

WORKING WITH THE CARBONATATION PROCESS UNDER SOUTH AFRICAN CONDITIONS

By J. RAULT.

In 1920 Mr. William A. Campbell, managing director of the Natal Estates Ltd., writing in the *Journal of Industries*,² spoke of the first application of a process of sugar manufacture new to Natal, namely, the carbonatation process of cane juice clarification. It was stated that:—

1. The viscous impurities naturally present in juices from the Uba cane grown under Natal climatic conditions were extremely refractory to clarification, slowed down all factory operations, required a disproportionate plant for the work done, and caused heavy sugar losses, with consequent poor crystal recovery; these losses being further aggravated when attempting to produce a white sugar for direct consumption.
2. Most of the difficulties encountered in the treatment of these refractory juices had been eliminated or at least minimised by the new process.
3. A higher percentage of sugar and a better quality sugar than previously was now obtained from a given quantity of juice.
4. South African material, namely, limestone and coke, was used instead of the previously employed lime, sulphur and phosphoric acid, i.e. an important consideration in the national economy and an insurance in war-time against short supply from overseas.

The high cost of the chemical bill, as a result of heavy transport cost, was unfavourably commented upon and declared to be the greatest deterrent to the adoption of the process by other milling concerns.

In 1923, F. Maxwell⁶ commented favourably on the carbonatation process for white sugar production, based his case for its adoption in South Africa on the experience of Java with the de Haan's modification, backed by a few results of the Mount Edgecombe factory communicated to him by the present writer.

Farnell,⁴ in a paper resuming his investigations of the colloids of cane juices and syrups in Natal and Mauritius, refers to the carbonatation process and the work done by Rault on the comparative removal of gum, wax and nitrogenous matter by carbonatation versus sulphitation.

Very little has been published on the process in our Proceedings of the South African Sugar Technologists' Association, apart from what can be gathered

from the annual summary of factory results, or occasional answers to questionnaires under the anonymous designation of factory No. 1.

The reason for this mild "blackout" may be explained by the dissimilarity of the process from the usual South African processes offering no common basis for discussion and forcing us to solve our particular problems by a slow process of self-help devoid of the benefit of exchange of experience with friendly neighbours.

