INVESTIGATIONS OF UNDETERMINED LOSSES AT PONGOLA

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Abstract

A review of the history of undetermined loss at Pongola showed that the problem began in about 1974 and grew steadily. Major factory modifications took place just prior to 1974 but the dominant cane variety also changed at this time and the use of variety NCo 376 increased in parallel with the undetermined loss. The loss has shown a seasonal trend with maxima at the beginnings and ends of seasons.

Weekly measurements of sucrose lost from the stage between mixed juice and syrup showed that usually more than 50%, and sometimes more than 80%, of the undetermined loss occurred from this stage. When particularly high loss occurred however it was mainly from a stage after syrup. Very high losses invariably occurred only in the late season. In the laboratory, composite weekly samples of late season molasses evolved gas at a rate which correlated with the factory undetermined loss. Some samples of late season syrups lost sucrose at unexpectedly high rates when heated at 70°C for 48 hours, and the losses correlated with those measured in the factory. No such correlations were evident for mid-season samples.

Introduction

The problem of undetermined loss at Pongola (PG) reached a climax in the 1981-82 season with a loss for the season of 3,57%. The value of this excessive loss, when compared with the industrial average of 1,55%, was about R750 000. This warranted a detailed investigation of the problem and so a team involving C. G. Smith Sugar, Ltd., Central Board and SMRI was set up.

History

Before 1972 the undetermined loss at PG was generally below the industrial average, with the mean annual losses between 1954 and 1972 being 1,24% for PG and 1,30% for the industrial average. Since 1974 the PG undetermined loss has always been above the industrial average and has steadily increased (Figure 1).

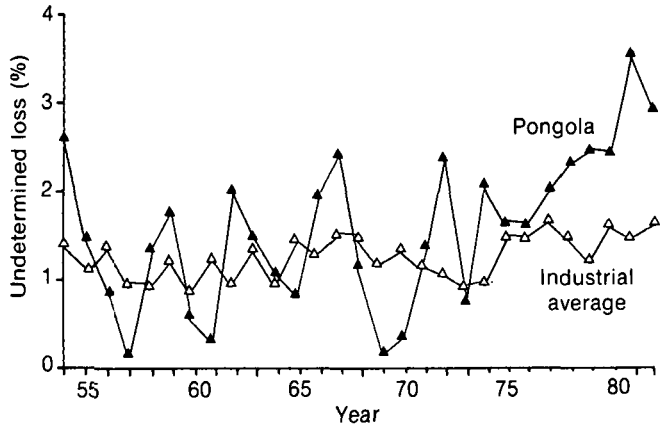

FIGURE 1 Annual undetermined losses for Pongola and industrial average. (Data are based on pol. Prior to 1976 sucrose in molasses was determined by Lane and Eynon, whilst before 1972 sucrose in mixed juice was by Jackson and Gillis).

Major factory modifications took place between 1972 and 1974. These included the installation of a diffuser, two long-tube Kestners, increased pan capacity, independent vacuum pumps for each pan, consolidated pan supply tanks, an additional Oliver filter and an SRI clarifier to replace the three Dorr clarifiers.

