FACTORY BALANCE SYMPOSIUM

A FACTORY CHLORIDE BALANCE

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Abstract

A chloride balance in a raw sugar factory has been established. This balance compares favourably with the sucrose balance and could be used to detect chemical losses of sucrose. The use of chloride ions as tracers to detect losses in particular areas of the factory has been partially successful. The techniques of sampling and analysis are briefly described, and suggestions are made towards improving the accuracy of this work.

1. Introduction

The discussion following the paper "Potentiometric Determination of Chlorides in Molasses",[1] at the 1969 congress, included lively comment on the use of chloride as the basis for factory balance calculations. The concept had been used with success by Davies and his co-workers[2] in 1941 for investigating the magnitude of sugar losses in the evaporators and pan station, and by Virginio Zanni[3] in 1951, but little work has been reported since that time.

With the introduction of a quick and accurate potentiometric method for determining chloride, a routine factory chloride balance becomes a practical possibility. Chlorides are not subject to chemical destruction and, in common with other highly soluble inorganic ions can only be lost in the factory by physical removal, either through entrainment or absorption. A mass balance for chlorides over the factory should theoretically yield results approaching 100% recovery, and should definitely prove more reliable than a balance based on sucrose or total non sucrose, both of which may be altered chemically during the process.

Work has been carried out during the past season in an attempt to establish chloride balances in a raw sugar factory. This work has been partially successful in that a number of occasions a recovery of almost 100% was recorded over periods of two to three weeks.

At the same time at other factories, chloride was used as a tracer to pinpoint sugar losses. This programme has highlighted a number of unusual features.

The results so far are sufficiently promising to warrant continued and intensified investigation of the overall factory balance. If the techniques can be improved sufficiently to produce 100% confidence in the chloride balance, the adoption of the balance as a routine feature of factory control may help to explain some of the anomalies of present comparative factory data.

2. Experimental

2.1 *Chloride Analysis*

The Metrohm Potentiograph (E.436) as described by Comrie[1] was used throughout this work. $AgNO_3$ of either 0.05 or 0.1N strengths was titrated automatically against the various factory products, the end point being determined from the resultant millivolt curve.

The factory products which were analysed were as follows:

(i) Mixed Juice: 15 to 20 g of juice was accurately weighed and diluted to 50 ml for titration. The samples were not filtered before analysis and were therefore representative of the mixed juice as weighed.

(ii) Clear Juice: as for mixed juice.

(iii) Filter Cake: A number of techniques were used for this determination, none of them completely satisfactorily. At some mills it was possible to make a slurry of cake with distilled water, and titrate directly into this. At other factories this method did not give satisfactory results, and it was necessary to digest the cake with water or dilute HNO_3 and titrate the extract. Another technique used which gave a slightly improved titration curve was to add a known amount of chloride to the slurry before titrating.

(iv) Syrup: 4 g of syrup were weighed and diluted to about 50 ml.

(v) Sugar: 40 g of sugar were weighed and diluted to constant volume.

(vi) Molasses: 1 g of molasses was weighed and diluted to constant volume.

(vii) Stock: All products in stock were analysed for chloride at the beginning and end of each balance period.

(viii) Milk of Lime: 150 g of supernatant liquor was weighed, neutralized with HNO_3 and titrated directly.

2.2 *Sampling Frequency*

2.2.1. When a complete factory balance was being established the various points sampled were as follows:

(i) Mixed Juice and Syrup: Both were continuously sampled and composited for each shift.

(ii) Filter Cake: An hourly catch sample was composited for each shift.

(iii) Sugar: Each truck was sampled and composited weekly.

(iv) Molasses: Each scale tip was sampled and composited weekly.
No preservative was used at all.

2.2.2. During shorter tracer experiments, mixed juice, clear juice, filter cake and syrup were sampled either continuously where possible or frequent catch samples were composited for a period of four hours. In one experiment samples were composited for one week. Formaldehyde was used as a juice preservative, both mercuric chloride and basic lead acetate being obviously unsuitable.

2.3 *Sampling Methods*

2.3.1. The normal factory samples of mixed juice, syrup, filter cake, sugar and final molasses were used.

2.3.2. All intermediate products in stock were sampled for chloride analysis. The difficulties encountered in sampling the stock were:

(i) Syrup samples were not truly representative due to liquidation of the evaporators. The sample received was taken from the top of the tank and was usually of a very low brix.

(ii) Difficulties were encountered when compositing low grade massecuites and the crystal content of the final sample for analysis (1 g) fluctuated considerably. This produced substantial variations in the chloride content of massecuites. A maximum of 17 730 ppm chloride and a minimum 16 400 ppm were determined on one massecuite.

(iii) The inaccuracy of determining accurately the quantity of solids in stock led to further errors.

