showed a loss of 7 units in the Hübl figure and of 25 units in the viscosity figure. An almond oil, similarly exposed for a longer time, decreased from 98.1 to 84.6 in the Hübl figure and from 167.8 to 123.8 in the viscosity figure. On the other hand, a sample of lard oil exposed until the Hübl figure fell from 73.3 to 66.7 showed no appreciable change in the viscosity figure; and in several samples of cottonseed, maize and linseed oils, in which atmospheric oxidation had caused losses of 10 to 40 units in the Hübl figures, the decrease in the viscosity figure was in no case greater than 4 units.

SUMMARY.

Olive and almond oils yield soap solutions of considerably greater viscosity than those obtained from the other more common fatty oils.

This "viscosity figure" is apparently higher in the better than in the poorer grades of olive oil.

The lowering of the viscosity figure by admixture of other oils furnishes an additional method for the detection of adulteration in olive and almond oils. As suggested by Blasdale, it will be especially useful for the detection of lard oil for which we have no specific test. The characteristic high viscosity figure of olive or of almond oil may be largely lost on sufficiently long exposure of the oil to air at ordinary temperature.

While the reason for the high figures shown by olive and almond oils cannot be stated, it appears probable that the explanation is to be found in the quantitative relations of the fatty acids present, rather than in the presence of any peculiar constituent, and that interesting results might be obtained from a study of the viscosities of the soap solutions of pure fatty acids and their mixtures.

QUANTITATIVE LABORATORY,
June, 1903.

[CONTRIBUTION FROM THE DEPARTMENT OF FOOD AND DRUG INSPECTION OF THE MASSACHUSETTS STATE BOARD OF HEALTH.]

THE DETERMINATION OF COMMERCIAL GLUCOSE IN MOLASSES, SYRUPS, AND HONEY.

BY ALBERT E. LEACH.

Received May 28, 1903.

THE importance of a ready method for the determination of so common an adulterant as commercial glucose is obvious. Several of the states have in their food laws a provision requiring that so-called "compound" foods (such as molasses containing commercial glucose), to be legally sold, must have formulas setting forth the names and percentages of their ingredients. In such cases it becomes virtually a necessity for the analyst to be able to verify, within certain limits, such formulas and thus guard against fraudulent and misleading statements thereon.

Unfortunately, from the variability in composition of commercial glucose, there can obviously be no accurate method for its determination, especially in complex, saccharine products in which one is likely to find it, and which themselves contain components common to glucose.

In enforcing the food laws conservatively, a formula on a package expressing the name and percentage of ingredients in the food contained therein is not condemned, because it differs by a few per cent. only from the analyst's findings, nor should the presence of a mere trace of an adulterant, which may be accidental, serve as a basis for a complaint in court. It is only when substantial deviations from a manufacturer's formula are found, or the presence of enough of the adulterant is evident to make it an object for fraudulent use, that the analyst should condemn it. In cases like these, an approximate method must be used, but preferably one, the adaptability of which to the purpose in hand has been well tested by long trial.

In molasses, syrups, honey, and similar preparation wherein commercial glucose is present as an adulterant, it has been the practice of the writer for upwards of ten years in the prosecution of such cases under the food laws of Massachusetts to calculate the approximate amount of commercial glucose present by assuming 175 as the direct polarization of a normal weight of the glucose, 26.048 grams, made up to 100 cc. and polarized in a 200 mm. tube. From this was naturally developed the formula which was long afterwards included by the writer in the methods outlined by him as an associate referee for the analysis of saccharine products, and later incorporated in the provisional food methods of the Association of Official Agricultural Chemists,[1] as follows:

$$G = \frac{(a - S)\,100}{175},$$

where G = per cent. of commercial glucose in the sample,

[1] Bureau of Chemistry, Bulletin 65, p. 48.

a = direct polarization of sample, and S = per cent. of cane-sugar as calculated from direct and invert polarization according to Clerget. This method and formula have been severely criticised by Mr. Edward Gudeman,[1] who regards the use of a single assumed factor as unwarranted, and one object of the present paper is to reply to his criticism and to show how satisfactorily the method has proved in practice. The formula applies more particularly to molasses and maple-syrup, as stated in the provisional methods. For honey, the same factor, 175, is used, but more accurate results are obtainable by dividing the polarization of the sample at 87° by this factor.

When asked in court, as one is sure to be in a contested case, the amount of commercial glucose in the sample, it is convenient to reply that *at least* such and such an amount must necessarily be present. By assuming in the above formula the highest figure that is apt to be found as the direct polarization of commercial glucose (175), it is obvious that the resulting value for S is the lowest or minimum one, thus giving the benefit of any doubt to the defendant. In other words, it is perfectly safe to allege that amount of glucose.

Realizing the uncertain composition of commercial glucose due to variations in amount of dextrose, maltose, dextrine and water present therein, the above method of calculation was long regarded by the writer as of doubtful value on theoretical grounds, and was not published for several years after being used. Continued opportunities were furnished, however, for comparison of the results thus calculated with actual conditions prevailing, such for example as occasional recipes furnished by defendants in court showing how the products were mixed, and one could hardly fail to be impressed by the striking and almost invariable similarity in results. Longer experience produced greater confidence, and it was thought best to examine a number of samples of commercial glucose obtained in a variety of ways from confectioners, mixers of compound honey and molasses, and jelly and jam manufacturers, with a view to ascertain the grades actually used by them and hence best suited for their purpose. It was found that considerable uniformity prevailed in the character of the glucose employed for different uses, especially as regards polarization. Thus seven

[1] Proceedings of 19th Annual Convention of the Association of Official Agricultural Chemists, Bur. of Chem., Bull. 73, p. 65.

samples, obtained in 1893, 1898 and 1901 from as many different manufacturers of compound molasses and honey or from dealers supplying them, polarized as follows:[1] 173°, 171°, 176°, 175°, 178°, 168° and 177°, the average being 174°. The writer's experience is not in accord with Mr. Gudeman's, who states that he has never known the manufacturer of glucose to furnish special or uniform grades for special purposes. In this connection the following is in point from the letter of a kind friend who obtained, through a broker, samples of glucose for analysis and who quotes from the broker's letter: "I am sending you two samples of glucose from the factory with the following advice: *42° Bé. is the grade used for syrup and honey, the XXXXX is the grade used for chewing-gum.*" The latter grade was found to polarize at 184.6°.