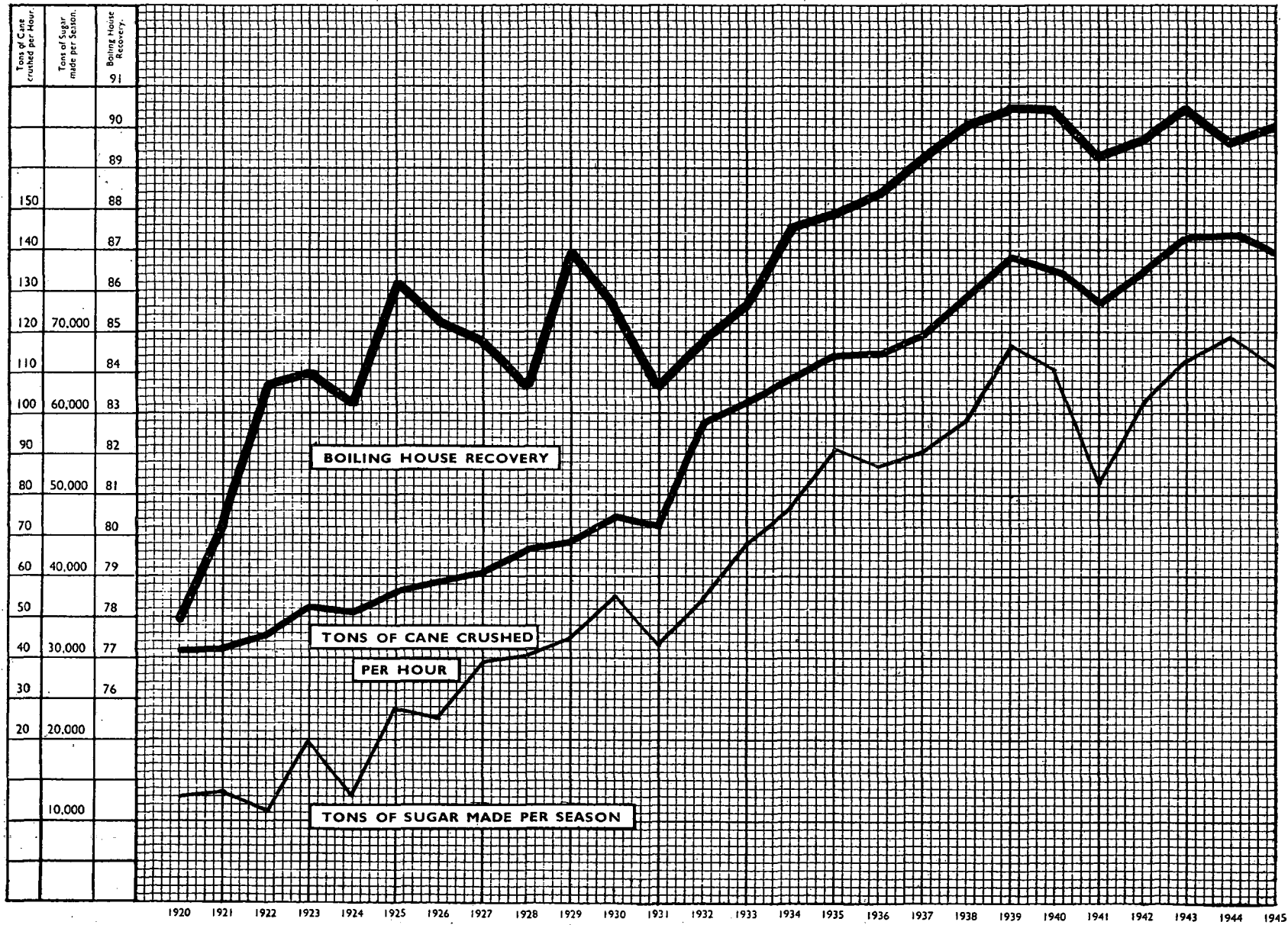
Over 25 years have elapsed since the decision to adopt the carbonatation process was taken by Mr. W. A. Campbell, at a time when this move was regarded as a risky undertaking, in view of the lack of technical data, the cost of the process, and the very inadequate plant for its efficient practice.

During that period a revolution in cane planting has been accomplished by the introduction of new varieties. Our agricultural practice has considerably improved through increased mechanization, intensive fertilization and green manuring, irrigation, etc. South African factories have modernized their equipment. Scientific methods of control in all its various aspects and co-operative investigational work from the South African technologists have enriched in a marked degree the pool of knowledge of sugar technology and consequently contributed to the efficiency of the industry.

A glance at the accompanying graph of the Natal Estates Ltd., Mount Edgecombe, results, shows the upward trend of progress, coincident with the adoption of the carbonatation process.

The following table shows a few landmarks of seasonal progress for field returns, hourly throughput of the mills, mill extraction, boiling house recovery, overall recovery, tons of cane to sugar and output of sugar.

Year.	Per cent Uba cane.	Tons cane per acre.	Tons cane crushed per hour.	Mill ex-traction.	Boiling house recovery.	Over-all recovery.	Tons cane to sugar.	Sugar output for season.
1920	100.0	20.8	40.2	88.10	77.91	68.63	11.18	12,891
1923	100.0	21.6	52.9	91.91	85.20	77.77	9.47	20,093
1928	100.0	18.6	64.7	93.70	83.65	79.63	9.13	30,220
1933	100.0	24.9	102.7	94.21	85.81	80.84	8.62	40,481
1934	95.1	29.8	109.4	94.20	87.57	82.49	9.29	48,818
1937	66.7	30.5	119.6	94.80	89.33	84.69	8.11	55,349
1943	7.6	44.0	143.3	94.96	90.52	85.88	8.87	66,236
1944	6.4	39.6	143.7	94.86	89.72	85.11	8.45	69,265
1945	4.9	36.7	139.3	94.78	90.06	85.36	8.31	65,964



26 Years results, working with the Carbonatation Process at Natal Estates Ltd., Mount Edgcombe, Natal

Quality of Product.

