# THE EFFECT OF REDUCED FREQUENCY OF SAMPLING AND ANALYSIS AT SUGAR MILL LABORATORIES

By P. MELLET and A. DUNSMORE
*Sugar Milling Research Institute*


## Abstract

A comprehensive survey was undertaken at fifteen South African sugar mill laboratories to establish if consolidation of the frequency of sampling and analysis could be achieved without impairing good factory control. By merely cutting down on what was not strictly necessary, it was concluded that the number of analyses could be reduced by 25%. Furthermore, if the day shift performs the bulk of the analyses while the other two shifts concentrate only on essential shift analyses and the compositing of samples, then an additional 12–15% reduction on the number of analyses could be attained. This would reduce the number of staff required per day to twelve or thirteen for raw house control plus an additional two people where a white-end refinery is attached to the raw house. The possible savings in labour and the use of chemicals could be about R20 000 per annum per factory.


## Introduction

During the latter half of the 1981/82 season SMRI undertook a survey of nine South African sugar mill laboratories. Aspects considered were the type and frequency of analysis performed, the labour used to perform these analyses and the cost of chemicals. The nine factories chosen to give a good cross section of the whole industry included factories with milling tandems, diffusers, tandem plus diffuser and also factories with and without back-end refineries. The results of the survey showed that there was a wide variation in the number of analyses and also the manpower utilised per laboratory, e.g. laboratory staff varied in number from 16 to 35 leading to substantial differences in costs. It thus became obvious that there is ample scope to rationalise laboratory analyses and to attempt to lay down specific recommendations in so far as sampling, number and frequency of analyses and optimum staffing per laboratory are concerned.

## Procedure

*The Tongaat-Hulett Group Laboratories*

The SMRI was represented in discussions with the Tongaat-Hulett Group of factories where sampling, analyses and frequency of analysis in mill laboratories were considered. A manual was compiled with the aim of ensuring standardisation in both sampling techniques and analytical frequency throughout the Group.[1] It was stressed that the recommendations be seen as the minimum required for good factory control, and when processes differed, the requirements would change. In general it was envisaged that a reduction of 25% in the number of analyses could be achieved (excluding analysis of traces of sugar in condensates, and the analysis of boiler and feed waters).

*The C. G. Smith Group Laboratories*

With regard to the C. G. Smith Group, SMRI was requested to visit the individual laboratories and assess the needs of each one. An analysis programme agreeing fairly closely with that drawn up for the Tongaat-Hulett mills was suggested to NB, PG and UK, while the other three C. G. Smith mills, i.e. IL, SZ and GH were keen to cut the number of analyses even further. This resulted in the concept of doing mainly day shift analyses and this was introduced at SZ where staff complements employed by the two night shifts were reduced to three each, and during these shifts the analysts performed only those analyses essential for process control. At the same time samples of other products were taken and, where applicable, were preserved and/or composited to be analysed by the day shift.

*Analyses for Raw House Control*

The suggested programme of analyses is attached to this report (Appendix 1) and a blank column is provided so that individual laboratories can enter their figures to compare their existing procedure of analyses with the number of analyses suggested. It can be seen that an additional 12–15% reduction in the number of analyses compared with that of the Tongaat-Hulett analysis programme can be attained.

*Sample Preservation and Compositing*

To enable a laboratory to operate effectively as suggested in the attached programme, certain precautions must be taken. Special attention must be paid to sampling and sub-sampling techniques so that composites are truly representative for the period covered. In this respect two factors are of prime importance:

(a) continuous sampling must be implemented where possible and done in such a way that the sample taken will be proportional to the mass of product produced. The latter is not always possible and in these cases suitable intermittent samples must be taken at preset frequencies;

(b) having achieved this, adequate steps must be taken to store the samples in such a way that there will be no change in their composition, e.g. by deterioration or contamination. It is strongly recommended that all liquid samples, i.e. from clear juice to syrup, A-, B- and C-molasses, remelt and imbibition water be sampled by means of continuous/semi-continuous means. Details of type of sampling equipment are not discussed here but will be covered in a future report. All samples having a brix of less than 65° must be adequately preserved as prescribed in the Laboratory Manual for South African Sugar Factories.[2]

It will be necessary for each factory laboratory to purchase a small deep freeze and have facilities to prepare samples for deep freezing. Since most of the analytical procedures are self-explanatory, only some analyses will be discussed in detail.

