XXI.—NEW PROCESS FOR THE RAPID ESTIMATION OF PURE SUGAR IN RAW AND REFINED COMMERCIAL SUGARS.

BY P. CASAMAJOR.

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Part First.

The process for the estimation of sugar, which I propose to describe this evening, is based on a happy idea of M. Dumas. It cannot but be a matter of surprise that, although this idea was published several years ago, it has never been taken up and studied by the numerous chemists whose business it is to analyze sugar.

As an introduction to the subject, I will recall to your memories the main points relating to the process of M. Dumas, and, as this process was suggested to its eminent author, while studying that of Payen, we may also say a few words of the latter.

In 1846, Payen published a process for determining the amount of pure sugar in commercial sugars, which seems to conform more closely to the ordinary processes of chemical analysis than is the case with those which have been more generally adopted for testing sugars. This process consists in washing commercial sugars with alcohol previously saturated with pure sugar. The object of this operation was to wash out everything except the pure sugar, and this, after being freed from the sugar-saturated alcohol by washing with absolute alcohol, could be dried and weighed, as is done with other substances submitted to chemical analysis. Payen found, however, that the quantity of sugar obtained in this way was always less than the actual quantity present. He seems afterwards to have paid very little attention to this process, and apparently did not attach much importance to it.

It was reserved for Dr. Scheibler, of Berlin, in these latter years, to discover that the result obtained by the process of Payen was not the quantity of pure sugar present, but that it represents the *rendement*, or quantity of sugar obtainable in refining. It cannot but be a matter of surprise that Dr. Scheibler did not distinctly claim this discovery, instead of pretending that he had, in a manner, re-discovered the process of Payen, "which had fallen into the domain of history," when, indeed, he had only attached to it cumbersome paraphernalia, which really added nothing to the value of the process.

Payen used two alcoholic solutions saturated with sugar, and finally absolute alcohol, to wash out the last traces of the sugar-saturated solutions. The first solution was obtained by taking alcohol at 85 per cent., and adding to this 5 per cent. of strong acetic acid. This mixture was saturated with sugar. I believe that this addition of 5 per cent. of acetic acid was to decompose the sucrates, which were a great bugbear to the sugar chemists of those days. This addition of acetic acid fulfills, however, a useful purpose, as it seems to make the mixture better able to remove the impurities of gummy sugar.

This first solution of Payen was the one adopted by M. Dumas, and here is the process which he proposed for analyzing commercial sugars:

If we introduce an alcohometer of Gay-Lussac in the first solution of Payen, we find that it sinks to a point which, if we operate at 15° C., corresponds to 74 per cent. If we take a certain volume of this solution and stir it up in a glass with a sufficient quantity of a sugar to be analyzed, we are able to ascertain, by again placing the alcohometer in the solution, that this has taken up something from the commercial sugar, for the alcohometer no longer indicates 74 per cent., but a lower degree, corresponding to a greater density. The process which M. Dumas proposed was this: Take 100 cubic centimeters of the first solution of Payen, and 50 grammes of sugar; agitate the mixture in a glass; filter the solution, and observe the alcohometric degree corresponding to 15° C. For every per centage of sugar less than 100, you will find that the solution indicates 1 per cent. less than 74.

A simpler way of stating the same thing is to note that the difference between 100 and 74 is 26, and, if the above proposition is correct, we can obtain the percentage of sugar by adding 26 to the alcohometric degree. Thus, if the alcohometer indicates 68, the percentage of sugar is $68 + 26 = 94$.

The only account I have seen of this process is in L'Abbé Moigno's *Saccharimétrie Physique, Chimique et Mélassimétrique*; this is translated in the *American Chemist* of February, 1871, p. 302. This description may also be found *verbatim* in a recent work of M. Maumené on sugar.

The account of the process of M. Dumas, as found in the above paper, states that, for sugars having 87 per cent. or more of pure sugar, the results agree very closely with those of the saccharometer, even within $\frac{1}{10}$ of 1 per cent. For sugars of lower grade, the results obtained were not satisfactory.

