	15.1	1.06176	8.54
10.2	1.04097	5.78	15.2	1.06219	8.59
10.3	1.04139	5.83	15.3	1.06262	8.65
10.4	1.04180	5.89	15.4	1.06306	8.71
10.5	1.04222	5.95	15.5	1.06349	8.76
10.6	1.04264	6.00	15.6	1.06392	8.82
10.7	1.04306	6.06	15.7	1.06436	8.88
10.8	1.04348	6.12	15.8	1.06479	8.93
10.9	1.04390	6.17	15.9	1.06522	8.99
11.0	1.04431	6.23	16.0	1.06566	9.04
11.1	1.04473	6.29	16.1	1.06609	9.10
11.2	1.04515	6.34	16.2	1.06653	9.16
11.3	1.04557	6.40	16.3	1.06696	9.21
11.4	1.04599	6.46	16.4	1.06740	9.27
11.5	1.04641	6.51	16.5	1.06783	9.33
11.6	1.04683	6.57	16.6	1.06827	9.38
11.7	1.04726	6.62	16.7	1.06871	9.44
11.8	1.04768	6.68	16.8	1.06914	9.49
11.9	1.04810	6.74	16.9	1.06958	9.55
12.0	1.04852	6.79	17.0	1.07002	9.61
12.1	1.04894	6.85	17.1	1.07046	9.66
12.2	1.04937	6.91	17.2	1.07090	9.72
12.3	1.04979	6.96	17.3	1.07133	9.77
12.4	1.05021	7.02	17.4	1.07177	9.83
12.5	1.05064	7.08	17.5	1.07221	9.89
12.6	1.05106	7.13	17.6	1.07265	9.94
12.7	1.05149	7.19	17.7	1.07309	10.00
12.8	1.05191	7.24	17.8	1.07358	10.06
12.9	1.05233	7.30	17.9	1.07397	10.11

Degrees Brix.	Specific Gravity.	Degrees Baumé.	Degrees Brix.	Specific Gravity.	Degrees Baumé.
18.0	1.07441	10.17	23.0	1.09686	12.96
18.1	1.07485	10.22	23.1	1.09732	13.02
18.2	1.07530	10.28	23.2	1.09777	13.07
18.3	1.07574	10.33	23.3	1.09823	13.13
18.4	1.07618	10.39	23.4	1.09869	13.19
18.5	1.07662	10.45	23.5	1.09915	13.24
18.6	1.07706	10.50	23.6	1.09961	13.30
18.7	1.07751	10.56	23.7	1.10007	13.35
18.8	1.07795	10.62	23.8	1.10053	13.41
18.9	1.07839	10.67	23.9	1.10099	13.46
19.0	1.07884	10.73	24.0	1.10145	13.52
19.1	1.07928	10.78	24.1	1.10191	13.58
19.2	1.07973	10.84	24.2	1.10237	13.63
19.3	1.08017	10.90	24.3	1.10283	13.69
19.4	1.08062	10.95	24.4	1.10329	13.74
19.5	1.08106	11.01	24.5	1.10375	13.80
19.6	1.08151	11.06	24.6	1.10421	13.85
19.7	1.08196	11.12	24.7	1.10468	13.91
19.8	1.08240	11.18	24.8	1.10514	13.96
19.9	1.08285	11.23	24.9	1.10560	14.02
20.0	1.08329	11.29	25.0	1.10607	14.08
20.1	1.08374	11.34	25.1	1.10653	14.13
20.2	1.08419	11.40	25.2	1.10700	14.19
20.3	1.08464	11.45	25.3	1.10746	14.24
20.4	1.08509	11.51	25.4	1.10793	14.30
20.5	1.08553	11.57	25.5	1.10839	14.35
20.6	1.08599	11.62	25.6	1.10886	14.41
20.7	1.08643	11.68	25.7	1.10932	14.47
20.8	1.08688	11.73	25.8	1.10979	14.52
20.9	1.08733	11.79	25.9	1.11026	14.58
21.0	1.08778	11.85	26.0	1.11072	14.63
21.1	1.08824	11.90	26.1	1.11119	14.69
21.2	1.08869	11.96	26.2	1.11166	14.74
21.3	1.08914	12.01	26.3	1.11213	14.80
21.4	1.08959	12.07	26.4	1.11259	14.85
21.5	1.09004	12.13	26.5	1.11306	14.91
21.6	1.09049	12.18	26.6	1.11353	14.97
21.7	1.09095	12.24	26.7	1.11400	15.02
21.8	1.09140	12.29	26.8	1.11447	15.08
21.9	1.09185	12.35	26.9	1.11494	15.13
22.0	1.09231	12.40	27.0	1.11541	15.19
22.1	1.09276	12.46	27.1	1.11588	15.24
22.2	1.09321	12.52	27.2	1.11635	15.30
22.3	1.09367	12.57	27.3	1.11682	15.35
22.4	1.09412	12.63	27.4	1.11729	15.41
22.5	1.09458	12.68	27.5	1.11776	15.46
22.6	1.09503	12.74	27.6	1.11824	15.52
22.7	1.09549	12.80	27.7	1.11871	15.58
22.8	1.09595	12.85	27.8	1.11918	15.63
22.9	1.09640	12.91	27.9	1.11965	15.69

Degrees Brix.	Specific Gravity.	Degrees Baumé.	Degrees Brix.	Specific Gravity.	Degrees Baumé.
28.0	1.12013	15.74	33.0	1.14423	18.50
28.1	1.12060	15.80	33.1	1.14472	18.56
28.2	1.12107	15.85	33.2	1.14521	18.61
28.3	1.12155	15.91	33.3	1.14570	18.67
28.4	1.12202	15.96	33.4	1.14620	18.72
28.5	1.12250	16.02	33.5	1.14669	18.78
28.6	1.12297	16.07	33.6	1.14718	18.83
28.7	1.12345	16.13	33.7	1.14767	18.89
28.8	1.12393	16.18	33.8	1.14817	18.94
28.9	1.12440	16.24	33.9	1.14866	19.00
29.0	1.12488	16.30	34.0	1.14915	19.05
29.1	1.12536	16.35	34.1	1.14965	19.11
29.2	1.12583	16.41	34.2	1.15014	19.16
29.3	1.12631	16.46	34.3	1.15064	19.22
29.4	1.12679	16.52	34.4	1.15113	19.27
29.5	1.12727	16.57	34.5	1.15163	19.33
29.6	1.12775	16.63	34.6	1.15213	19.38
29.7	1.12823	16.68	34.7	1.15262	19.44
29.8	1.12871	16.74	34.8	1.15312	19.49
29.9	1.12919	16.79	34.9	1.15362	19.55
30.0	1.12967	16.85	35.0	1.15411	19.60
30.1	1.13015	16.90	35.1	1.15461	19.66
30.2	1.13063	16.96	35.2	1.15511	19.71
30.3	1.13111	17.01	35.3	1.15561	19.76
30.4	1.13159	17.07	35.4	1.15611	19.82
30.5	1.13207	17.12	35.5	1.15661	19.87
30.6	1.13255	17.18	35.6	1.15710	19.93
30.7	1.13304	17.23	35.7	1.15760	19.98
30.8	1.13352	17.29	35.8	1.15810	20.04
30.9	1.13400	17.35	35.9	1.15861	20.09
31.0	1.13449	17.40	36.0	1.15911	20.15
31.1	1.13497	17.46	36.1	1.15961	20.20
31.2	1.13545	17.51	36.2	1.16011	20.26
31.3	1.13594	17.57	36.3	1.16061	20.31
31.4	1.13642	17.62	36.4	1.16111	20.37
31.5	1.13691	17.68	36.5	1.16162	20.42
31.6	1.13740	17.73	36.6	1.16212	20.48
31.7	1.13788	17.79	36.7	1.16262	20.53
31.8	1.13837	17.84	36.8	1.16313	20.59
31.9	1.13885	17.90	36.9	1.16363	20.64
32.0	1.13934	17.95	37.0	1.16413	20.70
32.1	1.13983	18.01	37.1	1.16464	20.75
32.2	1.14032	18.06	37.2	1.16514	20.80
32.3	1.14081	18.12	37.3	1.16565	20.86
32.4	1.14129	18.17	37.4	1.16616	20.91
32.5	1.14178	18.23	37.5	1.16666	20.97
32.6	1.14227	18.28	37.6	1.16717	21.02
32.7	1.14276	18.34	37.7	1.16768	21.08
32.8	1.14325	18.39	37.8	1.16818	21.13
32.9	1.14374	18.45	37.9	1.16869	21.19

Degrees Brix.	Specific Gravity.	Degrees Baumé.	Degrees Brix.	Specific Gravity.	Degrees Baumé.
38.0	1.16920	21.24	43.0	1.19505	23.96
38.1	1.16971	21.30	43.1	1.19558	24.01
38.2	1.17022	21.35	43.2	1.19611	24.07
38.3	1.17072	21.40	43.3	1.19663	24.12
38.4	1.17132	21.46	43.4	1.19716	24.17
38.5	1.17174	21.51	43.5	1.19769	24.23
38.6	1.17225	21.57	43.6	1.19822	24.28
38.7	1.17276	21.62	43.7	1.19875	24.34
38.8	1.17327	21.68	43.8	1.19927	24.39
38.9	1.17379	21.73	43.9	1.19980	24.44
39.0	1.17430	21.79	44.0	1.20033	24.50
39.1	1.17481	21.84	44.1	1.20086	24.55
39.2	1.17532	21.90	44.2	1.20139	24.61
39.3	1.17583	21.95	44.3	1.20192	24.66
39.4	1.17635	22.00	44.4	1.20245	24.71
39.5	1.17686	22.06	44.5	1.20299	24.77
39.6	1.17737	22.11	44.6	1.20352	24.82
39.7	1.17789	22.17	44.7	1.20405	24.88
39.8	1.17840	22.22	44.8	1.20458	24.93
39.9	1.17892	22.28	44.9	1.20512	24.98
40.0	1.17943	22.33	45.0	1.20565	25.04
40.1	1.17995	22.38	45.1	1.20618	25.09
40.2	1.18046	22.44	45.2	1.20672	25.14
40.3	1.18098	22.49	45.3	1.20725	25.20
40.4	1.18150	22.55	45.4	1.20779	25.25
40.5	1.18201	22.60	45.5	1.20832	25.31
40.6	1.18253	22.66	45.6	1.20886	25.36
40.7	1.18305	22.71	45.7	1.20939	25.41
40.8	1.18357	22.77	45.8	1.20993	25.47
40.9	1.18408	22.82	45.9	1.21046	25.52
41.0	1.18460	22.87	46.0	1.21100	25.57
41.1	1.18512	22.93	46.1	1.21154	25.63
41.2	1.18564	22.98	46.2	1.21208	25.68
41.3	1.18616	23.04	46.3	1.21261	25.74
41.4	1.18668	23.09	46.4	1.21315	25.79
41.5	1.18720	23.15	46.5	1.21369	25.84
41.6	1.18772	23.20	46.6	1.21423	25.90
41.7	1.18824	23.25	46.7	1.21477	25.95
41.8	1.18877	23.31	46.8	1.21531	26.00
41.9	1.18929	23.36	46.9	1.21585	26.06
42.0	1.18981	23.42	47.0	1.21639	26.11
42.1	1.19033	23.47	47.1	1.21693	26.17
42.2	1.19086	23.52	47.2	1.21747	26.22
42.3	1.19138	23.58	47.3	1.21802	26.27
42.4	1.19190	23.63	47.4	1.21856	26.33
42.5	1.19243	23.69	47.5	1.21910	26.38
42.6	1.19295	23.74	47.6	1.21964	26.43
42.7	1.19348	23.79	47.7	1.22019	26.49
42.8	1.19400	23.85	47.8	1.22073	26.54
42.9	1.19453	23.90	47.9	1.22127	26.59

Degrees Brix.	Specific Gravity.	Degrees Baumé.	Degrees Brix.	Specific Gravity.	Degrees Baumé.
48.0	1.22182	26.65	53.0	1.24951	29.31
48.1	1.22236	26.70	53.1	1.25008	29.36
48.2	1.22291	26.75	53.2	1.25064	29.42
48.3	1.22345	26.81	53.3	1.25120	29.47
48.4	1.22400	26.86	53.4	1.25177	29.52
48.5	1.22455	26.92	53.5	1.25233	29.57
48.6	1.22509	26.97	53.6	1.25290	29.63
48.7	1.22564	27.02	53.7	1.25347	29.68
48.8	1.22619	27.08	53.8	1.25403	29.73
48.9	1.22673	27.13	53.9	1.25460	29.79
49.0	1.22728	27.18	54.0	1.25517	29.84
49.1	1.22783	27.24	54.1	1.25573	29.89
49.2	1.22838	27.29	54.2	1.25630	29.94
49.3	1.22893	27.34	54.3	1.25687	30.00
49.4	1.22948	27.40	54.4	1.25744	30.05
49.5	1.23003	27.45	54.5	1.25801	30.10
49.6	1.23058	27.50	54.6	1.25857	30.16
49.7	1.23113	27.56	54.7	1.25914	30.21
49.8	1.23168	27.61	54.8	1.25971	30.26
49.9	1.23223	27.66	54.9	1.26028	30.31
50.0	1.23283	27.72	55.0	1.26086	30.37
50.1	1.23334	27.77	55.1	1.26143	30.42
50.2	1.23389	27.82	55.2	1.26200	30.47
50.3	1.23444	27.88	55.3	1.26257	30.53
50.4	1.23499	27.93	55.4	1.26314	30.58
50.5	1.23555	27.98	55.5	1.26372	30.63
50.6	1.23610	28.04	55.6	1.26429	30.68
50.7	1.23666	28.09	55.7	1.26486	30.74
50.8	1.23721	28.14	55.8	1.26544	30.79
50.9	1.23777	28.20	55.9	1.26601	30.84
51.0	1.23832	28.25	56.0	1.26658	30.89
51.1	1.23888	28.30	56.1	1.26716	30.95
51.2	1.23943	28.36	56.2	1.26773	31.00
51.3	1.23999	28.41	56.3	1.26831	31.05
51.4	1.24055	28.46	56.4	1.26889	31.10
51.5	1.24111	28.51	56.5	1.26946	31.16
51.6	1.24166	28.57	56.6	1.27004	31.21
51.7	1.24222	28.62	56.7	1.27062	31.26
51.8	1.24278	28.67	56.8	1.27120	31.31
51.9	1.24334	28.73	56.9	1.27177	31.37
52.0	1.24390	28.78	57.0	1.27235	31.42
52.1	1.24446	28.83	57.1	1.27293	31.47
52.2	1.24502	28.89	57.2	1.27351	31.52
52.3	1.24558	28.94	57.3	1.27409	31.58
52.4	1.24614	28.99	57.4	1.27467	31.63
52.5	1.24670	29.05	57.5	1.27525	31.68
52.6	1.24726	29.10	57.6	1.27583	31.73
52.7	1.24782	29.15	57.7	1.27641	31.79
52.8	1.24839	29.20	57.8	1.27699	31.84
52.9	1.24895	29.26	57.9	1.27758	31.89

