

Ethyl Selenocyanide, C_2H_5SeCN .—This compound, which appears to be new, was prepared from 16.1 grams of potassium selenocyanide and 15 grams of ethyl bromide in alcohol solution. The reaction is complete in a few minutes, and it is best then to add water without distilling off the alcohol. The oil is somewhat volatile in alcohol vapor and also in that of ether. On distilling at 741 mm. pressure it practically all boiled at 172° . It formed a pale yellow oil with a highly disagreeable odor. A nitrogen determination gave :

	Calculated for C_2H_5NSe .	Found.
Nitrogen.....	10.44	10.38

When this was warmed with thiobenzoic acid it gave off a gas or vapor, with the odor of hydrogen cyanide, and an oil and a solid were obtained. These products were not investigated.

AN IMPROVED METHOD FOR THE RAPID ESTIMATION OF SUGAR IN BEETS.

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INTRODUCTION.

EXPERIENCE has abundantly shown that both the good and the bad properties of any individual beet are largely hereditary and may be transmitted to succeeding generations of beets. The success of the beet-sugar industry in recent times has been due to a very large extent to the building up of a race of high-grade beets by means of the selection of individual beets having high sugar content for the production of the seed for later use. Long-continued and careful efforts in this direction have resulted in the production of beets of highly satisfactory sugar-producing qualities. This condition is more or less abnormal, however, and the tendency is toward a retrogression or reversion to the original state. In order to prevent this and to maintain the present high state of perfection, constant care in the selection of only the best beets for mothers for seed production is necessary. Not only is it essential to select those beets which possess the best form of leaves and root, but care must also be taken that only those whose roots are of high sugar content are used. The sugar content of individual beet roots under the same conditions of growth varies

widely. It is obvious, therefore, that a knowledge of the sugar content of each beet which is selected is of the utmost importance. The necessity of some simple, rapid, and accurate method for testing small samples taken from individual beets is therefore apparent. Moreover, the rapid development of the beet sugar industry in this country of late, emphasizes the need of some such rapid method adapted to factory control.

DISCUSSION OF PRESENT METHODS.

Many of the methods already suggested for the determination of sugar in beets are very satisfactory as far as accuracy is concerned, but all are more or less complicated and require the use of expensive apparatus and great care in manipulation. Furthermore, a large force of laboratory assistants is necessary in order to make the process of any of the known methods of analysis rapid enough for control work in the selection of mother beets. Dr. François Sachs, of Brussels, Belgium, in a paper read before the second International Congress of Applied Chemistry held in Paris, July 27 to August 5, 1896, gives a very clear and concise statement of the present methods for the analysis of sugar beets and suggests certain modifications and improvements in them. Inasmuch as this article has not yet appeared in American chemical literature, we quote the more pertinent portions of it below:¹

“We may divide the methods which have been proposed, for this purpose, into three groups:

“1. The indirect methods based upon the analysis of the juice.

“2. The alcoholic methods.

“3. The aqueous methods.

“The indirect methods no longer have any more than an historic interest, and their use, particularly in France, is gradually disappearing, more rational methods taking their place.

“It has long been believed that the use of alcohol is indispensable for the exact determination of the percentage of sugar contained in the beets. Even to-day this appears to be the opinion held by most German sugar chemists. In fact, when one removes the pulp of the sugar beet, extracts with alcohol, and washes the exhausted pulp with water, the aqueous solution thus obtained turns a ray of polarized light distinctly to the right. Hence, it has been concluded that alcohol is necessary to perfect

¹ Translation made by Division of Chemistry, Department of Agriculture, Washington, D. C.

the solution of optically active bodies, such as sugar. But it has been demonstrated, notably by the work of two Belgian chemists, Chevron and Droixhe, that the precipitation of these active bodies (which appear to be pectic bodies) is complete when subacetate of lead is added to their aqueous solution.

“It is necessary to conclude, after what has been said, that the alcohol and aqueous methods ought to give the same results with the same beets. This is exactly what has been observed by Pellet, Petermann, Weisberg, and others. The objection has been made that Petermann did not obtain in all of his experiments absolutely identical results, but the differences are so small that they may well be attributed to errors in sampling. Moreover, subsequent experiments made with greater care have given results still more concordant. It may seem that the results obtained by the aqueous diffusion method are too high by about 0.1 per cent., because the hydration of the marc is not taken into account, but this same error must occur when the alcoholic diffusion methods are used.

“On the other hand, the results obtained by the cold aqueous diffusion methods may be too high if the analyst neglects to completely eliminate the air imprisoned in the fine pulp, as has been frequently observed, notably by Wojcki in Russia, and by Nassou at Gembloux. When all of the common errors are eliminated, in the application of the two methods, no sensible difference between the results have been observed, either in Belgium or in France. In Germany, on the contrary, most of the chemists generally continue to affirm that the alcoholic methods give more exact results than the aqueous methods, and that for certain abnormal beets the differences may be very considerable.

