

# EXPERIMENTS ON THE TESTING OF BAGASSE.

By J. BRUNIQUET AND E. P. HEDLEY.

CHAIRMAN: Before asking Mr. Bruniquet to read the next paper, I wish to say right off that there is no intention of altering the present method of testing bagasse. What we want to do is to make up in our minds exactly what we mean by those methods, and to remove any ambiguity.

Mr. J. BRUNIQUET then read the following paper:—

The instructions which the Committee for the Standardisation of Chemical Control issue for the analysis of the bagasse discharged from the last mill, some of the chemists contend are vague. The point at issue reads: "The water is brought to the boil and allowed to simmer for 30 minutes. . . . Cool with condenser still attached to about 70° C."

In some factories the sample for analysis is drawn off, by means of the cock, as soon as the boiling has ceased, in others the cooling is effected by standing in

water for various lengths of time, no regard being paid to the final temperature. One place even contends that the lid of the bagasse basket is not sufficiently heavy to keep the bagasse entirely immersed in the water, and have substituted a lead disc of about 1 kilo weight for the basket lid, thus keeping the bagasse under the water for the full 30 minutes of boiling.

To assist in the solution of these simple questions the following experiments were performed:—

A number of sticks of cane were crushed in a laboratory mill, the sticks being passed through the mill three times. The sticks were then chopped up to about the size of ordinary mill bagasse. They were then placed in the bagasse boiler and boiled for the requisite 30 minutes. At the end of that time a sample was drawn off and cooled under the tap (column A); the boiler was then cooled to 70°, 60° and 50° C. (column B) and its pol. read:—

TABLE I.  
Water-cooled Bagasse Extract.

No.	A.	..	B.			NOTES.
			70° C.	60° C.	50° C.	
1.	7.50	..	—	7.60	7.65	{ Time of cooling, 5 mins. to 60° C., 10 mins. to 50° C. Temperature of cooling water = 25° C.
2.	8.10	..	—	8.10	8.20	
3.	6.50	..	—	6.45	6.65	{ Temperature of cooling water = 26° C. Cooling too rapidly to get a sample of extract at 70° C. Time of cooling, 5 mins. to 60° C., 10 mins. to 50° C.
4.	7.00	..	—	7.00	7.05	
5.	6.70	..	6.70	6.65	6.70	{ Temperature of cooling water = 25.5° C. Cooled in paraffin tin of water to 70° C. and in running water to 60° C. and 50° C. Time of cooling, 4 mins. to 70° C., 9 mins. to 60° C., 12 mins. to 50° C.
6.	6.60	..	6.70	6.65	6.70	
7.	6.50	..	—	6.50	6.70	{ Temperature of cooling water = 24° C. Time of cooling 6 mins. to 60° C., 13 mins. to 50° C.
8.	6.40	..	—	6.45	6.45	
9.	6.20	..	—	6.25	6.25	{ Temperature of cooling water = 24.5° C. Time of cooling, 7 mins. to 60° C., 13 mins. to 50° C.
10.	6.30	..	—	6.30	6.45	
11.	6.50	..	6.50	6.50	6.60	{ Temperature of cooling water = 24.5° C. Time of cooling, 5 mins. to 70° C., 7 mins. to 60° C., 13 mins. to 50° C.
12.	6.50	..	6.50	6.55	6.65	
13.	6.60	..	6.50	6.60	6.60	{ Temperature of cooling water = 26° C. Time of cooling, 6 mins. to 70° C., 10 mins. to 60° C., 14 mins. to 50° C.
14.	7.00	..	—	7.00	7.10	
15.	6.90	..	6.90	6.95	6.95	{ Temperature of cooling water = 3° C. Time of cooling, 4 mins. to 70° C., 8 mins. to 60° C., 13 mins. to 50° C.
16.	6.80	..	—	6.70	6.80	
17.	6.30	..	6.50	6.40	6.40	{ Temperature of cooling water = 26° C. Time of cooling, 3 mins. to 70° C., 6 mins. to 60° C., 11 mins. to 50° C.
18.	6.50	..	6.45	6.50	6.50	
19.	6.35	..	—	6.40	6.40	{ Temperature of cooling water = 27° C. Time of cooling, 3 mins. to 70° C., 6 mins. to 60° C., 11 mins. to 50° C.
20.	6.40	..	6.40	6.40	6.45	

In all, some twenty experiments were performed, and it will be noted that it makes practically no difference whether the sample for analysis is drawn off at the conclusion of the boiling, or when the temperature reaches 70° C. The method of cooling adopted consisted in sinking the bagasse boiler into a paraffin tin full of water and taking off the sample when the desired temperatures were reached. Very little time is consumed in reaching the desired temperatures.