It is now generally admitted that the carbonation process, by the energetic elimination of a large part of the juice impurities, puts on the market a final product of outstanding value, possessing qualities of its own such as crystalline structure, clarity, brilliancy, very low sulphur dioxide and thermophilic bacteria content, fitness for aerated waters, keeping quality on storage, unrivalled by the product of other juice processes.

A compliment to the value of the process comes from a source not usually favourably disposed towards methods of refining other than bonechar, when we read in one of the most candid and informative books on sugar refining, from the pen of Oliver Lyle,⁷ that the incomparable high standard of Tate & Lyle's granulated, which has set a model to the sugar world, could not be maintained if it did not use a mild form of carbonation as part of its process.

On the other hand, Honig of Java⁸ maintains that by carbonation methods, a granulated sugar equal in quality to standard refined, can be made without further treatment with bone-black or vegetable carbon.

With our present system, and the conditions of the South African market, the production of No. 1 Edgcombe refined constitutes an attempt to refine sugar liquors right at the earliest stage possible, as it comes from the cane, together with a reprocessing of our own washed raw sugars from the second and third boilings.

The Process.

Carbonation relies on the mass action of lime dispersed in the juice at various concentrations or pH, subsequently neutralised by CO₂ gas for the flocculation, absorption and precipitation of organic and mineral impurities present in the juice in the state of coarse suspensions, colloidal dispersion or true solutions, which are partly or wholly removed in the lime carbonate filter cake.

A limekiln working continuously is an essential part of a carbonation plant. We will not touch on this part of our process, which is interesting enough for a comprehensive paper, and much of it can be found in text books.

Filtration rates become the touchstone of successful juice treatment, and a carbonation factory, in which all the juice must be filtered faultlessly at some stage previous to evaporation, develops consequently a "filtration complex" where the advantages derived from a high juice handling capacity is of greater and more immediate economic importance than the striving after the optimum conditions laid down by obscure theories on the exhaustibility of final molasses.

In the successful adaptation of the process to our special South African conditions, the following factors have had a far-reaching influence on the technique of our factory and consequently on the ultimate percentage recovery of our final product.

1. An instability in the hourly throughput of canes, which, far from being static and providing a basis for standardising our equipment for an optimum capacity, has by successive leaps and bounds increased by 300 per cent. and thrown out of balance one or other station of the manufacturing process in the course of the past 25 years.

2. Fluctuations in the ratio of refined sugars to raw sugars for export or for the local market, viz., in the course of the same campaign a 100 per cent. refined sugar production was carried out for a continuous period of seven weeks, for supplying a depleted market in June, to be followed by months of 70 per cent. refined and 30 per cent. raws; whilst on a few occasions our refined juices had to be turned out into a cargo sugar for export. For the 1942 season, 85 per cent. of our whole production was a No. 1 refined.

3. A rising standard in the quality of the No. 1 refined sugar, with special reference to colour, as set by the bone-char and vegetable-char refineries.

