Chemical Control.

The analysis of the flue gases by the orsat or any other suitable apparatus gives the composition of the gases and the temperature is recorded by means of the pyrometer; any defect in the admission of air in the draught and in the setting of the dampers is brought to notice at once and the necessary alterations can be made, because the flue gases should contain a maximum of carbon di-oxide, indicating the complete combustion of the fuel. The result will be steady steam pressure, regular and full day's work of the factory.

(12) Necessary Outfit for the Control.

Besides the usual laboratory instruments including bottles, graduated flasks, crucibles, basins, etc., the laboratory must have very delicate and sensitive balances, a perfect polariscope; tested brix and specific gravity hydrometers; a refractometer, which is necessary for the control of the fabrication, because its use has the advantages over the brix or the specific gravity spindles of speed, easy manipulation, less quantity of sample for determination and more correct information. A flue gas apparatus, as the orsat, and a pyrometer for the temperature of the gases, a small hand-driven centrifugal to test the massecuites and treacles and so necessary for the detection of the degree of exhaustion of treacles added to the massecuites in crystallisers, when these machines are worked as such and not as mere coolers. A powerful hand or belt driven small mill or a disintegrator and a strong press for the analysis of samples of cane.

From a well equipped laboratory under honest, intelligent and clever European chemists and assistants, who understand their work, who can interpret and apply analyses, will come reliable and correct information which should improve the fabrication for the benefit of the mill-owners and of the sugar industry in general.

DISCUSSION ON THE PAPER.

Mr. Lomeau stated they had all listened with interest to Mr. de Froberville's paper. As he noticed a number of very capable chemists in the audience he would like to take the opportunity of suggesting to them the desirablity of getting a uniform control if possible. This had been done in some countries with a measurable amount of success and he believed it was a step in the right direction if they could get together and devise some form of uniform control. The actual working out of the purity differed according to the various methods employed and if they could have some uniform control to apply to all the factories it would be very valuable indeed.

The Chairman in thanking Mr. de Froberville for his excellent paper, stated that he thought it would be a very difficult matter for chemists or any scientists to express technical subjects in such a way as to be easily understood by those who had very little knowledge of the subject. He had to admit however that Mr. de Froberville had succeeded in doing so, and where a more technical paper would perhaps have been of little interest to him because he would not have understood it, he had been able to follow Mr. de Froberville with very great interest. (Applause).

THE SULPHO-DEFECATION PROCESS IN NATAL.

(Paper by L. E. Rouillard, La Mercy.)

Every newcomer to South Africa connected with the sugar industry cannot help thinking as soon as he learns of the average recovery of sucrose from the usual sulphitation process, that he is going to improve matters and make a name for himself. I cannot say that this was my impression when I first joined the industry, as then it had no chemical history, and I was probably the first chemist called upon to deal with Uba juice, but I shall never forget the disappointment I experienced after calculating my first weekly return.

The purity and sucrose content during that particular year were extremely good, everything seemed to have worked normally during the manufacture, but the recovery was such that it was impossible not to doubt the correctness of my figures. I thought the polariscope was out of order, that the samples had been badly taken, that the juice was leaking out somewhere or that it had been purposely thrown out of the factory. After a time I was compelled to believe that nothing was wrong except the refractory nature of the Uba juice, and it seemed quite plain to me that for certain reasons the sulphitation process was not suited to, or was not properly applied, to this particular cane juice.

As the position to-day has not so materially altered as to make us feel satisfied that the results leave nothing to be desired, I wish to express my views

Sulpho-Defecation Process.

on the subject, to invite criticism, with the hope that some good may result from a free discussion on this important question.

The Sulpho-defecation process or more simply the sulphitation process, for those who are not acquainted with the manufacture of sugar, may be briefly described as follows:—

The mixed juice flowing from the various mills is pumped through a sulphur apparatus where it absorbs a certain quantity of sulphurous acid gas and the bleached acid juice is treated with lime up to the desired point. It is then heated to coagulate the albuminoids which are thrown down along with the insoluble compounds formed by the action of lime in combination with the acids in the juice. Boiling is either carried out in open defecators and the juice allowed to settle in the same receptacles; or the juice is heated in super-heaters under pressure, the steam separated by an apparatus known as a steam separator, and subsequently the juice is boiled and skimmed in open shallow vessels known as eliminators and finally sent to subsiders and allowed to settle.