After making a great number of tests with the solution used by M. Dumas, I am in a position to state that it is not possible, even with sugars of high grade, to obtain results at all approaching those of the optical saccharometer, when we operate on such sugars as we have in this market. The sugars to which the process was applied in France, may have been beet sugar of about the same grade and similar composition. With our cane sugar, both raw and refined, the differences between the results of the saccharometer and those of the alcohometer were sometimes as high as 3 or even 4 per cent. for sugars above 87 per cent. As to beet sugars, I regret to say that, since I undertook these researches, I have not been able to obtain any samples to form an idea of their behaviour with this process of M. Dumas.

At any rate, as nearly one-half of the raw sugars that come to this market stand below 87 per cent., there seemed to be little use in a process which was declared to be inapplicable to sugars of low grade. I found, however, after trying the process several times, that, although the results obtained were mostly unfavorable, it was impossible to dismiss it entirely, for, upon reflecting upon these results, I found that many questions arose which required to be solved, and, on their solution, I based the hope of modifying this process so as to apply it to the analysis of cane sugars of all grades.

In considering commercial sugars, with the view of applying to them the process of M. Dumas, there are several points which experience has shown to be worthy of attention. Several other points, which have taken a great deal of my time, were found to be of no importance whatever in the study of this question.

To understand the points that are of importance in this inquiry, we may consider that a commercial sugar is composed of four classes of substances :

1st. Pure sugar.

2d. Water.

3d. Soluble impurities, or other soluble substances, besides pure sugar.

4th. Sand, earth and other insoluble substances.

We may now examine each class separately.

1st. The behaviour of pure sugar with the saturated alcoholic solution used by M. Dumas, is easily understood. This solution being incapable of dissolving any further quantity of sugar, its density cannot be affected by the addition of pure sugar alone.

2d. An addition of water lowers the alcoholic degree. When an excess of sugar is present, the alcohol, diluted with water, is able to dissolve an additional quantity of sugar, which still further increases its density. To ascertain the quantities of pure sugar which alcohol of various strengths will hold, I took several saturated solutions and introduced them in the tube of the saccharometer. The results of the observations thus obtained are given in Table No. 1. In the first column of this table is the percentage of alcohol before being saturated with sugar; in the second column is the indication of the alcohometer for the same alcohol, after this has been saturated; in the third column is the direct reading of the optical saccharometer (Ventzke's), when the saturated solution is placed in its tube. Finally, in a fourth column, I have placed the number of grammes of sugar in 100 cubic centimeters of the saturated alcohol. The quantities in this fourth column are calculated from those in the third, by taking 0.26 gr. of sugar as corresponding to one degree of the saccharometer scale.

TABLE No. 1.

DEGREE OF THE ALCOHOMETER BEFORE SATURATION.	DITTO AFTER SATURATION.	DEGREE OF THE SACCHAROMETER.	GRAMMES OF SUGAR IN 100 CC.
92.	91.	2.2	0.572
87.	82.3	......	......
85.	79.5	12.2	3.17
84.	78.	14.6	3.80
82.4	74.	19.3	5.02
80.	67.62	26.3	6.83
75.	48.55	50.	13.01
50.	sp. gr. 1.14	192.	49.9

We may notice, in the above table, that if we compare alcohol of 85 per cent. with alcohol of 80 and 75 per cent., that a fall of 5 per cent. from 85 to 80, answers to a difference of 11.88 per cent. in the corresponding saturated solutions, while a fall of 5 per cent. from 80 to 75 corresponds to a difference of 19.07 per cent. in their saturated solutions.

These facts are of prime importance in this inquiry, and we will have occasion in the sequel to draw from them consequences worthy of attention.

3d. *Soluble Impurities.*—To study the effect of soluble impurities on alcohol saturated with sugar, I prepared quite a large quantity of inverted sugar by heating with hydrochloric acid. The acid was afterwards thrown down as chloride of lead, and a small quantity of lead which remained in solution was precipitated with carbonate of soda, but not enough of this reagent was used to make the solution alkaline. This solution was evaporated over a water bath, and the result was, on cooling, a very stiff gummy substance of light yellow color. The presence of a slight excess of sodic chloride did not interfere with the usefulness of the product, as it merely acted as the representative of the soluble impurities of commercial sugar, and these always contain soluble salts.