Degrees Brix.	Specific Gravity.	Degrees Baumé.	Degrees Brix.	Specific Gravity.	Degrees Baumé.
58.0	1.27816	31.94	63.0	1.30777	34.54
58.1	1.27874	32.00	63.1	1.30837	34.59
58.2	1.27932	32.05	63.2	1.30897	34.65
58.3	1.27991	32.10	63.3	1.30958	34.70
58.4	1.28049	32.15	63.4	1.31018	34.75
58.5	1.28107	32.20	63.5	1.31078	34.80
58.6	1.28166	32.26	63.6	1.31139	34.85
58.7	1.28224	32.31	63.7	1.31199	34.90
58.8	1.28283	32.36	63.8	1.31260	34.96
58.9	1.28342	32.41	63.9	1.31320	35.01
59.0	1.28400	32.47	64.0	1.31381	35.06
59.1	1.28459	32.52	64.1	1.31442	35.11
59.2	1.28518	32.57	64.2	1.31502	35.16
59.3	1.28576	32.62	64.3	1.31563	35.21
59.4	1.28635	32.67	64.4	1.31624	35.27
59.5	1.28694	32.73	64.5	1.31684	35.32
59.6	1.28753	32.78	64.6	1.31745	35.37
59.7	1.28812	32.83	64.7	1.31806	35.42
59.8	1.28871	32.88	64.8	1.31867	35.47
59.9	1.28930	32.93	64.9	1.31928	35.52
60.0	1.28989	32.99	65.0	1.31989	35.57
60.1	1.29048	33.04	65.1	1.32050	35.63
60.2	1.29107	33.09	65.2	1.32111	35.68
60.3	1.29166	33.14	65.3	1.32172	35.73
60.4	1.29225	33.20	65.4	1.32233	35.78
60.5	1.29284	33.25	65.5	1.32294	35.83
60.6	1.29343	33.30	65.6	1.32355	35.88
60.7	1.29403	33.35	65.7	1.32417	35.93
60.8	1.29462	33.40	65.8	1.32478	35.98
60.9	1.29521	33.46	65.9	1.32539	36.04
61.0	1.29581	33.51	66.0	1.32601	36.09
61.1	1.29640	33.56	66.1	1.32662	36.14
61.2	1.29700	33.61	66.2	1.32724	36.19
61.3	1.29759	33.66	66.3	1.32785	36.24
61.4	1.29819	33.71	66.4	1.32847	36.29
61.5	1.29878	33.77	66.5	1.32908	36.34
61.6	1.29938	33.82	66.6	1.32970	36.39
61.7	1.29998	33.87	66.7	1.33031	36.45
61.8	1.30057	33.92	66.8	1.33093	36.50
61.9	1.30117	33.97	66.9	1.33155	36.55
62.0	1.30177	34.03	67.0	1.33217	36.60
62.1	1.30237	34.08	67.1	1.33278	36.65
62.2	1.30297	34.13	67.2	1.33340	36.70
62.3	1.30356	34.18	67.3	1.33402	36.75
62.4	1.30416	34.23	67.4	1.33464	36.80
62.5	1.30476	34.28	67.5	1.33526	36.85
62.6	1.30536	34.34	67.6	1.33588	36.90
62.7	1.30596	34.39	67.7	1.33650	36.96
62.8	1.30657	34.44	67.8	1.33712	37.01
62.9	1.30717	34.49	67.9	1.33774	37.06

Degrees Brix.	Specific Gravity.	Degrees Baumé.	Degrees Brix.	Specific Gravity.	Degrees Baumé.
68.0	1.33836	37.11	73.0	1.36995	39.64
68.1	1.33899	37.16	73.1	1.37059	39.69
68.2	1.33961	37.21	73.2	1.37124	39.74
68.3	1.34023	37.26	73.3	1.37188	39.79
68.4	1.34085	37.31	73.4	1.37252	39.84
68.5	1.34148	37.36	73.5	1.37317	39.89
68.6	1.34210	37.41	73.6	1.37381	39.94
68.7	1.34273	37.47	73.7	1.37446	39.99
68.8	1.34335	37.52	73.8	1.37510	40.04
68.9	1.34398	37.57	73.9	1.37575	40.09
69.0	1.34460	37.62	74.0	1.37639	40.14
69.1	1.34523	37.67	74.1	1.37704	40.19
69.2	1.34585	37.72	74.2	1.37768	40.24
69.3	1.34648	37.77	74.3	1.37833	40.29
69.4	1.34711	37.82	74.4	1.37898	40.34
69.5	1.34774	37.87	74.5	1.37962	40.39
69.6	1.34836	37.92	74.6	1.38027	40.44
69.7	1.34899	37.97	74.7	1.38092	40.49
69.8	1.34962	38.02	74.8	1.38157	40.54
69.9	1.35025	38.07	74.9	1.38222	40.59
70.0	1.35088	38.12	75.0	1.38287	40.64
70.1	1.35151	38.18	75.1	1.38352	40.69
70.2	1.35214	38.23	75.2	1.38417	40.74
70.3	1.35277	38.28	75.3	1.38482	40.79
70.4	1.35340	38.33	75.4	1.38547	40.84
70.5	1.35403	38.38	75.5	1.38612	40.89
70.6	1.35466	38.43	75.6	1.38677	40.94
70.7	1.35530	38.48	75.7	1.38743	40.99
70.8	1.35593	38.53	75.8	1.38808	41.04
70.9	1.35656	38.58	75.9	1.38873	41.09
71.0	1.35720	38.63	76.0	1.38939	41.14
71.1	1.35783	38.68	76.1	1.39004	41.19
71.2	1.35847	38.73	76.2	1.39070	41.24
71.3	1.35910	38.78	76.3	1.39135	41.29
71.4	1.35974	38.83	76.4	1.39201	41.33
71.5	1.36037	38.88	76.5	1.39266	41.38
71.6	1.36101	38.93	76.6	1.39332	41.43
71.7	1.36164	38.98	76.7	1.39397	41.48
71.8	1.36228	39.03	76.8	1.39463	41.53
71.9	1.36292	39.08	76.9	1.39529	41.58
72.0	1.36355	39.13	77.0	1.39595	41.63
72.1	1.36419	39.19	77.1	1.39660	41.68
72.2	1.36483	39.24	77.2	1.39726	41.73
72.3	1.36547	39.29	77.3	1.39792	41.78
72.4	1.36611	39.34	77.4	1.39858	41.83
72.5	1.36675	39.39	77.5	1.39924	41.88
72.6	1.36739	39.44	77.6	1.39990	41.93
72.7	1.36803	39.49	77.7	1.40056	41.98
72.8	1.36867	39.54	77.8	1.40122	42.03
72.9	1.36931	39.59	77.9	1.40188	42.08

Degrees Brix.	Specific Gravity.	Degrees Baumé.	Degrees Brix.	Specific Gravity.	Degrees Baumé.
78.0	1.40254	42.13	83.0	1.43614	44.58
78.1	1.40321	42.18	83.1	1.43682	44.62
78.2	1.40387	42.23	83.2	1.43750	44.67
78.3	1.40453	42.28	83.3	1.43819	44.72
78.4	1.40520	42.32	83.4	1.43887	44.77
78.5	1.40586	42.37	83.5	1.43955	44.82
78.6	1.40652	42.42	83.6	1.44024	44.87
78.7	1.40719	42.47	83.7	1.44092	44.91
78.8	1.40785	42.52	83.8	1.44161	44.96
78.9	1.40852	42.57	83.9	1.44229	45.01
79.0	1.40918	42.62	84.0	1.44298	45.06
79.1	1.40985	42.67	84.1	1.44367	45.11
79.2	1.41052	42.72	84.2	1.44435	45.16
79.3	1.41118	42.77	84.3	1.44504	45.21
79.4	1.41185	42.82	84.4	1.44573	45.25
79.5	1.41252	42.87	84.5	1.44641	45.30
79.6	1.41318	42.92	84.6	1.44710	45.35
79.7	1.41385	42.96	84.7	1.44779	45.40
79.8	1.41452	43.01	84.8	1.44848	45.45
79.9	1.41519	43.06	84.9	1.44917	45.49
80.0	1.41586	43.11	85.0	1.44986	45.54
80.1	1.41653	43.16	85.1	1.45055	45.59
80.2	1.41720	43.21	85.2	1.45124	45.64
80.3	1.41787	43.26	85.3	1.45193	45.69
80.4	1.41854	43.31	85.4	1.45262	45.74
80.5	1.41921	43.36	85.5	1.45331	45.78
80.6	1.41989	43.41	85.6	1.45401	45.83
80.7	1.42056	43.45	85.7	1.45470	45.88
80.8	1.42123	43.50	85.8	1.45539	45.93
80.9	1.42190	43.55	85.9	1.45609	45.98
81.0	1.42258	43.60	86.0	1.45678	46.02
81.1	1.42325	43.65	86.1	1.45748	46.07
81.2	1.42393	43.70	86.2	1.45817	46.12
81.3	1.42460	43.75	86.3	1.45887	46.17
81.4	1.42528	43.80	86.4	1.45956	46.22
81.5	1.42595	43.85	86.5	1.46026	46.26
81.6	1.42663	43.89	86.6	1.46095	46.31
81.7	1.42731	43.94	86.7	1.46165	46.36
81.8	1.42798	43.99	86.8	1.46235	46.41
81.9	1.42866	44.04	86.9	1.46304	46.46
82.0	1.42934	44.09	87.0	1.46374	46.50
82.1	1.43002	44.14	87.1	1.46444	46.55
82.2	1.43070	44.19	87.2	1.46514	46.60
82.3	1.43137	44.24	87.3	1.46584	46.65
82.4	1.43205	44.28	87.4	1.46654	46.69
82.5	1.43273	44.33	87.5	1.46724	46.74
82.6	1.43341	44.38	87.6	1.46794	46.79
82.7	1.43409	44.43	87.7	1.46864	46.84
82.8	1.43478	44.48	87.8	1.46934	46.88
82.9	1.43546	44.53	87.9	1.47004	46.93

Degrees Brix.	Specific Gravity.	Degrees Baumé.	Degrees Brix.	Specific Gravity.	Degrees Baumé.
88.0	1.47074	46.98	93.0	1.50635	49.34
88.1	1.47145	47.03	93.1	1.50707	49.39
88.2	1.47215	47.08	93.2	1.50779	49.43
88.3	1.47285	47.12	93.3	1.50852	49.48
88.4	1.47356	47.17	93.4	1.50924	49.53
88.5	1.47426	47.22	93.5	1.50996	49.57
88.6	1.47496	47.27	93.6	1.51069	49.62
88.7	1.47567	47.31	93.7	1.51141	49.67
88.8	1.47637	47.36	93.8	1.51214	49.71
88.9	1.47708	47.41	93.9	1.51286	49.76
89.0	1.47778	47.46	94.0	1.51359	49.81
89.1	1.47849	47.50	94.1	1.51431	49.85
89.2	1.47920	47.55	94.2	1.51504	49.90
89.3	1.47991	47.60	94.3	1.51577	49.94
89.4	1.48061	47.65	94.4	1.51649	49.99
89.5	1.48132	47.69	94.5	1.51722	50.04
89.6	1.48203	47.74	94.6	1.51795	50.08
89.7	1.48274	47.79	94.7	1.51868	50.13
89.8	1.48345	47.83	94.8	1.51941	50.18
89.9	1.48416	47.88	94.9	1.52014	50.22
90.0	1.48486	47.93	95.0	1.52087	50.27
90.1	1.48558	47.98	95.1	1.52159	50.32
90.2	1.48629	48.02	95.2	1.52232	50.36
90.3	1.48700	48.07	95.3	1.52304	50.41
90.4	1.48771	48.12	95.4	1.52376	50.45
90.5	1.48842	48.17	95.5	1.52449	50.50
90.6	1.48913	48.21	95.6	1.52521	50.55
90.7	1.48985	48.26	95.7	1.52593	50.59
90.8	1.49056	48.31	95.8	1.52665	50.64
90.9	1.49127	48.35	95.9	1.52738	50.69
91.0	1.49199	48.40	96.0	1.52810	50.73
91.1	1.49270	48.45	96.1	1.52884	50.78
91.2	1.49342	48.50	96.2	1.52958	50.82
91.3	1.49413	48.54	96.3	1.53032	50.87
91.4	1.49485	48.59	96.4	1.53106	50.92
91.5	1.49556	48.64	96.5	1.53180	50.96
91.6	1.49628	48.68	96.6	1.53254	51.01
91.7	1.49700	48.73	96.7	1.53328	51.05
91.8	1.49771	48.78	96.8	1.53402	51.10
91.9	1.49843	48.82	96.9	1.53476	51.15
92.0	1.49915	48.87	97.0	1.53550	51.19
92.1	1.49987	48.92	97.1	1.53624	51.24
92.2	1.50058	48.96	97.2	1.53698	51.28
92.3	1.50130	49.01	97.3	1.53772	51.33
92.4	1.50202	49.06	97.4	1.53846	51.38
92.5	1.50274	49.11	97.5	1.53920	51.42
92.6	1.50346	49.15	97.6	1.53994	51.47
92.7	1.50419	49.20	97.7	1.54068	51.51
92.8	1.50491	49.25	97.8	1.54142	51.56
92.9	1.50563	49.29	97.9	1.54216	51.60

Degrees Brix.	Specific Gravity.	Degrees Baumé.	Degrees Brix.	Specific Gravity.	Degrees Baumé.
98.0	1.54290	51.65	99.0	1.55040	52.11
98.1	1.54365	51.70	99.1	1.55115	52.15
98.2	1.54440	51.74	99.2	1.55189	52.20
98.3	1.54515	51.79	99.3	1.55264	52.24
98.4	1.54590	51.83	99.4	1.55338	52.29
98.5	1.54665	51.88	99.5	1.55413	52.33
98.6	1.54740	51.92	99.6	1.55487	52.38
98.7	1.54815	51.97	99.7	1.55562	52.42
98.8	1.54890	52.01	99.8	1.55636	52.47
98.9	1.54965	52.06	99.9	1.55711	52.51
			100.0	1.55785	52.56

II.