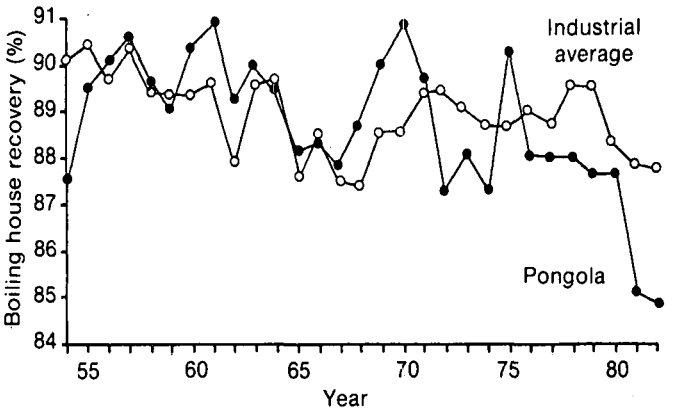

FIGURE 2 Annual boiling house recoveries for Pongola and industrial average

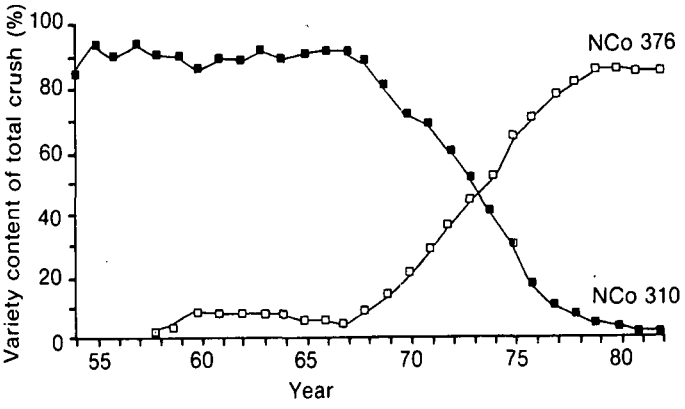

FIGURE 3 Cane variety contents of annual crushes at Pongola

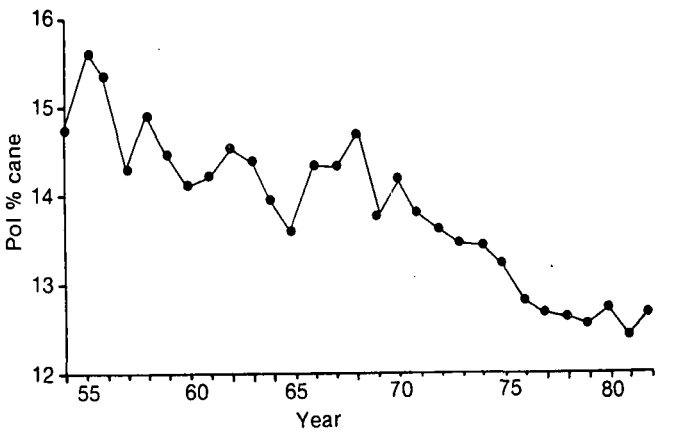

FIGURE 4 Historical trend in pol % cane at Pongola

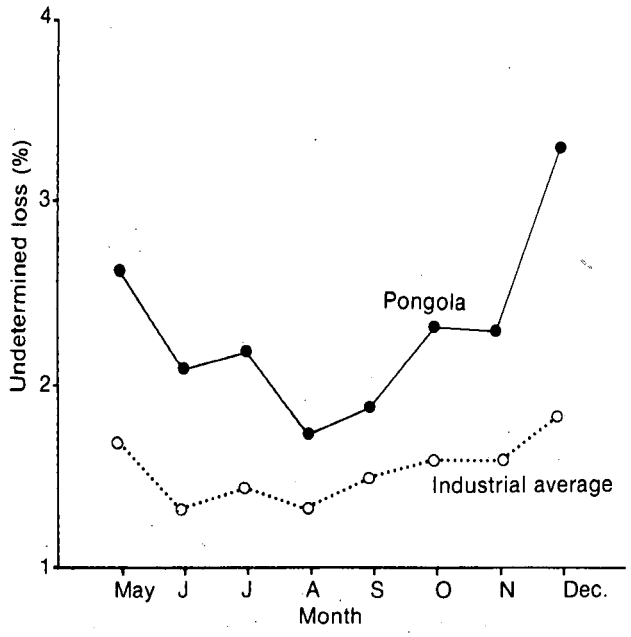

FIGURE 5 Seven-year (1975-81) mean undetermined losses for different months

The fact that boiling house recoveries (Figure 2) and undetermined losses changed from comparatively good to comparatively poor when major factory modifications took place might suggest that the changes in performance were caused by the factory modifications. This could however be a misleading conclusion because other significant changes took place at about the same time as the factory modifications. For example the cane variety NCo 376 replaced NCo 310 as the dominant variety (Figure 3). One probable consequence of this variety change was that the pol % cane decreased steadily as the new variety increased (Figure 4). A more subtle consequence was that the nitrogen intake to the factory, per unit of sucrose produced, probably increased by about 17%. This can be calculated from data on nitrogen and sucrose contents of the two varieties when grown in an experiment at PG (Hellman[2]).

Since 1976 the annual mean purity of mixed juice at PG has always been below the industrial average, with the mean difference for the period 1976 to 1982 being 1,02%. This comparatively low purity at PG was not evident before 1976 and it may also be a reflection of the change in cane variety.

The annual increases in undetermined loss since 1976 cannot be attributed to an increased rate of factory throughput because this has been almost static since 1976.

The seasonal trend in undetermined loss at PG has tended to follow a pattern of steady decline until mid-season, followed by a distinct increase towards the end of season. This trend has been much less pronounced in the industrial averages (Figure 5) but has been evident at Malelane (ML) where undetermined loss has also been high. PG and ML have particularly high reducing sugar ratios in mixed juice and the seasonal trend in this ratio tends to run parallel with the undetermined loss.

Between 1972 and 1981 the undetermined loss problem at PG was the subject of 11 reports covering a range of investigations. These included studies of analytical accuracy, inversion in the refinery and losses in entrainment, effluent and weekend stock. None of the reports gave a clear indication of the likely cause of the problem.

Investigation Strategy

In the absence of a clear indication of the cause of high undetermined loss it was decided that the first phase of the investigation should be aimed at locating the factory stage(s) from which loss was occurring. This was to be done by measuring sucrose balances across the following three sections of the factory:-

(i) mixed juice to syrup
(ii) syrup to refinery intake
(iii) refinery intake to warehouse.

Laboratory investigations into the mechanism of the loss were planned as a second phase for application only after areas of loss had been located.

Factory Investigations

Methods

The monitoring points involved in the investigation together with a broad indication of the relative quantities passing each point are shown in Table 1.

TABLE 1

Monitoring points and their throughputs

Monitoring point	Approximate sucrose throughput relative to mixed juice (%)
Mixed juice scale	100 (3 000 t/wk)
Clear juice scale	100
Filter cake weigher	0,15
Syrup scale	98
Melt scale (rawhouse to refinery)	105-115
Orifice plate (sweetwater to melter)	7
Return syrup scale (refinery to rawhouse)	15-20
Weighbridge (warehouse returns)	0-2
Molasses scale	9-14
Refined sugar scale	79-85
Parshall flume (effluent)	0,5
Annubars and weir (cooling water)	0,3

The clear juice scale was installed as a check scale for mixed juice and it can be used for either function.

Magnetic flowmeters were initially used to measure melt and return-syrup entering and leaving the refinery. These proved unreliable and had to be replaced with scales. Samples of the melt and return-syrup were drawn by gear pumps which were activated by each scale tip.