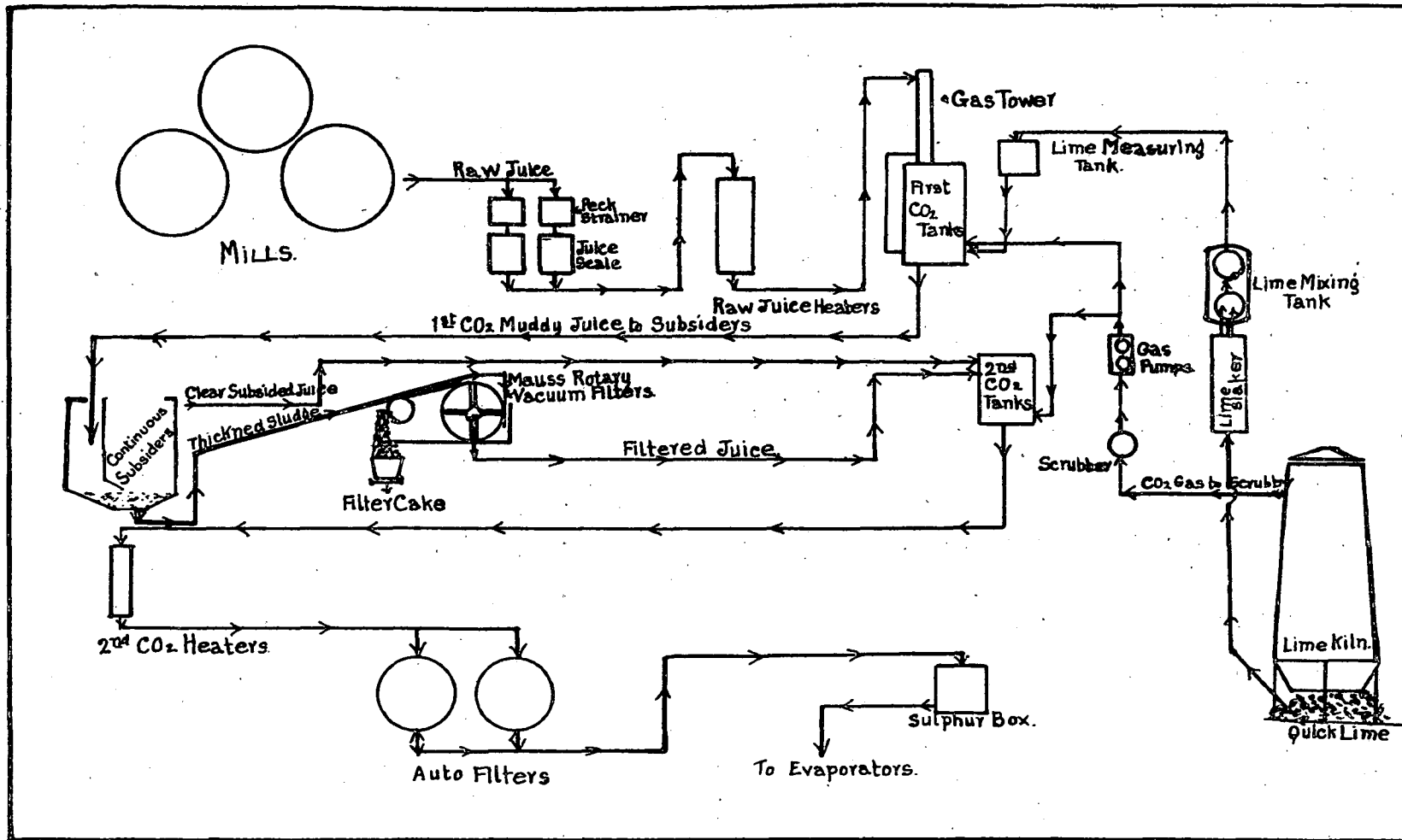
4. The general trend towards expensive labour and the necessity of simplifying our process of manufacture in view of economising labour.

That our peculiar climatic conditions, with its recurrent dry and cold spells, rather than the much maligned Uba variety, is the cause of our processing difficulties, is admitted by all technologists who have had local experience. For this reason, so far no factory has succeeded or is willing to clarify their juices by the simple lime defecation process of other cane sugar lands, notwithstanding the practical elimination of the Uba from our fields.