*CORRECTIONS FOR TEMPERATURE IN DETERMINATIONS
BY THE SPECIFIC GRAVITY HYDROMETER.*

(CASAMAJOR.)

II.

Normal Temperature: 15.0° C.		Normal Temperature: 17.5° C.	
Temperature in Degrees Centigrade.	Add to the Reading of the Hydrometer.	Temperature in Degrees Centigrade.	Add to the Reading of the Hydrometer.
9.90	-0.0005	7.5	-0.0010
15.00	0.0000	13.0	-0.0005
18.20	+0.0005	17.5	0.0000
20.75	0.0010	20.2	+0.0005
23.20	0.0015	23.0	0.0010
25.30	0.0020	25.0	0.0015
27.30	0.0025	27.0	0.0020
29.40	0.0030	29.0	0.0025
31.20	0.0035	31.0	0.0030
32.80	0.0040	32.5	0.0035
34.50	0.0045	34.7	0.0040
36.10	0.0050	36.2	0.0045
37.60	0.0055	37.4	0.0050
38.80	0.0060	39.0	0.0055
40.40	0.0065	40.5	0.0060
41.60	0.0070	42.0	0.0065
42.90	0.0075	43.4	0.0070
44.20	0.0080	44.2	0.0075
45.00	0.0083	45.0	0.0080

III.

*CORRECTIONS FOR TEMPERATURE IN DETERMINATIONS
BY THE BRIX HYDROMETER.*

Normal Temperature = 17.5° C.

(STAMMER.)

III.

Degree Centigrade.	DEGREE BRIX OF THE SOLUTION.												
	0	5	10	15	20	25	30	35	40	50	60	70	75
	The degree read is to be <i>decreased</i> by —												
0°	0.17	0.30	0.41	0.52	0.62	0.72	0.82	0.92	0.98	1.11	1.22	1.25	1.29
5	0.23	0.30	0.37	0.44	0.52	0.59	0.65	0.72	0.75	0.80	0.88	0.91	0.94
10	0.20	0.26	0.29	0.33	0.36	0.39	0.42	0.45	0.48	0.50	0.54	0.58	0.61
11	0.18	0.23	0.26	0.28	0.31	0.34	0.36	0.39	0.41	0.43	0.47	0.50	0.53
12	0.16	0.20	0.22	0.24	0.26	0.29	0.31	0.33	0.34	0.36	0.40	0.42	0.46
13	0.14	0.18	0.19	0.21	0.22	0.24	0.26	0.27	0.28	0.29	0.33	0.35	0.39
14	0.12	0.15	0.16	0.17	0.18	0.19	0.21	0.22	0.22	0.23	0.26	0.28	0.32
15	0.09	0.11	0.12	0.14	0.14	0.15	0.16	0.17	0.16	0.17	0.19	0.21	0.25
16	0.06	0.07	0.08	0.09	0.10	0.10	0.11	0.12	0.12	0.12	0.14	0.16	0.18
17	0.02	0.02	0.03	0.03	0.03	0.04	0.04	0.04	0.04	0.04	0.05	0.05	0.06
	The degree read is to be <i>increased</i> by —												
18	0.02	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.02
19	0.06	0.08	0.08	0.09	0.09	0.10	0.10	0.10	0.10	0.10	0.10	0.08	0.06
20	0.11	0.14	0.15	0.17	0.17	0.18	0.18	0.18	0.19	0.19	0.18	0.15	0.11
21	0.16	0.20	0.22	0.24	0.24	0.25	0.25	0.25	0.26	0.26	0.25	0.22	0.18
22	0.21	0.26	0.29	0.31	0.31	0.32	0.32	0.32	0.33	0.34	0.32	0.29	0.25
23	0.27	0.32	0.35	0.37	0.38	0.39	0.39	0.39	0.40	0.42	0.39	0.36	0.33
24	0.32	0.38	0.41	0.43	0.44	0.46	0.46	0.47	0.47	0.50	0.46	0.43	0.40
25	0.37	0.44	0.47	0.49	0.51	0.53	0.54	0.55	0.55	0.58	0.54	0.51	0.48
26	0.43	0.50	0.54	0.56	0.58	0.60	0.61	0.62	0.62	0.66	0.62	0.58	0.55
27	0.49	0.57	0.61	0.63	0.65	0.68	0.68	0.69	0.70	0.74	0.70	0.65	0.62
28	0.56	0.64	0.68	0.70	0.72	0.76	0.76	0.78	0.78	0.82	0.78	0.72	0.70
29	0.63	0.71	0.75	0.78	0.79	0.84	0.84	0.86	0.86	0.90	0.86	0.80	0.78
30	0.70	0.78	0.82	0.87	0.87	0.92	0.92	0.94	0.94	0.98	0.94	0.88	0.86
35	1.10	1.17	1.22	1.24	1.30	1.32	1.33	1.35	1.36	1.39	1.34	1.27	1.25
40	1.50	1.61	1.67	1.71	1.73	1.79	1.79	1.80	1.82	1.83	1.78	1.69	1.65
50	2.65	2.71	2.74	2.78	2.80	2.80	2.80	2.80	2.79	2.70	2.56	2.51
60	3.87	3.88	3.88	3.88	3.88	3.88	3.88	3.90	3.82	3.70	3.43	3.41
70	5.18	5.20	5.14	5.13	5.10	5.08	5.06	4.90	4.72	4.47	4.35
80	6.62	6.59	6.54	6.46	6.38	6.30	6.26	6.06	5.82	5.50	5.33

IV.

FACTORS.

Arranged for Specific Gravity Determinations.

Calculated for Wiechmann: Sugar Analysis, from the data given in Table I.

$$\text{Factor} = \frac{26.048}{\text{Degree Brix} \times \text{Specific Gravity}}$$

IV.

Specific Gravity.	Factor.	Specific Gravity.	Factor.	Specific Gravity.	Factor.	Specific Gravity.	Factor.
1.0950	1.053	1.0980	1.023	1.1010	0.990	1.1040	0.959
1.0955	1.047	1.0985	1.013	1.1015	0.985	1.1045	0.955
1.0960	1.042	1.0990	1.008	1.1020	0.981	1.1050	0.950
1.0965	1.037	1.0995	1.004	1.1025	0.976	1.1055	0.946
1.0970	1.033	1.1000	1.000	1.1030	0.972	1.1060	0.942
1.0975	1.028	1.1005	0.944	1.1035	0.968		

V.

FACTORS.

Arranged for Brix determinations.

Calculated for Wiechmann: Sugar Analysis, from the data given in Table I.

$$\text{Factor} = \frac{26.048}{\text{Degree Brix} \times \text{Specific Gravity}}$$

VI.

*ESTIMATION OF PERCENTAGE OF SUGAR BY WEIGHT
IN WEAK SUGAR SOLUTIONS*

Tucker: Manual of Sugar Analysis.

Abridged from a table calculated by

OSWALD.

VI.

Degree Brix.	Specific Gravity.	READING OF THE SACCHARIMETER.									
		1	2	3	4	5	6	7	8	9	10
0.0	1.0000	.260	.521	.781	1.042	1.302	1.563	1.823	2.084	2.344	2.605
0.5	1.0019	.260	.520	.780	1.040	1.300	1.560	1.820	2.080	2.340	2.600
1.0	1.0039	.259	.519	.778	1.038	1.297	1.557	1.816	2.076	2.335	2.595
1.5	1.0058	.259	.518	.777	1.036	1.295	1.554	1.813	2.072	2.331	2.590
2.0	1.0078	.258	.517	.775	1.034	1.292	1.551	1.809	2.068	2.326	2.585
2.5	1.0097	.258	.516	.774	1.032	1.290	1.548	1.806	2.064	2.322	2.580
3.0	1.0117	.257	.515	.772	1.029	1.287	1.545	1.802	2.060	2.317	2.575
3.5	1.0137	.257	.514	.771	1.028	1.285	1.542	1.799	2.056	2.313	2.570
4.0	1.0157	.256	.513	.769	1.026	1.282	1.539	1.795	2.052	2.308	2.565
4.5	1.0177	.256	.512	.768	1.024	1.280	1.536	1.792	2.048	2.304	2.559
5.0	1.0197	.255	.511	.766	1.022	1.277	1.533	1.788	2.044	2.299	2.554
5.5	1.0213	.255	.510	.765	1.020	1.275	1.530	1.785	2.040	2.295	2.549
6.0	1.0237	.254	.509	.763	1.018	1.272	1.527	1.781	2.036	2.290	2.544
6.5	1.0257	.254	.508	.762	1.016	1.270	1.524	1.778	2.032	2.285	2.539
7.0	1.0278	.253	.507	.760	1.014	1.267	1.521	1.774	2.027	2.281	2.534
7.5	1.0298	.253	.506	.758	1.012	1.265	1.518	1.771	2.023	2.276	2.529
8.0	1.0319	.252	.505	.757	1.010	1.262	1.515	1.767	2.019	2.272	2.524
8.5	1.0339	.252	.504	.756	1.008	1.260	1.512	1.763	2.015	2.267	2.519
9.0	1.0360	.251	.503	.754	1.006	1.257	1.509	1.760	2.011	2.263	2.514
9.5	1.0380	.251	.502	.753	1.004	1.255	1.506	1.757	2.007	2.258	2.509
10.0	1.0410	.250	.501	.751	1.002	1.252	1.503	1.753	2.003	2.254	2.504
10.5	1.0422	.250	.500	.750	1.000	1.250	1.500	1.750	1.999	2.249	2.499
11.0	1.0443	.249	.499	.748	.998	1.247	1.497	1.746	1.995	2.245	2.494
11.5	1.0464	.249	.498	.747	.996	1.245	1.494	1.743	1.991	2.240	2.489
12.0	1.0485	.248	.497	.745	.994	1.242	1.491	1.739	1.987	2.236	2.484
12.5	1.0506	.248	.496	.744	.992	1.240	1.488	1.735	1.983	2.231	2.479
13.0	1.0528	.247	.495	.742	.990	1.237	1.484	1.732	1.979	2.227	2.474
13.5	1.0549	.247	.494	.741	.988	1.235	1.482	1.728	1.975	2.222	2.469
14.0	1.0570	.246	.493	.739	.986	1.232	1.479	1.725	1.971	2.218	2.464
14.5	1.0591	.246	.492	.738	.984	1.230	1.476	1.722	1.967	2.213	2.459
15.0	1.0613	.245	.491	.736	.982	1.227	1.473	1.718	1.963	2.209	2.454
15.5	1.0635	.245	.490	.735	.980	1.225	1.470	1.714	1.959	2.204	2.449
16.0	1.0657	.244	.489	.733	.978	1.222	1.467	1.711	1.955	2.200	2.444
16.5	1.0678	.244	.488	.732	.976	1.220	1.464	1.708	1.951	2.195	2.439
17.0	1.0700	.243	.487	.730	.974	1.217	1.461	1.704	1.948	2.191	2.434
17.5	1.0722	.243	.486	.729	.972	1.215	1.458	1.701	1.944	2.186	2.429
18.0	1.0744	.242	.485	.727	.970	1.212	1.455	1.697	1.940	2.182	2.424
18.5	1.0765	.242	.484	.726	.968	1.210	1.452	1.694	1.936	2.178	2.420
19.0	1.0787	.241	.483	.724	.966	1.207	1.449	1.690	1.932	2.173	2.415
19.5	1.0810	.241	.482	.723	.964	1.205	1.446	1.687	1.928	2.169	2.410
20.0	1.0833	.240	.481	.721	.962	1.202	1.443	1.683	1.924	2.164	2.405
20.5	1.0855	.240	.480	.720	.960	1.200	1.440	1.680	1.920	2.160	2.400
21.0	1.0878	.239	.479	.718	.958	1.197	1.437	1.676	1.916	2.155	2.395
21.5	1.0900	.239	.478	.717	.956	1.195	1.434	1.673	1.912	2.151	2.390
22.0	1.0923	.238	.477	.715	.954	1.192	1.431	1.669	1.908	2.146	2.385
22.5	1.0946	.238	.476	.714	.952	1.190	1.428	1.666	1.904	2.142	2.380
23.0	1.0969	.237	.475	.712	.950	1.187	1.425	1.662	1.900	2.137	2.375

VII.

"HUNDRED POLARIZATION"

(SCHEIBLER.)

VII.

Degrees read.	Instead of 13.024 g. there must be taken		Degrees read.	Instead of 13.024 g. there must be taken		Degrees read.	Instead of 13.024 g. there must be taken	
	Grams.	Difference.		Grams.	Difference.		Grams.	Difference.
82.0	15.883	2.859	86.0	15.144	2.120	90.0	14.471	1.447
1	864	840	1	127	103	1	455	431
2	844	820	2	109	085	2	439	415
3	825	801	3	092	068	3	423	399
4	806	782	4	074	050	4	407	383
5	778	763	5	057	033	5	391	367
6	768	744	6	039	015	6	375	351
7	748	724	7	022	1.998	7	359	335
8	729	705	8	005	981	8	344	320
9	710	686	9	14.987	963	9	328	304
83.0	692	668	87.0	970	946	91.0	312	288
1	673	649	1	953	929	1	296	272
2	654	630	2	936	912	2	281	257
3	635	611	3	919	895	3	265	241
4	616	592	4	902	878	4	249	225
5	598	574	5	885	861	5	234	210
6	579	555	6	868	844	6	218	194
7	560	536	7	851	827	7	203	179
8	542	518	8	834	810	8	187	163
9	523	499	9	817	793	9	172	148
84.0	505	481	88.0	800	776	92.0	157	133
1	486	462	1	783	759	1	141	117
2	468	444	2	766	742	2	126	102
3	450	426	3	750	726	3	111	087
4	431	407	4	733	709	4	095	071
5	413	389	5	717	693	5	080	056
6	395	371	6	700	676	6	065	041
7	377	353	7	683	659	7	050	026
8	358	334	8	667	643	8	034	010
9	340	316	9	650	626	9	019	0.995
85.0	322	298	89.0	634	610	93.0	004	980
1	304	280	1	617	593	1	13.989	965
2	286	262	2	601	577	2	974	950
3	268	244	3	585	561	3	959	935
4	251	227	4	568	544	4	944	920
5	233	209	5	552	528	5	929	905
6	215	191	6	536	512	6	915	891
7	197	173	7	520	496	7	900	876
8	179	155	8	503	479	8	885	861
9	162	138	9	487	463	9	870	846

VII.

