

needed for the completion of the digestion was considerably lengthened.

The Gunning modification of the Kjeldahl digestion process was also tested but the foaming resulting from the employment of the large quantities of potassium sulphate rendered the use of this modification impracticable.

By the employment of either mercuric oxide or metallic mercury, however, the oxidation is effected rapidly and completely and the subsequent precipitation of the phosphoric acid is easily accomplished.

NOTES ON THE ESTIMATION OF CRUDE FIBER IN SUGAR CANE.

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Received February 25, 1894.

THERE is no part of the analytical work connected with the chemical control of diffusion process sugar house so unsatisfactory as that of the estimation of the so-called "crude fiber" in the cane, or that portion of the stalk which is insoluble in water. Duplicate analyses, manipulated with great care, show wide variations. The average of such variations for 100 samples taken consecutively was 0.736 per cent. Since the percentage of fiber is used as a factor in calculating the percentage of sucrose on the weight of the cane it becomes highly desirable that either a better method of fiber estimation be devised, or a more accurate and quick method of estimating the percentage of the sucrose directly on the weight of the cane. After a systematic and thorough investigation of the subject I have come to the conclusion that a very accurate method of fiber estimation for technical purposes is not feasible, on account of the wide variation in fiber content in different parts of the cane stalk.

Extraction of the Soluble Bodies from the Chips.—A study was first made of the extraction of the sugars and other soluble bodies in the chips as follows: Twenty grams of chips taken from the comminuter were placed in a beaker, and nine volumes of water of varying temperatures added. The diffusates were polarized every fifteen minutes until the last two readings were the same.

The water in the beakers was kept as nearly as possible at the temperature of the water when added.

Results:

At	23° C.,	diffusion complete in	65	minutes.
"	60° C.,	"	"	" 45-50 "
"	75-80° C.,	"	"	" 30-35 "

After pouring off the diffusate as completely as possible the above process was repeated in order to see if there was any appreciable difference in the rate of diffusion for the second application of water, but there was none.

From the polariscope readings of the completely diffused solutions the percentage of extraction of sucrose was calculated for the first two applications of water, and found to be seventy-five to eighty-five per cent. Assuming eighty per cent. for the average of each application of water, and twelve per cent. as the average amount of sucrose in the chips, after five complete diffusions of each sample there would remain in the chips only 0.0384 per cent. of sucrose, which is practically nil. Theoretically, ninety per cent. of the sucrose should be removed for each complete diffusion. In quite a number of cases the final diffusate was tested for sugars by means of the α naphthol reaction, which invariably showed only a very low percentage of these bodies.

The coagulation point of the water soluble albuminoids by heat was ascertained by gradually raising the temperature of the first diffusates till coagulation began, and found to be between 80 and 90° C. It is therefore unnecessary to begin the extraction by the addition of cold water, then warm water, etc., as is usually done. Since the diffusion is so much more rapid at high temperatures it is advantageous to begin by adding water at 75° C. for the first two diffusions, and boiling water for the last three, letting stand each time for thirty minutes. This can be safely done, as it will be seen, without fear of coagulating the albuminoids. This method was followed throughout the season.

Losses in the Preparation of the Samples.—In preparing samples for the estimation of the fiber in different parts of the cane, a portion of a cane stalk cut at the internodes with a sharp knife was rapidly weighed, ten grams cut off from both ends into a tared beaker, and the stalk reweighed, and was found to sus-

tain a greater loss than the ten grams cut off. This process was repeated in four cases, giving similar results.

Results:

No. 1.	Difference in weight after cutting off ten grams,	10.115
" 2.	" " " " " " " " " "	10.161
" 3.	" " " " " " " " " "	10.100
" 4.	" " " " " " " " " "	10.102

The chips were cut off in semi-circular pieces about $\frac{1}{4}$ of an inch in diameter. Sample 1, as soon as weighed, was cut finer with a pair of sharp scissors and rapidly weighed. It sustained a further loss of 0.107 gram, which added to the first loss makes a total of 0.22 per cent. while preparing the sample. These losses could not have been due in the main to evaporation, for the whole time occupied in preparing sample No. 1 was not over twenty minutes. Then Nos. 2, 3, and 4, were cut off early in the morning when the samples of 10 grams each lost by evaporation, for one hour, on an average 0.079 gram. The loss was more likely a mechanical one of the juice due to the rupture of the cells by the knife while cutting off the chips. If so, this loss would be augmented by the cutter and comminuter in the mill, especially so when the knives are dull.

Loss by Evaporation from the Chips.—According to well-known laws of physics the amount of evaporation varies with time of exposure, surface area exposed, and the temperature and humidity of the atmosphere. The fineness of the chips, which increases the surface, affects the rate of evaporation, probably also the ratio of the juice to the fiber. Upon the latter point I made no experiments. A piece of cane stalk, about an inch in diameter, was cut at the internodes, weighed, placed in the shade and reweighed at the end of each hour. The temperature at 12 o'clock 78° F., day dry.

Results:

12-1 P. M., loss by evaporation.....	0.48 gram.
1-2 " " " "	0.56 "
2-3 " " " "	0.40 "
3-4 " " " "	0.49 "
4-5 " " " "	0.52 "

Average loss each hour..... 0.49 gram.

This evaporation was mainly from the open ends and is therefore largely independent of the length of the piece of stalk.

Ten grams of chips were placed in a beaker in the room and weighed every hour, with the following results:

No. 1.	From 10:30-11.30	A. M.,	loss by evaporation,	0.086	gram.
" 2.	" "	" "	" "	0.073	" "
" 1.	" 11.30-12.30	" "	" "	0.269	" "
" 2.	" "	" "	" "	0.266	" "

The morning was very damp, there being a heavy fog which did not clear away until nearly 10 o'clock. From these results it would appear that quite an error may come from evaporation of the chips while the samples are accumulating at the mill, which often requires an hour or more. Add to this the mechanical loss from cutting the chips by the machine, and the error is sufficient to make the fiber appreciably high.