Flowmeters on sweetwater also proved problematic and so the sweetwater circuit was simplified and the flow measured across an orifice plate. A signal from the differential pressure cell across the plate controlled the speed of a motor which drove a gear pump to collect samples. The samples were drawn from a "break pot" in which sweetwater was at atmospheric pressure. (Sweetwater under slight pressure was of sufficiently low viscosity to be forced through the gear pump at a rate which was not proportional to the pump speed). A peristaltic pump was used initially to draw the sweetwater sample from a pressurised point but it required considerable maintenance.

Effluent flow was monitored continuously with a Parshall flume and associated sonic level detector at a point before run-off from the diffuser area enters the drains. A signal from the integrator on the flow recording device activated a peristaltic pump which drew samples of effluent.

Cooling water entering and leaving the factory was measured by annubar devices. Signals from the integrators periodically activated solenoid valves through which samples of inlet and outlet water were collected.

A scale for weighing refined sugar was installed for the project but proved unreliable because of a tendency to overflow. Refined sugar production has therefore been monitored by a system of bag counts, using electronic counters, whilst efforts to improve the reliability of the scale continue.

Raw sugar was bagged only occasionally and in small quantities which were monitored by bag counts.

Warehouse returns were collected in tote bags and weighed on the main weighbridge.

Sample cooling devices of the type used by Central Board were used for the syrup, melt and sweetwater samples. Mercuric chloride preservative was added to the sample receivers, including those for effluent and cooling water. The samples were collected and analysed hourly. Sugar traces in effluent and cooling water were determined colorimetrically using the phenol-sulphuric acid method. Composite weekly samples of mixed juice, clear juice and syrup were analysed by gas chromatography and stored at −20°C for any subsequent laboratory studies.

Sampling of high brix syrup through a gear pump was unsuccessful at first because of crystal formation in the pump and its feed pipe. This problem was overcome by fitting a 'T' piece to the feed pipe close to the pump. Syrup from a stirred tank above the scale flowed permanently by gravity across the 'T' and thereby prevented crystallisation by keeping the pump and pipe warm.

Before factory start-up the mixed juice and syrup scales were checked against each other by connecting them in series and pumping 830 tons of water through them. The difference between the two scale readings was only 0,13% which was well within the 0,5% tolerance allowed for scales.

Saccharate liming was not used during the period of the investigations reported because it would have introduced complications of syrup recirculation.

Results and Discussion

Effluent and entrainment: During the 1982–83 season losses in effluent increased steadily to an alarming level of almost 28 tons per week, representing an undetermined loss of 0,8% (Figure 6). At this stage (week 17) hosepipes were banished and there was an immediate decline in the losses. A sump was built to collect spillages for return to process and this came into operation in week 21. Losses since then have been about 6 tons per week and have persisted at this level throughout the following season.

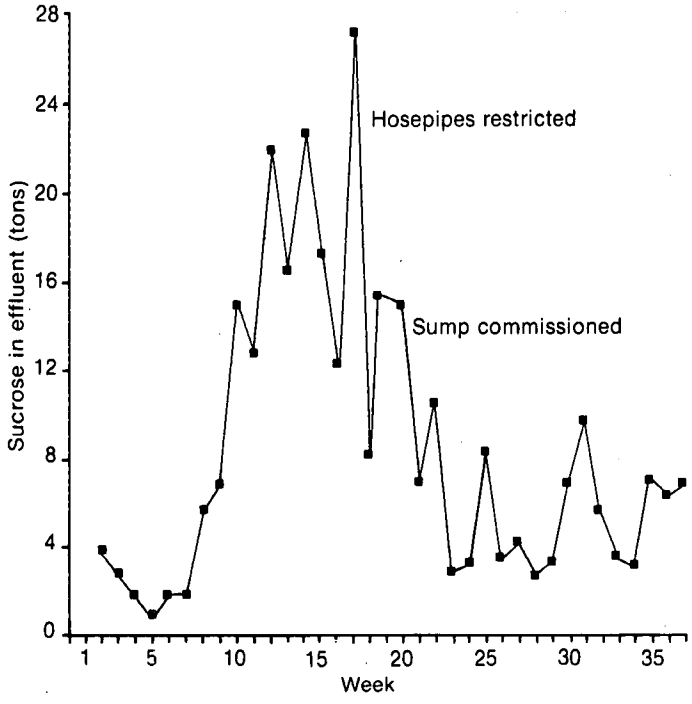

FIGURE 6 Weekly losses in effluent in the 1982–83 season

Losses in cooling water were generally less than 4 tons per week. Occasional peaks of about 10 tons per week coincided with known entrainment problems.

The possibility of entrainment into the V1 stream which is injected into the diffuser has been of concern because the pol factor (mixed juice: DAC) has regularly exceeded 101 since mid 1982. Sugar traces were detected in samples of the V1 stream but these decreased to very low levels when the entrainment separator was modified and overhauled, but the pol factor remained high.

Chloride balance: Weekly composite samples of syrup and molasses were analysed for chloride so that a chloride balance could be established in an attempt to detect any gross physical losses. The analyses were done by potentiometric titration. The balance could not start from mixed juice because the mixed juice samples are contaminated with chloride from the mercuric chloride preservative, and the collection of special unpreserved samples was impractical. Syrup was considered stable enough to allow the preservative to be withheld until after a sub-sample for chloride analysis had been collected. The use of hydrochloric acid for cleaning filter cloths in the refinery was banned.

