On-Line COD Measurement at Lactose NZ

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1. Introduction

The use of on-line COD measurement at Lactose New Zealand (LNZ) is an integral part of the LNZ loss monitoring system. This paper deals with the loss monitoring in our two liquid effluent streams. Loss calculations are based on the COD values that are then converted into a lactose percentage. These values are corrected for density and multiplied by the flow rate to give the lactose loss rate.

Loss monitoring has been carried out at LNZ to:

- Obtain financial data on losses within LNZ's operation.
- Aid in the prevention of losses.
- Provide data for justification of process improvement projects.
- Validate process parameters such as drain times, flush times, etc. - especially after plant upgrades.
- Identify problems and to assist in reducing them.
- Support the control and improvement of environmental issues.
- Determine the impact of the process on the environment and provide data for the design of processes to reduce the impact.

The two primary goals for the loss monitoring system at LNZ were:

I. Detect losses
II. Quantify losses

The previous system was labour intensive and unreliable. It used automatic samplers set up on various waste streams. The samples were then analysed in the laboratory for lactose content. A more responsive and cost effective system was required.

LNZ's specific systems requirements were identified as follows:

- The method must be capable of directly or indirectly measuring lactose.
- The system must not be adversely affected by the presence of diatomaceous earth, carbon, proteins, CIP chemicals or colour compounds in the sample stream.
- There must be rapid feedback from the system to allow operators to take action.
- The method must be reported continuously to allow any incidents or problems to be investigated.
- The system must have low maintenance and have high reliability. The factory runs 24 hours a day and so must the monitoring system.
- There must be minimal operator supervision of the system.

2. Technologies

Numerous methods were investigated to determine how to best monitor the lactose losses in the two effluent streams. These were

Updating the status quo

A carousel sampling system with laboratory analysis of the sample's lactose content.

Infra Red

Concentration based on the absorption of light by the dissolved particles or reflection of light by the suspended particles in the sample.

On-Line COD

Determines the chemical oxidation demand of the sample. There is then a correlation between COD and Lactose percentage.

On-Line BOD

Determines the Biological oxidation demand of the sample. There is then a correlation between BOD and Lactose percentage.

On-Line TOC

Determines the Total Organic Carbon content in the sample. There is then a correlation between TOC and Lactose percentage.

3. Instruments Considered

Various instruments were investigated and the purchasing decision was based upon the following rationale.

NB.

The reasoning listed is based on LNZ's research (and understanding) of the suitability of each technology or instrument to LNZ particular processes and conditions. It does not imply in any way that any instrument is poorly made or designed and does not imply that any technology or instrument is inferior or superior to any other.

LAR COD Analyser

This instrument uses OH- radicals to oxidise the sample. The radicals are produced by electrodes. A sample is drawn into the measuring chamber and the COD is determined. The sample is then expelled and the chamber is cleaned ready for the next sample. The response time is very quick (quoted at 2 minutes). The measurement of the COD is a batch process. There were no LAR COD units in operation in New Zealand. The instrument offered appeared to be less robust when compared to the STIP COD analyser.

STIP COD Analyser

This instrument uses ozone to oxidise the sample. The ozone is generated by the instrument from bottled oxygen. A sample is drawn into the reaction chamber on a continuous basis and mixed. There is a three minute lag time due to the volume in the reaction chamber. An operational unit in a Hobart water treatment facility gave positive references. The instruments appeared to be quite robust and suited for industrial uses.

Wedgewood IR

An in line measuring instrument that could be set up to measure dissolved solids or suspended solids - not both at once. The instrument was not recommended by the suppliers after clarification of the processing environment - it would have been badly affected by the other stream components.

LECO Near IR

A laboratory style instrument that was not set up to be used in an industrial setting and under industrial conditions.

On-Line BOD

After investigation the instrument was not recommended by the supplier as the biomass could be affected by pH swings.

NOTE

This note added by Rob Dexter of DCM 13-5-99-

The Stip BOD analyser and the Stip COD analyser can be fitted with a pH control loop added to prevent these pH swings in the actual sample taken into the analyser reaction chamber. Lactose installed one after initial start-up problems. Either analyser would work well for Lactose..

On-Line TOC

Suitable for lower levels of organic carbon but not capable of reading the concentration ranges required by LNZ. → check with S. Peter

Status Quo - Carousal Sampler

This system was very labour intensive and produced historical results (potentially up to 30 hours after an event). The sampling and testing lead to unreliable results and did not produce the accuracy required.

4. Decision

Considering the above information it was decided to purchase a STIP Phoenix - 1010 on-line COD analyser.