These factors made it difficult to determine accurately the quantity of chloride in stock and no doubt led to an undetermined chloride loss or gain.

3. Results

3.1 *Example of a Chloride Balance*

TABLE I

Empangeni Chloride Balance

18/11/1969 – 30/11/1969

	Metric Tons		Metric Tons
Mixed Juice	48.662	Sugar	1.541
Lime	0.010	Molasses	42.699
Stock (17.11.69)	22.986	Filter Cake	0.194
		Stock (30.11.69)	26.772
		Undm. Loss	0.452
	71.658		71.658

Undm. Cl^- loss % Cl^- in mixed juice = 0.93%

TABLE II

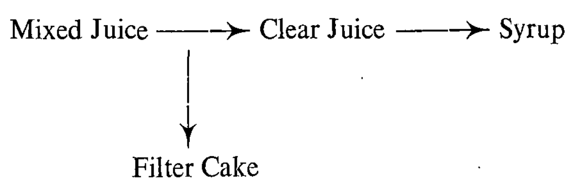
3.2 *Undetermined Losses at Empangeni*

No. of weeks Date	2 4.11.69/ 18.11.69	2 18.11.69/ 30.11.69	2 30.11.69/ 14.12.69	5 14.2.69/ 18.1.70	Total 11 4.11.69/ 18.1.70
Type of loss Undm Cl^--loss/gain % Cl^- in m. juice	0.88% gain	0.93% loss	8.27% loss	0.29% gain	1.33% loss
Undm Suc. loss/gain % Suc. in m. juice	2.55% loss	1.14% loss	2.73% loss	1.33% loss	1.76% loss
Undm Solids loss/gain % solids in c. juice	5.85% loss	4.41% loss	5.17% loss	1.06% loss	3.35% loss
Undm N. Suc. loss/gain % N. Suc in c. juice	21.70% loss	19.77% loss	17.17% loss	0.28% gain	11.15% loss

3.3 *Chloride tracer experiments at IL and TS*

At these two factories an intensive investigation was carried out in the clarification and evaporation stations. Mixed juice, clear juice and syrup were sampled continuously, or as nearly continuously as was possible, and analysed four-hourly for chloride. Filter cake was sampled hourly and a four-hourly composite was analysed.

All calculations were based on the assumption that there was no loss of chloride from the system:



except that which was removed in the filter cake. Thus, by determining the tons of chloride in the mixed juice and the filter cake, the tons of chloride in the clear juice and syrup could be calculated. From these figures, and the ratios of Pol/chloride, Brix/chloride and reducing sugar/chloride, the tons of pol, Brix and reducing sugars respectively could be deduced.

Specimen calculation

(*a*)	*Mixed Juice:*		Tons of mixed juice	13 391.207
	% Pol	12.07	Tons of Pol	1 616.319
	% Brix	14.45	Tons of Brix	1 935.029
	% red. sug	0.46	Tons of red. sug.	61.466
	% chloride	0.088	Tons of chloride	11.843
(*b*)	*Filter Cake*		Tons of filter cake	459.430
	% pol	1.49	Tons of pol	6.845
			Tons of Brix (based on purity of m.j.)	8.195
			Tons of red. sug. (based on red. sug/sucrose ratio in m.j.)	0.231
	% chloride =	0.026	Tons of chloride	0.119

(c) *Clear Juice*
pol/CI = 136.74
Therefore tons pol = (11.843 - 0.119) x 136.74
= 1 603.139
Brix/CI = 160.07 tons Brix = 11.724 x 160.07
= 1 876.661
reg. sug/CI = 46.18 tons red. sug. = 11.724 x 46.18
= 54.141

(d) *Syrup*
pol/CI = 136.12
Therefore tons pol = 11.724 x 136.12
= 1 595.871
Brix/CI = 159.39 tons Brix = 1 868.688
red. sug/CI = 41.25 tons red. sug. = 48.361

loss of pol in clarification = (1 616.319 - 6.845) - 1 603.139
= 6.335 tons
loss of pol in evaporation = 1 603.139 - 1 595.871
= 7.368 tons
loss of brix in clarification = (1 935.029 - 8.195) - 1 876.661
= 50.173 tons
loss of brix in evaporation = 1 876.661 - 1 868.668
= 7.973 tons
purity of loss in clarification = 12.63
purity of loss in evaporation = 92.41
loss of reducing sugars in clarification = (61.466 - 0.231) - 54.141
= 7.094 tons
loss of reducing sugars in evaporation = 54.141 - 48.361
= 5.780 tons

The results of two week runs at IL and TS are tabulated below in table III.