The weekly chloride balances varied widely but the mean value was close to that of a perfect balance (Figure 7). The wide fluctuations between weeks 29 and 30 were caused by a known faulty molasses scale. The results gave no indication of a physical loss from the factory between syrup and molasses but the method lacked the necessary precision and was abandoned after the first season.

Checks on mixed juice scale: The high pol factor, together with the fact that the mixed juice scale is subjected to vibration from the shredder led to the suspicion that, under dynamic conditions, the mixed juice scale overweighs by 1 to 2% (the earlier checks against the syrup scale could be done only when the shredder was inoperative).

To test this possibility a second mixed juice weighing station was constructed and operated in series with the main mixed juice scale, all analyses being done by Central Board staff. A number of comparison checks were done and none revealed any significant discrepancy between the scales. The most recent comparison, extending over a full week, showed a difference in tons pol of only 0,08%.

Sectional Balances: As from the 14th week of the 1982-83 season balances across the mixed juice-syrup stage have been attained with reasonable regularity and confidence. Balances across the other two stages have proved much more difficult, reasonable success being achieved only towards the end of the 1983-84 season after considerable testing and modification of equipment.

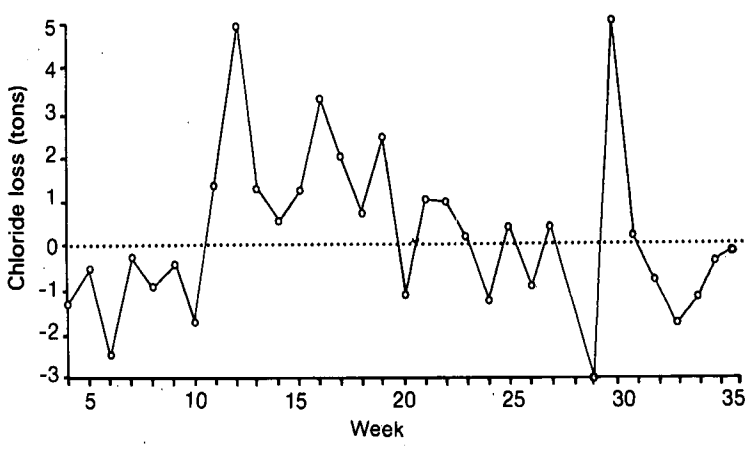

FIGURE 7 Weekly chloride balance between syrup and molasses

Results of the mixed juice-syrup balances for 1982-83 are summarised in Figure 8 and Table 2. Between weeks 14 and 31 more than 50% of the total undetermined loss could be accounted for in the mixed juice-syrup stage, with a figure for some weeks exceeding 80%. During the last six weeks of the season, when the mixed juice purity was dropping rapidly and the undetermined losses were high, the loss between mixed juice and syrup averaged only 30% of the total undetermined loss for this period. The loss for the mixed juice-syrup stage remained fairly constant when expressed as a percentage of throughput.

TABLE 2

Losses between mixed juice and syrup for different periods during the 1982–83 season

Week no's	14–19	20–25	26–31	32–37
No. of valid records	5	4	4	4
Mean weekly total undetermined loss (t)	91	78	78	120
Mean weekly MJ-syrup loss (t)	46	62	45	33
Mean difference (& SD)	45(20	16(22)	33(13)	87(46)
Mean MJ-syrup loss (a) % of suc. in cane	1,30	1,76	1,54	1,25
(b) % of undet. loss (& SD)	52(15)	83(18)	58(14)	30(9)

The finding that most of the undetermined loss takes place from the mixed juice-syrup stage was confirmed in the 1983–84 season and again it was found that there was a seasonal effect. Towards mid-season a high proportion of the undetermined loss (up to 82%) could be accounted for in the mixed juice-syrup stage but this proportion declined towards the beginning and end of season when the purity of mixed juice was also relatively low (Figure 9).

These figures have been calculated from pol analyses done at the mill but corrected for sucrose content by applying the pol:sucrose ratio as determined by Central Board on composite weekly samples. The pol:sucrose ratio was consistently higher for syrup than for mixed juice (because fructose is destroyed during evaporation) so this correction is important.

Limited success was achieved in measuring balances from mixed juice to clear juice. The results were too erratic to be really useful. It is suspected that, prior to week 10, occasional by-passing of the clear juice scale might have caused overestimation of loss. The later results in Figure 9 suggest that clarification and filtration are responsible for 32 to 59% of the loss which occurs within the mixed juice-syrup stage.

The glucose:sucrose ratio for clear juice was always very similar to that for mixed juice. In fact, in the 1983–84 season the difference between the two was exactly zero when the overall weekly mean was computed. Each week however syrup had a higher glucose:sucrose ratio than mixed juice. For the 1983–84 season the mean weekly increase in glucose content between mixed juice and syrup was 0,21% on sucrose. This shows that at least 0,38% of the sucrose inverts between clear juice and syrup. An unknown amount of glucose is decomposed during evaporation so this figure is an underestimate of the loss.

Despite the unchanged glucose:sucrose ratio between mixed juice and clear juice the sucrose purity of clear juice was consistently lower (0,5 to 1,0 units) than that of mixed juice. This might suggest that sucrose decomposition took place between mixed juice and clear juice but, with very few exceptional weeks, there was a gain in purity (usually more than 0,5 units) between clear juice and syrup. This anomalous purity gain (detected by GC sucrose analysis) casts doubt on the real cause and meaning of the measured purity drop between mixed juice and clear juice.