Degrees read.	Instead of 13.024 g. there must be taken		Degrees read.	Instead of 13.024 g. there must be taken		Degrees read.	Instead of 13.024 g. there must be taken	
	Grams.	Difference.		Grams.	Difference.		Grams.	Difference.
94.0	13.855	0.831	96.0	13.567	0.543	98.0	13.290	0.266
1	841	817	1	553	529	1	276	252
2	826	802	2	538	514	2	263	239
3	811	787	3	524	500	3	249	225
4	797	773	4	510	486	4	236	212
5	782	758	5	496	472	5	222	198
6	767	743	6	482	458	6	209	185
7	753	729	7	468	444	7	196	172
8	738	714	8	455	431	8	182	158
9	724	700	9	441	417	9	169	145
95.0	710	686	97.0	427	403	99.0	156	132
1	695	671	1	413	389	1	142	118
2	681	657	2	399	375	2	129	105
3	666	642	3	385	361	3	116	092
4	652	628	4	372	348	4	103	079
5	638	614	5	358	334	5	089	065
6	623	599	6	344	320	6	076	052
7	609	585	7	331	307	7	063	039
8	595	571	8	317	293	8	050	026
9	581	557	9	303	279	9	037	013
						100.0	024	000

VIII.

ESTIMATION OF PERCENTAGE OF SUGAR BY WEIGHT:

FOR USE WITH SOLUTIONS PREPARED BY ADDITION OF 1/10 VOLUME
BASIC ACETATE OF LEAD.

For Soleil-Ventzke Polariscopes.

SCHMITZ.

VIII.

PER CENT BRIX FROM 0.5 TO 12.0.		Polariscope Degrees	PER CENT BRIX AND								
Tenths of a Degree.	Per Cent Sucrose.		10.5	11.0	11.5	12.0	12.5	13.0	13.5	14.0	14.5
			1.0422	1.0443	1.0464	1.0485	1.0506	1.0528	1.0549	1.0570	1.0592
0.1	0.03	1	0.28	0.27	0.27	0.27	0.27	0.27	0.27	0.27	0.27
0.2	0.06	2	0.55	0.55	0.55	0.55	0.54	0.54	0.54	0.54	0.54
0.3	0.08	3	0.82	0.82	0.82	0.82	0.82	0.81	0.81	0.81	0.81
0.4	0.11	4	1.10	1.10	1.09	1.09	1.09	1.09	1.08	1.08	1.08
0.5	0.14	5	1.37	1.37	1.36	1.36	1.36	1.36	1.35	1.35	1.35
0.6	0.17	6	1.64	1.64	1.64	1.64	1.63	1.63	1.62	1.62	1.62
0.7	0.19	7	1.92	1.91	1.91	1.91	1.90	1.90	1.89	1.89	1.89
0.8	0.22	8	2.19	2.19	2.18	2.18	2.18	2.17	2.17	2.16	2.16
0.9	0.25	9	2.47	2.46	2.46	2.45	2.45	2.44	2.44	2.43	2.43
		10	2.74	2.74	2.73	2.73	2.72	2.71	2.71	2.70	2.70
		11	3.02	3.01	3.00	3.00	2.99	2.99	2.98	2.97	2.97
		12	3.29	3.28	3.28	3.27	3.26	3.26	3.25	3.24	3.24
		13	3.56	3.56	3.55	3.54	3.54	3.53	3.52	3.51	3.51
		14	3.84	3.83	3.82	3.82	3.81	3.80	3.79	3.78	3.78
		15	4.11	4.11	4.10	4.09	4.08	4.07	4.06	4.06	4.05
		16	4.39	4.38	4.37	4.36	4.35	4.34	4.33	4.33	4.32
		17	4.66	4.65	4.64	4.63	4.62	4.62	4.61	4.60	4.59
		18	4.93	4.93	4.91	4.91	4.90	4.89	4.88	4.87	4.86
		19	5.21	5.20	5.19	5.18	5.17	5.16	5.15	5.14	5.13
		20	5.49	5.47	5.46	5.45	5.44	5.43	5.42	5.41	5.40
0.1	0.03	21	5.76	5.75	5.74	5.73	5.71	5.70	5.69	5.68	5.67
0.2	0.05	22	6.03	6.02	6.01	6.00	5.99	5.97	5.96	5.95	5.94
0.3	0.08	23	6.31	6.30	6.28	6.27	6.26	6.24	6.23	6.22	6.21
0.4	0.11	24	6.58	6.57	6.56	6.54	6.53	6.52	6.50	6.49	6.48
0.5	0.13	25	6.86	6.84	6.83	6.82	6.80	6.79	6.78	6.76	6.75
0.6	0.16	26	7.13	7.12	7.10	7.09	7.07	7.06	7.05	7.03	7.02
0.7	0.19	27	7.41	7.39	7.38	7.36	7.35	7.33	7.32	7.30	7.29
0.8	0.21	28	7.68	7.66	7.65	7.63	7.62	7.60	7.59	7.57	7.56
0.9	0.24	29	7.96	7.94	7.92	7.91	7.89	7.87	7.86	7.84	7.83
		30	8.23	8.21	8.20	8.18	8.16	8.15	8.13	8.11	8.10
		31	8.50	8.49	8.47	8.45	8.44	8.42	8.40	8.39	8.37
		32	8.78	8.76	8.74	8.73	8.71	8.69	8.67	8.66	8.64
		33	9.05	9.03	9.02	9.00	8.98	8.96	8.94	8.93	8.91
		34	9.33	9.31	9.29	9.27	9.25	9.23	9.22	9.20	9.18
		35	9.60	9.58	9.56	9.54	9.53	9.51	9.49	9.47	9.45
		36	9.88	9.86	9.84	9.82	9.80	9.78	9.76	9.74	9.72
		37	10.15	10.13	10.11	10.09	10.07	10.05	10.03	10.01	9.99
		38		10.40	10.38	10.36	10.34	10.32	10.30	10.28	10.26
		39		10.68	10.66	10.64	10.61	10.59	10.57	10.55	10.53

VIII.

CORRESPONDING SPECIFIC GRAVITY.											Polariscope Degrees.
15.0	15.5	16.0	16.5	17.0	17.5	18.0	18.5	19.0	19.5	20.0	
1.0613	1.0635	1.0657	1.0678	1.0700	1.0722	1.0744	1.0766	1.0788	1.0811	1.0833	
0.27	0.27	0.27	0.27	0.27	0.27	0.27	0.27	0.27	0.27	0.26	1
0.54	0.54	0.54	0.54	0.53	0.53	0.53	0.53	0.53	0.53	0.53	2
0.81	0.81	0.80	0.80	0.80	0.80	0.80	0.80	0.79	0.79	0.79	3
1.08	1.08	1.07	1.07	1.07	1.07	1.06	1.06	1.06	1.06	1.06	4
1.35	1.34	1.34	1.34	1.34	1.33	1.33	1.33	1.32	1.32	1.32	5
1.62	1.61	1.61	1.61	1.60	1.60	1.60	1.59	1.59	1.59	1.58	6
1.88	1.88	1.88	1.87	1.87	1.86	1.86	1.86	1.85	1.85	1.85	7
2.15	2.15	2.15	2.14	2.14	2.13	2.13	2.12	2.12	2.12	2.11	8
2.42	2.42	2.41	2.41	2.40	2.40	2.39	2.39	2.38	2.38	2.37	9
2.69	2.69	2.68	2.68	2.67	2.67	2.66	2.65	2.65	2.64	2.64	10
2.96	2.95	2.95	2.94	2.94	2.93	2.92	2.92	2.91	2.91	2.90	11
3.23	3.22	3.22	3.21	3.20	3.20	3.19	3.18	3.18	3.17	3.17	12
3.50	3.49	3.49	3.48	3.47	3.46	3.46	3.45	3.44	3.44	3.43	13
3.77	3.76	3.75	3.75	3.74	3.73	3.72	3.72	3.71	3.70	3.69	14
4.04	4.03	4.02	4.02	4.01	4.00	3.99	3.98	3.97	3.97	3.96	15
4.31	4.30	4.29	4.28	4.27	4.26	4.26	4.25	4.24	4.23	4.22	16
4.58	4.57	4.56	4.55	4.54	4.53	4.52	4.51	4.50	4.49	4.48	17
4.85	4.84	4.83	4.82	4.81	4.80	4.79	4.78	4.77	4.76	4.75	18
5.12	5.11	5.10	5.09	5.08	5.06	5.05	5.04	5.03	5.02	5.01	19
5.39	5.38	5.36	5.35	5.34	5.33	5.32	5.31	5.30	5.29	5.28	20
5.66	5.65	5.63	5.62	5.61	5.60	5.59	5.58	5.56	5.55	5.54	21
5.93	5.91	5.90	5.89	5.88	5.87	5.85	5.84	5.83	5.82	5.80	22
6.20	6.18	6.17	6.16	6.14	6.13	6.12	6.11	6.09	6.08	6.07	23
6.46	6.45	6.44	6.43	6.41	6.40	6.39	6.37	6.36	6.35	6.33	24
6.73	6.72	6.71	6.69	6.68	6.67	6.65	6.64	6.63	6.61	6.60	25
7.00	6.99	6.97	6.96	6.95	6.93	6.92	6.90	6.89	6.88	6.86	26
7.27	7.26	7.24	7.23	7.21	7.20	7.18	7.17	7.15	7.14	7.13	27
7.54	7.53	7.51	7.50	7.48	7.47	7.45	7.44	7.42	7.40	7.39	28
7.81	7.80	7.78	7.77	7.75	7.73	7.72	7.70	7.68	7.67	7.65	29
8.08	8.06	8.05	8.03	8.02	8.00	7.98	7.97	7.95	7.93	7.92	30
8.35	8.33	8.32	8.30	8.28	8.27	8.25	8.23	8.21	8.20	8.18	31
8.62	8.60	8.58	8.57	8.55	8.53	8.51	8.50	8.48	8.46	8.45	32
8.89	8.87	8.85	8.84	8.82	8.80	8.78	8.76	8.75	8.73	8.71	33
9.16	9.14	9.12	9.10	9.09	9.07	9.05	9.03	9.01	8.99	8.97	34
9.43	9.41	9.39	9.37	9.35	9.34	9.31	9.30	9.28	9.26	9.24	35
9.70	9.68	9.66	9.64	9.62	9.60	9.58	9.56	9.54	9.52	9.50	36
9.97	9.95	9.93	9.91	9.89	9.87	9.85	9.83	9.81	9.79	9.77	37
10.24	10.22	10.20	10.18	10.15	10.13	10.11	10.09	10.07	10.05	10.03	38
10.51	10.49	10.46	10.44	10.42	10.40	10.38	10.36	10.34	10.32	10.29	39

VIII.

PER CENT BRIX FROM 11.5 TO 22.5.		Polariscope Degrees.	PER CENT BRIX AND					
Tenths of a Degree.	Per Cent Sucrose		11.5 1.0464	12.0 1.0485	12.5 1.0506	13.0 1.0528	13.5 1.0549	14.0 1.0570
		40	10.93	10.91	10.89	10.86	10.84	10.82
0.1	0.03	41		11.18	11.16	11.14	11.12	11.09
0.2	0.05	42		11.46	11.43	11.41	11.39	11.36
0.3	0.08	43			11.71	11.68	11.66	11.64
0.4	0.11	44			11.98	11.95	11.93	11.91
0.5	0.13	45			12.25	12.23	12.20	12.18
0.6	0.16	46				12.50	12.47	12.45
0.7	0.19	47					12.74	12.72
0.8	0.21	48					13.02	12.99
0.9	0.24	49						13.26
		50						
		51						
		52						
		53						
		54						
PER CENT BRIX FROM 23.0 TO 24.0.		55						
		56						
		57						
Tenths of a Degree.	Per Cent Sucrose	58						
		59						
0.1	0.03	60						
0.2	0.05	61						
0.3	0.08	62						
0.4	0.10	63						
0.5	0.13	64						
0.6	0.16	65						
0.7	0.18	66						
0.8	0.21	67						
0.9	0.23	68						
		69						
		70						
		71						
		72						
		73						
		74						
		75						
		76						
		77						
		78						
		79						
		80						

VIII.

PER CENT BRIX FROM 11.5 TO 22.5		Polariscope Degrees.	PER CENT BRIX AND					
Tenths of a degree.	Per cent Sucrose.		18.0 1.0744	18.5 1.0766	19.0 1.0788	19.5 1.0811	20.0 1.0833	20.5 1.0855
0.1	0.03	40	10.64	10.62	10.60	10.58	10.56	10.54
0.2	0.05	41	10.91	10.89	10.87	10.85	10.82	10.80
0.3	0.08	42	11.18	11.16	11.13	11.11	11.09	11.07
0.4	0.11	43	11.45	11.42	11.40	11.38	11.35	11.33
0.5	0.13	44	11.71	11.69	11.66	11.64	11.62	11.59
0.6	0.16	45	11.98	11.96	11.93	11.91	11.88	11.86
0.7	0.19	46	12.25	12.22	12.20	12.17	12.15	12.12
0.8	0.21	47	12.51	12.49	12.46	12.44	12.41	12.39
0.9	0.24	48	12.78	12.75	12.73	12.70	12.67	12.65
		49	13.05	13.02	12.99	12.97	12.94	12.91
		50	13.31	13.29	13.26	13.23	13.20	13.18
		51	13.58	13.55	13.52	13.50	13.47	13.44
		52	13.85	13.82	13.79	13.76	13.73	13.70
		53	14.11	14.08	14.05	14.03	14.00	13.97
		54	14.38	14.35	14.32	14.29	14.26	14.23
		55	14.65	14.62	14.59	14.56	14.53	14.50
		56	14.91	14.88	14.85	14.82	14.79	14.76
		57	15.18	15.15	15.12	15.09	15.06	15.02
		58	15.45	15.42	15.38	15.35	15.32	15.29
		59	15.71	15.68	15.65	15.62	15.58	15.55
		60	15.98	15.95	15.92	15.88	15.85	15.82
0.1	0.03	61	16.25	16.21	16.18	16.15	16.11	16.08
0.2	0.05	62	16.52	16.48	16.45	16.41	16.38	16.35
0.3	0.08	63	16.78	16.75	16.71	16.68	16.64	16.61
0.4	0.10	64	17.05	17.01	16.98	16.94	16.91	16.87
0.5	0.13	65	17.32	17.28	17.24	17.21	17.17	17.14
0.6	0.16	66		17.55	17.51	17.47	17.44	17.40
0.7	0.18	67		17.81	17.78	17.74	17.70	17.67
0.8	0.21	68			18.04	18.00	17.97	17.93
0.9	0.23	69			18.31	18.27	18.23	18.19
		70				18.53	18.50	18.46
		71					18.76	18.72
		72					19.03	18.99
		73						19.25
		74						19.52
		75						19.78
		76						
		77						
		78						
		79						
		80						

VIII.

