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## NOTE.

Reply to Dr. Ewell's "Notes on the Paper by Hiltner and Thatcher.—In an article recently published in this Journal, we outlined a modification of the Sachs-Le Docte modification of Pellet's "instantaneous aqueous diffusion methods for the estimation of the sugar content of beets." In the succeeding number of the Journal, Dr. E. E. Ewell published some notes on our paper<sup>1</sup> criticizing the proposed modification in several points. Inasmuch as Dr. Ewell has apparently fallen into some of the errors which we specifically warned against in our previous article, it would seem that a further word of comment is necessary. This reply has been delayed by the withdrawal of Mr. Thatcher from this laboratory and by the many distractions of the summer vacation season, but may not be out of place at this time.

<sup>1</sup> This Journal, **23**, 432.

The object for which the work reported in our previous article was undertaken, was an investigation of the possibility and feasibility of doing away with the necessity of weighing an exact definite weight of beet pulp for each analysis, and using instead any known weight of pulp with its proportionate amount of water and lead subacetate solution. When we began the work we were unaware that such a device had ever been suggested, since Walawski's work had not yet been published in any of the journals on file in our library. Subsequent correspondence with the Division of Chemistry at Washington, secured for us a copy of a translation of the paper of Dr. Sachs from which we quoted freely in our article. After we had completed our work and a copy of the report of it had gone into the hands of the printer, a personal letter from Dr. G. L. Spencer informed us that he had for some time past been making use of a modification of the method in question, by which any desired weight of pulp might be proportionately diluted, clarified, and polarized. The idea, although original with ourselves, was therefore not new, but inasmuch as our modification differed in some essentials from the commonly adopted methods, and in some important respects from Walawski's process, particularly as to practical details-its publication was considered advisable.

Dr. Ewell criticizes the suggested modification because it is based upon a correction for the volume of water in the beet rather than the volume of juice, or liquid in the beet. It is admitted at the outset, of course, that either the Sachs-Le Docte assumption of an average juice factor or our assumption of an average water factor will give results varying somewhat from the truth in individual cases. Which of these two assumptions is the more desirable as a basis for a rapid method for technical analysis depends, therefore, upon two things : first, the probable error that may be introduced in individual analysis, and, second, the ease with which a factor to accurately represent the average of the beet to be analyzed may be determined.

Researches during the past ten years by sugar-house and experiment-station chemists show that the amount of *marc*, or insoluble matter, in the beet often rises to 8 per cent. and sometimes even to 10 per cent. of the weight of the beet. A very large number of direct determinations of this factor made at this laboratory during the past seven years shows that in immature or

TABLE I. INSOLUBLE MATTER (MARC) IN BEETS.						
Year.	Number o determinatio		Minimum. Per cent.	Average. Per cent.		
1892	15	8.4	2.5	4.I		
1894	6	4.6	2.2	3.3		
1895	14	7.0	2.4	3.7		
1896	7	3.6	2.0	2.4		
1897	IO	5.9	2.2	3.5		
1898	8	4.0	2.0	2.9		

low-grade beets, it sometimes falls as low as 2 per cent. This is shown in the following table :

Hence the assumption of 5 per cent. of insoluble solids, or 95 per cent. of juice, as an average for the calculation of the volume occupied by this juice must produce a significant error in many cases. The tediousness and extreme difficulty of a direct determination of the amount of insoluble matter in beets, and the unreliability of the results obtained by an indirect estimation of this factor, make it almost impossible to control the work of any given season or portion of a season by a determination of the correct average juice factor of the beets to be analyzed.

Furthermore, the calculation of the volume occupied by a definite weight of juice by the ordinary methods is subject to several errors. In the first place the usual method of determining the specific gravity of the juice by means of the Brix spindle gives erroneous results because the spindle is calibrated in solutions of pure sugar. Investigations carried on in this laboratory through several seasons show that the results obtained in this way are almost invariably too high, the percentage of total solids obtained by a direct determination, or indicated by an accurate determination of the specific gravity of the juice, being always less than that indicated by the spindle. As is shown in the following table, the real coefficient of purity is always higher than that obtained by calculation from the Brix spindle and polariscope readings. In other words, the amount of non-sugars in solution is less than that generally accepted and the volume of the resulting solution is, therefore, less.

		Differences between real and apparent purity.		
Year.	Number of determinations,	Maximum difference, Per cent.	Minimum difference. Per cent.	Average difference, Per cent.
1892	13	+ 9.10	+ 3.30	+6.2
1894	6	6.30	+3.30	+4.6
1895	15	-9.13	- 2.96	+5.2
1896	10	+5.97	+1.20	+3.4
1897	14	+5.97	+2.34	+3.8
1898	ģ	+4.80	+ I.70	+ 3.3

TABLE II. REAL VERSUS APPARENT PURITY.

It is evident, then, that the only correct basis from which to calculate the volume of any given weight of juice is a direct determination of the specific gravity of the juice, or of the total solids in solution in it. This latter is the same determination upon which we base our correction for water content.