The effect of dissolving inverted sugar in alcohol saturated with sugar, was to lower the alcohometric degree, and this lowering was progressively greater, as the quantity of inverted sugar was increased. Thus, 5 grammes of inverted sugar lowered the alcohometric degree 11.63 per cent., and 10 grammes lowered it 24.5 per cent., while, if the decrease had been strictly proportional to the quantity of inverted sugar, 10 grammes should have given 11.63 × 2 = 23.26 per cent. We may note, however, that the lowering of the alcoholic degree by inverted sugar, which here stands as the representative of the soluble impurities, although not exactly proportional to the quantity added, is more nearly so than is the case with equivalent quantities of water, as we may see in Table No. 1.

4th. *Insoluble Impurities.*—These have no effect whatever on the density of an alcoholic solution saturated with sugar. With clean, dry sand, as well as with pure, dry sugar, no effect takes place in the density of the alcoholic solution. When insoluble substances occur in appreciable quantities, they should be deducted from the indication of the alcohometer.

It must be understood that in the experiments which are related above with water and inverted sugar, an excess of pure, dry sugar was always present. Otherwise the results obtained would not have been applicable to the analysis of sugars.

Although four classes of substances have been mentioned as worthy of consideration, only water and the soluble impurities have any influence in the results obtained by the process of M. Dumas. If a certain weight of water lowered the alcohometric degree to exactly the same extent as an equal weight of soluble impurities, the process proposed by M. Dumas could, by taking a less weight of sugar for 100 cc of solution, be made to yield accurate results; but this does not happen, and if we have, for instance, a sugar containing 94 per cent. of pure sugar, the other constituents may be either 6 per cent. of water or 6 per cent. of impurities, or any sum of impurities and water amounting altogether to 6 per cent. In each of these cases we may obtain a different result. On the other hand, as the addition of a certain quantity of water gives very different results, according to the strength of the alcohol solution, we can conceive that, by a series of experiments, we may succeed in obtaining a solution of such strength, that, within the limits which correspond to commercial sugars, the results in lowering the alcoholic degree may be practically the same for a certain quantity of water, as for the same quantity of soluble impurities.

Following up this idea, I made a great many experiments, to ascertain whether it was not possible, by raising or lowering the alcohometric degree of a saturated sugar solution, to obtain exactly the strength that would give the required result. In this connection, I may mention, that at an early stage of my experiments, I discarded the use of acetic acid, and I operated with mixtures of alcohol and water only. I found that the solution used by M. Dumas could be replaced by alcohol of 82.4 per cent., saturated with sugar. I tried other solutions, some stronger and others weaker, and I was not long in finding that the bad results obtained with either the solution of M. Dumas or with alcohol of 82.4, saturated with sugar, were much aggravated when the solutions had less alcoholic strength. By a series of experiments, I finally came to the conclusion that a solution of 87 per cent. alcohol would give the most satisfactory results. Alcohol of this strength, when saturated with sugar, stands at 82.3 by the alcohometer. This solution answered very well with sugars of quite high grade, but when I came to apply it to gummy sugars, the impurities would not dissolve, and the results obtained were always a great deal too high. I sought to remedy the evil by taking alcohol at 94 per cent., and lowering the degree with acetic acid instead of water, because, as I have said, acetic acid has a favorable action in dissolving gummy impurities, but I did not succeed in effecting this object, and, much to my disappointment, I had to give up, for a time at least, the project of applying the idea of M. Dumas to the analysis of cane sugars.

Part Second.

I had given up the hope of success, when, on passing before No. 71 Maiden Lane, I saw at a window a number of bottles, labelled

Diamond Methal, which proved to be methylic alcohol. I determined to try if methylic alcohol would answer better than the ethylic, and purchased a gallon of the product. This stood at 92.5 degrees by the alcohometer, and even at this strength, the gummiest sugar could be cleansed by mixing with this spirit and stirring the mixture. On saturating it with sugar, and placing the solution in the tube of a Ventzke saccharometer, the indication was 1.7, which is less than corresponds to ethylic alcohol of the same density.