CORRESPONDING SPECIFIC GRAVITY.							Polariscope Degrees.
21.0 1.0878	21.5 1.0900	22.0 1.0923	22.5 1.0946	23.0 1.0969	23.5 1.0992	24.0 1.1015	
10.52	10.49	10.47	10.45	10.43	10.41	10.38	40
10.78	10.76	10.74	10.71	10.69	10.67	10.65	41
11.04	11.02	11.00	10.97	10.95	10.93	10.90	42
11.31	11.28	11.26	11.24	11.21	11.19	11.17	43
11.57	11.55	11.52	11.50	11.47	11.45	11.42	44
11.83	11.81	11.78	11.76	11.73	11.71	11.69	45
12.09	12.07	12.05	12.02	12.00	11.97	11.94	46
12.36	12.33	12.31	12.28	12.26	12.23	12.21	47
12.62	12.60	12.57	12.54	12.52	12.49	12.47	48
12.88	12.86	12.83	12.81	12.78	12.75	12.73	49
13.15	13.12	13.09	13.07	13.04	13.01	12.99	50
13.41	13.39	13.36	13.33	13.30	13.27	13.25	51
13.68	13.65	13.62	13.59	13.56	13.53	13.51	52
13.94	13.91	13.88	13.85	13.82	13.79	13.77	53
14.20	14.17	14.14	14.11	14.08	14.06	14.02	54
14.47	14.44	14.41	14.38	14.35	14.32	14.29	55
14.73	14.70	14.67	14.64	14.61	14.58	14.55	56
14.99	14.96	14.93	14.90	14.87	14.84	14.81	57
15.26	15.23	15.19	15.16	15.13	15.10	15.07	58
15.52	15.49	15.46	15.42	15.39	15.36	15.33	59
15.78	15.75	15.72	15.69	15.65	15.62	15.59	60
16.05	16.01	15.98	15.95	15.91	15.88	15.85	61
16.31	16.28	16.24	16.21	16.18	16.14	16.11	62
16.57	16.54	16.51	16.47	16.44	16.40	16.37	63
16.84	16.80	16.77	16.73	16.70	16.66	16.63	64
17.10	17.07	17.03	17.00	16.96	16.92	16.89	65
17.37	17.33	17.29	17.26	17.22	17.19	17.15	66
17.63	17.59	17.56	17.52	17.48	17.45	17.41	67
17.89	17.86	17.82	17.78	17.74	17.71	17.67	68
18.16	18.12	18.08	18.04	18.00	17.97	17.93	69
18.42	18.38	18.35	18.31	18.27	18.23	18.19	70
18.68	18.65	18.61	18.57	18.53	18.49	18.45	71
18.95	18.91	18.87	18.83	18.79	18.75	18.71	72
19.21	19.17	19.13	19.09	19.05	19.01	18.97	73
19.48	19.44	19.40	19.35	19.31	19.27	19.23	74
19.74	19.70	19.66	19.62	19.57	19.53	19.49	75
20.00	19.96	19.92	19.88	19.84	19.80	19.75	76
20.27	20.22	20.18	20.14	20.10	20.06	20.01	77
	20.49	20.45	20.40	20.36	20.32	20.27	78
	20.75	20.71	20.66	20.62	20.58	20.54	79
		20.97	20.93	20.88	20.84	20.80	80

IX.

POUNDS SOLIDS PER CUBIC FOOT IN SUGAR SOLUTIONS

Calculated for Wiechmann, Sugar Analysis, from the following data taken from Everett: Physical Units and Constants.

1 cubic centimeter of water at 17.5° C. weighs 0.9987605 gram.

1 cubic foot = 28316 cubic centimeters.

1 kilogram = 2.2046212 lbs.

Hence 1 cubic foot of water at 17.5° C. weighs 62.3487 lbs.

FORMULÆ.

- I. $62.3487 \times \text{Specific Gravity of Sugar solution.}$
II. $\frac{\text{Result obtained by I.} \times \text{Degree Brix}}{100}$
= Pounds Solids per Cubic Foot.

IX.

Degree Baumé.	Degree Brix.	Specific Gravity.	Lbs. solids in 1 cu. ft.	Degree Baumé.	Degree Brix.	Specific Gravity.	Lbs. solids in 1 cu. ft.
0.0	0.0	1.00000	0.000	26.5	47.7	1.22019	36.289
0.5	0.9	1.00349	0.563	27.0	48.7	1.22564	37.215
1.0	1.8	1.00701	1.130	27.5	49.6	1.23058	38.056
1.5	2.6	1.01015	1.638	28.0	50.5	1.23555	38.903
2.0	3.5	1.01371	2.212	28.5	51.5	1.24111	39.852
2.5	4.4	1.01730	2.791	29.0	52.4	1.24614	40.712
3.0	5.3	1.02091	3.374	29.5	53.4	1.25177	41.677
3.5	6.2	1.02454	3.960	30.0	54.3	1.25687	42.552
4.0	7.0	1.02779	4.486	30.5	55.2	1.26200	43.434
4.5	7.9	1.03146	5.081	31.0	56.2	1.26773	44.421
5.0	8.8	1.03517	5.680	31.5	57.2	1.27351	45.418
5.5	9.7	1.03889	6.283	32.0	58.1	1.27874	46.322
6.0	10.6	1.04264	6.891	32.5	59.1	1.28459	47.335
6.5	11.5	1.04641	7.503	33.0	60.0	1.28989	48.254
7.0	12.4	1.05021	8.119	33.5	61.0	1.29581	49.283
7.5	13.2	1.05361	8.671	34.0	61.9	1.30117	50.217
8.0	14.1	1.05746	9.296	34.5	62.9	1.30717	51.264
8.5	15.0	1.06133	9.926	35.0	63.9	1.31320	52.319
9.0	15.9	1.06522	10.560	35.5	64.9	1.31928	53.384
9.5	16.8	1.06914	11.199	36.0	65.8	1.32478	54.350
10.0	17.7	1.07309	11.842	36.5	66.8	1.33093	55.432
10.5	18.6	1.07706	12.491	37.0	67.8	1.33712	56.523
11.0	19.5	1.08106	13.144	37.5	68.8	1.34335	57.624
11.5	20.4	1.08509	13.801	38.0	69.8	1.34962	58.735
12.0	21.3	1.08914	14.464	38.5	70.7	1.35530	59.742
12.5	22.2	1.09321	15.132	39.0	71.7	1.36164	60.871
13.0	23.1	1.09732	15.804	39.5	72.7	1.36803	62.009
13.5	24.0	1.10145	16.482	40.0	73.7	1.37446	63.158
14.0	24.9	1.10560	17.164	40.5	74.7	1.38092	64.316
14.5	25.8	1.10979	17.852	41.0	75.7	1.38743	65.484
15.0	26.7	1.11400	18.545	41.5	76.7	1.39397	66.662
15.5	27.6	1.11824	19.243	42.0	77.7	1.40056	67.850
16.0	28.5	1.12250	19.946	42.5	78.8	1.40785	69.169
16.5	29.4	1.12679	20.655	43.0	79.8	1.41452	70.378
17.0	30.3	1.13111	21.369	43.5	80.8	1.42123	71.598
17.5	31.2	1.13545	22.088	44.0	81.8	1.42798	72.829
18.0	32.1	1.13983	22.812	44.5	82.8	1.43478	74.070
18.5	33.0	1.14423	23.543	45.0	83.9	1.44229	75.447
19.0	33.9	1.14866	24.278	45.5	84.9	1.44917	76.710
19.5	34.8	1.15312	25.020	46.0	85.9	1.45609	77.985
20.0	35.7	1.15760	25.766	46.5	87.0	1.46374	79.398
20.5	36.6	1.16212	26.519	47.0	88.0	1.47074	80.695
21.0	37.6	1.16717	27.362	47.5	89.1	1.47849	82.134
21.5	38.5	1.17174	28.127	48.0	90.1	1.48558	83.454
22.0	39.4	1.17635	28.897	48.5	91.2	1.49342	84.919
22.5	40.3	1.18098	29.674	49.0	92.3	1.50130	86.397
23.0	41.2	1.18564	30.456	49.5	93.3	1.50852	87.753
23.5	42.2	1.19086	31.333	50.0	94.4	1.51649	89.256
24.0	43.1	1.19558	32.128	50.5	95.5	1.52449	90.773
24.5	44.0	1.20033	32.929	51.0	96.6	1.53254	92.303
25.0	44.9	1.20512	33.737	51.5	97.7	1.54068	93.850
25.5	45.9	1.21046	34.641	52.0	98.8	1.54890	95.413
26.0	46.8	1.21531	35.462	52.5	99.9	1.55711	96.987

X.

*FACTORS FOR THE CALCULATION OF CLERGET
INVERSIONS.*

Calculated for Wiechmann, Sugar Analysis, by the
formula:

$$\text{Factor} = \frac{100}{142.66 - \frac{t}{2}}$$

X.

Temperature.	Factor.	Temperature.	Factor.
10°	0.7257	21°	0.7567
11	0.7291	22	0.7595
12	0.7317	23	0.7624
13	0.7344	24	0.7653
14	0.7371	25	0.7683
15	0.7397	26	0.7712
16	0.7426	27	0.7742
17	0.7454	28	0.7772
18	0.7482	29	0.7802
19	0.7510	30	0.7833
20	0.7538		

XI.

DETERMINATION OF TOTAL SUGAR.

German Government: Law of July 9, 1887.

XI.

Mgr. Sucrose.	Mgr. Copper.	Mgr. Sucrose.	Mgr. Copper.	Mgr. Sucrose.	Mgr. Copper.	Mgr. Sucrose.	Mgr. Copper.
40	79.0	73	145.2	106	208.6	139	269.1
41	81.0	74	147.1	107	210.5	140	270.9
42	83.0	75	149.1	108	212.3	141	272.7
43	85.2	76	151.0	109	214.2	142	274.5
44	87.2	77	153.0	110	216.1	143	276.3
45	89.2	78	155.0	111	217.9	144	278.1
46	91.2	79	156.9	112	219.8	145	279.9
47	93.3	80	158.9	113	221.6	146	281.6
48	95.3	81	160.8	114	223.5	147	283.4
49	97.3	82	162.8	115	225.3	148	285.2
50	99.3	83	164.7	116	227.2	149	286.9
51	101.3	84	166.6	117	229.0	150	288.8
52	103.3	85	168.6	118	230.9	151	290.5
53	105.3	86	170.5	119	232.8	152	292.3
54	107.3	87	172.4	120	234.6	153	294.0
55	109.4	88	174.3	121	236.4	154	295.7
56	111.4	89	176.3	122	238.3	155	297.5
57	113.4	90	178.2	123	240.2	156	299.2
58	115.4	91	180.1	124	242.0	157	300.9
59	117.4	92	182.0	125	243.9	158	302.6
60	119.5	93	183.9	126	245.7	159	304.4
61	121.5	94	185.8	127	247.5	160	306.1
62	123.5	95	187.8	128	249.3	161	307.8
63	125.4	96	189.7	129	251.2	162	309.5
64	127.4	97	191.6	130	252.9	163	311.3
65	129.4	98	193.5	131	254.7	164	313.0
66	131.4	99	195.4	132	256.5	165	314.7
67	133.4	100	197.3	133	258.3	166	316.4
68	135.3	101	199.2	134	260.1	167	318.1
69	137.3	102	201.1	135	261.9	168	319.9
70	139.3	103	202.9	136	263.7	169	321.6
71	141.3	104	204.8	137	265.5	170	323.3
72	143.2	105	206.7	138	267.3		

XII.

DETERMINATION OF INVERT SUGAR VOLUMETRIC METHOD.

(Using Fehling's Solution.)

5 grams to 100 cubic centimeters.

Divide 1.00 by the number of cubic centimeters used of above solution and multiply result by 100.

XII.

Number of c.c. used.	Per Cent of Invert Sugar.	Number of c.c. used.	Per Cent of Invert Sugar.	Number of c.c. used.	Per Cent of Invert Sugar.	Number of c.c. used.	Per Cent of Invert Sugar.
1	100.00	26	3.85	51	1.96	76	1.32
2	50.00	27	3.70	52	1.92	77	1.30
3	33.33	28	3.57	53	1.89	78	1.28
4	25.00	29	3.45	54	1.85	79	1.27
5	20.00	30	3.33	55	1.82	80	1.25
6	16.67	31	3.23	56	1.79	81	1.23
7	14.29	32	3.13	57	1.75	82	1.22
8	12.50	33	3.03	58	1.72	83	1.20
9	11.11	34	2.94	59	1.69	84	1.19
10	10.00	35	2.86	60	1.67	85	1.18
11	9.09	36	2.78	61	1.64	86	1.16
12	8.33	37	2.70	62	1.61	87	1.15
13	7.69	38	2.63	63	1.59	88	1.14
14	7.14	39	2.56	64	1.56	89	1.12
15	6.67	40	2.50	65	1.54	90	1.11
16	6.25	41	2.44	66	1.52	91	1.10
17	5.88	42	2.38	67	1.49	92	1.09
18	5.55	43	2.33	68	1.47	93	1.08
19	5.26	44	2.27	69	1.45	94	1.06
20	5.00	45	2.22	70	1.43	95	1.05
21	4.76	46	2.17	71	1.41	96	1.04
22	4.55	47	2.13	72	1.39	97	1.03
23	4.35	48	2.08	73	1.37	98	1.02
24	4.17	49	2.04	74	1.35	99	1.01
25	4.00	50	2.00	75	1.33	100	1.00

XIII.