“I shall not stop to discuss the hot aqueous diffusion methods which give good results, but which have not the desired simplicity. The cold aqueous diffusion, designed by our colleague, M. Pellet, is much more practical. However, as has already been said, this method as it is generally applied requires the complete elimination of the air imprisoned in the pulp, and therefore demands especial care. Moreover, the introduction of the pulp by means of a funnel into a flask with a more or less narrow neck, is an operation which it is very desirable to dispense with.

“It is these considerations that led us to modify the ordinary methods by introducing the pulp into a large ungraduated vesse

and adding to it at once by means of a pipette, the proper quantity of lead subacetate and water. This method was announced two years ago (April 24, 1894) at the meeting of the Association of Belgian Chemists. We then incorrectly attributed the original idea to Wojjcki. We wish to say now that it should be credited to Kaiser and Lewenberg.

“Kaiser¹ proposed to use 26.048 grams of the finely rasped pulp with the addition of 76 cc. of water and lead subacetate solution. He used a special correction in the case of very rich beets.

“Lewenberg² directed the use of 26.048 grams of the finely rasped pulp, 3 cc. of lead subacetate and 73.8 cc. of water.

“Wojjcki³ designed a simple apparatus, by means of which he demonstrated that in the ordinary method even as much as 3.1 cc. of air is left in the flask.

“Walawski⁴ proposed to take any convenient weighed quantity of the beet pulp and to add to it 3.6 times its weight of water and lead subacetate solution. This modification does not appear to us to be a desirable one.

“We have in our turn simplified somewhat the form of the apparatus used. We have found it convenient to use for the normal weight, 26.048 grams of the beet pulp, about 5 cc. of lead subacetate solution, and a sufficient quantity of water to bring the total volume of the liquid contained in the pipette to 77 cc. We tried this method and the results were not satisfactory. We found that it was necessary to shake the mixture for a very long time in order to obtain a complete distribution of the sugar throughout. We then decided to modify the method more radically and for that purpose constructed a pipette, having a capacity of 177 cc., which enabled us to obtain the conditions recommended for the ordinary method. Working in this manner we obtained perfectly satisfactory results. We have never found that more than three minutes are necessary to obtain a complete distribution of the sugar throughout the mixture.”

OBJECTIONS TO PRESENT METHODS.

Any of the methods now in use will give fairly accurate and

¹ *Deutsche Zuckerindustrie*, 1893, p. 413.

² *Dodatek*, 1892.

³ *Gazeta Cukrowicza*, 1893, p. 313; *Oester. Zeitschrift*, 1894, p. 146.

⁴ *Gazeta Cukrowicza*, 1894, p. 268; *Oester. Zeitschrift*, 1895, p. 1117.

satisfactory results if carefully carried out. They all possess in general, however, certain inherent sources of error, the inaccuracies resulting from which become greater and greater as carefulness of manipulation is sacrificed to speed. The several sources of error which may be mentioned here are as follows: First, the pulp itself is susceptible to changes in composition during weighing, both through the evaporation of its water and through pressure exerted by the appliances used in transferring the pulp from the dish containing it to the weighing capsule. In rapid work, where pincers or some similar appliance must be used to transfer small portions of the pulp either to or from the weighing dish in order to obtain the exact normal weight, the change in composition of the pulp thus handled becomes quite significant. Moreover, as in all cases where only a portion of the sample is used for analysis (the normal weight or some fraction or multiple thereof), unless great care is taken the portion which is weighed out does not accurately represent an average of the whole sample. Second, the amount of lead subacetate to be used in clarifying the juice varies, and too much or too little of the reagent is liable to be added, with a resulting effect upon the polarization value of the solution. Third, the sample of pulp occupies some space in the flask and the flask does not, therefore, contain exactly 100 cc. of liquid (*i. e.*, sugar solution). This error is compensated for in most cases by using flasks which are graduated at 101.3 cc., the 1.3 cc. additional being adopted as the average value for the volume occupied by the pulp. This requires, of course, specially constructed flasks, and the figure thus arbitrarily adopted is more or less far from the truth in individual cases. Fifth, as has been noted in the article quoted above, the pulp mechanically encloses considerable air, and the error due to the change in volume of solution thus produced may in some cases become very significant. Finally, the volume to which the solution is made is always measured in graduated flasks, the calibration of which is not always accurately done.

Moreover, in all the present methods of analysis as employed in this country, there are certain steps in the process which require considerable care and the manipulation of which requires much time. These interfere seriously with rapid work, and the speed of the operation is consequently quite limited. In the first place, a certain definite weight of pulp must be obtained. If

this is done with a tolerable degree of exactness considerable time is consumed. Next, the pulp must be transferred to a flask having a somewhat narrow neck. Owing to the tendency of the particles of pulp to agglomerate, this is a difficult and tedious operation at best. In the third place, the volume of the solution must be made up to the mark on the neck of the flask with care. Since all aqueous solutions of beet juice froth badly while the water is being added, some precaution to break this froth is generally necessary and it is with difficulty that the process is carried out quickly.