Another series of experiments were performed by comparing the pol resulting from boiling the bagasse (A) *without* a cover on the basket, (B) with a cover on the basket, and (C) *with* a kilo weight on the basket lid.

TABLE II.

A.	B.	C.
6.15	6.15	6.10
6.30	6.35	6.35
6.20	6.20	6.25

The results of this series should dispose of the idea that any weight is required to keep the bagasse in the hot solvent, as it will be noticed that the polys are practically the same under each of the three conditions.

A final series was carried out in which the boiler was allowed to cool in air with the condenser attached. The results are given in Table III. "A" is the sample drawn off at the end of the boiling; "B" is the sample cooled by air to temperatures 70°, 60° and 50° C.

TABLE III.

No.	A.			B.			NOTES.
	70° C.	60° C.	50° C.	70° C.	60° C.	50° C.	
1.	7.00	..	7.35	7.40	7.40		{ Room temperature = 24° C. Time of cooling, 40 mins. to 70° C., 1 hour to 60° C., 1 hour 40 mins. to 50° C.
2.	6.60	..	6.90	7.00	7.10		{ Room temperature = 26.5° C. Time of cooling, 61 mins. to 70° C., 1 hour 35 mins. to 60° C., 2 hrs. 15 mins. to 50° C.
3.	6.70	..	7.00	7.05	7.20		{ Room temperature = 25° C. Time of cooling, 45 mins. to 70° C., 1 hour 7 mins. to 60° C., 2 hours to 50° C.
4.	6.60	..	6.90	6.95	7.20		{ Room temperature = 28° C. Time of cooling, 56 mins. to 70° C., 1 hour 31 mins. to 60° C., 2 hours 21 mins. to 50° C.
5.	6.00	..	6.30	6.40	6.55		{ Room temperature = 35° C. Time of cooling, 1 hour 5 mins. to 70° C., 1 hour 42 mins. to 60° C., 3 hours 5 mins. to 50° C.

In these results it will be seen that the pol of the sample rises the longer the bagasse is in contact with the hot water, but it will also be noticed that the time during which the temperature remains high is very long, and it is probable that the rise in pol is due not so much to extracted sugars as to other dextro-rotatory bodies, such as hemicellulose, dextrans, etc.

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CHAIRMAN: Here we have another paper on which I feel sure you have opinions of your own. Bagasse testing is a thing which, like the poor, is always with us. We are always, in my experience, always to rack our minds to find out what our bagasse test should be. Therefore I feel sure you have your particular ideas and opinions and experiments which you will like to impart to this meeting. I would like to mention that these results bear out certain other results which were obtained earlier in the year and which I think Mr. Jacobs might be able to confirm, and that is on a certain occasion at this mill the first method "A" was the one in normal use. In order to check it "B" was reverted to and the same results found as shown here,

that with rapid cooling by water there was no difference, but when the methods in Table II. "B" were used—that is, slow cooling in air—a difference of 0.4 was found, which is very similar to the difference found here. At the Experiment Station the difference was about 0.3. I hope I have my figures and facts correct on that.

Mr. H. M. JACOBS: I think the figures quoted by Mr. Moberly are pretty well correct. This is a good example of how careful one ought to be in adopting these methods of analysis. There was some dispute as to what was meant at that time by "cool with condenser attached." My own personal reading is that if it had been intended that the boiler was to be cooled in air it would have said "allow to cool," but the method saying "cool with condenser attached," I take it, it naturally means cool with water. When a chemist is told to cool anything he cools it with water as quickly as possible. When he is told to "allow it to cool" he does it in the atmosphere. However, these experiments were carried out at the mill and with the results as quoted by Mr. Moberly and confirmed by the Experiment Station.