*DETERMINATION OF INVERT-SUGAR.
GRAVIMETRIC METHOD.*

(Using Fehling's Solution.)

HERZFELD, HILLER. MEISSL.

XIII.

<i>S : I.</i>	<i>Z = 200</i> mgr.	175 mgr.	150 mgr.	125 mgr.	100 mgr.	75 mgr.	50 mgr.
0 : 100	56.4	55.4	54.5	53.8	53.2	53.0	53.0
10 : 90	56.3	55.3	54.4	53.8	53.2	52.9	52.9
20 : 80	56.2	55.2	54.3	53.7	53.2	52.7	52.7
30 : 70	56.1	55.1	54.2	53.7	53.2	52.6	52.6
40 : 60	55.9	55.0	54.1	53.6	53.1	52.5	52.4
50 : 50	55.7	54.9	54.0	53.5	53.1	52.3	52.2
60 : 40	55.6	54.7	53.8	53.2	52.8	52.1	51.9
70 : 30	55.5	54.5	53.5	52.9	52.5	51.9	51.6
80 : 20	55.4	54.3	53.3	52.7	52.2	51.7	51.3
90 : 10	54.6	53.6	53.1	52.6	52.1	51.6	51.2
91 : 9	54.1	53.6	52.6	52.1	51.6	51.2	50.7
92 : 8	53.6	53.1	52.1	51.6	51.2	50.7	50.3
93 : 7	53.6	53.1	52.1	51.2	50.7	50.3	49.8
94 : 6	53.1	52.6	51.6	50.7	50.3	49.8	48.9
95 : 5	52.6	52.1	51.2	50.3	49.4	48.9	48.5
96 : 4	52.1	51.2	50.7	49.8	48.9	47.7	46.9
97 : 3	50.7	50.3	49.8	48.9	47.7	46.2	45.1
98 : 2	49.9	48.9	48.5	47.3	45.8	43.3	40.0
99 : 1	47.7	47.3	46.5	45.1	43.3	41.2	38.1

XIV.

SOLUBILITY OF SUCROSE IN WATER.

(From 0° to 100° C.)

HERZFELD.

XIV.

Temp. ° C.	Percentage Sucrose.	Temp. ° C.	Percentage Sucrose.	Temp. ° C.	Percentage Sucrose.
0	64.18	34	69.38	68	75.80
1	64.31	35	69.55	69	76.01
2	64.45	36	69.72	70	76.22
3	64.59	37	69.89	71	76.43
4	64.73	38	70.06	72	76.64
5	64.87	39	70.24	73	76.85
6	65.01	40	70.42	74	77.06
7	65.15	41	70.60	75	77.27
8	65.29	42	70.78	76	77.48
9	65.43	43	70.96	77	77.70
10	65.58	44	71.14	78	77.92
11	65.73	45	71.32	79	78.14
12	65.88	46	71.50	80	78.36
13	66.03	47	71.68	81	78.58
14	66.18	48	71.87	82	78.80
15	66.33	49	72.06	83	79.02
16	66.48	50	72.25	84	79.24
17	66.63	51	72.44	85	79.46
18	66.78	52	72.63	86	79.69
19	66.93	53	72.82	87	79.92
20	67.09	54	73.01	88	80.15
21	67.25	55	73.20	89	80.38
22	67.41	56	73.39	90	80.61
23	67.57	57	73.58	91	80.84
24	67.73	58	73.78	92	81.07
25	67.89	59	73.98	93	81.30
26	68.05	60	74.18	94	81.53
27	68.21	61	74.38	95	81.77
28	68.37	62	74.58	96	82.01
29	68.53	63	74.78	97	82.25
30	68.70	64	74.98	98	82.49
31	68.87	65	75.18	99	82.73
32	69.04	66	75.38	100	82.79
33	69.21	67	75.59		

XV.

DETERMINATION OF DEXTROSE.

From E. Wein, Tabellen zur Quantitativen Bestimmung
der Zuckerarten.

F. ALLIHN.

XV.

Mgr. Copper.	Mgr. Dextrose.	Mgr. Copper.	Mgr. Dextrose.	Mgr. Copper.	Mgr. Dextrose.	Mgr. Copper.	Mgr. Dextrose.
10	6.1	58	29.8	106	54.0	154	78.6
11	6.6	59	30.3	107	54.5	155	79.1
12	7.1	60	30.8	108	55.0	156	79.6
13	7.6	61	31.3	109	55.5	157	80.1
14	8.1	62	31.8	110	56.0	158	80.7
15	8.6	63	32.3	111	56.5	159	81.2
16	9.0	64	32.8	112	57.0	160	81.7
17	9.5	65	33.3	113	57.5	161	82.2
18	10.0	66	33.8	114	58.0	162	82.7
19	10.5	67	34.3	115	58.6	163	83.3
20	11.0	68	34.8	116	59.1	164	83.8
21	11.5	69	35.3	117	59.6	165	84.3
22	12.0	70	35.8	118	60.1	166	84.8
23	12.5	71	36.3	119	60.6	167	85.3
24	13.0	72	36.8	120	61.1	168	85.9
25	13.5	73	37.3	121	61.6	169	86.4
26	14.0	74	37.8	122	62.1	170	86.9
27	14.5	75	38.3	123	62.6	171	87.4
28	15.0	76	38.8	124	63.1	172	87.9
29	15.5	77	39.3	125	63.7	173	88.5
30	16.0	78	39.8	126	64.2	174	89.0
31	16.5	79	40.3	127	64.7	175	89.5
32	17.0	80	40.8	128	65.2	176	90.0
33	17.5	81	41.3	129	65.7	177	90.5
34	18.0	82	41.8	130	66.2	178	91.1
35	18.5	83	42.3	131	66.7	179	91.6
36	18.9	84	42.8	132	67.2	180	92.1
37	19.4	85	43.4	133	67.7	181	92.6
38	19.9	86	43.9	134	68.2	182	93.1
39	20.4	87	44.4	135	68.8	183	93.7
40	20.9	88	44.9	136	69.3	184	94.2
41	21.4	89	45.4	137	69.8	185	94.7
42	21.9	90	45.9	138	70.3	186	95.2
43	22.4	91	46.4	139	70.8	187	95.7
44	22.9	92	46.9	140	71.3	188	96.3
45	23.4	93	47.4	141	71.8	189	96.8
46	23.9	94	47.9	142	72.3	190	97.3
47	24.4	95	48.4	143	72.9	191	97.8
48	24.9	96	48.9	144	73.4	192	98.4
49	25.4	97	49.4	145	73.9	193	98.9
50	25.9	98	49.9	146	74.4	194	99.4
51	26.4	99	50.4	147	74.9	195	100.0
52	26.9	100	50.9	148	75.5	196	100.5
53	27.4	101	51.4	149	76.0	197	101.0
54	27.9	102	51.9	150	76.5	198	101.5
55	28.4	103	52.4	151	77.0	199	102.0
56	28.8	104	52.9	152	77.5	200	102.6
57	29.3	105	53.5	153	78.1	201	103.2

XV.

Mgr. Copper.	Mgr. Dextrose.	Mgr. Copper.	Mgr. Dextrose.	Mgr. Copper.	Mgr. Dextrose.	Mgr. Copper.	Mgr. Dextrose.
202	103.7	250	129.2	298	155.4	346	182.1
203	104.2	251	129.7	299	156.0	347	182.6
204	104.7	252	130.3	300	156.5	348	183.2
205	105.3	253	130.8	301	157.1	349	183.7
206	105.8	254	131.4	302	157.6	350	184.3
207	106.3	255	131.9	303	158.2	351	184.9
208	106.8	256	132.4	304	158.7	352	185.4
209	107.4	257	133.0	305	159.3	353	186.0
210	107.9	258	133.5	306	159.8	354	186.6
211	108.4	259	134.1	307	160.4	355	187.2
212	109.0	260	134.6	308	160.9	356	187.7
213	109.5	261	135.1	309	161.5	357	188.3
214	110.0	262	135.7	310	162.0	358	188.9
215	110.6	263	136.2	311	162.6	359	189.4
216	111.1	264	136.8	312	163.1	360	190.0
217	111.6	265	137.3	313	163.7	361	190.6
218	112.1	266	137.8	314	164.2	362	191.1
219	112.7	267	138.4	315	164.8	363	191.7
220	113.2	268	138.9	316	165.3	364	192.3
221	113.7	269	139.5	317	165.9	365	192.9
222	114.3	270	140.0	318	166.4	366	193.4
223	114.8	271	140.6	319	167.0	367	194.0
224	115.3	272	141.1	320	167.5	368	194.6
225	115.9	273	141.7	321	168.1	369	195.1
226	116.4	274	142.2	322	168.6	370	195.7
227	116.9	275	142.8	323	169.2	371	196.3
228	117.4	276	143.3	324	169.7	372	196.8
229	118.0	277	143.9	325	170.3	373	197.4
230	118.5	278	144.4	326	170.9	374	198.0
231	119.0	279	145.0	327	171.4	375	198.6
232	119.6	280	145.5	328	172.0	376	199.1
233	120.1	281	146.1	329	172.5	377	199.7
234	120.7	282	146.6	330	173.1	378	200.3
235	121.2	283	147.2	331	173.7	379	200.8
236	121.7	284	147.7	332	174.2	380	201.4
237	122.3	285	148.3	333	174.8	381	202.0
238	122.8	286	148.8	334	175.3	382	202.5
239	123.4	287	149.4	335	175.9	383	203.1
240	123.9	288	149.9	336	176.5	384	203.7
241	124.4	289	150.5	337	177.0	385	204.3
242	125.0	290	151.0	338	177.6	386	204.8
243	125.5	291	151.6	339	178.1	387	205.4
244	126.0	292	152.1	340	178.7	388	206.0
245	126.6	293	152.7	341	179.3	389	206.5
246	127.1	294	153.2	342	179.8	390	207.1
247	127.6	295	153.8	343	180.4	391	207.7
248	128.1	296	154.3	344	180.9	392	208.3
249	128.7	297	154.9	345	181.5	393	208.8

XV.

Mgr. Copper.	Mgr. Dextrose.	Mgr. Copper.	Mgr. Dextrose.	Mgr. Copper.	Mgr. Dextrose.	Mgr. Copper.	Mgr. Dextrose.
394	209.4	412	219.9	430	230.4	447	240.4
395	210.0	413	220.4	431	231.0	448	241.0
396	210.6	414	221.0	432	231.6	449	241.6
397	211.2	415	221.6	433	232.2	450	242.2
398	211.7	416	222.2	434	232.8	451	242.8
399	212.3	417	222.8	435	233.4	452	243.4
400	212.9	418	223.3	436	233.9	453	244.0
401	213.5	419	223.9	437	234.5	454	244.6
402	214.1	420	224.5	438	235.1	455	245.2
403	214.6	421	225.1	439	235.7	456	245.7
404	215.2	422	225.7	440	236.3	457	246.3
405	215.8	423	226.3	441	236.9	458	246.9
406	216.4	424	226.9	442	237.5	459	247.5
407	217.0	425	227.5	443	238.1	460	248.1
408	217.5	426	228.0	444	238.7	461	248.7
409	218.1	427	228.6	445	239.3	462	249.3
410	218.7	428	229.2	446	239.8	463	249.9
411	219.3	429	229.8				

XVI

DETERMINATION OF LEVULOSE.

HÖNIG AND JESSER.

XVI.

Mgr. Levulose.	Mgr. Copper.	Mgr. Levulose.	Mgr. Copper.	Mgr. Levulose.	Mgr. Copper.
10	13.73	95	170.03	180	315.33
15	23.23	100	178.88	185	323.53
20	32.69	105	187.69	190	331.67
25	42.12	110	196.47	195	339.81
30	51.50	115	205.25	200	347.91
35	60.85	120	213.90	205	355.97
40	70.15	125	222.56	210	363.99
45	79.42	130	231.19	215	371.98
50	88.65	135	239.77	220	379.92
55	97.85	140	248.32	225	387.83
60	107.10	145	256.84	230	395.70
65	116.12	150	265.32	235	403.53
70	125.20	155	273.76	240	411.32
75	134.24	160	282.16	245	419.03
80	143.24	165	290.48	250	426.73
85	152.22	170	298.85		
90	161.14	175	307.09		

XVII.

*DENSITY OF WATER AT THE TEMPERATURES FROM 0°
TO 50° CENTIGRADE, RELATIVE TO ITS DENSITY AT
4° CENTIGRADE.*

ROSETTI.

Based on results obtained by Kopp, Despretz, Hagen,
Matthiessen, Rosetti.

XVII.

Temperature: Degrees Centi- grade.	Density of Water relative to its Density at 4° C.	Temperature: Degrees Centi- grade.	Density of Water relative to its Density at 4° C.
0°	0.99987	25°	0.99712
1	0.99993	26	0.99687
2	0.99997	27	0.99660
3	0.99999	28	0.99633
4	1.00000	29	0.99605
5	0.99999	30	0.99577
6	0.99997	31	0.99547
7	0.99993	32	0.99517
8	0.99989	33	0.99485
9	0.99982	34	0.99452
10	0.99975	35	0.99418
11	0.99966	36	0.99383
12	0.99955	37	0.99347
13	0.99943	38	0.99310
14	0.99930	39	0.99273
15	0.99916	40	0.99235
16	0.99900	41	0.99197
17	0.99884	42	0.99158
18	0.99865	43	0.99118
19	0.99846	44	0.99078
20	0.99826	45	0.99037
21	0.99805	46	0.98996
22	0.99783	47	0.98954
23	0.99760	48	0.98910
24	0.99737	49	0.98865
		50	0.98819

XVIII.

TABLE OF FACTORS.

MORSE.

XVIII.