Another letter from a molasses mixer, who labels his product correctly under the law, runs as follows: "*Only one kind of glucose* is used in this kind of mixture, a sample of which we send you, called in the trade mixing or "M" glucose; the gravity we believe is somewhere about 42° Bé."

The molasses mixer naturally prefers the grade of glucose which, by its consistency, is best adapted without further treatment for mixture with his product. If it is too heavy in body, it must first be watered, and again if too thin it is equally unfit for direct use. Commercial glucose of 42° Bé. in density is admirably suited to his purpose, and this is the grade that more often polarizes from 170° to 175°. A number of samples obtained from confectioners and makers of compound jellies and jams were found to polarize from 150° to 157°.

Out of eleven samples of commercial glucose used for a variety of purposes in food and obtained from manufacturers of these foods, but one sample was found to polarize above 178° (the "XXXXX" brand above referred to), and none below 150°. Doubtless wider variations may possibly occur in commercial glucose of different densities as found on the market, but the point made here is that only such grades as are adapted for the particu-

[1] Figures being expressed in terms of 26.048 grams glucose made up to 100 cc. with water and polarized in a 200 mm. tube on the cane-sugar scale of the S. & H. instrument. It should be noted that in polarizing glucose, or molasses and syrups adulterated therewith, a 100 mm. tube should be used and the necessary correction applied, in order to bring the high reading within the limits of the scale. For exact data as to clarification of sample and other precautions, the reader is referred to Bull. 65, Bureau of Chemistry, p. 47.

lar purpose in hand are used by the food manufacturer, and these come within certain particular limits.

Mr. Gudeman's deductions regarding the composition and variations of glucose show his familiarity with its manufacture, but he makes no mention of the effect of maltose as one of the important reducing sugars present, though he apparently makes allowance for it in a process which he suggests as an alternative to the provisional method. In this process he estimates the reducing sugars before and after inversion, and again after hydrolyzing with malt extract, calculating by difference the dextrose resulting from the hydrolysis of the non-reducing substances. In making the latter calculation, he assumes a factor which can be true only of a fixed proportion of maltose to dextrine, a condition which cannot, of course, be depended on. Another source of error in Gudeman's method lies in the fact that his results are expressed in percentage of dry substance, leaving one in the dark as to the actual amount of glucose present as an adulterant, unless one assumes also a definite percentage of water in the glucose. A determination of dry grape-sugar or dextrose in molasses or honey is of no importance whatever to the enforcer of the food law.

Mr. Gudeman seeks to prove the unreliability of the provisional method by applying it to various hypothetical mixtures of grape-, cane- and invert-sugar, for which the method never was intended and could not, under any possible conditions, be used. The provisional method applies solely to molasses and syrups as found on the market, and to the commercial glucose used as an adulterant. No ambiguity need attend the use of the term commercial glucose in this country. The product has too important a place in trade not to be a pretty well understood article. By it is not meant a mere haphazard mixture of dextrose, maltose and dextrine, but a well-defined series of graded products of varying density, depending on the degree of conversion, but which, as the result of long experience in manufacture, are fairly uniform, so that the product of a given grade or density has a polarization varying between narrow limits. While the confectioner from his multiplicity of products can use various grades, the honey and molasses mixer is limited to the use of a special grade best suited to his use.

In carrying out the provisional method, commercial glucose is considered as just as much of an entity as milk, for example. To cite a parallel case, it has long been a successful practice for the food analyst to approximately calculate the amount of added water in milk by using an assumed constant for a factor, as for instance, the per cent. of solids not fat or total solids, in spite of the well-known wide variation in the normal constituents in milk. It is equally legitimate to adopt a constant for calculation of commercial glucose, based on such a uniform factor as the polarization seems to be in such cases.

Not the least of the advantages of the method is its simplicity, requiring only a direct and invert reading of the sample, which must be done in any event before deciding on the presence of commercial glucose. The method has never been claimed to be exact, but continued experience shows it to yield results much nearer the truth than was at first supposed possible.

Molasses, table syrup, and honey, put up in packages having incorrect formulas thereon, are included in the lists of adulterated brands, which, under the law, the Massachusetts Board of Health is obliged to publish monthly. The calculation of glucose in these cases is always based on the use of 175 as a factor, and experience has shown that manufacturers who doubt the findings of the Board are not slow to challenge its results.

Finally, the method has never been discredited, after long usage in the Massachusetts courts, where, of all places, in closely contested cases it is naturally subject to any criticism that may reasonably be brought against it.

PURIFICATION AND ESTIMATION OF IODINE.[1]

By Abraham Gross.

Received July 7, 1903.

In view of the importance of iodine in the arts and of the apparent difficulty in obtaining it absolutely pure, a discussion of this subject does not appear untimely and may prove of some value. Very few methods have been published in the direction of iodine purification and but one finds favorable mention. This is the method used by Stas in his researches on the atomic weight of iodine, and consists in dissolving iodine in a solution of potassium iodide, precipitating the iodine with water, drying over calcium nitrate, and subliming the dried mass. This method is criticized

[1] Read before the Pittsburg Section, June 18, 1903.