These first trials were very encouraging, and those that followed were not less so. Without recounting now the experiments that were made, I may state that, by the use of methylic alcohol, I finally succeeded in obtaining, with an alcohometer, results that agree very closely with those of the optical saccharometer, with cane sugars of all classes, from the highest to the lowest.

The explanations already given concerning the use of ethylic alcohol of various strengths, saturated with sugar, and the effects that water and the soluble impurities have in lowering their alcohometric degrees, render superfluous any account of similar experiments with hydrate of mythyl. I may briefly state that, after making a great number of trials, I found that methylic alcohol of 83½ degrees of the alcohometer,* when saturated with sugar, stands at 77.1 degrees, and that this solution is the one that has given the most accurate results.

This saturated solution is quite easily obtained by taking methylic alcohol standing at 83½ degrees by the alcohometer, and saturating it with sugar, by the process which Numa Grar suggested to Payen. This consists in shaking up the alcohol with powdered sugar. Grar made his powdered sugar by grinding it in a mortar in presence of alcohol. I have generally used dry *extra-powdered* sugar. When this cannot be had, grinding in a mortar is an excellent way of getting the sugar in fine particles, which are very necessary for obtaining a saturated solution in a short time. When very finely powdered sugar is shaken up with either ethylic or methylic alcohol, the finest particles are dissolved almost immediately, while the coarser

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*Since this paper was written, I have found the following interesting coincidence : The alcohometric degree 83½. corresponds to a specific gravity of 0.85315 (Prof. MacCulloch's tables for Tralles' alcohometer). On consulting Dr. Ure's table for methylic alcohol (Phil. Mag. [3], XIX, 51). it may be seen that specific gravity 0.8531, corresponds to 87 per cent. of methylic alcohol. Now, in the first part of this paper, I have said that ethylic alcohol of 87 per cent., saturated with sugar, gives the results which agree most closely with those of the optical saccharometer.

grains fall to the bottom, leaving the liquid quite clear after standing a minute or two. When the alcohol clears up in this way, it is an indication that it is not saturated, and an additional quantity of sugar should be shaken up with it. This is repeated until the mixture remains cloudy for at least two or three minutes after adding finely powdered sugar. When this takes place, the liquid is saturated, and a further addition of sugar will not increase its density. By operating in this way, we may obtain saturated alcohol in 10 or 15 minutes, while, by hanging sugar crystals in the solution, I have not been able to obtain the maximum of density in less than two days. When we are about to use the solution, it is advisable to shake it up a little while before using it, and, after the sugar has been deposited, to take the alcohometric degree. This degree cannot be higher than that shown by previous observations, but it may become lower, if the bottle is not tightly closed, as the alcohol evaporates, leaving a residuum containing a greater proportion of water. There is no use in placing chloride of calcium tubes on the cork of the bottle in the vain hope that, in this way, water may be absorbed from the atmosphere. The lowering of the alcoholic degree takes place principally by the evaporation of the spirit, and nothing can replace tight stoppers. The tin cans, with screw caps, that are used for refined petroleum, are well adapted for holding methylic alcohol, and of these, Coleman's *elevated swinging cans* are the most convenient for holding our standard solution.

When on testing the saturated alcoholic solution, we find that the degree is lower than required, we may raise it by adding more alcohol. The addition of alcohol causes the precipitation of a part of the sugar in solution, so that the quantity of alcohol to be added should be somewhat less * than the quantity to be added to a mixture of alcohol and water, of which we desire to raise the degree by the addition of stronger alcohol.

If we have a certain volume, V, of alcohol and water, whose alcohometric degree is d, and we wish to raise this degree to D, with strong alcohol of degree $\triangle$, if the volume of the latter alcohol to be added is called x, we shall have

$$Vd + x \triangle = (V + x) D,$$

whence

$$x = \frac{V (D - d)}{\triangle - D}$$

If we have 1000 cc of alcohol at 81, and we wish to raise the degree to $83\frac{1}{2}$, with alcohol of 92 per cent., then $d = 81$, $D = 83\frac{1}{2}$, $V =$

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* I usually take 0.8 x.