An ideal method for the rapid determination of sugar in samples of beet pulp would, therefore, be one in which the entire sample as received from the drill would be used, the pulp itself would not be transferred from the dish into which it is first received, and the proportion of lead subacetate to the amount of pulp used would be constant. The article quoted above describes the recent attempts by European chemists to modify Pellet's method in order to conform more closely to these conditions. The modifications suggested obviate some of the objectionable features of the original method, but give more or less unsatisfactory results. In order to ascertain and to eliminate, if possible, the sources of error in the methods suggested by Dr. Sachs, and in order to take advantage of the more desirable features of these methods, the present work was undertaken.

THE PROPOSED METHOD. THEORETICAL CONSIDERATIONS.

In the following discussion the factors required by the Schmidt and Haensch polariscope are used exclusively. The principles involved are general, however, and the method of analysis may be used with any other make of instrument, the factors being changed accordingly.

It is evident that, in order that the scale of the instrument shall correctly show the percentage of sugar in the sample taken, a certain relation of weight of pulp taken to volume of solution must always be maintained; namely, 26.048 : 100. It is not necessary that the weight taken shall always be 26.048 grams. It may be any fraction or any multiple of this, but the volume of solution must be correspondingly decreased or increased, and the flask in which the solution is made up to volume must be calibrated accordingly. Hence, all methods of analysis now

in use require that the weight of pulp to be taken shall be some simple multiple or fraction of the normal weight, in order that the required volume may be 200 cc., 100 cc., or 50 cc., these being the volumes at which flasks are usually graduated. The 100 cc. of liquid in the solution required by the normal weight of pulp is made up of two components, namely, the water originally present in the juice of the sample and the water added to complete the proper volume. If, now, the quantity of water in the pulp be known, the residual quantity which must be added to give the correct volume of solution, can easily be determined. This amount of water may then be added directly to the pulp in the containing vessel and the necessity for transferring the pulp to a graduated flask and adding water to the mark be obviated. Furthermore, there would then be no necessity for taking just the normal weight of pulp or an even multiple or fraction thereof, since it is only necessary to preserve the ratio of weight to volume mentioned (that is, 26.048 : 100 or 1 gram : 3.839 cc.) in order to preserve the conditions required by the polariscope. In other words, any weight of pulp might be taken and sufficient water added to complete the volume of the solution in cubic centimeters to 3.839 times the weight of the pulp taken.

Not all beets contain the same percentage of water. It is a well-known fact, however, that in general this percentage does not vary through very wide limits. In most cases as the percentage of sugar in the beet increases the percentage of non-sugars decreases, or in other words the total solid matter in the beet does not change as rapidly as does the sugar content. A long series of determinations made at this Station during several years shows that for beets of from 8 per cent. to 15 per cent. sugar content the water content almost always falls between 80 per cent. and 84.5 per cent. Since such wide limits of sugar content, as those mentioned, would include most beets to be analyzed during any season or during a particular period of analysis, it would be possible to assume an average factor for water content which would vary from the true amount of water in any individual beet by not more than 2 per cent., except in very rare cases. A simple calculation will show that this maximum of error in the water factor will not change the dilution of the sugar solution sufficiently to cause a perceptible difference in the polariscopic reading. For example, 2 per cent. of 26.048 equals 0.52.

An error of 2 per cent. in the assumed water factor would then result in the addition of 0.52 gram, or cubic centimeters of water too much or too little. The resulting volume will therefore be 100 ± 0.52 cc., or it will be increased or decreased by 0.52 per cent. of itself, and the polariscopic reading will be correspondingly affected. A beet which should give a polariscopic reading of 14 will therefore read 0.52 per cent. of 14, or 0.07, too high or too low, an error scarcely appreciable in any ordinary polariscope. If in exceptional cases the error in the water factor should rise to 4 per cent. or even to 5 per cent., the corresponding error in the polariscopic reading would amount to less than 0.2 per cent., or in other words an extraordinarily poor beet having an unusually high percentage of water will give a reading slightly too low, or an exceptionally good beet may give a reading slightly too high, but in no possible case would the error due to this cause amount to more than 0.2. In analyses for the selection of mother beets it is customary to double the dilution in order to obtain a larger filtrate from a small sample of pulp, and then double the polariscopic reading. In case this should be done, the possible error due to variation of water content from the factor assumed would be diminished one-half, and in no possible case could there be an error amounting to 0.1 from this source.

