AN ACCURATE COMMERCIAL METHOD FOR THE ANALYSIS OF SUGAR BEETS.[1]

By David L. Davoll, Jr.

Received September 6, 1906.

The indirect analysis of the sugar-beet by the juice method and the use of the transposing factor so much in vogue in the United States is open to serious objections, for while it does not affect the price paid by the factory, provided a reasonable control is kept over the factor by frequent direct analyses, it is bound to work injustice to individual farmers, while at the same time unduly favoring others.

It has been our experience that the factor progressively diminishes as the harvesting and the beet-storing season advances, varying from 0.95 in early September to 0.88 in late January.

Frozen beets offer serious obstacles to the indirect method and this condition may occur in most northern states any time after November 1st.

A factory slicing 1,000 to 1,200 tons of beets per day must be in a position to make in its receiving laboratory from 400 to 500 analyses per day of ten working hours, and with the minimum working force. In good weather 500 ton houses must handle this number.

The practical workings of the much recommended instantaneous aqueous method have not been such as to secure its adoption, and about 125 samples a day is the limit to the number that may be analyzed with any degree of accuracy by one chemist and assistant in one day, using the ordinary method of hot water digestion in a flask. The latter method is outlined as follows:

"Ten cc. to 16 cc. of solution of lead subacetate of 54.3 Brix are placed in a 201.2 cc. sugar flask and 52.096 grams of shredded beet are introduced by means of a glass rod assisted by a spray of water; the volume is finally completed to about 190 cc. A little ether is added to beat down the foam and the whole heated in a water-bath at 80° for thirty minutes, rotating the flask from time to time to promote extraction and facilitate the escape of air bubbles. Water is occasionally added so that the volume is completed to the mark at the end of thirty minutes. Cool to

[1] Read before the Sixth Congress of Applied Chemistry, Rome, Italy, April, 1906.

room temperature, add ether to dissipate any remaining foam, dilute with water to the mark, mix thoroughly and filter."

The criticisms of this method are the following: The introduction of the beet pulp into the flask is painstaking and time-consuming, a medium fineness of a shredded nature giving most trouble. The rotation to secure proper extraction and get rid of occluded air, when properly attended to, requires much attention and the repeated addition of ether, while careless operators, in practice, neglect rotation, particularly when in a hurry. Where 26.048 grams are used in 200.6 cc. dilution, the manipulation is more satisfactory, but the use of 200 mm. tubes requires the objectionable doubling of the reading. A double normal should always be taken in commercial work, being more representative of the original sample, since perfectly intimate mixing cannot be counted upon where the chemist is dependent upon the unscientific helper to prepare the sample. Rapid and uniform cooling cannot be obtained without stirring the flask contents, which is difficult. After adding the final quantity of water, intimate mixing of the water content, which is absolutely essential, is rendered difficult by the presence of the pulp.

The author has overcome the objections to the method of hot water digestion mentioned above by substituting a beaker for the flask, and finally completing the mass to a certain weight, *i. e.*, 209.2 grams, instead of volume. A definite weight of water, and therefore a fixed volume of water also, cannot be added to a normal or multiple thereof of shredded beet, unless it first be ascertained that the diminishing volume of the definite weight of beet, due to increased specific gravity of the constituent juice with rise of sugar percentage, is without influence upon the polarization within the limits of accuracy of the saccharimeter. The Kaiser-Sachs modification of Pellet's instantaneous diffusion directs the weighing of a quantity of water, *i. e.*, 172 grams, where a normal of pulp is used, into the counterpoised flask containing the lead solution. In a modification of this same instantaneous method by cold digestion, Fr. Sachs and A. LeDocte, while measuring 5 cc. solution of lead subacetate and 177 cc. water upon 26.048 grams of shredded beet contained in a copper capsule, in effect use the principle of weighing.

If a strictly representative juice could be expressed from the beet it would be possible to ascertain its specific gravity and thus

calculate the gross weight of pulp, lead subacetate solution and water necessary to complete the volume to 201.2 cc. for beets of different sugar percentages. Obtaining the total solids in the beet and from this subtracting the marc, while giving the real Brix or total solids in the juice, would not give the Brix by "spindling," having its equivalent in specific gravity, for well-known reasons.

Ordinarily the quantity of lead subacetate solution to be used may vary according to the condition of the beet, whether it be fresh and normal or unripe, spoiled, frozen or has been preserved in silos. At the moment of adding the lead subacetate the chemist can scarcely be expected to detect any of these conditions and will be guided considerably by the season, by experimentation with various quantities and noting the effect of lead solution upon the clear filtrate. The first step in the investigation was to ascertain how much would be the weight of 52.096 grams of pulp, 14 cc. of lead subacetate solution and water when mixed, digested, cooled to 20° (the average room temperature) and diluted to exactly 201.2 cc.; or if the weight varied, to establish that variation. Sixty-one individual analyses were made in the following manner: The weights of the clean, dry, 201.2 cc. flasks were carefully ascertained and the capacities in Mohr cubic centimeters determined. This latter was found to vary between 200.76 cc. and 201.2 cc. which, while influencing the weight of the contents and is of importance in the experiment, does not affect the polarization.

The several analyses were then made in the usual manner of the hot water digestion, the flask carefully wiped outside and, with its contents, weighed and the polarization made. The weight of flask contents and the polarization were then corrected to 201.2 cc. capacity at 20°. The weight of the flask contents was found to vary between 207.67 grams (for 11.9 per cent. beets) and 210.08 grams (for 17.9 per cent. beets) with the average of 209.2. This latter may be taken, therefore, as the correct weight to which the mass may be adjusted in water digestion for beets in the receiving laboratory or for *cossettes* at the diffusion battery. It might be remarked here that the beets are purposely selected so as to get as many types as possible, as regards shape, size, degree of ripeness, sugar contents and frozen. The selection rep-

resents the pick of 500 to 600 tons passing along the conveyor to the automatic scale.