Mr. BECHARD: In what I might call parallel experiments I made some years ago, by reversing the

process—that is, getting up to the boil in a long time—I found that the more sugar you have in the bagasse the more the difference, which is not proportional at all. If you get a bagasse with only 2% to 2.5% of sugar you get very little increase at all, practically so small that you cannot measure it; but when you get a bagasse with 6% to 7% of sugar the difference then is very accentuated.

Mr. JACOBS: I take it you mean raising the boiler to boiling-point from cold water.

Mr. BECHARD: Yes, taking a long time to get to your boiling-point.

Mr. JACOBS: Do you say, then, that with normal bagasse having a normal low sucrose, there is no difference?

Mr. BECHARD: The differences are smaller, if measurable.

Mr. JACOBS: That does not confirm my experience. The longer the hot solution is in contact with the bagasse, even with a normal bagasse, the sucrose is always higher.

Mr. RAULT: Seeing that the opinions of the two previous speakers, based on practical experience, do not agree, and also that the tests carried out by Mr. Bruniquel as recorded in Table III. of his paper shows a continuous rise in polarisation of bagasse through prolonged contact of the cooling liquor, it becomes imperative that the Experiment Station carry out more tests with the object of finding out the minimum time of boiling required to reach a constant polarisation of bagasse.

We are not all of one mind about this minimum time, and it is right that such an investigation should likewise be carried out at the Experiment Station, and I suggest that this work be carried out in the course of next season, when bagasse from the various mills will be available. It is possible that the result of this investigation may raise some controversial point about the payment of cane, if our methods of sucrose determination have to be modified; nevertheless, it is right that we should come to a finality and adopt the correct method.

Mr. BRUNIQUEL: With regard to the rises, about 0.3 to 0.4 in the air-cooled bagasse, you cannot ascribe that definitely to sucrose. We have nothing to prove that. Mr. Dymond in his paper last year could not prove that it was sucrose and not some dextro-rotatory body.

Mr. BIJOUX: There is also the question of the pH of the bagasse and of the water the bagasse was boiled in. These are important.

Mr. BRUNIQUEL: Tank water was used to boil these bagasse samples, and of course soda was added to it. There was no inversion taking place.

Mr. BIJOUX: I think you will have a difference if the solution is acid or not.

CHAIRMAN: With reference to the minimum time of boiling, some experiments were carried out last year by Mr. Christianson at Gledhow in which bagasse was boiled for varying times, and tested at different points of boiling, and we found in almost every case a rising curve which flattened out suddenly for a bit and then rose again. My interpretation of that was that the first rising part of the curve was the removal of sucrose, and the second rising part of the curve was largely but not necessarily entirely due to the slower hydrolysis of parts of the fibre. I don't know whether Mr. Christianson can remember the details of time in that experiment and when the flat part did occur.

Mr. CHRISTIANSON: Quoting from memory, I think it took about 45 minutes on the average before the constant was reached and after about an hour it began to rise again.

Mr. JACOBS: On that point Prinsen Gereligs, in the 1924 edition of the Cane Sugar Book, says that dextro-rotatory substances begin to dissolve after 10 minutes boiling. He says that very definitely.

Mr. RAULT: In asking for these tests to be carried out, I had in mind what I just read in Dr. Maxwell's book concerning the reliability of milling control. It is mentioned that the Javan laboratories boil their bagasse for fully one hour, against our "allowed to simmer for 30 minutes." We know that chemical control in Java is of the highest order and that methods are only enforced after thorough investigation from the Experiment Station.

Mr. BRUNIQUEL: We did some tests of bagasse boiling with and without sodium carbonate, as the Java method does not mention the addition of sodium carbonate. We boiled them both for an hour and there was no difference at all. There does not seem to be any inversion up to an hour, even without sodium carbonate.

Mr. BECHARD: I am afraid I can't agree that without sodium carbonate you get the same results. Some years ago I had quite a little bit of unpleasantness over the same matter. I tested some samples for pH after boiling, and I found a difference of 5.1% to 5.2%, and duplicating those with sodium carbonate gave a figure of 6.8%. I found a large difference, from 0.7 to 1 degree of pol—that is following the ordinary method of 30 minutes boiling.