It is possible, then, to assume a factor for the water content which will represent the true amount of water present in the beet so closely as to produce no appreciable error in the polariscope reading. If we represent this factor by x , the ratio of weight of pulp taken to the volume of water necessary to be added in order to bring the total volume up to the proper amount would be represented by $26.048 : 100 - 26.048x$. Having assumed, then, a value for x , this ratio may readily be calculated, and a table arranged to show the exact volume of water to be added to any given weight of pulp in order to bring the total water of the mass up to the correct volume for that weight. For example, if we assume 82 per cent. as this factor, the formula would then become $26.048 : 100 - (26.048 \times 0.82)$, or $26.048 : 78.64$, or $1 : 3.019$. Upon this basis, then, the volume of water to be added in any particular case would be 3.019 times the weight of pulp taken. A table could then be prepared showing the amount of water to be added for any particular weight of pulp taken. In case the samples are to be obtained by means of a boring rasp,

as recommended below, this table would not need to extend over more than 10 grams, between the limits of 5 and 15 grams, since all the samples obtained by the boring rasp will usually fall between these limits of weight. The following arrangement of the table has been found to be very satisfactory :

Grams.→	5	6	7	8	9	10 etc.
↓	cc.	cc.	cc.	cc.	cc.	cc.
0.00	15.10	18.12	21.14	24.16	27.18	30.20
0.02	15.16	18.18	21.20	24.22	27.24	30.26
0.04	15.22	18.24	21.26	24.28	27.30	30.32
0.06	15.28	18.30	21.32	24.34	27.36	30.38
0.08	15.34	18.36	21.38	24.40	27.42	30.44
0.10	15.40	18.42	21.44	24.46	27.48	30.50
etc.						

Such a table will show at a glance the desired volume to be added. For example, 5 grams of pulp would require the addition of 15.10 cc. of water ; 9.08 grams would require 27.42 cc., etc.

DETERMINATION OF AVERAGE WATER CONTENT.

For the purpose of obtaining figures from which to decide upon the number to be assumed as the water factor in calculating the quantity of water to be added to the pulp, a direct determination by taking samples from as many beets as possible should be made. The beets taken for this purpose should be of as widely different character and grown under as different conditions as are those which are subsequently to be analyzed by this process. A convenient method for determining the water content is as follows : The beet should be split in half longitudinally, and thin slices cut from one of the exposed surfaces from various parts of the beet until about 15 grams are obtained. These thin slices should be received on a weighed watch-glass or other similar dish, covered to prevent evaporation, and their weight immediately determined. The watch-glass should then be placed in a water oven and dried until the loss in weight in one hour is not more than 5 milligrams. From the total loss in weight the percentage of water originally contained may be calculated.

A simple determination of the total solid matter in the juice of the beet by the ordinary method, using a Brix spindle and calculating this value back to that for the original beet by means of the usual coefficient for marc, will not suffice if an accurate

result be required. Variations in the relative amounts of insoluble solids (marc) are too great. Furthermore, the fact that the Brix spindle is calibrated in solutions of pure sugar gives rise to an error which has been found to amount to as much as $1\frac{1}{2}$ per cent. in some cases, and its reading is nearly always from 0.5 to 0.8 higher than the figure obtained by a direct determination of the total solids in the juice.

During the investigations upon the applicability of the proposed method made at this laboratory this season, the actual water content of some sixty beets was determined. These beets were taken from fields in five different sections of the state, and from each field beets of widely different appearance were selected. The following table shows the results of these determinations :

TABLE I. WATER CONTENT OF BEETS.

Sugar in beet. Per cent.	Number of analyses.	Water content.		
		Maximum. Per cent.	Minimum. Per cent.	Average. Per cent.
8 to 10	11	86.04	82.72	84.64
10 to 12	30	85.22	80.56	83.05
12 to 14	13	83.63	79.91	82.10
Above 14	1	81.25
Not determined	6	85.16	80.54	82.69
—				
Total, 61		Average, 82.74		

That the above results represent closely the normal condition of beets for any year and that the water content, to a very striking degree, is independent of the season, of weather, and of the kind of cultivation, is shown in the following table. These data were compiled from results obtained at the Nebraska Experiment Station. Excepting for the year 1898, the samples analyzed were taken from fields on the Station farm. Each year the beets were grown in a different field. The character of the soil in these fields differs considerably. Moreover the meteorological records for these successive years show a decided lack of uniformity in climatic conditions. The figures given for 1898 represent the composition of mature beets grown that year at Ames, Nebraska, where the character of the soil, the rain-fall, etc., are wholly different from that at Lincoln.

TABLE II.

Year.	No. of analyses.	Sugar in beet.			Percentage of water.		
		Max.	Min.	Average.	Max.	Min.	Average.
1892	9	14.2	10.8	12.7	83.70	80.75	81.74
1894	7	13.8	10.6	11.9	83.66	80.24	82.00
1895	9	10.3	7.3	8.6	85.64	83.64	84.51
1896	5	13.5	11.1	11.8	84.14	82.11	83.27
1897	10	13.8	10.8	12.3	83.65	80.26	81.89
1898	9	15.5	11.8	13.6	82.98	79.33	81.21

In spite of this diversity of conditions that affect the growth and development of the beet, the differences in the percentages of water contained are not large, as the above table shows. The grand average is 82.43 per cent. The largest percentage observed was 85.64, and the smallest, 79.33. The former was from an abnormally poor beet with a sugar percentage of 7.5 while the latter was from a high-grade beet containing 14.1 per cent. sugar. With these two exceptions the range of values is from 80.24 per cent. to 84.51 per cent.