Brix balances across the mixed juice-syrup stage were erratic in the first season, with the figures based on hourly analyses done at the mill always showing less loss than those based on analyses of composite weekly samples. In the 1983–84 season reasonable agreement was achieved between the two sets of analyses. The brix losses moved in sympathy with the sucrose losses and were often about 85% of the sucrose loss, seldom more. Without being able to assess accurately brix addition and removal due to clarification, filtration and volatilisation it is doubtful whether brix balances have sufficient accuracy to be really meaningful in the mixed juice-syrup stage.

The sucrose balances measured across the mixed juice-syrup stage suggest that two phenomena are involved in undetermined losses in PG. One operates permanently in the mixed juice-syrup stage and it accounts for most of the loss when mixed juice purity is high. When the purity declines an additional loss mechanism becomes pronounced outside the mixed juice-syrup stage. Whilst this latter mechanism was operating at the end of the 1983–84 season a balance across all three sections of the factory was attained with confidence for the first time. It indicated the following losses:-

Total loss	5,94%
Loss in mixed juice-syrup stage	2,33%
Loss in syrup-refinery stage	3,29%
Loss in refinery-warehouse stage	0,32%

This balance needs confirming but it supports the suspicion that the high losses measured at the ends of seasons are occurring mainly in the rawhouse after syrup.

Laboratory Studies

Weekly composite samples of factory products were subjected to laboratory studies in an attempt to elucidate the loss mechanism which occurred when very high losses were recorded.

Methods

Gas evolution from syrups and molasses was measured by placing either 10 g of syrup or 2 g of molasses into a 12 mℓ bottle fitted with a rubber bung through which a graduated 1 mℓ pipette protruded. Air space above the sample was filled with liquid paraffin and the bottle was then immersed in a water bath at 70°C. Any gas given off from the sample collected against the rubber bung and displaced some paraffin up the pipette so that volume changes could be measured.

Sucrose stability in syrups was measured by accurately weighing about 0,25 g of syrup into a weighed 20 ml screw-capped bottle and then exposing the bottle to a selected temperature (usually 70°C for 48 hours). The results were erratic if bottles were not scrupulously clean. Unheated controls were stored at −20°C. Any evaporation or accidental dilution which occurred during heating or freezing was not critical because the entire sample was diluted and reweighed before analysis. Analyses for sucrose, glucose and fructose were done by high performance liquid chromatography using a Waters 'Sugar Pak' column with aqueous $Na_2CaEDTA$ (50 ppm) (Merck No. 8439) as solvent and a maximum sucrose concentration of 0,7% (above this the response of the refractive index detector was not linear). A twin pen recorder and dual level signal attenuator were used to obtain glucose and fructose peaks of adequate height whilst maintaining the sucrose peak on scale.

The syrups were diluted slightly before testing to adjust all their sucrose concentrations to 50–52%.