The main difference between these two systems is that in the first case the juice is heated under or up to the boiling point and in settling separates into three layers, scums on the top, clear juice in the middle and mud at the bottom. In the second case it is heated above the boiling point and all the mud is thrown down in settling, leaving nothing but clear juice above or a very slight layer of froth on the surface.

Whatever system is adopted the clear juice after settling is sent to the evaporators for concentration into syrup and the muddy portion sent to special tanks where it is treated with lime if necessary, filtered through filter presses and ultimately mixed with the clarified juice on its way to the evaporators. On leaving the evaporators the syrup is allowed to settle in tanks, the clear portion being drawn into the vacuum pans where crystallization takes place and the muddy portion clarified separately or returned to the defecators.

When crystallisation has taken place to a sufficient extent in the vacuum pans the thick mass of sugar known as Massecuite is discharged into crystallizers, which consist of either cylindrical or U shaped tanks, with revolving paddle intended to keep the massecuite in motion to prevent it from solidifying in cooling. The sugar crystals are finally separated from the mother liquor (molasses) by centrifugal force. This is, in few words, the process as a whole.

Attention must now be directed more in detail to the treatment of the juice by what is known as sulphitation. The process cannot be said to be based on any theory demonstrable by figures. It is not a chemical reaction in the sense that a definite proportion of a base unites with a definite proportion of an acid to form a salt. If we knew that a juice of a certain composition, say of a certain acidity, required a certain treatment sugar manufacture would be different from what it actually is, and you must, if I may so express myself, feel your way all the time. It requires long practice and considerable experience rather than theoretical knowledge to successfully make sugar.

The method generally adopted is to add lime and sulphurous acid until a point is reached when the solids in the juice separate from the liquid, in other words up to the point when the juice settles. As the raw material may constantly differ in composition it is useless to expect by adding measured quantities of chemicals over a stated time to obtain calculable results. At times it appears quite an easy matter to secure a perfectly clear juice, but suddenly for some unknown reason it is impossible to obtain anything closer than a milky juice and again sometimes the juice will not settle at all, or more correctly will settle very badly in spite of the long experience of the man in charge. Generally speaking this situation usually occurs when you have a visitor who has come specially to have a look at the juice.

If obtaining a juice which settles rapidly and well is one of our main objects it is far from being the only essential in the manufacture of white sugar.

There are so many other factors to consider that the clearest juice is not always a positive indication that the finished article will be of the best quality. It is, however, usually found when inferior sugar is produced from clear juice that some mistake in the treatment of scums or at some other stage in the manufacture is the cause. It is generally admitted that Uba juice does not subside or does not filter freely with the ordinary treatment adopted in other countries, dealing with other varieties of cane, and usually more lime has to be used followed by more sulphurous acid than is the case elsewhere. Whether the lime is added before or after sulphitation does not appear to matter, as one method succeeds no better than the other in removing the impurities and it seems that the point of importance is the application of chemicals in excess to secure the desired results.

As I have already said the process is more or less empirical. It must not be supposed, however, that the settling point can only be obtained by the addition of a certain proportion of chemicals. The amount of lime and sulphurous acid used may vary within very wide limits and the settling will occur as it were in stages instead of as a continuous process. The difficulty is to strike the exact point which will give the best clarification.

In the manufacture of raw sugar where the colour of the finished product is immaterial a perfect subsidation is not so essential although it must not be overlooked. As a rule in order to reduce expenses the smallest possible quantity of lime should be added, provided filtration is not interfered with; in fact with varieties of cane other than Uba a very limited quantity of lime is often the only chemical used. With Uba cane, however, this system has not

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given satisfactory results. The great viscosity of the final products due to the non-elimination of the gums is the principal objection to it and sulphitation along with liming cannot be dispensed with; in other words a small proportion of lime being insufficient to eliminate the gums more lime has to be added which requires to be neutralised by sulphurous acid. This neutralisation is essential to avoid the formation of a very viscous product caused by the action of lime on glucose in an alkaline medium at a high temperature.