All of these figures indicate that for beets, most of which contain between 8 per cent. and 14 per cent. of sugar, 83 per cent. might be assumed as an average water factor without there being a variation in any individual case of more than 3 per cent. As has been shown above, this variation will not produce an error of more than 0.1 in the polariscope reading. In a more favorable season, or portion of a season, when the beets average better, the average water content would be lower. For use in analysis of beets which have from 12 per cent. to 16 per cent. of sugar, the factor 82 per cent. or 81.5 per cent. would probably be found to be more nearly correct. In case unusually good beets are to be analyzed, for example, beets that have previously been carefully sorted out for use as mother beets for seed production, a still lower factor—80 per cent. or 78 per cent., depending upon the richness of the beets—would be selected.

From the above discussion it will be seen that the addition to the pulp of 3.6 times its weight of water and lead subacetate solution suggested by Walawski is erroneous and would give rise to grave inaccuracies. For example, 3.6 times the normal weight (26.048) equals 93.8. 100 cc. minus 93.8 cc. equals 6.4 cc., the water presumed to be present in the beet pulp. Since 6.4 cc. or grams of water is only 25 per cent. of the weight of the sample taken, the error in the supposition is apparent.

In the same way Kaiser's proposition to add 76 cc. of water and lead subacetate solution to the normal weight of pulp would yield erroneous results, since in this case it is supposed that 24 cc. (or approximately 92 per cent. of water) is present in the beet pulp, an unknown condition. Lewenberg's modification, increasing the volume of water and lead subacetate to be added to 76.8 cc., supposes the presence of 23.2 cc., or 89.2 per cent. of water in the beet pulp. This percentage of water is still much higher than that obtained by direct determination, and would introduce a corresponding error.

Dr. Sachs' method requires the addition of 77 cc. of water and lead subacetate solution to the normal weight. In this case allowance is made for 23 cc., or 88.3 per cent. of water, in the beet pulp. This high water factor may be the source of the unsatisfactory results which he first obtained and which he attributed to incomplete diffusion of the sugar present. The experience and observation of many analysts show that the diffusion of sugar is perfect and practically instantaneous, if the pulp is sufficiently fine, when only the ordinary or normal dilution is made. This makes unnecessary the double dilution of the solution (with its attendant increase of error in the polariscopic reading) as proposed by Dr. Sachs. The error due to the incorrect value for the water coefficient would of course be diminished one-half by the double dilution, and this possibly explains the more satisfactory results that were obtained thereby.

THE PROPOSED METHOD. DETAILS OF MANIPULATION.

This being a method depending upon cold aqueous diffusion of the sugar in the beet, it is absolutely essential that the sample to be analyzed, be reduced to an impalpable pulp in order that the diffusion of the sugar throughout the solvent may be complete. The boring rasp devised by Messrs. Kiel & Dollé has been found to be well adapted for this purpose.

The sample of pulp, after being rasped to a sufficient degree of fineness, is received directly into a tared capsule of the form suggested by Le Docte¹ and the weight of the pulp ascertained. For this purpose a balance which is sensitive to one centigram is sufficiently accurate, since it is not necessary to know the weight with more exactness than the nearest centigram. The process

¹ Spencer's "Handbook for Chemists of Beet-Sugar Houses", page 151.

of weighing may be facilitated by the use of a gram rider on the beam of the balance, in place of the usual fractional weights.

As soon as the weight of the pulp is ascertained, the volume of water to be added to this weight is read off from the table previously prepared as indicated above. Since the solution must be clarified as well as made up to definite volume, the water to be added should contain sufficient lead subacetate to clarify the mixture. For this purpose, water containing 3 per cent. by volume of a solution of lead subacetate of 54.3° Brix, or specific gravity 1.257, has been recommended and has been found to give excellent results. If it be desired to use acetic acid as recommended by Pellet, this may also be added to the water. For rapid work, the solution thus prepared should be contained in a reservoir connected with a burette having a two-way connection, which will automatically fill to the zero mark. The capsule containing the pulp is then held under this automatic burette and the volume of liquid which was ascertained from the table is discharged into the capsule. A quarter turn of the stop-cock, or proper adjustment of the pinch-cocks, closes the discharge and connects the burette with the reservoir so that it may fill to the zero, ready for the next sample.

The capsule containing the pulp thus properly diluted is then covered with a light disc of wood or glass, inclosed in a sheet of rubber so that it will fit closely to the top of the capsule and make a water-tight covering. The capsule is then grasped between the thumbs and forefingers in such a way as to press the cover down closely, and shaken vigorously. The sugar is diffused uniformly throughout the solution, practically instantaneously if the sample has been properly prepared. The cover may be coated with vaseline before use, and if slipped to one side, not lifted, in removing, it is in readiness for another determination.