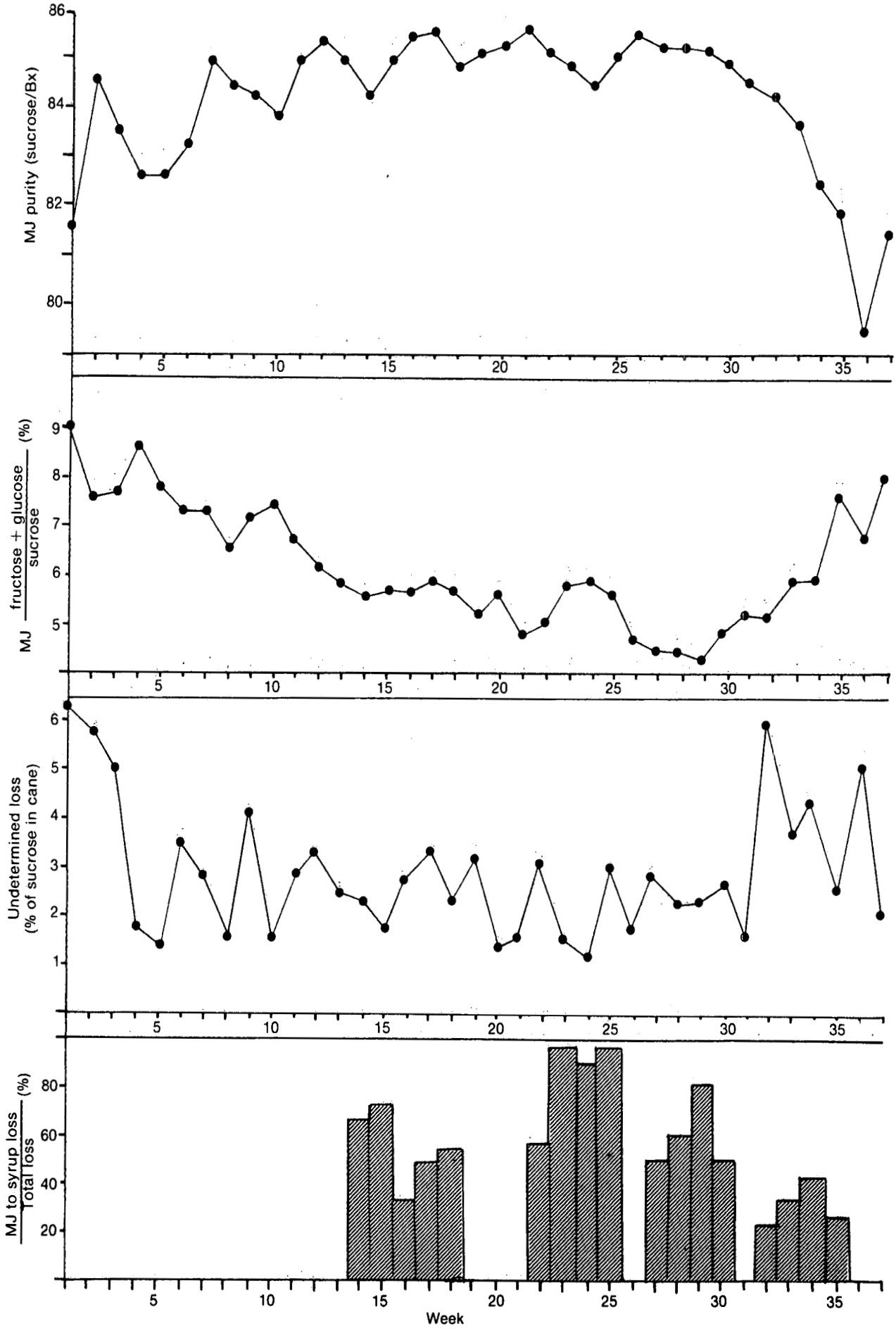

FIGURE 8 Weekly mixed juice purity, reducing sugar ratio, undetermined loss and loss in the mixed juice-to-syrup stage for the 1982–83 season

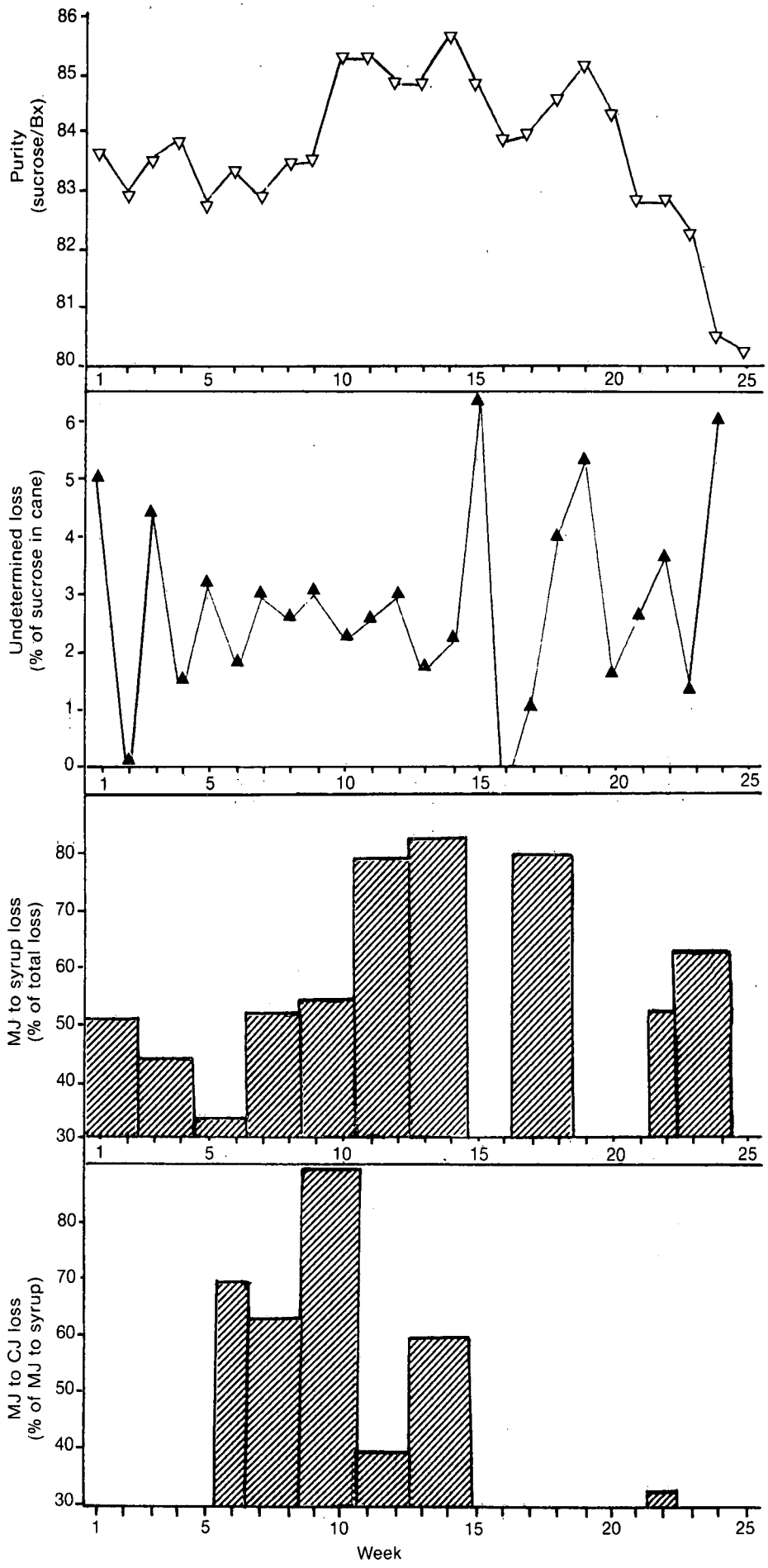

FIGURE 9 Weekly mixed juice purity, undetermined loss, mixed juice-to-syrup loss and mixed juice-to-clear juice loss for the 1983–84 season

Results and Discussion

All the molasses samples produced gas when first heated but the mid-season samples soon ceased gassing, whilst the end of season samples continued at a steady rate for at least 74 hours. Results for weeks 29–36 of the 1982–83 season and the relation of the gas production from these end of season samples to the undetermined loss measured at the factory are shown in Figure 10. No such relationship existed for the mid-season samples.