1000 and $\Delta=92$, and the volume of alcohol of 92 to be added, is

$$x=\frac{1000\times 2.5}{8.5}=294.1 \text{ cc.}$$

If the addition of alcohol has been too great, we may diminish the degree by adding water very cautiously, and stirring up the mixture with an excess of sugar. To obtain the quantity of water, we may use the above formula, but we must note that $\Delta=0$, and, as both numerator and denominator have become negative quantities, we may change the signs, and we shall have :

$$x=\frac{V\,(d-D)}{D}$$

These particulars have been entered into, because of the great importance of obtaining saturated solutions which do not deviate too widely from 77.1 degrees of the alcohometer. Too much attention cannot be given to this, as otherwise it is not possible to obtain correct results.

Next in importance, is the weight of commercial sugar to be taken for 100 cubic centimeters of the methylic solution saturated with sugar. In the process proposed by M. Dumas, 50 grammes of sugar are stirred with 100 cubic centimeters of his standard solution. At the outset of the experiments on this process, I found that one obvious cause of error was the imperfect solubility of the impurities of commercial sugars in this standard solution. I was led then to grind the sugar down in a mortar as thoroughly as possible, so as to break up all the lumps and crush the large crystals, to allow everything to dissolve that would. I used for this quite a heavy pestle, of a size quite disproportionate to that of the mortar. By this thorough crushing, much less sugar is used than M. Dumas proposed. It is an easy matter to determine the quantity of sugar to be used in a test. At first we may take an arbitrary quantity, and note the result, which may be corrected by the following considerations. The lowering of the alcohometric degree depends, as we have said, on the water and the soluble impurities present in the sugar. If we take a certain weight of sugar, say 45 grammes, we may find by the alcohol process that the result is 91.5 per cent. of sugar. If we test the same sugar by the optical saccharometer and find 93 per cent. of sugar, we know that the alcohol process has given us too low a result, and this because the solution was too dense. Our result shows in the sugar : $100-91.5=8.5$ of impurities and water, while we ought to have had $100-93=7$. We must then, to obtain 93, take a weight equal to $\frac{45\times 7}{8.5}=37.05$ grammes.

After trying many experiments with solutions of different strengths, it was found that each solution required a different weight. For the saturated solution of 77.1 degree of the alcohometer, which is our standard solution, the weight is 39.6 grammes for 100 cubic centimeters of the solution.

Instead of using 100 cubic centimeters, I have, for a long time, used only 50. To be able to use a cylinder in which this volume would give indications, I needed to use alcohometers of quite small diameter. These were made by Mr. H. Weinhagen; they are very well graduated and very satisfactory in every way.

For 50 cubic centimeters of standard solution, the proper weight is half of the one for 100 cubic centimeters = 19.8 grammes. To show how this was obtained, the following table, No. 2, is given. In the first column are numbers designating the sugars that were tested; in the second column is the per centage of sugar as obtained by the optical saccharometer; in the third column is the result by the methylic alcohol process, when we take 19 grammes as the weight of the sugar tested; in the fourth column is the quantity that should have been taken, instead of 19 grammes, to obtain the same result as by the optical saccharometer. These weights were obtained by the calculation already given.

TABLE No. 2.

SUGAR.	DEGREE OF SACCHAROMETER.	RESULT BY METHYLIC ALCOHOL. Weight, 19 gr.	WEIGHT TO GIVE RESULTS IN 2D COLUMN.
			gr.
1	91.6	91.9	19.7
2	84.8	85.36	19.66
3	91.7	92.1	19.96
4	81.7	82.46	19.8
5	88.8	89.1	19.7
6	96.3	96.6	20.6

From the results in the 4th column, the weight adopted was 19.8 gr.

By using this weight, with 50 cubic centimeters of standard solution, the following results were obtained in the case of 15 consecutive tests of raw and refined sugars:

TABLE NO. 3.