The mixture is then poured on a dry filter and the remainder of the operation carried out as usual. The use of the Pellet continuous-flow observation tube materially shortens the time required for the polariscopic reading.

EXPERIMENTAL WORK.

The method thus described was used in the analysis of some sixty beets in this laboratory last fall. In every case a large sample of pulp from each beet was obtained, thoroughly

mixed to insure uniformity of composition, and then divided, one portion being analyzed by the new method and the other by the Pellet hot aqueous diffusion method—using double the normal weight of pulp and making the volume up to 202.6 cc. The beets were selected from the five different fields mentioned above, and the results obtained show that they varied in composition through wide limits. The water factor assumed in preparing the table for the analyses was 82 per cent. The beets did not prove to be so rich in sugar content as was expected, however, and the factor 83 per cent. would probably have represented more closely the average water content of the beets analyzed. This latter figure would probably have reduced somewhat the error in the results obtained for the very low-grade beets. The results of the analyses are shown in the following table:

TABLE III.—COMPARATIVE RESULTS OBTAINED BY THE NEW METHOD.

No.	Pulp taken. Grams.	Water added. cc.	Sugar by new method. Per cent.	Sugar by hot diffusion. Per cent.	Extent of error.
1	17.33	52.34	6.2	6.25	— 0.05
2	7.61	22.95	13.6	13.7	— 0.1
3	23.34	70.50	11.9	11.9	..
4	11.88	35.88	13.1	13.05	+ 0.05
5	16.79	50.70	11.45	11.3	+ 0.15
6	12.27	37.06	11.85	11.85	..
7	25.08	75.74	5.5	5.9	— 0.4
8	18.60	56.17	10.1	10.2	— 0.1
9	23.37	70.57	10.8	10.7	— 0.1
10	19.79	59.75	8.9	8.9	..
11	13.54	40.89	12.55	12.55	..
12	25.07	75.70	11.4	11.45	— 0.05
13	13.96	42.20	14.05	14.05	..
14	16.30	49.23	12.2	12.3	— 0.1
15	9.94	30.85	11.45	11.5	..
16	23.70	71.57	12.2	12.15	+ 0.05
17	22.79	68.80	10.0	9.95	+ 0.05
18	11.98	36.18	8.4	8.7	— 0.3
19	12.55	37.89	7.9	7.9	..
20	18.25	55.10	8.55	8.7	— 0.15
21	24.89	75.14	12.35	12.4	— 0.05
22	24.82	74.96	11.45	11.4	— 0.05
23	17.78	53.68	10.0	9.95	+ 0.05
24	14.13	42.67	12.5	12.6	— 0.1
25	13.12	39.62	6.5	6.7	— 0.2
26	17.59	53.12	11.0	10.9	— 0.1

No.	Pulp taken. Grams.	Water added. cc.	Sugar by new method. Per cent.	Sugar by hot diffusion. Per cent.	Extent of error.
27	21.85	66.00	10.0	9.9	+ 0.1
28	19.80	59.80	11.1	11.1	..
29	24.12	72.84	11.4	11.4	..
30	17.02	51.04	12.0	12.1	- 0.1
31	11.11	33.58	11.1	11.2	- 0.1
32	23.71	71.57	13.5	13.5	..
33	23.73	71.72	12.5	12.6	- 0.1
34	23.26	70.55	12.5	12.5	..
35	23.33	70.46	9.35	9.4	- 0.05
36	24.02	72.54	10.8	10.8	..
37	22.23	67.15	9.35	9.5	- 0.15
38	24.56	74.17	6.9	7.2	- 0.3
39	16.91	51.07	11.9	11.9	..
40	15.78	47.65	10.55	10.5	- 0.05
41	16.90	51.04	10.0	9.85	+ 0.15
42	18.32	55.33	12.45	12.55	- 0.1
43	26.04	78.64	10.5	10.5	..
44	20.00	60.40	10.4	10.45	- 0.05
45	13.96	42.16	11.6	11.6	..
46	16.18	48.86	11.85	11.9	- 0.05
47	17.07	51.56	10.95	11.1	- 0.15
48	15.21	45.93	7.1	7.05	+ 0.05
49	16.19	48.90	10.8	10.9	- 0.1
50	25.28	76.35	12.95	13.0	- 0.05
51	19.96	60.28	10.4	10.35	+ 0.05
52	20.00	60.40	11.0	11.0	..
53	13.56	40.95	9.9	9.8	+ 0.1
54	14.72	44.45	8.6	8.6	..
55	13.92	42.04	11.5	11.4	+ 0.1
56	18.52	55.93	8.0	8.2	- 0.2

Of these results only five (Nos. 7, 18, 20, 27, and 56) show a variation from those obtained by the hot aqueous diffusion, greater than might be obtained from duplicate samples analyzed by the same method. The five beets which gave results too low by 0.2 per cent., or more, are all very low-grade beets from which low results might be expected owing to the increased dilution of the solution to be polarized, because of the unusual amount of water present in the beets. It is generally admitted that the hot aqueous diffusion method of analysis gives scientifically accurate results if properly carried out. Since the new method gives results which compare so favorably with those obtained by this process, its accuracy and the fact that it is based on correct principles are established.