The raw sugar factories, in order to work smoothly and also to avoid throwing difficulties at a later stage on the refinery, are commonly using amounts of lime, sulphur and phosphoric acid that would be considered excessive for sulphitation factories manufacturing plantation whites.

The same basic cause, together with the four factors enumerated above, have contributed to put up the limestone consumption of our factory to a level which would be judged wasteful by Javan technologists experienced in the treatment of tropical cane juices, or by beet technologists who in the last decade have successfully reduced their chemical bill by adopting pre-liming methods.

Laboratory investigation on the treatment of raw cane juice with increasing amounts of lime, has given a curve of pH where two definite zones of improved clarity are noticeable, namely, between pH 7.2 to 8.5, and also pH 10.2 to 10.6, indicating different optimum iso-electric points of colloid flocculation.



Flow sheet of Clarification by the Carbonatation Process at Natal Estates Ltd, Mount Edgewcombe, Natal.

We have not been able to confirm that preliminary or progressive liming to an optimum pH confers the advantages claimed by the beet technologist to the subsequently prepared carbonated juices, in the way of filtration rates and lime economy.¹

Our inadequate tank space for time of contact before gassing and the absence of a reliable automatic pH recorder, may be responsible for this unsatisfactory finding, which will be further investigated.

Contrary to the experience of Java,⁵ we have derived no material benefit in lime economy by using de Haan's modification of liming and gassing.

We have adopted the simultaneous liming and gassing on account of the absolute failure met with, at the earliest attempts with heavy liming in one operation, followed by gassing, when the excessive formation of the gelatinous and frothy hydro-sucro-carbonate of lime increased the time of preparation of one tank to two hours.

We rely entirely on the progressive physical and chemical action of incremental doses of lime carbonated in the juice at low alkalinity in order to obtain a high non-sugar elimination and high filtration rates. 12 to 14 per cent. of 20° Baume lime-cream on volume of juice is our normal practice for safe work at the filters.

The volume of lime neutralised per unit of time, i.e. efficiency of gas absorption, is of capital importance. This has been found to be retarded by too low a pH at gassing, although not to the same disastrous extent as in the case of excessive alkalinity.

Our efforts have concentrated on the design of carbonatation tanks, where the gas is split up into bubbles by a combination of distribution pipes and perforated baffles, together with lime admission at various points in view of a homogeneous reaction, not easily obtainable in tanks with 10 feet of juice.

Much as we would like to preserve our already low reducing sugars, we have not been able to maintain consistently a high throughput by working our liming and gassing at temperatures under 55°C., and in view of the definite improvement in settling and filtering rate the temperature of the raw juice going to carbonatation is kept near 70°C.

The subsequent exposure of reducing sugars to alkaline reactions during settling does not help reducing sugar preservation.

On the score of exhaustion of final molasses, our system is open to criticism, with its formation of harmful reducing sugar decomposition products. No froth fermentation is, however, noticeable in our last massecuites, which are dropped somewhat hot from the pan, namely, 70-75°C.

The quality of our refined sugar is nevertheless a challenge to the hitherto accepted dogma of dark colour formation with high-temperature alkaline cane juice clarification.

We propose to read, at a later date, a contribution to the study of loss of sugar in molasses as revealed by the percentage composition of the non-sugars of various factory molasses for a number of years. Data on pH curves, gas absorption, speed of settling and filtration, reducing sugars destruction, elimination of ash, gum, wax and organic non-sugars, have been studied in great detail and will also form the subject of short papers.

It is sufficient to state that, with the high elimination of non-sugars during clarification, the average rise in purity from raw juice to clarified juice is 5.06 degrees for the past 25 years, with an optimum rise in excess of 6 degrees for two complete seasons.

Very seldom has the weight of molasses exceeded 3 per cent. cane, and for the past twelve seasons this figure has been reduced to 2.5 per cent. cane.