It has been shown that rapid deterioration of prepared cane samples takes place[3] and analysis of such samples must be done as soon as possible after collection. Imbibition water is not normally analysed for pol, but the presence of sugars may have a serious effect on the pol of mixed juice. It is therefore suggested that pol measurements of imbibition water be taken every shift.

Continuous recording of pH, brix and temperature, for instance, should be carried out where possible and only occasional laboratory measurements should be made to check the accuracy of these recordings, e.g. temperatures of diffuser stage juices, pH recording of limed juice, clear juice, syrup and filtrate and brix recording of syrup should be read from gauges, etc.

Analysis of massecuites must be done on catch samples as soon as possible after the sample has been collected, because settling of the mother liquor occurs on standing and complete remixing of crystal and molasses becomes extremely difficult.

Should the number of strikes become too many for individual analysis, every second or third strike should be analysed. Composites of strikes should never be prepared.[4]

The terms "mud" and "filter feed" must be well defined, i.e. "mud" is the product coming out of the clarifier and "filter feed" is that which goes on to the filter after the addition of any secondary products like lime, bagacillo, flocculant, etc.

For the purpose of this report the analysis of boiler feed and boiler waters has not been considered since treatment and analysis are specified by industrial boiler water treatment firms. In addition water, such as tail pipe water, condensates, etc. have not been considered due to the fact that each factory differs in layout and hence in the control procedures adopted.

*Refinery Control*

The following types of refining processes are in operation in South Africa:

(a) Carbonatation plus ion exchange (HR)
(b) Phosphatation plus Talofloc plus ion exchange (NB)
(c) Carbonatation followed by light sulphitation (ML, PG and GH)
(d) Phosphatation plus Talofloc (EN)
(e) Sulphitation (UF).

Analyses concerning Talofloc and ion exchange have not been covered in this investigation so that the attached programme deals only with analyses involved in carbonatation/sulphitation and phosphatation plants.

*Analyses for Refinery Control*

Only some analyses will be discussed in detail at this stage; a complete programme of refinery analyses is also attached to this report (Appendix 2).

(a) *Affinated sugar*
Since no local refineries affinate prior to melting raw sugar, no analysis needs to be performed.

(b) *Brix, conductivity ash/brix and reducing sugars*
It was found that some refineries monitor these constituents closely from raw melt through to sugar(s), cake and run off(s). The high frequency at which the products are analysed is unnecessary under normal operating conditions and these parameters should be analysed only as indicated in the schedule.

(c) *Continuous sampling and preservation of samples*
As for the raw house it is strongly recommended that continuous/semi-continuous sampling be carried out wherever possible. All samples with a brix of less than 65° must be preserved with mercuric chloride and deep freeze facilities must be available for storage of samples.

*Other South African Sugar Factories*

Apart from the six Tongaat-Hulett mills and the six C. G. Smith mills dealt with so far, ML, UC and UF were also visited and similar programmes of analyses were recommended. Only GD and EN factories were not surveyed.

*Manpower Employed and Chemicals Used*

As mentioned earlier the number of laboratory staff per mill per day at present varies from 16 to 35. A slight increase in the number of day shift analysts is necessary to handle the increased number of samples. Figure 1 illustrates a typical labour distribution in a factory laboratory, while Figure 2 gives the layout of the proposed system. It can be seen that substantial savings could be obtained by the proposed system and a labour force of about 13 per day would be required for raw house control while an additional 2 people per day should suffice when a back-end refinery is attached to the raw house.

Additional savings could be obtained by considering the distribution of the working hours of the three shifts. Better productivity would probably be obtained by having the day shift from 08h00 to 17h00 and the two night shifts from 16h00 to 00h00 and from 00h00 to 08h00 respectively. In financial terms, all this means that the cost of running a laboratory will be reduced by as much as half in certain cases.