APPLICATION OF THE METHOD TO THE ANALYSIS OF MOTHER BEETS.

The principles of the method as outlined above permit the use of certain modified forms of the apparatus employed which will decrease to a very considerable extent the time required for making a single analysis and hence increase the number of analyses which may be made in a day. The most important of these modifications are described below.

Balance—For very rapid work, an automatic and self-registering balance, to be used in determining the weight of the sample of pulp, is very desirable. The new form of balance recently put upon the market by Kaehler and Martini would serve very well for this purpose, since the weight of any substance placed upon the pan of the balance is indicated on the dial face directly without the use of weights. With such a balance, all the weighings necessary for 1,200 or 1,500 analyses per day could probably be made by two assistants, one to wipe the capsules and place them on the balance pan, and the other to read and record the weights on the slips accompanying the samples. In the absence of such a balance an ordinary chemical balance of the heavier forms, sensitive to one centigram, may be used. The use of a one-gram rider on the beam in the place of the ordinary fractional weights will facilitate the weighing.

Capsules.—When large numbers of samples are to be analyzed in a very short time, the capsules into which the sample is received must all be of the same weight or tare. This can be attained by using metal capsules with a small button of tin or solder attached to the side or bottom. By filing or scratching this soft button, the weight of the capsule can be changed at will, and an equal tare for the entire set be obtained. Aluminum is a very desirable metal to be used for the construction of these capsules, since it affords the necessary rigidity and at the same time the very desirable feature of light weight. Capsules of this material are practically indestructible and are subject to only very slight changes in weight. They should be so constructed as to have a capacity of about 100 cc., and should weigh 25 to 30 grams. A shape similar to that of the ordinary porcelain crucible has been found to be a very satisfactory one for these capsules. They may, however, be made with straight walls and a

flat bottom with rounded corners.¹ The edge, upon which the cover is to be placed in agitating the contents, should be ground flat and may be reinforced by a ring of thicker metal if sufficient rigidity cannot be obtained otherwise. An equal number of lids which will fit the capsules fairly closely should be provided, in order that the pulp may be protected from evaporation during the time which elapses between the boring of the beet and the weighing of the sample. The lid should be removed at the instant the capsule is set on the balance pan, so that the pre-arranged tare may not be disturbed. When 1,000 to 1,500 analyses per day are to be made, at least 100 of these capsules should be provided.

Burette.—A 100 cc. burette, preferably one graduated in tenths of cubic centimeters, should be used. One graduated in fifths may be employed, but more care in reading is then necessary. The burette should be provided with an overflow at the zero mark² so that it will fill automatically to that point when connected with the reservoir containing the solution to be added. The manner of using the burette has been indicated in describing the details of the method. When used in this way, any desired volume of liquid can be quickly and accurately discharged.

The other apparatus necessary is identical with that required by any other method of analysis. A complete list of such apparatus has been published in Bulletin No. 60 of the Nebraska Experiment Station. The details of the method of obtaining the sample and of labeling the beet have been thoroughly discussed in the same bulletin.

THE ANALYTICAL OPERATION.

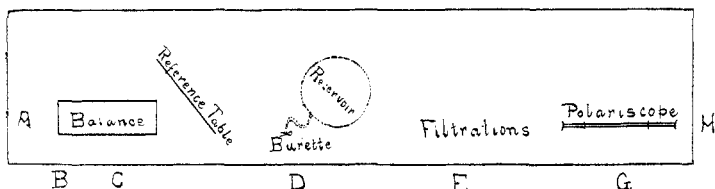
The details of the analytical operations may best be understood by reference to the following figure, which represents a laboratory table equipped for this work. This table should be about 14 feet in length and 3 feet in width. This size of table will permit independent action on the part of the operators, and at the same time avoid the necessity of any movement from one place to another.

The capsules containing the samples of pulp are brought from the drill and placed on a table at A. The first assistant, stationed at B, takes one of the capsules, wipes off the outside, removes the

¹ See design in Spencer's "Hand-book for Chemists of Beet-Sugar Houses", p. 181.