With a view to economy in labour, cloth and equipment, and to reduce sugar losses, we have been the pioneers in South Africa in discarding the plate and frame presses, which have been replaced by rotary vacuum self-discharging Mauss filters working in conjunction with settler-thickeners of our own design.

In this system the muddy juice from the first carbonatation tanks, limed and carbonated to a pH of 9.5 to 10.0, is settled in a continuous settler working on the upward mud filtration principle, where 60 to 65 per cent. of clear juice is withdrawn and the bottoms continuously delivered in the form of a thickened sludge containing 25 to 30 per cent. filter cake to the Mauss rotary vacuum filters.

Vacuum filtration with only 15 to 17 inches suction postulates for continuity of operations muddy juices of extremely good filtrability, and consequently this method of handling our large filter cake production has influenced our technique of carbonatation.

It is surprising to find that the total effective filtering area of our battery of 15 filters is only 675 sq. ft., out of which 10 filters or 450 sq. ft. are sometimes sufficient to take care of 18 tons of filter cake per hour, washed to a sucrose content of less than 1 per cent., equivalent to 4.5 sq. ft. of filtering area per ton of cane per hour—a removal of 53 lbs. of wet cake per sq. ft. per hour. At maximum efficiency these machines may remove 80 lbs. of wet cake (50 per cent. moisture) per sq. ft. per hour.

The mixture of decanted juice from the settler and of filtered juice from the Mauss, both at a pH of 9.5 to 10.0, is subjected to a second carbonatation by a continuous gassing system where the alkaline reaction is brought down to a pH 8.2 to 8.4 (faint pink colour to phenolphthalein solution).

No good purpose is served by further reducing the alkalinity, as this would not precipitate more lime and the alkalinity is due to potash.

The cloudy second carbonatation juice is raised to 80°C. and the small amount of fine precipitate is filtered in pressure leaf filters of the Auto-Suchar type.

We aim at producing a very bright liquor from these excellent filters, and in view of the fine nature of the precipitate, a pre-coating of Hyfflosupercel is often necessary, and the pressure is seldom allowed to rise over 25 lbs. per sq. in.

The pH of the bright filtered liquor is finally brought down to strict neutrality by a touch of sulphur dioxide before it enters the evaporator, without further filtration, as the formation of soluble potassium sulphite does not produce any cloudiness.

(All the various stages are shown in the attached flow sheet.)

In the early days of our inexperience with the process, before the introduction of pH control, this sulphured juice was strongly bleached, to an acidity equivalent to 250 milligrams sulphur dioxide per litre. We have since found that a pH reaction of 7.0 to 7.2 is quite safe for obtaining a high degree of whiteness in sugar, whilst obviating the risks of inversion losses and corrosion of evaporator tubes.

The auto-filters may or may not be used for syrup filtration from the evaporators. They also provide an up-to-date machine for the recovery of decolorising carbon if treatment by this decoloriser becomes necessary.

From this stage, the boiling house work is on parallel lines with all cane sugar factories, except in so far as the production of a high percentage of refined sugars enforces recrystallisation of the second and third sugars.

In this cursory survey of our conditions of work, for the sake of brevity, no reference has been made to our attempts at sulpho-carbonatation, single carbonatation, continuous carbonatation, and the uses of Norit, Carboraffin, etc.

Our present methods have been found practical and workable. They may not necessarily be the best, and they may be altered in the near future.

In view of our special difficult conditions, it is gratifying to compare our efficiency for the past eight years with that of the average of the 53 Javan factories³ working by the carbonatation process, before the war, as indicated in the juice sucrose balance sheet.

	N.E.L.	Java.
Lost in molasses per cent. sucrose in juice ...	7.46	7.20
Lost in filter cake per cent. sucrose in juice .	0.54	0.50
Lost in undetermined per cent. sucrose in juice...	1.91	1.80
Recovered in sugars per cent. sucrose in juice	90.09	90.30
Total in juice per cent. sucrose in juice ...	100.00	—

In the first ten years of its adoption up to 1929, 188,367 tons of refined sugar was manufactured; and from 1930 up to the war period this figure increased to 304,752 tons; whilst during the six years of war up to the last season 249,540 tons of refined sugar were produced.