DESIGNATION OF THE SUGAR.	PER CENTAGE OF PURE SUGAR BY SACCHAROMETER.	DITTO BY METHYLIC ALCOHOL.	DIFFERENCE.
Muscavado......	82.4	82.3	—.1
"	91.5	91.7	.2
Refined..........	95.	95.1	.1
Molasses.........	86.5	87.2	.7
Centrifugal......	96.5	96.2	—.3
Refined..........	96.3	96.6	.3
"	92.3	92.1	—.2
"	91.3	90.9	—.4
"	84.4	83.7	—.7
"	91.6	91.5	—.1
"	84.8	84.7	—.1
Raw............	91.7	91.8	.1
Refined.........	81.7	81.7	.0
Muscavado......	88.7	88.7	.0
Centrifugal......	96.3	96.5	.2

The greatest deviation shown in this table, is in the case of two sugars, in which the difference is 0.7. This is not due to impurities affecting the saccharometer, as I found by inverting the solution of the sugar in each case. In one of the sugars I found quite a number of soft lumps, which showed that the sugar was very uneven. In the other sugar nothing was found to account for the discrepancy.

The alcohometers used in these tests are those of Tralles, which are used by the United States Government. These do not differ materially from those of Gay-Lussac. They are more apt to differ from one another on account of defective construction, than they should do from Gay-Lussac's instrument.

Alcohometers, like other areometers, are very unreliable, unless placed in the hands of those who understand their use. We may, however, use alcohometers that are not strictly accurate, if only the divisions of the scale bear to one another the proper proportion, and if the displacement of the scale is not too great. This does not apply to the preparation of the standard solution, which should stand correctly at 77.1, which degree of the alcohometer of Tralles corresponds to specific gravity 0.87.

When we use an alcohometer that is properly graduated, and we have the standard solution standing at 77.1, we should add 22.9 to the degree of the alcohometer at 15° C., to obtain the per centage of sugar. If the alcohometer, through being defective, should not stand at 77.1° for 0.87 specific gravity, but at 76.5, the difference between this latter number and 100, being 23.5, this is the number to be added to the reading of the areometer at 15° C., to obtain the percentage of sugar in the sample under examination.

I have said that cane sugars of all grades, from the highest to the lowest, have given satisfactory results by this process. This assertion is borne out by the examples in Table No. 3. I wish, however, to call attention to this, that I do not comprise, under the head of sugars, melados or molasses. As to melados, those that have drained out and are comparatively dry, may be classed as sugars, but wet melados give very poor results. This may be very easily understood, if we remember what was said of the action of water in lowering the alcohometric degree of a saturated solution, as exemplified in Table No. 1. I have little doubt, however, that by experimenting with saturated solutions of methylic hydrate of higher alcoholic degree, we may obtain a solution specially adapted for testing wet melados. It might even be possible, with methylic hydrate of still higher alcohometric degree, to test molasses within certain limits of density. As an operator can always tell beforehand, whether what he has to test is properly a sugar, a wet melado or a sample of molasses, he could always use the saturated solution which was the best adapted to his sample.*

From past experience, with saturated solutions of various strengths, I may venture the opinion that methylic alcohol of 87 degrees of the alcohometer, saturated with sugar, would make a good standard solution for wet melados. For molasses of density, about 40° Baumé, methylic alcohol of 90½ degrees (alcohometer), saturated with sugar, would not, as far as I can judge without trying it, be far from the proper strength. These are merely hints thrown out to chemists who are interested in testing sugar. Should I not work out these sugges-

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*With melados and molasses, as well as with very low sugars, the direct test seldom gives the true per centage of sugar. It is necessary to invert, to obtain the correct result, and when we invert a solution that has been imperfectly decolorized, it becomes so red, after inversion, that it is a difficult matter to see through the tube. With chloride of tin and filtration over bone black, it is possible to improve the color of the solutions, but even these often fail. The bone black should be dry and not too great a quantity should be used. On account of these difficulties with dark solutions, experiments to establish the strength of the standard solution and the weight of sugar, may have to be made entirely with refined syrups or with low grade refined sugars, to which 5 or 6 per cent. additional of water should be added.

tions, I hope that others may find time to study them. The advantages of a process that allows a commercial saccharine product to be tested in about five minutes, are too great to be neglected. The weights of melado or molasses to be taken, would have to be determined, as was done for the weight 19.8 grammes, as shown in Table No. 2.