² A burette similar to that offered by Kaehler and Martini (Catalogue No. 2170) has proved to be satisfactory for this purpose.

lid, and places the capsule on the pan of the balance. The weigher C notes the weight indicated by the balance. From the reference table at his right, he ascertains the volume of water to be added to this weight of pulp and records this on the slip accompanying the sample. He then passes the capsule and its slip on to his right. In the meantime, B has prepared another



capsule and placed it on the balance pan. The operator at D then takes the weighed capsule, reads from the slip accompanying it the volume to be added, places the capsule under the automatic burette, and adds this volume of liquid. The same turn of the stop-cock which closes the discharge connects the burette with the reservoir and it is immediately filled, ready for the next sample. The capsule is at once passed on to the assistant at E, who covers it with the rubber cap, agitates it vigorously for a few seconds, and then pours the contents on a filter, which has been placed in a stemless funnel in proper position in a sugar beaker by the wash-boy stationed at J. The empty capsule is then passed across the table to the wash-boy at K, who cleans it, ready to be returned to the drill for another sample. After the filtration is complete, the assistant at G pours the filtrate into the funnel of the observation tube of the polariscope, places the accompanying slip before the observer, and passes the funnel and beaker across the table to the wash-boy at J. The latter cleans and dries them and prepares them for repeated use. The observer adjusts the polariscope, and while he is reading the scale and

recording the result on the slip before him, a new solution is poured into the observation tube by the assistant at G.

The process is thus continuous and is susceptible of great speed of manipulation. In case the ordinary form of balance is used instead of the automatic balance, a larger number of balances and assistants to operate them is, of course, necessary. By making the table wider, another row of operators might be stationed on the opposite side, thus doubling the working capacity without greatly increasing the laboratory space necessary. Care should be exercised that sufficient space for the sinks and for the wash-boys is always provided, however, since the proper cleansing and drying of the apparatus is of extreme importance.

APPLICATION OF THE NEW METHOD TO INDIRECT OR JUICE ANALYSES.

Despite the statement quoted above, that the indirect method of analysis is regarded as having "only an historic interest" it is still in very general use in this country as a means of determining the value, or purchase price, of sugar beets as they are delivered at the factories. It affords a convenient as well as fairly rapid and fairly accurate method of determining the average sugar content and purity coefficient of a composite sample consisting of quite a large number of beets of varying composition.

Several methods of obtaining a definite weight or definite volume of juice for analysis are in general use. In each of them the tendency of the somewhat viscous juice to retain bubbles of air, bits of pulp, etc., introduces more or less of inconvenience or error. The most accurate method for obtaining the desired weight of juice is by direct weighing, the so-called "gravimetric method." The same objections which were mentioned in connection with the discussion of the process of obtaining a definite weight of beet pulp are equally applicable to this procedure. It was deemed desirable, therefore, to test the accuracy of the new method when applied to the analysis of the juice of the beet.

For the purpose of calculating the volume of water and lead subacetate solution to be added, a water factor of 85 per cent. was adopted. Eight beets of different physical appearance were selected, and the juice from each obtained in the usual manner. The juice was thoroughly mixed to insure uniformity; one sample of 26.048 grams was weighed out and analyzed by the

“gravimetric method,” a second sample was measured in a sucrose pipette and the analysis completed as usual, and a third sample was analyzed by the new method. The results obtained are shown in the following table :

TABLE IV.—COMPARISON OF METHODS OF INDIRECT ANALYSIS.

Juice taken. Grams.	Liquid added. cc.	Sugar by new method. Per cent.	Sugar by gravimetric method. Per cent.	Sugar by pipette method. Per cent.
21.78	65.12	9.75	9.8	9.8
29.92	89.46	12.8	12.8	12.65
22.04	65.97 ¹	6.35	6.4	6.3
20.02	59.84	10.9	10.9	..
28.36	84.50	9.35	9.35	9.35
23.91	71.49	10.95	11.0	10.95
25.00	74.75	11.10	11.15	11.05
25.13	75.14	13.55	13.65	13.60

These few results are sufficient to show that the new method is applicable to indirect analysis. In point of accuracy it does not possess any great advantage over the sucrose pipette method if the latter is carefully carried out. It has the advantage that no specially constructed apparatus is necessary and it requires slightly less time for its manipulation than do any of the other methods.

UNIVERSITY OF NEBRASKA, LINCOLN, NEBRASKA,
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[CONTRIBUTION FROM THE NORTH CAROLINA AGRICULTURAL EXPERIMENT STATION.]

THE RATE OF NITRIFICATION OF SOME FERTILIZERS.

BY W. A. WITHERS AND G. S. FRAPS.

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THE value of any fertilizer depends on its availability to the plant, that is, the readiness with which it can be absorbed directly by the plant, or converted into forms which can be assimilated. Nitrogen can be assimilated by plants directly in four forms; *viz.*—(1) free nitrogen; (2) as certain organic compounds; (3) as ammonium salts; (4) as nitrates.

Free nitrogen can be assimilated from the air by a class of plants with the aid of organisms living in nodules on their roots. This method of assimilation is confined to the leguminosae, which includes clover, peas, beans, the peanut, vetch, etc.

¹ Used water factor 90 per cent. in calculation.