Summing up the production of refined, Government grade and cargo sugars, from its inception, we arrive at the respectable total of 1,075,999 tons of commercial product having been manufactured from cane juice treated by the carbonatation process in South Africa. Our national economy has been the richer by the use of 482,933 tons of limestone and 52,051 tons of coke obtained from our soil.

Well can we say, after the silver jubilee of its life, that the carbonatation process has merited its right of citizenship as a South African industry, and justly deserves the praises of Honig⁸ as "one of the cheapest, cleanest and most reliable processes in the sugar industry, ensuring standard quality of the sugar."

References.

- ¹ Baerts, F. (1934): Le prechaulage Dedek-Vasatko. Rapports III. Congres International Tech., Paris.
- ² Campbell, W. A. (1920): A New Method of Manufacture in Natal. Jour. of Ind., S.A., Oct. 954.
- ³ Deerr, Noel (1933): A Review of Java Factory Practice. I.S.J. 8.
- ⁴ Farnell, R. G. W. (1925): Investigation of the Colloids of Cane Juice and Syrup in Natal and Mauritius, I.S.J. 254.
- ⁵ Geerligs, P. (1915): Practical White Sugar Manufacture. 69.
- ⁶ Maxwell, F. (1923-4): The Case for the Carbonatation Process in the Manufacture of White Sugars. I.S.J. 572.
- ⁷ Lyle, Oliver (1941): Technology for Sugar Refinery Workers. 254.
- ⁸ Spencer and Meade (1945): Manual for Cane Sugar Manufacturers. 119.

THE PRESIDENT, in opening the paper for discussion, said that the Natal Estates had been the pioneers in introducing the carbonation process in South Africa more than twenty-five years ago, but it had been a pioneering move, the initiative of which had never been followed by the rest of the industry. The reason was probably that we knew too little of what was being done by Natal Estates in their unique process, and we were therefore very grateful to Mr. Rault for presenting this paper to us.

Mr. RAULT said that during the last few years they had so many visitors to the factory who enquired about the process, but no satisfactory answers could be given during the course of conversation. They had also been honoured by a visit of the leading technologists of the industry last season. There was therefore a great deal of interest taken in the carbonatation process and although not much was said in the past, the process was by no means a secret; in fact it was the standard process in beet factories

in other countries, but they had had to adapt it to South African conditions, and certain deviations from standard practice overseas had to be made. He had often been struck by the differences in technique as outlined in text books and as followed in actual practice. That, then, was the reason for writing this paper. After agreeing to submit a paper on the carbonatation process he wondered whether to deal with one aspect of it or to give a general and necessarily scanty review of the whole. He had decided on the latter and he had therefore tried to give their 25 years' experience in carbonatation in one short paper. He might, however, deal with certain interesting aspects of the process at a later date.

Mr. VIGER said he noticed in the flow sheet a gas absorption tower on top of the first carbonatation tank. He wanted to know if the pre-heated acid raw juice from the heaters went through this tower before entering the carbonatation tank. If this was the case, the acid hot juice would not absorb any carbon dioxide as it had not been previously limed. He was aware that the gas from the kilns contained about 30 to 40 per cent. carbon dioxide, could Mr. Rault tell him what the carbon dioxide content of the gas escaping from the first carbonatation tank was?

Mr. BOOTH endorsed and emphasised Mr. Rault's statement that juice filtration rates were the essence of successful juice treatment and the advantages derived from proper juice handling were of greater and more immediate economic importance than the striving after optimum conditions laid down in the obscure theories on the exhaustibility of final molasses. He thought filtration had been a neglected subject in our sugar factories. The Oliver type of filter was a strainer, not a true filter, and should be regarded as such even where sugars of 96° and 98° pol were produced.