Again, even though the volume of the juice in the sample taken be correctly determined, the figures are of no real value in determining the volume of water to be added, since the juice is clarified by the same process by which its volume is to be increased to the normal dilution, the percentage of solids in solution being materially diminished with its attendant effect upon the volume of the solution. A portion of the soluble non-sugars as well as a portion of the lead in the clarifying agent are removed from the solution by precipitation. The exact amount of the diminution in volume due to this cause in any particular case it is impossible to determine. It is obvious, however, that Dr. Ewell's carefully prepared tables, based upon the assumption that the juice in the pulp taken has the same specific gravity as would a solution which contained a percentage of pure sugar, equal to the apparent amount of total solids in the juice, and that the solution contains all the solids originally present in its constituents are not true to facts and lead to erroneous conclusions. Moreover, both of the errors introduced are such as would, in actual practice, tend to diminish the total volume of the solution and so in part, counteract the error due to the increase in volume caused by the sugar dissolved on the water of the juice.

It would seem, therefore, that both the theoretical consideration of the matter and the practical results obtained, and reported in our previous article, show that the assumption of an average water factor as a basis for a rapid working method is fully as satisfactory as that of an average juice content. The determination of the correct average factor to be used in any series of analyses is unquestionably much more easily made in the former case. The error in individual cases might be lessened by doubling the dilution as required by the Sachs-LeDocte modification, but as was pointed out in the previous article (see page 310), this has not been found necessary in ordinary work.

Dr. Ewell further criticizes our work "because their determinations of the water contained in beets, \* \* \* are higher than is indicated by previous results or determinations of the amount of filter contained in beets' and further states "that as little as 4 per cent. of marcis possible, but 2.61 per cent. is doubtful and 2.28 per cent, is still more so'' and supports his criticism by tables calculated from assumed conditions. The figures cited in Table I above are sufficient to show that the results recorded are not at all impossible, but, on the contrary accord very satisfactorily with the other observed facts concerning the beets with which we had to deal. The results which we have recorded on pages 308 and 309 in every case were those obtained by the best of the direct methods of determination and not by indirect calculations using It is but fair to add that, owing to very late arbitrary factors. warm Fall rains in Nebraska last year, the beets which were available for work at that time were very largely immature and unripe ones. It is hoped that in the near future, the applicability of the proposed method may be further tested on a better class of beets. The determinations of moisture recorded on page 309 of the original article were made on beets at all stages of growth from samples taken from July 15th to November 15th of each year, and, hence, as was stated, represent widely varying conditions of the beet.

It is a fact well known among chemists actively engaged in work with beets that the factors arbitrarily adopted to represent an average beet are often far from the real conditions to be met with in actual work and that percentages calculated from these arbitrarily assumed standards are by no means sufficiently reliable to afford a basis for criticism of results obtained in actual practice.

Our critic makes a number of logical deductions from certain computations that he has made, but we must beg to insist that he errs considerably in his premises. As a basis for his calculations on page 433 he makes an assumption as to purity coefficient (80 per cent.) which is quite untenable in view of the facts as ascertained by us. The coefficients of purity of the beets in question seldom reached 75 per cent., as our records show ; many analyses indicated a purity of less than 70 per cent., some indeed as low as 65 per cent. Were we to apply such factors as these in the same manner as Mr. Ewell has applied the factor 80 we would obtain most astounding figures. The danger in building too much on averages and on conventional factors is here very apparent.

Although a little remote from the main point in question we

wish finally to notice the fact that in making these computations for the tables on page 433, errors have been made aside from those just mentioned. The values there used, for average sugar content in the beets, were obtained by us by indirect analysis of the beets. The percentages of sugar in the juices were first determined and from these the percentages in the beets were estimated by deducting the conventional 5 per cent. for marc. Using these same figures in connection with other data. Mr. Ewell *calculates* the percentages of marc to range from 1.79 per cent. to The error also in attempting to calculate *soluble* 4.74 per cent. non-sugars in the beet and later the percentage of marc by employing an assumed factor (or even a definitely determined factor) of *purity of the juice* is quite apparent. (By factor of purity of the juice we mean, of course, the ratio of sugar in the juice to soluble total solids in the juice: and by soluble non-sugars the difference between total solids and sugar in the juice.) By thus confusing the values for the beet and those for the juice it is manifestly impossible to compute values for marc or other components of the root.

To conclude, we wish only to again draw attention to the facts stated on page 311, *et seq.*, of the current volume of this journal regarding the experimental part of our work and especially to Table III. where the results obtained by the proposed method are compared with those secured by Pellet's hot aqueous diffusion process (an acknowledged standard method). The results given certainly point plainly to the reliability of the method and indicate that the process is based on correct principles.

R. S. HILTNER AND R. W. THATCHER.

UNIVERSITY OF NEBRASKA, LINCOLN, NEB., September 9, 1901.