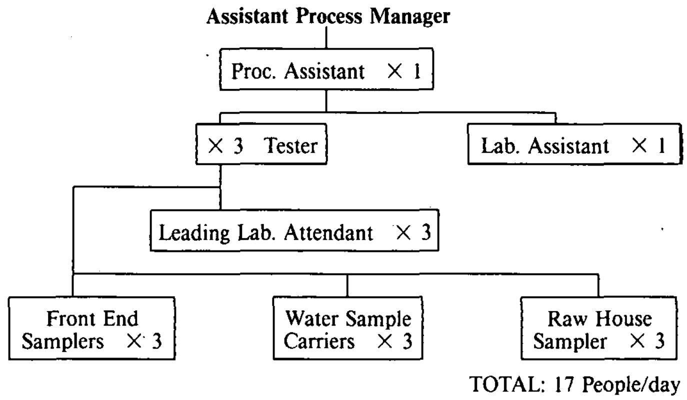


**FIGURE 1** Existing staffing in a mill laboratory

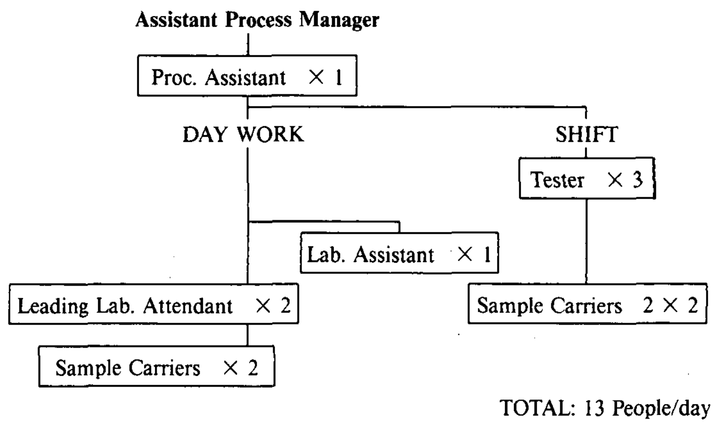


**FIGURE 2** Proposed staffing in a mill laboratory

The cost of chemicals is between R10 000 and R15 000 p.a., but because many of the analyses adjusted to lower frequencies do not involve the direct use of chemicals, i.e. temperature and pH measurements, it is not easy to assess what cost reduction could be obtained. Nevertheless it is confidently predicted that some savings in costs will materialise.

## Conclusions

Substantial savings can be achieved if a factory laboratory implements the suggested analysis programme. SZ and IL have already started this procedure and so far no major problems have been encountered. Every mill will of course have its own situation to consider and the final sampling, analyses and frequency of analysis will vary from mill to mill.

The initial capital outlay in providing automatic recorders for pH, temperature, brix, etc., and sampling equipment, will probably amount to several thousand Rand, but this should be recovered in a relatively short period due to savings of about R20 000 p.a. once the proposed analysis procedure is adopted.

### REFERENCES

1 Archibald, R. D. (Sept. 1982). Process sampling and analytical frequencies. Hulett Sugar Limited.
2 (Anon). (1977). Laboratory Manual for South African Sugar Factories. S.A. Sugar Technologists Association.
3 Koenig, M. F. S. (Aug. 1982). Possible compositing of shredded cane samples for preparation index determination. SMRI Technical Report No. 1319.
4 Mellet, P. (Aug. 1979). Sampling and analysis of factory products, with special reference to A-massecuite. SMRI Technical Report No. 1190.