THE PRESIDENT wanted Mr. Rault to tell the conference what the fertilizer value of carbonatation filter cake was as compared with sulphitation cake.

Mr. WHEELER asked for the quantity of carbonatation cake per ton of cane. He thought it was much greater than that produced by the sulphitation factories. More impurities were removed from the raw juice and the purity of clarified juice was therefore increased considerably. He was not sure, however, what the effect of this elimination of impurities was on the purity of the final molasses.

Mr. RAULT, in reply to these questions raised, said that Mr. Viger was quite correct in his reading of the flow sheet which indicated that the raw heated juice from the mills was pumped up a tower where it cascaded through a countercurrent to the waste

gas from the carbonatation tanks. This hot raw juice was unlimed and still of a pH of about 5.6 and consequently the carbon dioxide absorption was practically nil. The tower, however, served a very useful purpose. It was a prolongation of all the short exhaust gas pipes of the battery of carbonatation tanks which delivered into one main pipe connected to the bottom of this tower. Any alkaline juice carried up the waste gas pipes in the form of a froth met the incoming cascading raw acid juice and was thereby broken up and returned to the tank in the process of filling. In spite of maintaining a low pH during the first carbonatation there was still occasional foaming and this tower was an excellent save-all.

The average live gas from the lime kiln contained 30 to 35 per cent. carbon dioxide and the exhaust gases going up to the tower still had 12 to 18 per cent. carbon dioxide. In fact it was as rich in carbon dioxide as the live gas from the sulphur burners was in sulphur dioxide content and richer in carbon dioxide than the average gas from the bagasse chimney stack. When compared with the efficiency of gas absorption in the sulphitation factories their carbonatation gas system seems very wasteful. It should be remembered that a lime kiln produced not only the 44 parts of carbon dioxide for every 56 parts of quicklime strictly required for neutralisation of the lime produced, but also an additional 30 parts carbon dioxide from the coke burned. And this had to be wasted—it was a surplus—unless additional lime from an outside source was used in the juice. A 60 to 70 per cent. gas absorption was therefore the maximum required.

With reference to a query from Mr. Dymond, the speaker said that Tate and Lyle's "mild carbonatation" referred to in his paper consisted in the carbonating of barely half a per cent. lime on melt and consequently it was possible to gas this small amount of lime by means of weak concentration of carbon dioxide present in the flue gases on lines similar to those tried at Darnall on raw juice where from 2 to 3 grams of lime per litre of juice had to be neutralised. At the Natal Estates the flue gases could not possibly be used for treating the heavy amount of lime required for their process as the gas handling plant would be disproportionately cumbersome. Their experience was that a carbon dioxide content of less than 28 per cent. in the kiln gas invariably meant an insufficient liming and a slowing down of their filtration rate.

In the 1942 Proceedings some details as regards the comparative values of carbonatation and sulphitation filter cakes were given. As could be expected, carbonatation filter cake was higher in lime content. The phosphate, nitrogen and organic matter in the carbonatation cake was lower than that

of the sulphitation cakes because it was diluted more with lime. The cake was nevertheless valuable agriculturally and was used in the fields and usually applied to the lands with irrigation water.

The wet filter cake from the factory varied from 10 to 12 per cent. on cane. Larger quantities of impurities were eliminated in the carbonatation process of clarification than in sulphitation. Mineral impurities as well as non-sugars were eliminated and there was a definite drop in ash content from raw to clarified juice. The ash composition of clari-

fied juice in the carbonatation process was different to that of sulphitation process. Invert sugar was unfortunately also reduced. In the raw juice the reducing sugar ratio was about 3 per cent. and it fell to 2 per cent. in clarified juice and consequently the purity of their molasses was somewhat higher as a result of an unfavourable reducing sugar ash ratio, but the smaller quantity of molasses produced, resulting from the higher non-sugar elimination partly compensates for the higher sucrose content of molasses.