TABLE III

Clarification	TS	IL
pol lost % pol in mixed juice	1.10 gain	0.39 loss
brix lost % brix in mixed juice	1.49 loss	2.59 loss
red. sug. lost % red. sug. in mixed juice	18.24 loss	11.54 loss
% increase in chloride % non-sucrose	17.16	15.32
Evaporation	TS	IL
pol lost % pol in mixed juice	0.48 loss	0.46 loss
brix lost % brix in mixed juice	0.34 loss	0.41 loss
red. sug. lost % red. sug. in mixed juice	3.80 loss	9.40 loss

3.4. Another series of experiments were carried out in four factories simultaneously, using the normal factory analysis for pol, brix and reducing sugar, and compositing the routine factory samples for a week for chloride analysis. Each factory was studied for two weeks. The results are as follows:

TABLE IV

	Factory			
Clarification	A	B	C	D
pol lost % pol in mixed juice	1.41 loss	2.07 loss	0.15 loss	3.04 loss
brix lost % brix in mixed juice	2.87 loss	3.10 loss	0.51 loss	4.83 loss
r.s. lost % r.s. in mixed juice	6.27 gain	10.17 gain	0.45 gain	33.32 loss
% increase in chloride % non-sugars	10.11	9.34	3.00	15.51
Evaporation				
pol lost % pol in mixed juice	3.11 gain	3.01 gain	9.23 gain	4.83 gain
brix lost % brix in mixed juice	4.13 gain	2.19 gain	8.62 gain	4.61 gain
r.s. lost % r.s. in mixed juice	9.50 gain	0.15 gain	12.03 loss	0.81 gain

4. Discussion

In a raw sugar factory 95% of the chloride is removed in the final molasses, whereas the majority of sucrose is removed in sugar. Any inaccuracy caused by the weighing of final molasses will influence the chloride balance far more than the sucrose balance. This may be the reason why the determination of a chloride balance at Felixton was unsuccessful. Final Molasses at Felixton is weighed in a beam balance, which is probably not as accurate as the balance at Empangeni.

Investigation has shown that the chloride content of juices obtained from cane from different areas varies considerably. The chloride in juice from mixed variety of cane from Nkwaleni averaged over 1 500 ppm representing 5.3% of non-pol solids. Variety 376 cane from Empangeni contained 900 ppm chloride, representing 3% of non-pol solids. With this variation possible it is evident that the chloride content of mixed juice can fluctuate considerably over even short time intervals.

The compositing of mixed juice for chloride analysis was examined. Variation between the analysis of the composite and the average analysis of the individuals were significant. The technique of compositing is to be improved possibly with the addition of a juice preservative. Formaldehyde was found to have no effect on the chloride determination.

No record was kept of the amount of water added after the mixed juice scales. This water consisted mainly of condensate, very low in chloride, and had little or no effect on the chloride balance.

Greater accuracy of the chloride analysis can be achieved by using a larger sample weight and/or a reduction in the silver nitrate normality. This should improve the accuracy of the overall balance.

At Empangeni all syrup is accurately weighed, hence it was possible to establish a chloride balance after the syrup scales. The chloric recovery from syrup to sugar and final molasses was 99.8% over a period of 6 weeks 30/11/1969 – 18/1/1970. Over the same period the sucrose recovery was only 98.74%, an undetermined loss of 1.26%.

The work which was done at IL and TS showed results which could be related to some extent with the normal factory balance over the test period. The apparent gain in pol in clear juice at TS is not explained, but is possibly due to the fact that stock changes were not taken into account in this relatively short run. At IL the loss in pol was found represented approximately 50% of the undetermined loss for the test period.

The figures obtained at the other factories are much less reliable, owing to the rather extended periods over which samples were composited for chloride analysis. However, in spite of this there are some rather anomalous results which should be looked at in more detail. Factory C, for example, showed a very low increase in Cl % non-sucrose during clarification. Chloride being completely soluble, this figure should provide some indication of the insoluble non-sucrose removal in the filter-cake. The average for the six factories investigated was 11.74% increase in chloride % N-S, whereas at factory C it was only 3%. There is some indication here that the clarification station at this factory should be investigated.

Another example is the apparent reducing sugar loss at factory D. A loss of 33.32% in reducing sugars at this particular factory represented more than 200 tons. This loss was highly unlikely to have occurred in fact, and the indication is that the analysis is suspect. Here again further investigation is warranted.

5. Conclusion

If the technique of mixed juice compositing can be improved, representative stock stamples taken and molasses weighing perfected, a highly accurate chloride balance should be achieved.

This method of investigating a factory balance shows promise, particularly with regard to pinpointing chemical losses of sucrose in particular areas of the factory. The techniques which have been used in the investigations reported here were not sufficiently reliable to provide a 100% balance, but with suitable improvements in the current season it is hoped that the method can be perfected into a really useful tool in factory control.

6. Acknowledgements

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7. REFERENCES

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