Purity of Molasses.	Polarization of Sugar.			
	100	99.5	99.0	98.5
93.0	14.30	15.40	16.40	17.60
92.8	13.90	14.95	15.90	17.00
92.6	13.50	14.50	15.40	16.45
92.4	13.15	14.10	14.95	15.95
92.2	12.80	13.70	14.50	15.45
92.0	12.50	13.35	14.10	15.00
91.8	12.20	13.00	13.70	14.55
91.6	11.90	12.65	13.35	14.15
91.4	11.65	12.30	13.05	13.75
91.2	11.35	12.05	12.65	13.40
91.0	11.10	11.75	12.35	13.05
90.8	10.85	11.50	12.05	12.70
90.6	10.65	11.25	11.75	12.40
90.4	10.40	11.00	11.50	12.10
90.2	10.20	10.70	11.25	11.80
90.0	10.00	10.55	11.00	11.55
89.8	9.80	10.30	10.75	11.25
89.6	9.60	10.10	10.55	11.00
89.4	9.45	10.00	10.30	10.80
89.2	9.25	9.90	10.10	10.55
89.0	9.10	9.70	9.90	10.35
88.8	8.95	9.50	9.70	10.15
88.6	8.75	9.35	9.55	9.95
88.4	8.60	9.15	9.35	9.75
88.2	8.45	9.00	9.20	9.55
88.0	8.35	8.85	9.00	9.35
87.8	8.20	8.70	8.85	9.20
87.6	8.05	8.55	8.70	9.05
87.4	7.95	8.40	8.55	8.85
87.2	7.80	8.25	8.40	8.70
87.0	7.70	8.15	8.25	8.55
86.8	7.60	8.00	8.15	8.40
86.6	7.45	7.85	8.00	8.30
86.4	7.35	7.75	7.90	8.15
86.2	7.25	7.65	7.75	8.00
86.0	7.15	7.50	7.65	7.90
85.8	7.05	7.40	7.50	7.80
85.6	6.95	7.30	7.40	7.65
85.4	6.85	7.20	7.30	7.55
85.2	6.75	7.10	7.20	7.45
85.0	6.65	7.00	7.05	7.32

XIX.

COMPARISON OF THERMOMETRIC SCALES.

$$F = \frac{9}{5}C + 32 = \frac{9}{4}R + 32.$$

$$C = \frac{5}{9}(F - 32) = \frac{5}{4}R.$$

$$R = \frac{4}{9}(F - 32) = \frac{4}{5}C.$$

XIX.
CENTIGRADE, FAHRENHEIT, RÉAUMUR.

Centi- grade.	Fahren- heit.	Réaumur.	Centi- grade.	Fahren- heit.	Réaumur.	Centi- grade.	Fahren- heit.	Réaumur.
°	°	°	°	°	°	°	°	°
100	212	80	53	127.4	42.4	6	42.8	4.8
99	210.2	79.2	52	125.6	41.6	5	41	4
98	208.4	78.4	51	123.8	40.8	4	39.2	3.2
97	206.6	77.6	50	122	40	3	37.4	2.4
96	204.8	76.8	49	120.2	39.2	2	35.6	1.6
95	203	76	48	118.4	38.4	1	33.8	0.8
94	201.2	75.2	47	116.6	37.6	0	32	0
93	199.4	74.4	46	114.8	36.8	-1	30.2	-0.8
92	197.6	73.6	45	113	36	-2	28.4	-1.6
91	195.8	72.8	44	111.2	35.2	-3	26.6	-2.4
90	194	72	43	109.4	34.4	-4	24.8	-3.2
89	192.2	71.2	42	107.6	33.6	-5	23	-4
88	190.4	70.4	41	105.8	32.8	-6	21.2	-4.8
87	188.6	69.6	40	104	32	-7	19.4	-5.6
86	186.8	68.8	39	102.2	31.2	-8	17.6	-6.4
85	185	68	38	100.4	30.4	-9	15.8	-7.2
84	183.2	67.2	37	98.6	29.6	-10	14	-8
83	181.4	66.4	36	96.8	28.8	-11	12.2	-8.8
82	179.6	65.6	35	95	28	-12	10.4	-9.6
81	177.8	64.8	34	93.2	27.2	-13	8.6	-10.4
80	176	64	33	91.4	26.4	-14	6.8	-11.2
79	174.2	63.2	32	89.6	25.6	-15	5	-12
78	172.4	62.4	31	87.8	24.8	-16	3.2	-12.8
77	170.6	61.6	30	86	24	-17	1.4	-13.6
76	168.8	60.8	29	84.2	23.2	-18	0.4	-14.4
75	167	60	28	82.4	22.4	-19	-2.2	-15.2
74	165.2	59.2	27	80.6	21.6	-20	-4	-16
73	163.4	58.4	26	78.8	20.8	-21	-5.8	-16.8
72	161.6	57.6	25	77	20	-22	-7.6	-17.6
71	159.8	56.8	24	75.2	19.2	-23	-9.4	-18.4
70	158	56	23	73.4	18.4	-24	-11.2	-19.2
69	156.2	55.2	22	71.6	17.6	-25	-13	-20
68	154.4	54.4	21	69.8	16.8	-26	-14.8	-20.8
67	152.6	53.6	20	68	16	-27	-16.6	-21.6
66	150.8	52.8	19	66.2	15.2	-28	-18.4	-22.4
65	149	52	18	64.4	14.4	-29	-20.2	-23.2
64	147.2	51.2	17	62.6	13.6	-30	-22	-24
63	145.4	50.4	16	60.8	12.8	-31	-23.8	-24.8
62	143.6	49.6	15	59	12	-32	-25.6	-25.6
61	141.8	48.8	14	57.2	11.2	-33	-27.4	-26.4
60	140	48	13	55.4	10.4	-34	-29.2	-27.2
59	138.2	47.2	12	53.6	9.6	-35	-31	-28
58	136.4	46.4	11	51.8	8.8	-36	-32.8	-28.8
57	134.6	45.6	10	50	8	-37	-34.6	-29.6
56	132.8	44.8	9	48.2	7.2	-38	-36.4	-30.4
55	131	44	8	46.4	6.4	-39	-38.2	-31.2
54	129.2	43.2	7	44.6	5.6	-40	-40	-32

XIX.

FAHRENHEIT, CENTIGRADE, RÉAUMUR.

Fahren- heit.	Centi- grade.	Réaumur.	Fahren- heit.	Centi- grade.	Réaumur.	Fahren- heit.	Centi- grade.	Réaumur.
°	°	°	°	°	°	°	°	°
212	100	80	165	73.89	59.11	118	47.78	38.22
211	99.44	79.56	164	73.33	58.67	117	47.22	37.78
210	98.89	79.11	163	72.78	58.22	116	46.67	37.33
209	98.33	78.67	162	72.22	57.78	115	46.11	36.89
208	97.78	78.22	161	71.67	57.33	114	45.55	36.44
207	97.22	77.78	160	71.11	56.89	113	45	36
206	96.67	77.33	159	70.55	56.44	112	44.44	35.56
205	96.11	76.89	158	70	56	111	43.89	35.11
204	95.55	76.44	157	69.44	55.56	110	43.33	34.67
203	95	76	156	68.89	55.11	109	42.78	34.22
202	94.44	75.56	155	68.33	54.67	108	42.22	33.78
201	93.89	75.11	154	67.78	54.22	107	41.67	33.33
200	93.33	74.67	153	67.22	53.78	106	41.11	32.89
199	92.78	74.22	152	66.67	53.33	105	40.55	32.44
198	92.22	73.78	151	66.11	52.89	104	40	32
197	91.67	73.33	150	65.55	52.44	103	39.44	31.56
196	91.11	72.89	149	65	52	102	38.89	31.11
195	90.55	72.44	148	64.44	51.56	101	38.33	30.67
194	90	72	147	63.89	51.11	100	37.78	30.22
193	89.44	71.56	146	63.33	50.67	99	37.22	29.78
192	88.89	71.11	145	62.78	50.22	98	36.67	29.33
191	88.33	70.67	144	62.22	49.78	97	36.11	28.89
190	87.78	70.22	143	61.67	49.33	96	35.55	28.44
189	87.22	69.78	142	61.11	48.89	95	35	28
188	86.67	69.33	141	60.55	48.44	94	34.44	27.56
187	86.11	68.89	140	60	48	93	33.89	27.11
186	85.55	68.44	139	59.44	47.56	92	33.33	26.67
185	85	68	138	58.89	47.11	91	32.78	26.22
184	84.44	67.56	137	58.33	46.67	90	32.22	25.78
183	83.89	67.11	136	57.78	46.22	89	31.67	25.33
182	83.33	66.67	135	57.22	45.78	88	31.11	24.89
181	82.78	66.22	134	56.67	45.33	87	30.55	24.44
180	82.22	65.78	133	56.11	44.89	86	30	24
179	81.67	65.33	132	55.55	44.44	85	29.44	23.56
178	81.11	64.89	131	55	44	84	28.89	23.11
177	80.55	64.44	130	54.44	43.56	83	28.33	22.67
176	80	64	129	53.89	43.11	82	27.78	22.22
175	79.44	63.56	128	53.33	42.67	81	27.22	21.78
174	78.89	63.11	127	52.78	42.22	80	26.67	21.33
173	78.33	62.67	126	52.22	41.78	79	26.11	20.89
172	77.78	62.22	125	51.67	41.33	78	25.55	20.44
171	77.22	61.78	124	51.11	40.89	77	25	20
170	76.67	61.33	123	50.55	40.44	76	24.44	19.56
169	76.11	60.89	122	50	40	75	23.89	19.11
168	75.55	60.44	121	49.44	39.56	74	23.33	18.67
167	75	60	120	48.89	39.11	73	22.78	18.22
166	74.44	59.56	119	48.33	38.67	72	22.22	17.78

XIX.

Fah- ren- heit.	Centi- grade.	Réaumur.	Fah- ren- heit.	Centi- grade.	Réaumur.	Fah- ren- heit.	Centi- grade.	Réaumur.
°	°	°	°	°	°	°	°	°
71	21.67	17.33	33	0.55	0.44	- 4	-20	-16
70	21.11	16.89	32	0	0	- 5	-20.55	-16.44
69	20.55	16.44	31	- 0.55	- 0.44	- 6	-21.11	-16.89
68	20	16	30	- 1.11	- 0.89	- 7	-21.67	-17.33
67	19.44	15.56	29	- 1.67	- 1.33	- 8	-22.22	-17.78
66	18.89	15.11	28	- 2.22	- 1.78	- 9	-22.78	-18.22
65	18.33	14.67	27	- 2.78	- 2.22	-10	-23.33	-18.67
64	17.78	14.22	26	- 3.33	- 2.67	-11	-23.89	-19.11
63	17.22	13.78	25	- 3.89	- 3.11	-12	-24.44	-19.56
62	16.67	13.33	24	- 4.44	- 3.56	-13	-25	-20
61	16.11	12.89	23	- 5	- 4	-14	-25.55	-20.44
60	15.55	12.44	22	- 5.55	- 4.44	-15	-26.11	-20.89
59	15	12	21	- 6.11	- 4.89	-16	-26.67	-21.33
58	14.44	11.56	20	- 6.67	- 5.33	-17	-27.22	-21.78
57	13.89	11.11	19	- 7.22	- 5.78	-18	-27.78	-22.22
56	13.33	10.67	18	- 7.78	- 6.22	-19	-28.33	-22.67
55	12.78	10.22	17	- 8.33	- 6.67	-20	-28.89	-23.11
54	12.22	9.78	16	- 8.89	- 7.11	-21	-29.44	-23.56
53	11.67	9.33	15	- 9.44	- 7.56	-22	-30	-24
52	11.11	8.89	14	-10	- 8	-23	-30.55	-24.44
51	10.55	8.44	13	-10.55	- 8.44	-24	-31.11	-24.89
50	10	8	12	-11.11	- 8.89	-25	-31.67	-25.33
49	9.44	7.56	11	-11.67	- 9.33	-26	-32.22	-25.78
48	8.89	7.11	10	-12.22	- 9.78	-27	-32.78	-26.22
47	8.33	6.67	9	-12.78	-10.22	-28	-33.33	-26.67
46	7.78	6.22	8	-13.33	-10.67	-29	-33.89	-27.11
45	7.22	5.78	7	-13.89	-11.11	-30	-34.44	-27.56
44	6.67	5.33	6	-14.44	-11.56	-31	-35	-28
43	6.11	4.89	5	-15	-12	-32	-35.55	-28.44
42	5.55	4.44	4	-15.55	-12.44	-33	-36.11	-28.89
41	5	4	3	-16.11	-12.89	-34	-36.67	-29.33
40	4.44	3.56	2	-16.67	-13.33	-35	-37.22	-29.78
39	3.89	3.11	1	-17.22	-13.78	-36	-37.78	-30.22
38	3.33	2.67	0	-17.78	-14.22	-37	-38.33	-30.67
37	2.78	2.22	-1	-18.33	-14.67	-38	-38.89	-31.11
36	2.22	1.78	-2	-18.89	-15.11	-39	-39.44	-31.56
35	1.67	1.33	-3	-19.44	-15.56	-40	-40	-32
34	1.11	0.89						

XX.

*TABLES FOR CONVERTING CUSTOMARY AND METRIC
WEIGHTS AND MEASURES.*

UNITED STATES COAST AND GEODETIC SURVEY.

OFFICE OF STANDARD WEIGHTS AND MEASURES.

T. C. MENDENHALL, Superintendent.

WASHINGTON, D. C., 1890.

[Authorized Reprint.]

XX.