When we come to the manufacture of white sugar, clarification must be as perfect as possible and a far more thorough supervision must be exercised at every stage of the process than is necessary in the case of raw sugar.

Working according to the principle that a bulky precipitate tends to remove more impurities from the juice than a light precipitate, we conducted at La Mercy a number of experiments to ascertain, if possible, the limit up to which sulphitation could be carried to obtain a high grade sugar. The juice was sulphured with from one to four grammes of sulphurous acid per litre adding naturally an equivalent proportion of lime to reduce its acidity. Owing to the difficulty experienced with such heavy sulphitation as 4 grammes per litre we had recourse to a double sulphitation process by which the juice was limed and sulphured and subsequently pumped throught a second sulphitation column, and ultimately partly neutralised with lime. The subsidation under this treatment left nothing to be desired and the filtration was comparatively good although the scums were more or less slimy. All these processes, however, never produced what could be called a first rate white sugar. The sugar was certainly of good quality, but did not fetch the best market price. It must be remembered that the South African market is hard to please as sugar must come closely to refined sugar in order to attract buyers. Having failed to obtain the principal object in view it was naturally concluded that the relatively large precipitate was not sufficiently bulky to remove all the colouring matter from the juice and that the bleaching action of the sulphurous acid was too fugitive to be reliable whatever might be the initial quantity of the gas used. The process was continued and in addition the syrup was sulphured. The sugar produced was certainly of better quality than before but could not stand comparison with our best samples obtained with sulphitation in conjunction with phosphoric acid.

In spite of these unfavourable results it would be rash to positively declare that the best mill-white sugar cannot be secured by sulphitation alone and I am rather inclined to believe that the best manner of applying sulphurous acid has still to be found. It must be remembered that these experiments on a large scale are costly when not successful and can only be carried out for brief periods. Moreover, the various products cannot be separated to obtain the final results such as recovery and one is not always at liberty to do anything one likes. For all these reasons and in the presence of our difficulties, I have always thought that a small experimental factory would result in the solving of important problems and would have amply paid the industry had it been established long ago.

If we turn our attention for a moment to the Carbonatation Process, which has given such gratifying results to the directors of The Natal Estates, it is at once evident that the extremely large quantity of lime used, which becomes insoluble in the form of calcium carbonate or chalk by the addition of carbonic acid gas envelopes all the colouring matter, gums and so on which are ultimately and completely removed by filtration in bulk. This is the best proof we have that the purification of the juice is only to a certain extent a chemical reaction and is after all mainly a physical action. It cannot be supposed that such a large quantity of lime is required to form a chemical combination with the relatively minute proportion of colloidal substances in the juice. Their separation must be attributed to their physical inclusion in a thick precipitate, which is easily separated from the pure juice, avoiding thereby all the trouble experienced with the sulphitation process at the last stage.

In presence of the knowledge that the inclusion of all colouring matter in a bulky precipitate seems to be the main reason for the production of a high grade sugar and seeing that the granular precipitate of calcium sulphite is not sufficient to secure the elimination of certain chromogenes, the use of phosphoric acid has been more or less universally adopted as an addition to sulphitation and the voluminous precipitate produced by the combination of lime with phosphoric acid separates most if not all the colouring matter, which remains in the mud or filtration. Were it not a question of cost I am inclined to believe that good results would follow from filtration in bulk with sufficient phosphoric acid to secure free filtration.

The question remains whether it would be possible to obtain the quantity of sugar required in working neutral or faintly acid juice, thus avoiding the necessity of neutralizing acid molasses at the final stage which is to my mind one of the causes of our inferior recovery. If, as we know, the proportion of ash in final molasses is more or less constant it stands to reason that all the solids added will tend to increase the quantity of final molasses. It is certainly true that in theory sulphitation should not be carried out beyond the neutral stage but up to now no one, in this country, to my knowledge, has ever produced a superior white sugar who did not work the juice freely acid. The trouble experienced with calcium bi-sulphite under these conditions is extremely worrying but is one of the things which the manufacturer of white sugar must accept as an unavoidable expense.

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Sulpho-Defecation Process.

