VOLUMETRIC DETERMINATION OF SUGAR IN MILK.*

BY JOSEPH L. MAYER.

Shortly after Benedict published his "The Detection and Estimation of Glucose in Urine," I read a paper before the New York State Pharmaceutical Association, "Quantitative Estimation of Glucose in Urine." (Proc. N. Y. State Pharm. Assoc., 1914, page 298) in which I submitted the results of estimating glucose in urine volumetrically by means of Benedict's solution and gravimetrically by means of the method of Defren-O'Sullivan in which it was shown that both methods gave practically identical results, and since the volumetric method is shorter, attention was called to this advantage over the other method.

It then appeared to me that it would be a good plan to employ Benedict's solution for the quantitative determination of sugar in milk and to determine the accuracy of the method the following work was undertaken:

Twenty-five grammes of the milk (24.2 Cc.) were transferred to a 250-Cc. flask, 0.5 Cc. of a 30% solution of acetic acid were added and the contents well shaken. After standing for a few minutes, about 100 Cc. of boiling water was run in, the contents again shaken, 25 Cc. of alumina cream was next added, the flask shaken once more, and set aside for at least ten minutes. The supernatant liquid was then poured upon a previously wetted ribbed filter, and finally the whole contents of the flask were brought thereon, and the filtrate and washings made up to 250 Cc.

The alumina cream was prepared by dividing a cold, saturated, aqueous solution of alum into two unequal portions, to the larger of which there was added a slight excess of ammonia, then adding by degrees the remaining portion to a faint acid reaction.

The sugar in the clear solution was then determined by employing the following modification of Benedict's method which I recommended in a paper read at the Annual Meeting of the American Pharmaceutical Association (J. A. PH. A., May, 1914, page 687).

Into a 100-Cc. Erlenmeyer flask, with cord wrapped around the neck to prevent burning the fingers, there was added 25 Cc. of accurately measured Benedict's solution, a few grammes of cryst. sodium carbonate and the whole placed on the hot plate. When the solution was boiling the sugar solution was gradually added from a burette with sufficient slowness to allow the reaction to proceed, putting the flask back on the hot plate until the disappearance of color.

The sugar in this same solution was then determined gravimetrically by the following method of Defren-O'Sullivan (Leach, Food Inspection and Analysis, 2nd Edition, page 564):

Fifteen Cc. of Fehling's copper solution was mixed with 15 Cc. of the tartrate solution in a quarter liter Erlenmeyer flask and 50 Cc. distilled water was added, the flask and its contents were then placed in a boiling water bath and allowed to remain there for five minutes; there was then run rapidly from a burette into the hot liquor in the flask, 25 Cc. of the sugar solution to be tested (which should contain not more than one-half percent of reducing sugar). The flask was allowed to remain in the boiling water bath just fifteen minutes after the addition of the sugar

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solution, removed, and with the aid of a vacuum, the contents were rapidly filtered in a porcelain Gooch crucible containing a layer of prepared asbestos fiber about I Cm. thick, the Gooch with the asbestos having been previously ignited, cooled, and weighed. The cuprous oxide precipitate was thoroughly washed with boiling distilled water till the water ceased to be alkaline.

(The asbestos was of the long-fibered variety and was especially prepared as follows: It was boiled first with nitric acid (specific gravity 1.05 to 1.70), washing out the acid with hot water, then boiled with a 25 percent solution of sodium hydroxide, and finally the alkali was washed out with hot water. The asbestos was kept in a wide-mouthed bottle and transferred to the Gooch by shaking it up in the water and pouring it quickly into the crucible while under suction.)

The Gooch with its contents was dried in the oven, and finally heated to dull redness for fifteen minutes, during which the red cuprous oxide was converted into the black cupric oxide. After oxidation as above, the crucible was transferred to a desiccator, cooled, and quickly weighed. From the milligrammes of cupric oxide, the milligrammes of lactose were calculated from table accompanying the method.

The results by both methods were as follows:

Gravimetric	3.31%
Volumetric	3.28%

0.03% Difference

These results clearly indicate that the volumetric method of Benedict modified as above while rapid and easily applied is capable of yielding just as accurate results as the longer gravimetric method.

The Benedict's solution was prepared as follows:

Copper sulphate (pure, crystallized)	18 Gm.
Sodium carbonate (crystallized)	200 Gm.
Sodium or potassium citrate	200 Gm.
Sodium sulphocyanate	105 Gm.
5% Potassium ferrocyanide solution	5 mils
Distilled water to make	1000 mils

With the aid of heat dissolve the carbonate (half the amount of anhydrous salt may be used), citrate and sulphocyanate in enough distilled water to make about 800 mils; filter if necessary. Dissolve the copper sulphate in about 100 mils of distilled water, and pour this solution slowly, with constant stirring, into the other solution. Add the ferrocyanide solution—cool and dilute to exactly 1000 mils.

A reference to Benedict's original formula will show that it calls for 125 grammes of potassium sulphocyanate but since the war it has practically been impossible to obtain this salt and I have, therefore, employed the sodium salt in the above amount. This lot of Benedict's solution as well as all others employed in our laboratory was standardized by the method of the U. S. P. (page 558), as follows:

To a solution of 0.95 Gm. of pure cane sugar in about 50 mils of distilled water, 2 mils of hydrochloric acid were added and the whole heated at 70° C. for ten minutes. After neutralizing with sodium carbonate, the solution was diluted to 1000 mils.

The number of Cc. of this solution required to completely reduce 25 Cc. of Benedict's solution gave the factor.

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A STANDARD DOSAGE MEASURE.*

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Accuracy in the compounding of medicines is a first principle instilled into the mind of a pharmacist and so he realizes fully the importance of correct weights and measures. This is a matter that is now also receiving official attention in some of the States, where the scales, weights and measures of the pharmacists, the same as those of other merchants, are periodically inspected by a representative of the State or Municipal Department of Weights and Measures.

While in the past the apothecary may have used graduated measures of variable shapes and with indifferent and inaccurate markings, today he is supplied by the manufacturers with "standard graduates," the shapes and graduation of which have been standardized with the coöperation of the United States Bureau of Standards, and the use of which is already compulsory by statutes in some of the states.

Accuracy in the administration of medicines is equally as important as accuracy in their preparation. The physician calculates the amount of active medicament that he intends shall be given his patient in each dose of the prescription that he directs. To the trained physician the word dose has a well defined meaning, namely, the right amount to obtain the desired effect, no more and no less. So it is evident that if too much be given untoward effects or undesirable reactions may result and that if too little be given there may be expected a failure to produce such effect. In either case, the intent of the physician may be nullified with detriment to his patient. It is the height of inconsistency to invalidate the judgment, the professional knowledge and skill of the physician and the pharmacist and to make these useless by the careless or inaccurate administration of the medicines.

The inaccuracy of the ordinary dose measures has been so frequently decried that the variability and uncertainty of these should be common knowledge. The almost universal custom is for the physician to direct as a dose of a liquid medicine, either a teaspoonful, a dessertspoonful, a tablespoonful, or possibly so many drops. Spoons of all sizes and shapes are marketed by the various manufacturers without any attempt to standardize the content of those bearing the same designation. In the same household one teaspoon may hold 55 minims and another as much as 80 minims and as great a range of variation may be shown by the dessertspoons and the tablespoons. Another source of error in measuring with the ordinary metal spoons is what may be termed the personal equation. One person does not hold the spoon level, another gauges the spoon as full when it is not en-

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