Upon the basis of the weights, corrected as above stated, a calculation was made to see how much the polarizations would have been affected had the weight at the end of the analysis been made up to 209.2 grams in every case, *i. e.*, to the average weight and a constant quantity. The differences, partly plus and partly minus, in some instances showed no variation at all, in others inappreciable, and in all cases, with beets containing less than 17 per cent. sugar, they were well within the limits of accuracy of the polariscope.

The average of the minus differences was 0.023 per cent., and of the plus differences 0.03 per cent., with the four extreme results showing 0.09, 0.06, 0.06 and 0.079 per cent. In these four cases, as in forty-eight other analyses, single beets *only* were taken, while in the remaining nine analyses but two beets were united. This was for the purpose of detecting any great irregularities due to individuals, but in a receiving laboratory or cossette analysis, where very many beets are united for one test, any peculiarities would sink into insignificance and show no eccentricities in variation.

The author has, therefore, dispensed with the use of a flask altogether in the analysis of beets and cossettes by the method of hot water digestion during the last sugar-making season, substituting therefor a glass beaker, which is superior to a container of any other material because its weight can be kept constant.

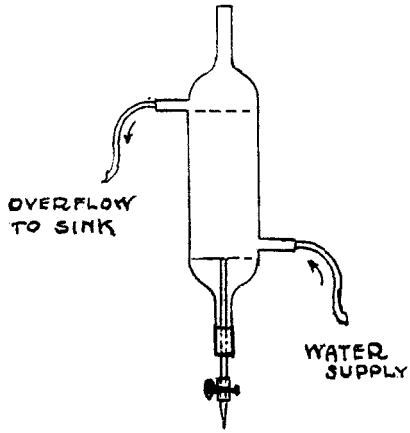
Many thousand individual analyses to determine the purchase price were made with accuracy, speed and cleanliness, and it was well demonstrated that from 600 to 700 analyses may be made by one chemist, one assistant and one wash boy in a day of ten working hours.

The preparation of the digestion beaker was as follows:

A quantity of 300 cc. Jena beakers was purchased, amounting to perhaps 200 in number; they were placed upon a balance, one at a time, and the heaviest selected. This was then provided with a $^{3}/_{16}$-inch tube of glass, sealed at both ends, provided with a rubber tip secured by copper wire, of suitable length to act as a stirrer. This heaviest beaker and its stirrer were then carefully weighed, and this weight was taken as the standard weight to which all flasks and their respective stirring rods were to be brought. A

metallic counterpoise was then made. In preparing the stirring rods for the other *lighter* beakers, metallic mercury was introduced into the hollow stirring rods, previous to sealing, in amount sufficient to bring them all up to the standard weight, *i. e.*, the weight of the heaviest and its stirrer, after which they were sealed. The beakers were then etched with consecutive numbers and the stirring rods as well to correspond.

The plan for handling the samples was as follows: After throwing the weighted pulp into the beaker, the weighing dish was rinsed free of traces of pulp into the beaker by means of an overflow pipette. This pipette can be home-made, and is best constructed from a glass condenser-jacket, as shown in the illustration, which practically explains itself. It is always filled



to the same level, but the quantity drawn off is regulated at the lower dotted line by sliding the glass tube up or down. The quantity to be drawn off is first determined by experiment and should be sufficient in amount to allow for evaporation during the half hour digestion at 80°. With the large bath in use this is a very uniform amount, and after cooling, the addition of from four to five drops of water completes the mass to 209.2 grams, plus the weight of the beaker and stirrer.

Two baths are necessary, one for digesting and one for cooling, and each should accommodate five baskets of ten beakers each at one time. The baskets are best made of galvanized iron perforated with holes, provided with handles which act as hangers

and having *double* brass wire strung across for keeping the beakers about $^3/_8$ of an inch apart. These two wires, side by side, passing between each beaker, prevent clashing and resulting breakage. The bottom is lined with two-ply rubber, perforated.

The farmer's tickets accompanying the sample are placed in a tin case having a large brass number soldered to it, and a corresponding number is upon a basket. The basket, placed with the number facing the chemist, is filled with beakers in a definite order, and the sample tickets are slipped into the corresponding tin box in a similar order or arrangement.

After digestion and cooling, the beaker is wiped outside (or allowed to drain and dry) and placed upon a balance, and with the contents brought with a few drops of water to the desired mass, *i. e.*, 209.2 grams plus the counterpoise. The analysis is then completed as usual.

This method will of course apply to sugar-cane and bagasse analyses, where, weight being applied instead of volume, the quantities may be proportionately increased and the difficulty in sampling thereby overcome.

CARO, MICHIGAN. March 10, 1906.

[CONTRIBUTION FROM THE BUREAU OF CHEMISTRY, U. S. DEPARTMENT OF AGRICULTURE].

THE RAPID DETERMINATION OF WATER IN BUTTER.

BY G. E. PATRICK.

Received August 11, 1906.

SINCE the laws regulating the percentage of water in butter have come into vogue there has grown up a persistent demand for some method whereby the butter-maker, the renovator and the butter merchant can easily and quickly determine the amount of water in his product or his commodity. The most exacting demand is that of the butter-maker in the creamery, or the churn-man in the renovating factory, who needs a method which will yield its results in a few minutes, while his butter-worker stands waiting with its load, further working to be dependent upon the result of the test.

The writer has studied this problem at intervals for two years or more and has at last found a method which is sufficiently accurate, and at the same time so simple in principle and so rapid