## APPENDIX I
## ANALYSIS PROPOSALS FOR RAW HOUSE CONTROL

| Sample | Method and point of sampling | Composites | Parameters | Frequency of analyses | Proposed No. analyses per day | Present No. analyses per day |
|---|---|---|---|---|---|---|
| 1. Prepared cane (mill or diff.) | From SICB hatch | No | Preparation Index | 2 per day (see Note 1) | 4 | |
| 2. Diffuser stage juices | Catch from stage pump | No | pH<br>Brix | 8 hourly<br>8 hourly | 3 x<br>3 x<br>(No. of cells) | |
| 3. Individual mill bagasse | Catch | No | Brix<br>Moisture | Hourly, each mill in turn | 24<br>24 | |
| 4. Imbibition water | Cont. | Yes | Pol | 8 hourly | 3 | |
| 5. Mixed juice (mill or diff.) | SICB cont. | Yes | Reducing sugars | Daily (see note 2) | 1 | |
| 6. Limed juice | Catch from pH controller | No | pH | 2 hourly (see note 3) | 12 | |
| 7. Muds | Catch from clarifier mud outlet | No | pH<br>Pol<br>Brix | 8 hourly<br>Daily<br>Daily | 3<br>1<br>1 | |
| 8. Filter feed (on to filter) | Catch from outlet of mixer | No | Mud solids<br>pH | 2 × weekly<br>As required | -<br>- | |
| 9. Filter cake | Catch from<br>(a) belt<br>(b) filter | No | Pol<br>Pol<br>Moisture | Hourly<br>Hourly<br>Daily | 24<br>24<br>1 | |
| 10. Filtrate | Cont. from return to mixed juice | Yes | pH<br>Bx, Pol<br>Mud solids | 8 hourly<br>8 hourly<br>Daily (see note 2) | 3<br>6<br>1 | |
| 11. Bagacillo | Catch | No | Particle size | As required | - | |
| 12. Clear juice | Cont. | Yes | pH<br>Bx, Pol, RS | 4 hourly (see note 3)<br>Daily (see note 2) | 6<br>3 | |
| 13. Syrup | Cont. | Yes | Bx<br>pH<br>Bx, Pol<br>RS, Starch | 4 hourly (Note 3)<br>4 hourly (Note 3)<br>Daily<br>Daily (see note 2) | 6<br>6<br>2<br>2 | |
| 14. Remelt | Cont. | Yes | Bx<br>Pol | 4 hourly<br>4 hourly (see note 2) | 6<br>6 | |
| 15. A-mass. at strike | Catch from pan or gutter | No | Bx, Pol<br>Nutsch Bx, Pol | Every 2nd strike<br>Daily | 16<br>2 | |
| 16. A-mass. before curing | Catch from Crystalliser | No | Nutsch<br>Bx, Pol | Daily | 2 | |
| 17. A-molasses | Cont. | Yes | Bx<br>Pol | 8 hourly<br>8 hourly | 3<br>3 | |
| 18. A-sugar | Cont. | Yes | Pol, Moist. % Fines, Col. SGS, Starch (see note 4) | 4 hourly<br>Daily<br>Weekly | 12<br>2 | |
| 19. B-magma | Catch from mixer | Yes | Bx, Pol | 4 hourly | 12 | |
| 20. B-seed | Catch from pan | No | Bx, Pol | As required | - | |
| 21. B-mass at strike | Catch from pan or gutter | No | Bx, Pol<br>Nutsch Bx, Pol | Every 3rd strike<br>Daily | 6<br>2 | |
| **NB**<br>On introduction of cont. pans | Sample from pan outlet | No | Bx, Pol<br>Nutsch Bx, Pol | 8 hourly<br>8 hourly | 6<br>6 | |
| 22. B-mass. before curing | Catch from crystalliser outlet | No | Nutsch Bx, Pol | Daily | 2 | |
| 23. B-molasses | Cont. | Yes | Bx, Pol | 8 hourly | 6 | |
| 24. C-magma | Catch from mixer | No | Bx, Pol | 2 hourly | 24 | |
| 25. C-seed | Catch from pan | No | Bx, Pol | As required | - | |
| 26. C-mass. at strike | Catch from pan or gutter | No | Bx, Pol<br>Nutsch Bx, Pol | Every strike<br>Every strike | 8<br>8 | |
| 27. C-mass. after cooling | Catch from crystalliser | No | Nutsch Bx, Pol | 12 hourly | 4 | |
| 28. C-mass. after re-heating | Catch from reheater outlet | No | Nutsch Bx, Pol | 4 hourly | 12 | |
| **NB** On introduction of continuous procedure follow plan for B-massecuite | | | | | | |
| 29. C-molasses | Cont. | Yes | Bx, Pol | 8 hourly | 6 | |
| 30. Injection water condensates, factory drains, etc.<br>Boiler waters | | | pH<br>Sugar traces<br>Full analysis as recommended by water treatment company | Hourly or as present<br>Hourly or as present | | |

NOTES

1. Preparation index to be done twice daily - 1st sample to be done at the beginning of the day shift, the second at the end of the day shift.
2. Composites must be kept in the deep freeze.
3. The four hourly analysis must be done to check the readings of the continuous recorder.
4. The % fines will be done by the "quick" method; the SGS must be done in full by the official method.