CUSTOMARY TO METRIC

LINEAR.				CAPACITY.			
Inches to Millimeters.	Feet to Meters.	Yards to Meters.	Miles to Kilometers.	Fluid Drams to Milliliters or Centimeters.	Fluid Ounces to Milliliters.	Quarts to Liters.	Gallons to Liters.
1 = 25.4000	0.304801	0.914402	1.60935	1 = 3.70	29.57	0.94636	3.78544
2 = 50.8001	0.609601	1.828804	3.21869	2 = 7.39	59.15	1.89272	7.57088
3 = 76.2001	0.914402	2.743205	4.82804	3 = 11.09	88.72	2.83908	11.35632
4 = 101.6002	1.219202	3.657607	6.43739	4 = 14.79	118.30	3.78544	15.14176
5 = 127.0002	1.524003	4.572009	8.04674	5 = 18.48	147.87	4.73180	18.92720
6 = 152.4003	1.828804	5.486411	9.65608	6 = 22.18	177.44	5.67816	22.71264
7 = 177.8003	2.133604	6.400813	11.26543	7 = 25.88	207.02	6.62452	26.49808
8 = 203.2004	2.438405	7.315215	12.87478	8 = 29.57	236.59	7.57088	30.28352
9 = 228.6004	2.743205	8.229616	14.48412	9 = 33.28	266.16	8.51724	34.06896

SQUARE.				WEIGHT.			
Sq. Ins. to Sq. Centimeters.	Square Feet to Sq. Decimeters.	Square Yards to Square Meters.	Acres to Hectares.	Grains to Milligrams.	Avoirdupois Ozs. to Grams.	Avoirdupois Lbs. to Kilograms.	Troy Ounces to Grams.
1 = 6.452	9.290	0.836	0.4047	1 = 64.7989	28.3495	0.45359	31.10348
2 = 12.903	18.581	1.672	0.8094	2 = 129.5978	56.6991	0.90719	62.20096
3 = 19.355	27.871	2.508	1.2141	3 = 194.3968	85.0486	1.36078	93.31044
4 = 25.807	37.161	3.344	1.6187	4 = 259.1957	113.3981	1.81437	124.41392
5 = 32.258	46.452	4.181	2.0234	5 = 323.9946	141.7476	2.26796	155.51740
6 = 38.710	55.742	5.017	2.4281	6 = 388.7935	170.0972	2.72156	186.62089
7 = 45.161	65.032	5.853	2.8328	7 = 453.5924	198.4467	3.17515	217.72437
8 = 51.613	74.323	6.689	3.2375	8 = 518.3914	226.7962	3.62874	248.82785
9 = 58.065	83.613	7.525	3.6422	9 = 583.1903	255.1457	4.08233	279.93133

CUBIC.			
Cubic Inches to Centimeters.	Cubic Feet to Cubic Meters.	Cubic Yards to Cubic Meters.	Bushels to Hectoliters.
1 = 16.387	0.02832	0.765	0.35242
2 = 32.774	0.05663	1.529	0.70485
3 = 49.161	0.08495	2.294	1.05727
4 = 65.549	0.11327	3.058	1.40969
5 = 81.936	0.14158	3.823	1.76211
6 = 98.323	0.16990	4.587	2.11454
7 = 114.710	0.19822	5.352	2.46696
8 = 131.097	0.22654	6.116	2.81938
9 = 147.484	0.25485	6.881	3.17181

1 chain	=	20.1169	meters.
1 square mile	=	259	hectares.
1 fathom	=	1.829	meters.
1 nautical mile	=	1853.27	meters.
1 foot	=	0.304801	meter, 9.4840158 log.
1 avoirdupois pound	=	453.5924277	grams.
15432.35639 grains	=	1	kilogram.

XX.

METRIC TO CUSTOMARY

LINEAR.				CAPACITY.				
Meters to Inches.	Meters to Feet.	Meters to Yards.	Kilometers to Miles.	Milliliters or Cu. Centimeters to Fluid Drams.	Centiliters to Fluid Ounces.	Liters to Quarts.	Dekaliters to Gallons.	Hektoliters to Bushels.
1 = 39.3700	3.28083	1.093611	0.62137	1 = 0.27	0.338	1.0567	2.6417	2.8375
2 = 78.7400	6.56167	2.187222	1.24274	2 = 0.54	0.676	2.1134	5.2834	5.6750
3 = 118.1100	9.84250	3.280833	1.86411	3 = 0.81	1.014	3.1700	7.9251	8.5125
4 = 157.4800	13.12333	4.374444	2.48548	4 = 1.08	1.352	4.2267	10.5668	11.3500
5 = 196.8500	16.40417	5.468056	3.10685	5 = 1.35	1.691	5.2834	13.2085	14.1875
6 = 236.2200	19.68500	6.561667	3.72822	6 = 1.62	2.029	6.3401	15.8502	17.0250
7 = 275.5900	22.96583	7.655278	4.34959	7 = 1.89	2.368	7.3968	18.4919	19.8625
8 = 314.9600	26.24667	8.748889	4.97096	8 = 2.16	2.706	8.4534	21.1336	22.7000
9 = 354.3300	29.52750	9.842500	5.59233	9 = 2.43	3.043	9.5101	23.7753	25.5375

SQUARE.				WEIGHT.			
Sq. Centimeters to Square Inches.	Square Meters to Square Feet.	Square Meters to Square Yards.	Hectares to Acres.	Milligrams to Grains.	Kilograms to Grains.	Hectograms (100 grams) to Ozs. Av.	Kilograms to Lbs. Avordupois.
1 = 0.1550	10.764	1.196	2.471	1 = 0.01543	15432.36	3.5274	2.20462
2 = 0.3100	21.528	2.392	4.942	2 = 0.03986	30864.71	7.0548	4.40924
3 = 0.4650	32.292	3.588	7.413	3 = 0.04630	46297.07	10.5822	6.61386
4 = 0.6200	43.055	4.784	9.884	4 = 0.06173	61729.43	14.1096	8.81849
5 = 0.7750	53.819	5.980	12.355	5 = 0.07716	77161.78	17.6370	11.02311
6 = 0.9300	64.583	7.176	14.826	6 = 0.09259	92594.14	21.1644	13.22773
7 = 1.0350	75.347	8.372	17.297	7 = 0.10803	108026.49	24.6918	15.43235
8 = 1.2400	86.111	9.568	19.768	8 = 0.12346	123458.85	28.2102	17.63697
9 = 1.3950	96.874	10.764	22.239	9 = 0.13889	138891.21	31.7466	19.84159

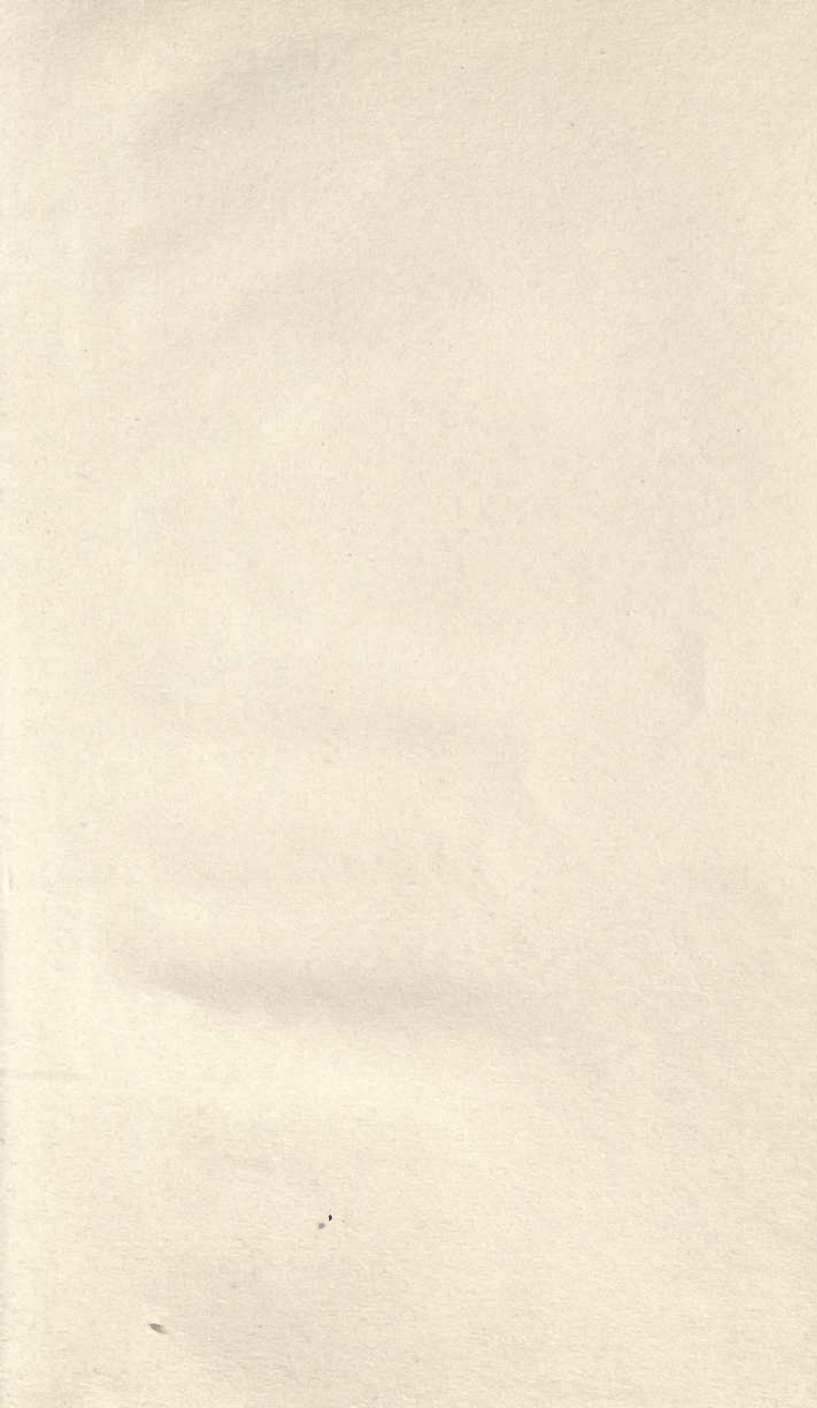
CUBIC.				WEIGHT.—(Continued.)		
Cu. Centimeters to Cubic Inches.	Cu. Decimeters to Cubic Inches.	Cubic Meters to Cubic Feet.	Cubic Meters to Cubic Yards.	Quintals to Lbs. Avordupois.	Millers or tonnes to Lbs. Av.	Grams to Ozs. Troy.
1 = 0.0610	61.023	35.314	1.308	1 = 220.46	2204.6	0.03215
2 = 0.1220	122.047	70.629	2.616	2 = 440.92	4409.2	0.06430
3 = 0.1831	183.070	105.943	3.924	3 = 661.38	6613.8	0.09645
4 = 0.2441	244.093	141.258	5.232	4 = 881.84	8818.4	0.12860
5 = 0.3051	305.117	176.572	6.540	5 = 1102.30	11023.0	0.16075
6 = 0.3661	366.140	211.887	7.848	6 = 1322.76	13227.6	0.19290
7 = 0.4272	427.163	247.201	9.156	7 = 1543.22	15432.2	0.22505
8 = 0.4882	488.187	282.516	10.464	8 = 1763.68	17636.8	0.25721
9 = 0.5492	549.210	317.830	11.771	9 = 1984.14	19841.4	0.28936

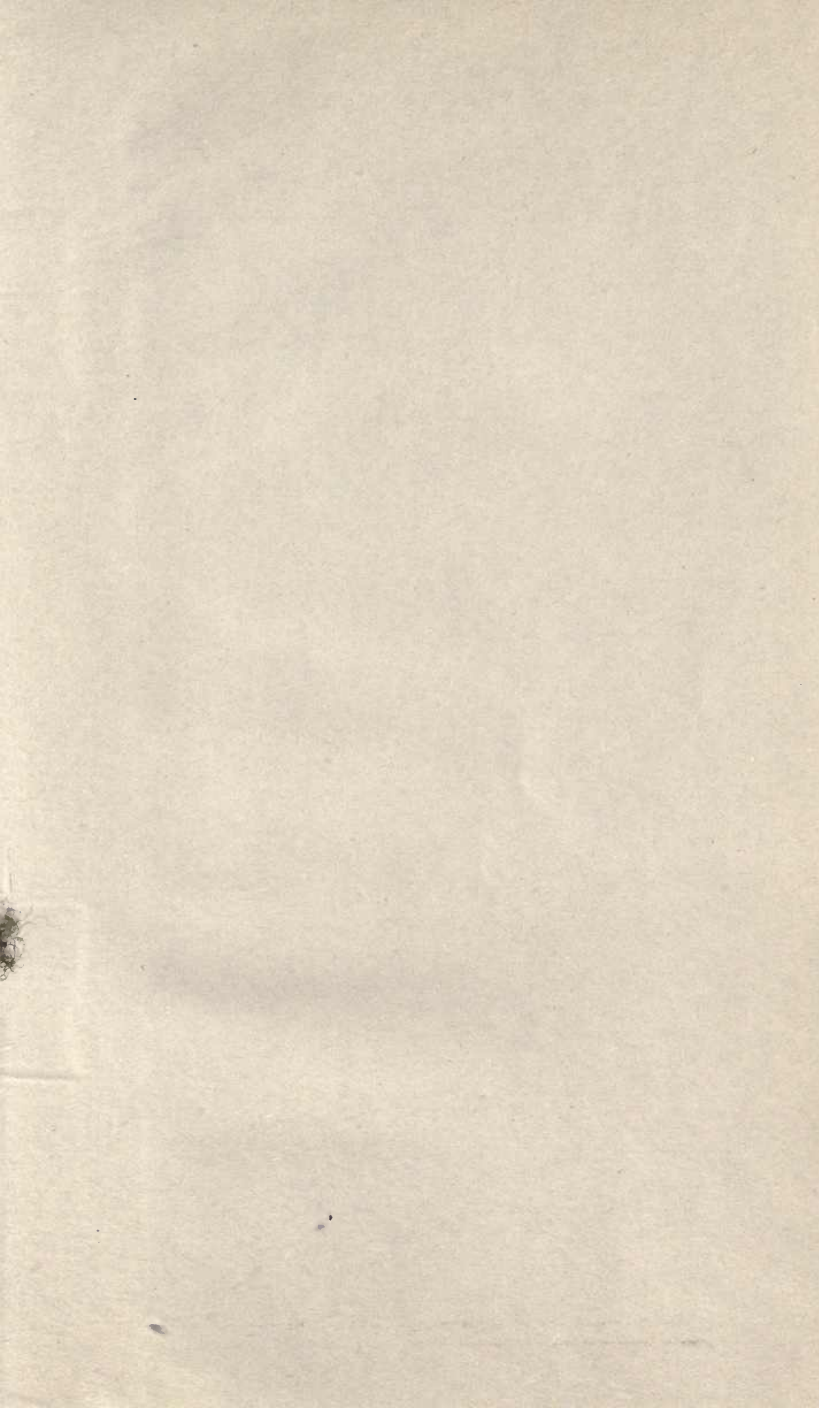
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