With syrups the gassing test gave a similar trend with mid-season samples producing less than 0,009 mℓ/g in 100 hours whereas syrups from weeks 28 to 36 of the 1982–83 season gave an amount of gas (up to 0,023 mℓ/g) which generally correlated well with the undetermined loss measured at the factory.

To check for sucrose degradation in the gassing molasses, four fresh samples were heated in duplicate at 70°C for 48 hours in sealed containers and the sucrose contents were then compared with unheated controls. Gas chromatography was used for the analyses and showed that 11 to 14% of the sucrose disappeared during heating. Addition of 3% glucose prior to heating had no measurable effect on sucrose degradation. Glucose and fructose formed during heating accounted for 42 to 62% of the sucrose which disappeared, the lowest figure being for the week which showed the greatest undetermined loss.

To check for seasonal changes in sucrose stability in syrups, a selection of composite weekly samples from the 1982–83 and 1983–84 seasons were heated at 70°C for 48 hours and the sucrose loss measured in the laboratory was compared with the undetermined loss (sucrose basis) measured at the factory.

The results were similar to those of the gassing tests in that for late season samples there was a significant correlation between sucrose loss measured in the laboratory and the undetermined loss reported from the factory. This correlation was not evident with mid-season samples. Results for late season samples from both seasons are shown in Figure 11.

The implication of these results is that the very high undetermined losses which are frequently observed at PG towards the ends of seasons are due to a factor which is already present at the syrup stage. The pH's of the syrups have been measured at 70°C and there is no indication that syrups showing higher losses have abnormal pH's.

For comparative purposes some late season samples from Umzimkulu were subjected to the heating test but none showed the unexpected high losses shown by some PG samples. The fact that the high losses at PG invariably occur when the purity is low suggests that some impurity is involved in the loss mechanism. The non-sucrose ratio at PG is appreciably higher than the industrial average (about 1,1 compared with 1,0) and is particularly high at the end of season, possibly because of the suspected reaction of sucrose with impurities. The correlation of gassing with undetermined loss suggests that a Maillard-type of reaction may be involved. Nitrogenous components are reactants in the Maillard reaction and so the observation that cane variety NCo 376 has a higher nitrogen:sucrose ratio than the variety which it replaced at PG in about 1974 is relevant, especially as undetermined losses increased as the new variety increased.

Thin layer chromatography was used for semi-quantitative analyses of amino acids in mixed juice, syrup and molasses from different weeks. A high loss week (36 of 1982–83) was compared with two low loss weeks and a clear difference was evident. In the high loss week there was an almost complete disappearance of amino acids at some stage between syrup and molasses whereas for the low loss weeks only minor reduction was evident. Thus amino acids are implicated in the loss reaction at PG. Their concentration in syrups from contrasting weeks was similar however so it seems that the reaction is triggered by something other than amino acid concentration.

A study of seasonal trends in amino acid concentrations revealed that, for both seasons studied, amino acids were readily detectable in molasses until the last 6 to 7 weeks of season (when the purity of mixed juice was declining and undetermined losses were generally high). This late season disappearance of amino acids was not evident in clear juice.

General Discussion and Conclusions

The undetermined loss problem at PG has not been solved but progress has been made in locating the areas of loss so that more detailed investigation can proceed. In response to the high losses measured between mixed juice and syrup, future investigations will concentrate on accurately measuring losses between mixed juice and clear juice. The clarifier is being modified to reduce the possibility of stagnant zones, and evaporator conditions are to be carefully monitored. It is perhaps significant that PG generally starts with the lowest brix mixed juice in the industry but produces the highest brix syrup.

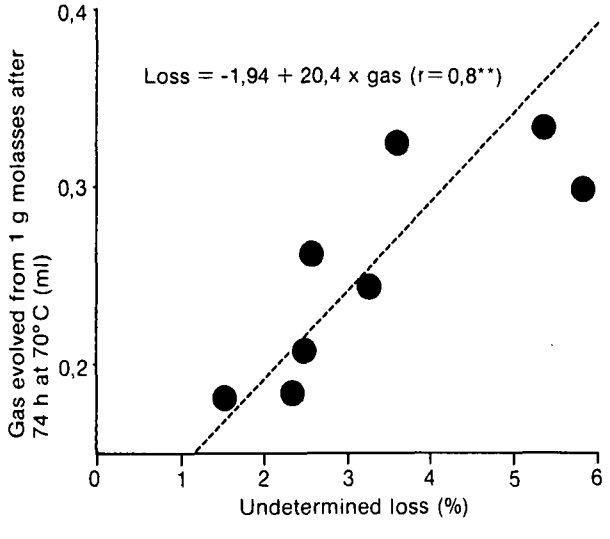

FIGURE 10 Comparison between late season undetermined losses measured at the factory and gas evolution from composite weekly molasses samples when heated in the laboratory