The Distribution of Fiber in the Cane.—These sources of error do not, of course, account for the differences in duplicate analyses. This error must be sought for in the difficulties in obtaining an average sample—in the variation in the fiber content in the different parts of the cane. To this end the following analyses were made: Ten grams of fiber were cut from the nodes, and ten grams from the contiguous internodes of the top, middle and butt of a stalk of purple cane, third year stubble of average size and weight, and the fiber estimated in these portions, giving the following results:

Top	{ Node..... 15.86 per cent.	Middle	{ Node..... 15.9 per cent.
	{ Internode 8.60 " "		{ Internode 8.0 " "
		Butt	{ Node..... 18.20 per cent.
			{ Internode 8.08 " "

It will be noticed that there is approximately twice the percentage of fiber in the nodes as in the internodes, the butt node showing the highest percentage.

Fiber content in the rind and pulp of portions of cane stalk taken from the top, middle, and butt, cut so as to include a node and internode. (a) purple variety, plant cane; (b) purple variety, third year stubble.

	Rind.	Pulp.	(b) Top.....	Rind.	Pulp.
(a) Top.....	26.0	8.7	Middle..	25.6	7.80
Middle ..	29.5	6.5	Butt.....	28.5	5.40
Butt	28.5	6.8		28.5	5.77

Stubble cane always has a higher fiber content than plant cane of the same variety. From the above it would appear that this difference resides more in the pulp than in the rind.

It is difficult to say just what is rind and what is pulp in the cane stalk. So a portion of cane, about five inches in length, was cut from the center of a stalk of plant cane, purple variety, just within the two nodes. Ten grams of the rind was pared off into one beaker, ten grams taken off all round from what remained, and so on until the center of the stalk was reached, and the fiber estimated in these separate portions with the following results:

True rind	33.9 per cent.
Inside rind or bast tissue	13.33 " "
Next to inside rind	6.79 " "
Next to middle.....	4.77 " "
Middle	4.13 " "

The diffusate from the inside bark (bast cells?) was quite yellow, and it is probable that these cells furnish the yellow coloring matter in the expressed juice.

A joint of cane was cut just without the nodes, and the true node separated from that portion penetrated by the rootlets of the eye. Then ten grams were cut off from both ends of what remained at right angles to the length of the stalk, and an average of what remained was taken, and the fiber estimated in these portions, with the following results:

PURPLE VARIETY, THIRD YEAR STUBBLE.

Nodes proper	21.3 per cent.
Root nodes	14.2 " "
Next to the above.....	14.13 " "
Remaining internode.....	11.9 " "

From the above data it will be seen that the fiber varies very widely in amount in different parts of the cane, from 4.13 per cent. in the pulp to 33.9 per cent. in the rind. The percentage of what was considered as rind in the above analyses was estimated, and as an average of four cases was approximately twenty-one per cent. the weight of the cane. With such a difference in the fiber content of the rind and pulp, and such a percentage of rind, it is no small wonder that the duplicate analyses could not be brought to agree. For the presence of a very small undue proportion of rind or pulp in the samples would make the duplicates disagree. Often the knives of the cutters are dull, and strips of rind, varying in length, get into the chips. These, by the motion of the carriers, tend to accumulate on top, and are easily gotten hold of in undue proportion in the taking of the

sample. In such cases it is more difficult to get an average sample than when the chips are fine and regular.

LOSS OF GOLD AND SILVER DURING SCORIFICATION ASSAY.

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Received October 6, 1893.

NO explanation of the following numerical results is required, beyond stating that the losses referred to represent total losses, that is to say, losses due to both volatilization and mechanical causes. The furnaces employed were "Battersea No. F," and the conditions under which the assays were made, such as heat of muffle, draught in muffle, and manipulations in general, were such as would obtain in careful practical work.

SILVER.				GOLD.			
Weight before cupelling.	Weight after cupelling.	Loss.	Per cent.	Weight before cupelling.	Weight after cupelling.	Loss.	Per cent.
210.765	206.360	4.405	2.09	338.030	335.025	3.005	0.888
543.165	535.645	7.52	1.38	349.020	348.200	0.820	0.234
206.360	200.325	6.035	2.92	335.025	334.365	0.660	0.197
535.645	523.330	12.315	2.29	348.200	346.900	1.30	0.373
200.325	196.720	3.905	1.79	334.365	333.120	1.245	0.372
523.330	514.765	8.565	1.63	346.900	345.790	1.11	0.319
196.720	191.733	4.987	2.53	332.575	331.725	0.85	0.255
514.765	503.950	10.815	2.10	345.790	344.15	1.64	0.474
191.733	187.820	3.913	2.03	331.725	330.600	1.125	0.338
434.180	424.925	9.255	2.13	344.965	344.265	0.70	0.202
187.820	184.525	3.295	1.80	330.600	329.900	0.70	0.211
424.925	419.975	4.95	0.95	334.650	343.960	0.69	0.206
184.525	180.560	3.965	2.14	329.900	329.130	0.77	0.233
419.975	410.430	9.545	2.27	220.635	220.200	0.435	0.197
410.430	403.365	7.075	1.72	329.130	328.860	0.27	0.082
403.365	394.550	8.815	2.18	220.200	219.835	0.365	0.165
Average loss..... 1.99 per cent.				Average loss..... 0.296 per cent.			
" " entire process.. 2.54 " "				" " entire process 0.87 " "			
" " by cupellation.. 1.99 " "				" " by cupellation 0.296 " "			
" " in scorification. 0.55 " "				" " in scorification 0.574 " "			