Correction for variations of temperature.—When we operate at temperatures different from 15° C. or 60° F., the corrections can be made by using either the tables of Gay-Lussac or those for the instrument of Tralles, which we owe to Prof. MacCulloch. I will, however, state that results that are sufficiently accurate, may be obtained by proceeding as follows :

For solutions which give, by the alcohometer, from 77 to 70, we may multiply the excess of temperature above 60° F. by 0.2, and deduct the product from the reading of the alcohometer. For degrees of the alcohometer between 70 and 60, we may multiply this excess by 0.205, and for indications below 60 degrees of the alcohometer, the excess of temperature above 60° F., must be multiplied by 0.21, and the product deducted. If a centigrade thermometer is used, the excess of temperature over 15° should be multiplied by 0.36 for solutions above 70 degrees of the alcohometer. For solutions between 60 and 70 degrees (alcohometer), the excess over 15° C. should be multiplied by 0.37, and for those below 60, this excess should be multiplied by 0.38. In all cases, the product should be deducted from the direct reading of the alcohometer. The results obtained in this way do not differ materially from those given by the tables.

There is another correction for variation of temperature, which relates to the volume of standard solution to be taken for a weight of sugar equal to 19.8 gr. As the temperature rises, a greater volume of standard solution should be taken. If we use a Farenheit thermometer we take

At 60° F.	50.	cc of	standard	solution
“ 70° F.	50.3	“	“	“
“ 80° F.	50.6	“	“	“
“ 90° F.	51.	“	“	“
“ 100° F.	51.3	“	“	“

If we use a centigrade thermometer, we should take

At 15° C.	50.	cc of	standard	solution
“ 20° C.	50.25	“	“	“
“ 25° C.	50.5	“	“	“
“ 30° C.	50.8	“	“	“
“ 35° C.	51.2	“	“	“
“ 40° C.	51.4	“	“	“

If it should be preferred to use always the same volume of 50 cc of standard solution, we may take the following weights for the temperatures of a centigrade thermometer

At 15° C.	19.8 grammes.
" 20° C.	19.7 "
" 25° C.	19.6 "
" 30° C.	19.5 "
" 35° C.	19.4 "
" 40° C.	19.3 "

In the following table may be found the same data concerning methylic alcohol of various strengths, saturated with sugar, as are given in table No. 1 for ethylic alcohol.

TABLE No. 4.

DEGREES OF THE ALCOHOMETER BEFORE SATURATION.	DITTO AFTER SATURATION WITH SUGAR.	DEGREE OF THE SACCHAROMETER (VENTZKE).	GRAMMES OF SUGAR IN 100 CC.
92.5	91.8	1.7	0.44
83.5	77.1	13.2	3.43
82.7	76.5	....	....
81.5	75.	....	....

From this table we may learn that the solution, saturated with sugar, standing at 77.1 by the alcohometer, should, when placed in the saccharometer tube, stand at 13.2. We also learn that the same saturated solution must contain 3.43 grammes of sugar in 100 cc. This is a useful guide for the quantity of extra powdered sugar that should be added to 1 liter of methylic alcohol of 83½°.*

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*From this table we may also learn that if we apply methylic alcohol to Payen's process, we need have no fear of leaving in the sugar a small quantity of the solution made by saturating with sugar methylic alcohol at 92.5 degrees. With ethylic alcohol, cane sugars are always left with too much impurity. Dr. Behr informs me, that on this account, he has never been able to obtain good results by this process. It is certainly worth while trying methylic alcohol. Cane sugars could be washed out with our standard solution, testing 77.1 (alcohometer), and the last particle of this could be removed by methylic alcohol 92.5, saturated with sugar. The small quantity of this latter remaining after filtration, would leave too little sugar to cause an appreciable error. The funnel with auxiliary vertical tube, of which I gave a description several years ago, is very well adapted to washing sugars out with alcohol, as it leaves them quite dry without the help of a filter pump (see *American Chemist*, Oct., 1875, Vol. vi, p. 122, and *Chemical News*, Vol. xxxii, No. 829, p. 183).