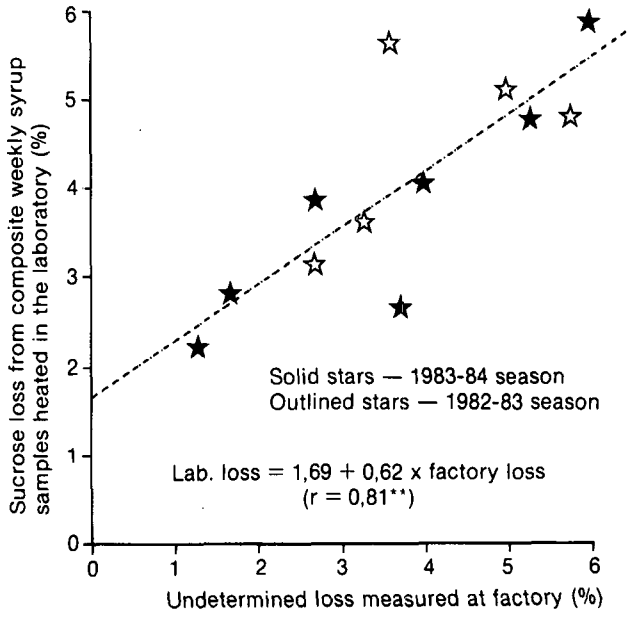

FIGURE 11 Comparison between late season undetermined losses measured at the factory and losses of sucrose from composite weekly syrup samples when heated in the laboratory at 70°C for 48 hours

The demonstration of unexpectedly high sucrose instability in some PG syrups emphasises the need for care with evaporation and for minimum recirculation of sucrose. Again it may be significant that since 1974, when the undetermined loss problem began, PG has always produced a greater volume of massecuite per ton brix than has the industry on average (annual means for 1974–82 are 1,86 and 1,71 m^3/ton respectively). Before 1970 PG produced relatively low volumes of massecuite/t Bx. The recent report (Anon[1]) that smut infestation of cane causes problems with massecuite exhaustion is interesting because smut is a problem in the PG area.

The suggestion that high losses at the end of season are due to high cooling water temperatures seems untenable because the end of season samples also gave highest losses in the laboratory where all samples were heated at identical temperatures. High cooling water temperatures would however accentuate any existing chemical degradation reaction.

Taken over a whole season the losses occurring outside the mixed juice-syrup stage are considerably less than those occurring within this stage. High priority is however attached to the elucidation of the mechanism of post-syrup loss because if this can be controlled then the overall losses at PG will be close to the industrial average. Furthermore, because the sucrose instability is detectable in syrup, is it conceivable that this mechanism operates to a limited extent in the mixed juice-syrup stage. The fact that the post-syrup loss varies so much suggests that it might be controllable. The more consistent loss occurring prior to syrup may be largely uncontrollable because of unavoidable thermal degradation.

Another reason for investigating phenomena which take place in the post-syrup stage at the end of season is that for this late season period there is a substantial increase in the amount of sucrose leaving the factory in molasses. For the last six weeks of the 1982–83 season the amount of sucrose in molasses averaged 13,5% of sucrose in mixed juice whereas for the previous 26 weeks it averaged 9,8%. Prior to the use of gas chromatography for analysis of molasses this loss in molasses was partially masked because of the low pol:sucrose ratio for PG molasses.

The combined effect of increased molasses production at the end of season together with its low pol:sucrose ratio often causes PG to show, at the end of the season, a lower undetermined loss when calculated on a sucrose basis than when calculated on a pol basis. Thus unlike most other mills PG's overall undetermined loss has been affected only slightly by the introduction of balances based on true sucrose. The past use of pol analyses has exaggerated the undetermined loss difference between PG and the industrial average. This is illustrated by the following annual undetermined loss figures:-

	1981–82		1982–83	
Loss basis	Pol	Suc	Pol	Suc
PG	3,57	3,59	2,92	3,04
Industrial average	1,55	2,04	1,62	1,91
Difference	2,02	1,55	1,30	1,13

If a conventional Maillard reaction caused the high loss at PG at the end of season then it should have been possible to detect an increase in glucose consumption at this time. The glucose loss between the syrup and molasses stages was however fairly constant throughout the season when expressed as a percentage of glucose in syrup. The mean weekly loss for the 1982–83 season was 49% (±6) whilst that for fructose was 11% (±6).

Laboratory studies will be necessary to develop a clearer comparison between the processing characteristics of cane from different weeks of the season and from different areas of the industry. A major problem in the elucidation of loss mechanisms is the need for extremely accurate and precise methods for measuring sucrose and for controlling and measuring pH at high temperatures. Progress in the development of such systems is being made at the SMRI. The collection of frozen weekly samples of factory products provides a useful base for future studies of variations in sucrose stability. Useful progress has already been made in the discovery that relatively poor sucrose stability could be detected in syrups from nearly all weeks which gave high undetermined loss.

Acknowledgements

All factory personnel at PG are thanked for their co-operation and their tolerance of disruptions caused by this project. Central Board staff provided invaluable assistance with weekly GC analyses and periodic analyses at PG. A variety of speciality analyses were kindly done by staff at SMRI.

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