Dr. HEDLEY: We are well aware that on these experiments one cannot base too much, but with the cane as prepared in the Experiment Station, where it is crushed and shredded, one is able to get an exceedingly good mixture. That, as every chemist knows, is one of the essential points in getting comparable analyses. I very much doubt whether it is possible to get two samples of bagasse alike in a mill, when the mill cane is taken. I have tried it several times when doing

milling tests at mills, and found quite a difference between two samples of bagasse taken from the bagasse roller and mixed and quartered into two samples. I think that possibly some differences are to be referred to the want of a good sample, but of course it stands to reason that it is necessary to add sodium carbonate to prevent inversion of the sugar which is there. We tried that to see whether we did get any difference, but we don't for one minute say you do not get it in other cases. It depends on the juice you have. We have no mill juice the same as in the mill; we had the juice from the cane which came straight from the field. With reference to Mr. Rault's remarks about the Experiment Station doing more experiments, that, of course, is just what the Experiment Station is for, but it is an exceedingly difficult thing for us to carry out experiments on bagasse when we have no bagasse; the bagasse is at the mills. We can only get crushed cane. I have considered this, as I think this paper lends itself to very great expansion. The mill bagasse analyses are in a very unsatisfactory state; there is no question but that they are not properly dried. The samples are put into your ovens and you get it fairly well dried, and then another sample is put into the same oven which has, say, 50% of moisture, and the first thing is that the water is distilled off the second sample which has just been put in and that moisture is absorbed by the first which is drying in the oven. I really question very much whether the correct moisture is returned to the engineer, and I think that in the coming year quite a lot of work can be done on this question. Maxwell's new book, which Mr. Rault referred to, goes very well into this question; but there are other people who have been working on it and have drawn attention to these difficulties in bagasse, and I think something should be done, but not at the Experiment Station only. Too often we say "the Experiment Station will do this." The Experiment Station is only too delighted to do anything it can for the Industry; but it could be better done at the mills, because they have got better facilities there. We form Committees, and these Committees bring forward annual reports which are so frequently made up at the end of the crop. We have arrived at the conclusion that possibly in the coming year we will work a Bagasse Committee or add it to the Fibre Committee and carry out an organised system of experiments. Possibly this year, with enthusiastic people like Mr. Bruniquel and Mr. Christianson and Mr. Rault and various other people who are always looking for work, we will give them a little more work

to do, and then can bring forward these papers next year. This is also Mr. Bruniquel's maiden effort, and I think he has done very well. (Applause.)

CHAIRMAN: There should be a definite outcome of this paper and we ought to know exactly in our minds what we mean by interpretation of official methods. It seems to be the general concensus of opinion that cooling with condenser attached means cooling in water. For myself, I should rather have liked to have seen the method specified as cooling for a definite time rather than cooling to a definite temperature, because these changes are evidently proportional to the amount of time of contact. However, the time of cooling is so rapid that probably it makes very little difference. Nevertheless, I do not think that would be a good thing to do at the present time, as we do not wish to alter the present official methods, but to define what we mean is a thing we are competent to do. I hope somebody will move a proposition to that effect, just amplifying our definition so as to give it an unambiguous meaning.

Dr. HEDLEY: I move as a formal resolution:—

"That the interpretation placed upon the instructions by the Technologists' Association is that the bagasse boiler be cooled in water until it reaches the temperature of 70° C."

CHAIRMAN: There is one difficulty arising out of that. Certain factories have these bagasse boilers with electric elements, and they cannot be dipped into a tank of water, but I have no doubt that could be overcome by the attachment of a water-jacket to the boiler. I think Dr. Hedley's proposal is a very sound one, as giving a proper interpretation of what it is intended to do.

Mr. BECHARD seconded the resolution, which on being put to the meeting was passed unanimously.

CHAIRMAN: The next paper on the list is the Annual Summary of Chemical Laboratory Reports from Natal Sugar Factories. This is not a very controversial paper, but more of a record. After publication of the programme I received a paper from Mr. Blacklock on the "Keeping Qualities of Natal Sugars." So I will change the programme slightly by asking Mr. Blacklock to read the paper.