Method of Procedure in Testing.—The sugar to be tested should not be weighed before every thing is ready, as otherwise it may dry out, after being weighed, and give too high a result. The mortar and pestle should be clean and dry, the cylinder should be filled with the standard solution to a line indicating 50 cubic centimeters, and a clean dry funnel should be ready with a paper filter in it. The weight of sugar to be taken is 19.8 grammes. This is transferred to the mortar as soon as weighed, and the standard solution should be poured in the mortar, and the sugars then ground with a heavy pestle until all lumps and large crystals are broken up.*

The mortar I have used for grinding sugar in presence of the standard solution, has a capacity of 180 cubic centimeters, and the pestle is of such a size, that it displaces 80 cubic centimeters in this mortar.

After all the lumps have been broken up, the contents of the mortar are poured on a filter standing over the cylinder, which previously held the standard solution. As there was some of this solution left in the cylinder, about 10 cubic centimeters of the filtered solution is allowed to run into the cylinder, after which the funnel is taken from the cylinder and held over the mortar long enough to shake up the liquid in the cylinder and pour it back on the filter. After this the funnel is placed again over the cylinder, the contents of the mortar are poured again on the filter, and from 30 to 35 cubic centimeters are allowed to run through the filter, which are generally sufficient for a test. In this filtered solution are placed, in succession, an alcohometer and a thermometer. To the alcohometric degree, corrected for temperature, is added the difference between 100 and the alcohometric degree of the standard solution. This sum represents the per centage of sugar.

Example.—The standard solution gives with the alcohometer 76.8, when corrected for temperature. The same alcohometer, placed in the filtered solution, marks 71 degrees, while a centigrade thermometer shows a temperature of 28°. According to what has been already

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*Persons who test raw centrifugal sugars by this process, must very soon see what *artificially colored sugars* consist in. This has been a vexatious question, occupying the attention of eminent chemists. By washing sugar crystals with alcohol, we may easily see that in the grains of some of them have been incorporated brown insoluble particles. In some cases these look like the scum from the defecation of the juice, and, in other samples, the coloring matter seems to be clay or some brown pigment This coloring matter seems generally to have been added in a very clumsy manner, as after washing the crystals in alcohol, it may be seen in lumps, specking the crystals with dark dots.

said, we take the difference between 28 and 15=13, and multiply this difference by 0.36, the product being 4.68. This quantity subtracted from 71, gives 66.32. The difference between 100 and 76.8 being 23.2, this quantity is added to 66.32, and the sum, 89.52, is the quantity of pure sugar in the sugar tested.

It has been suggested that the evaporation of methylic alcohol during the manipulations that have been described, must interfere with the accuracy of the results obtained, but this does not seem to be the case. It might be possible to avoid evaporation entirely by leaving out the operation of grinding the mixture of sugar and methylic alcohol in a mortar, but, in this case, the results obtained would be less accurate on account of the impurities left in the sugar. Beyond avoiding working in a draft, no precaution has been taken to prevent evaporation. One reason why the evaporation of the alcohol seems to have so little effect on the result, is that the operations of grinding and filtering occupy only a few minutes, during which no appreciable loss takes place.

XXII.—CONTRIBUTIONS FROM THE LABORATORY OF THE STEVENS INSTITUTE OF TECHNOLOGY.

BY ALBERT R. LEEDS.

I.—SOLUBILITY OF OZONE IN WATER.

SCHOENBEIN, in his numerous papers, taught that ozone is insoluble in water, and this statement was thence transferred to the accounts given of the properties of ozone by other writers, and finally adopted as a universally admitted fact in chemical text-books.*

WILLIAMSON (1845),† arrived at conflicting results with regard to the solubility of ozone and, it should be added, with reference to ozone itself. He ascribed the peculiar properties belonging to the the oxygen set free by electrolysis, to the admixture of a *peroxide or acid of hydrogen.* This body, according to Williamson, even when formed by the electrolysis of oxide of copper dissolved in sulphuric acid (in order that no hydrogen might be liberated at the same time), and after careful drying by calcium chloride, yielded when passed

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*"On Schoenbein's Ozone:" Faraday, Proc. Royal Inst., vol. i, p. 94. Elements of Chemistry: Miller, 1860, vol. ii, p. 23.

†"Some Experiments upon Ozone:" Phil. Mag., xxvii, p. 372. Chem. Soc. Mem., ii, p. 395. Liebig's Ann., liv, p. 127.