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.

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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.

by Lean and Whatmough,[1] who state that pure iodine can be obtained by heating cuprous iodide in dried air at 240° C. when the iodine is liberated.

Other investigations on this subject have been made by C. Meineke[2] and Z. Musset.[3] With a desire to obtain a reliable method for purifying iodine it was considered advisable to test the efficacy of the Stas method, and to compare the purity of the iodine thus produced with that obtained by other methods which suggested themselves at the time.

Accordingly, the following methods were tried: (1) The Stas method; (2) washing iodine with water, drying a portion over sulphuric acid, another portion over calcium nitrate and a third portion over calcium chloride; (3) mixing iodine with potassium iodide and drying over sulphuric acid.

Each of the above was then sublimed three times after drying.

The first method was carried out according to the directions of Stas, who states that to dissolve four kilos of iodine a solution of 1 kilogram of potassium iodide in 1 kilogram of water was required. These proportions were found inadequate, for, after standing some time with frequent shaking, a considerable portion of the iodine remained undissolved.

Several additions of potassium iodide and water were made, until complete solution was effected. This was accomplished when the proportions were 4 of iodine to 2 of potassium iodide and 2 of water.

The solution of iodine was then poured into water and allowed to stand until all of the iodine was precipitated. The supernatant fluid was poured off and the iodine washed with water until free from potassium iodide. It was then brought upon a filter, with sand as a filtering medium, and the water withdrawn by suction. The iodine was then transferred to a shallow dish and dried over calcium nitrate, which was replaced as soon as affected by the moisture. The drying continued twelve days. The dried sample was then sublimed three times by a process which will be described later.

In the next three methods, the iodine was washed with water fifteen times, and, after as much water as possible had been withdrawn by suction, was divided into three portions and dried, as

[1] *J. Chem. Soc.* (London), 1899.
[2] *Chem. Ztg.*, 16, 1219.
[3] *Ztschr. anal. Chem.*, 1891, p. 45.

previously mentioned, over sulphuric acid, calcium nitrate and calcium chloride respectively.

In conducting this experiment, two other points were in view: (1) To determine the best drying agent; (2) to ascertain whether any chlorine would be set free and absorbed by the iodine when the sample was dried over calcium chloride.

It may be mentioned here that the calcium nitrate and sulphuric acid were equally efficient as drying agents, but the inconvenience of constantly changing the calcium nitrate rendered it less desirable. Each of these samples was then sublimed three times.

The fifth sample was prepared by mixing iodine with pure potassium iodide, drying over sulphuric acid and subliming three times. The iodine obtained by this method was discarded, as a white residue remained after each sublimation. Apparently the potassium iodide was carried over with the iodine vapor.

PROCESS OF SUBLIMATION.

The iodine prepared by the methods previously described was placed in a piece of combustion tubing slightly inclined and resting on a support. Following the iodine was a plug of ignited asbestos to prevent the heated iodine from running down the tube. The lower end of the tube was connected to two tubes containing phosphoric anhydride and one containing calcium chloride. In this manner the air drawn through was perfectly dry. The upper end was covered by a bottle fitted to a rubber stopper to catch any vapor not previously condensed.

Through the stopper passed a glass tube which connected with an empty jar as a precautionary measure and in turn with a cylinder containing water and with the suction-pump. The suction was regulated by the bubbles produced in the cylinder. To prevent any accident from too sudden heating, a piece of iron pipe was placed over that part of the tube containing the asbestos and iodine and extending beyond for about 4 inches. This was protected from the glass by asbestos. A gentle heat was used and enough suction applied to condense the vapor sufficiently far away from the pipe to prevent any liquefaction of the sublimed crystals.

The succeeding two sublimations were conducted in precisely the same manner.

The following method proved itself highly satisfactory in the

determination of the purity of iodine. About 2 grams of the iodine were placed in a flask, 40 cc. of water, and about 4 grams of shot-zinc added. The flask was then shaken and allowed to stand with stopper inserted until the fluid was colorless. When all the iodine was taken up by the zinc, the solution was filtered into a half-liter flask, the residue and zinc hydroxide washed with hot water until free from iodine and the fluid made up to 500 cc. Fifty cc. of the solution were placed in a porcelain dish and titrated with silver nitrate, with potassium chromate as an indicator, until the end point, a slight brownish color, was reached.

The following results were obtained: Stas method—mean of five determinations gave 100.02 per cent.; second method—mean of five determinations gave 99.65 per cent. A sample of unpurified iodine gave, by this method, 98.83 per cent.

The iodine dried over the calcium chloride was tested for chlorine according to Fresenius,[1] but none was found.

For comparison, a sample of unpurified iodine was tested by the same method and the presence of the chlorine was indicated.

The following summary may be deduced from the foregoing research: (1) The Stas method gave the purest iodine; (2) sulphuric acid was the best drying agent; (3) the iodine was not contaminated when dried over calcium chloride; (4) the iodine, when pure, was satisfactorily determined when converted into zinc iodide and titrated with silver nitrate, with potassium chromate as an indicator.

A COLORIMETRIC METHOD FOR THE DETERMINATION OF SMALL QUANTITIES OF POTASSIUM.

By Lucian A. Hill.

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This method is primarily intended for use in the analysis of soil extracts and drainage waters where the potassium is in such small quantities that it is impracticable to use a gravimetric method. It has been used in this laboratory on a number of soil extracts, and has given very satisfactory results.

[1] "Qualitative Analysis."