Having always in view the necessity of improving our recovery we decided a couple of years ago to work according to the process recommended by M. Ch. Muller in a most interesting contribution in the "Bulletin de l'Association des Chemistes de Sucrerie de France." The main point which attracted our attention was that according to M. Muller certain cane yields a juice which does not settle properly on account of the presence of what he calls Silico Organic Colloidal compounds. He claims that by heating the green juice to 116 degrees C (240 degrees F.) with subsequent cooling to 45 degrees C. (115 degrees F.) before sulphuring and liming these products are completely decomposed. The Silico combines with the lime and is precipitated, the organic matter coagulates and the colloidal substances are destroyed by superheating. A third of the lime ordinarily used is sufficient to obtain perfect settling and sulphitation may even be dispensed with. Besides the complete elimination of the gums reduces almost completely the viscosity of the final products.

Clearly this seemed to be all that could be desired for our refractory Uba juice. I am sorry to say the experiment was negative and had to be stopped almost immediately because of the losses produced by inversion on account of the high temperature which with the means at our disposal could not be sufficiently reduced. After heating the juice to 116 degrees C. (240 degrees F.) it was found that the installation which had been erected at a small expense was not sufficient to cool the juice to the required extent viz: 450 degrees C. (213 degrees F.) and on account of the difficulty of improving our conditions we reluctantly gave up the experiment.

I mention this because during the time we actually tried the process subsidation was almost immediately perfect and the filtration through the filter presses the best I ever obtained. I believe that except in the case of old burnt cane containing acetic acid the process is certainly worth trying for those who have an excess of fuel and a relatively large supply of water.

In conclusion I may summarise my views thus:

1.—Judging from our results, simple neutralisation of the juice by lime has comparatively no effect on the gums and leads to extremely viscous final products which cannot be properly cured.

2.—Moderate sulphitation produces a better juice, but the gums are not sufficiently removed to secure the highest possible recovery.

3.—The most liberal initial sulphitation does not seem to clarify the juice to the extent necessary for the production of the best white sugar.

4.—Phosphoric Acid appears to materially help the removal of certain colouring matters in the juice.

5.—Although gums are removed to a larger extent by a bulky precipitate their removal is far from complete.

6.—The subsequent neutralisation of extremely acid molasses appears unfavourable to recovery by unduly increasing the quantity of final molasses.

7.—The great advantage of the carbonatation process over sulphitation is the complete removal of all objectionable impurities by filtration at the initial stage, leaving extremely fluid after products which do not require any further treatment.

DISCUSSION ON THE PAPER.

Mr. Kirkman asked whether Mr. Rouillard had any experience with the juices of the Java canes and Argentine canes in the country and how they compared with the Uba.

Mr. Rouillard replied that they were all mixed up with the Uba cane when sent to the mills and as there were such small quantities they were not noticeable.

Mr. Dymond stated that he had had the pleasure of spending a few days at Mount Edgecombe and he noticed that the action of lime on the juice at a temperature of about 50 degrees seemed to be one of the main points. After he returned he had found that by liming in the third and fourth mill-juices at a temperature of roughly between 45 and 50 degrees he had obtained a distinct rise of between 2 and 3 per cent in purity between the mixed juices and the clear juices—a rise which they did not obtain before.

Mr. Rouillard explained that in Java they always heated the juices up to 50 degrees before liming, and had found very appreciable results. He had done it also and had found a great difference in filtration, and that it was much better than working the cold juice. In Java they did it with the whole of the juice.

Mr. B. Pearce stated that he had the opportunity whilst in Hawaii of seeing an experiment carried out of liming in the fourth and fifth juices. Lime was added to the fourth mill juice, returned before the third, and then before the second mill. An experiment was made to find out the increase in the total recovery. This was raised from 87.67 when the juice was carried neutral to 92.60 when the juice was alkaline. The undetermined losses were reduced from 4.19 per cent. to .70 per cent.

Mr. Rouillard stated that when he came to this country about twenty five years ago they had the disease known as "gummy disease." He was told by one of the old planters that it was impossible to cook the sugar, that it would not crystallise. Finally they added a large quantity of lime before sulphuring to change the thing completely, and they managed to make sugar where they could not before. It was due to the action of the lime on the juice.

The Chairman after thanking Mr. Rouillard for his excellent paper, stated that Mr. Lomeau's suggestion would be put before the millers section of the Association and he hoped they would see their way to carry it out.