## APPENDIX 2
## ANALYSIS PROPOSALS FOR REFINERY CONTROL

| Sample | How sampled | Composited or not | Analyses required | Frequency | Proposed No. analyses per day | Present No. analyses per day |
|---|---|---|---|---|---|---|
| 1. Affinated sugar | – | – | Pol<br>CA | – | – | |
| 2. Raw melt | Cont. | Yes | Brix*<br>pH**<br>Colour<br>CA/Bx<br>RS | 4 hourly<br>4 hourly<br>1 daily<br>1 daily<br>1 daily | 6<br>6<br>1<br>2<br>1 | |
| 3. Limed melt | Catch | No | pH | 2 hourly | 12 | |
| 4. Carb. liq. | Catch | Yes | pH<br>Colour<br>CA/Bx | 2 hourly<br>8 hourly<br>Daily | 12<br>3<br>2 | |
| 5. Fine liq. (after sulphitation press) | Cont. | Yes | Bx<br>CA/Bx<br>Colour<br>pH | 4 hourly<br>Daily<br>8 hourly<br>2 hourly | 6<br>1<br>3<br>12 | |
| 6. Thick liq. | Cont. | Yes | Bx*<br>Colour<br>CA/Bx | 4 hourly<br>8 hourly<br>Daily | 6<br>3<br>1 | |
| 7. 1st mass. | Catch at centrifugal | No | CA/Bx | 4 hourly | 12 | |
| 8. 2nd mass. | Catch at centrifugal | No | CA/Bx | 4 hourly | 12 | |
| 9. 3rd mass. | Catch at centrifugal | No | CA/Bx | 8 hourly | 6 | |
| 10. 4th mass. | Catch | No | CA/Bx | Each strike | 4 | |
| 11. 1st run-off | Cont. | Yes | CA/Bx | 8 hourly | 6 | |
| 12. 2nd run-off | Cont. | Yes | CA/Bx | 8 hourly | 6 | |
| 13. 3rd run-off | Cont. | Yes | CA/Bx | 8 hourly | 6 | |
| 14. 4th run-off (return syrup) | Cont. | Yes | Pol<br>Bx<br>CA/Bx | 8 hourly<br>8 hourly<br>8 hourly | 3<br>3<br>6 | |
| 15. 1st sugar | Catch from centr. discharge (8 hourly) | Yes | Colour<br>CA | Daily | 1<br>1 | |
| 16. 2nd sugar | Catch | Yes | Colour<br>CA | Daily<br>Daily | 1<br>1 | |
| 17. 3rd sugar | Catch on curing | Yes | Colour<br>CA | Daily<br>Daily | 1<br>1 | |
| 18. 4th sugar | Catch | Yes | Colour<br>CA | Daily<br>Daily | 1<br>1 | |
| 19. Refined sugar | Cont. | Yes | Colour<br>Moisture | Daily<br>Per lot | 1<br>3 | |
| 20. Sweet water | Cont. 1 hourly | Yes | Bx<br>pH<br>Pol | 8 hourly<br>8 hourly<br>8 hourly | 3<br>3<br>3 | |
| 21. Filter cake | Catch | Yes | Pol<br>Moisture | 8 hourly<br>Daily | 3<br>1 | |
| 22. Cyclone waters | Catch | Yes | Bx | 2 hourly | 12 | |
| 23. Press water | Cont. | Yes | CA/Bx<br>Pol<br>Pol | Daily<br>Daily<br>Per cycle | 2<br>1<br>14 | |

* Continuous brix monitoring; check samples in laboratory every 4 h.
** Continuous pH monitoring; check samples in laboratory every 4 h.