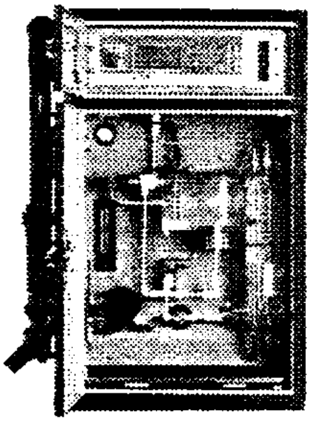

Figure 1 - STIP Phoenix 1010 On line Cod Analyser (image courtesy of Applied Instruments)

5. Operation

The instrument basic operation is outlined in Figures 2 & 3

<u>Sample Preparation</u>

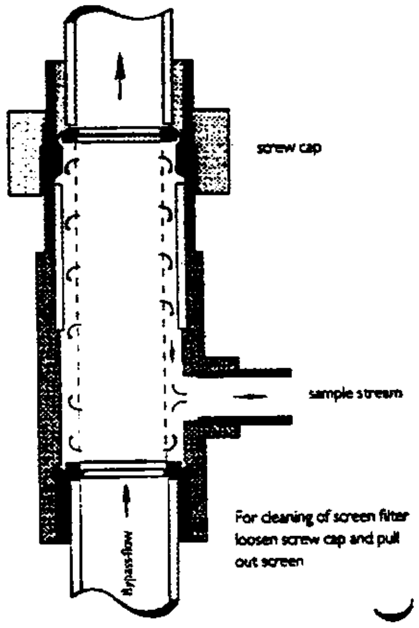

Figure 2 - STIP PA2 Sample Preparation Unit (image courtesy of Applied Instruments)

The effluent sample is drawn into the unit through a self cleaning filter unit. The filter mesh is 0.5mm and the filter is back flushed automatically (frequency of flush is operator adjustable). Pre-filtration is not required providing the effluent stream does not contain particles above 2 cm.

Method of Operation

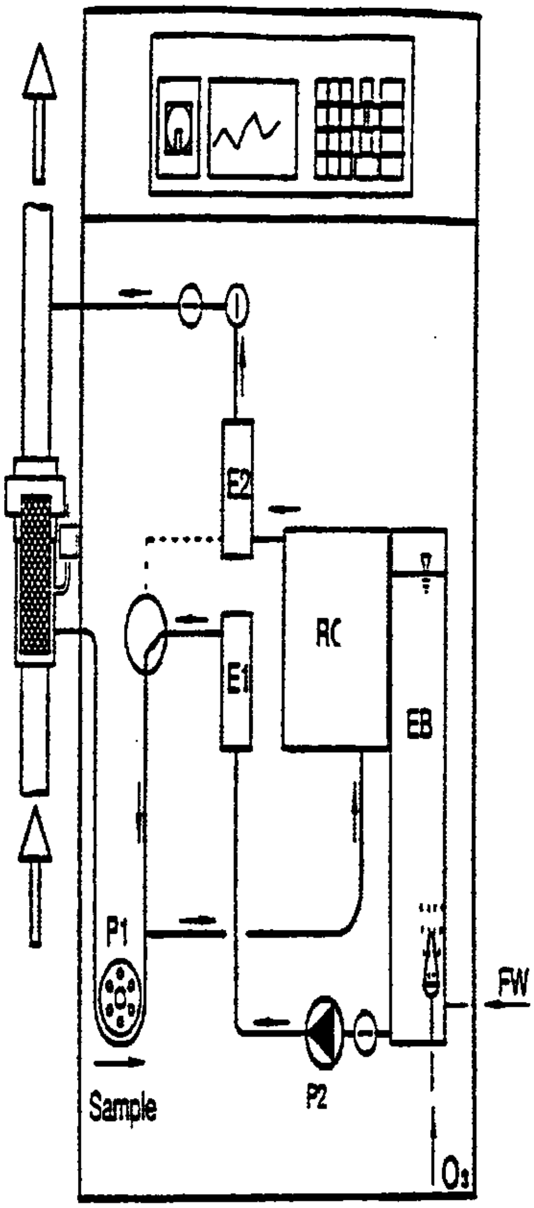

Figure 3 - STIP Phoenix 1010 (image courtesy of Applied Instruments)

a) Ozone is mixed with dilution water (FW) in the enrichment block (EB).
b) The resulting ozone concentration of the solution is measured by the first ozone probe (E1).
c) The waste water and the ozone enriched dilution water are mixed and transferred to the reaction chamber (RC). In the reaction chamber (RC) the waste water is oxidised by the ozone enriched dilution water.
d) The remaining ozone concentration is measured at the second ozone probe (E2).
e) The computer controls the ratios of mixing waste water (via P1) and dilution water (via P2) to ensure ozone consumption is low and constant. The resulting ozone surplus is constant and high.
f) From the ratios of P1 and P2 the COD is calculated.

6. Installation

The unit is located in a small shed just before the main effluent tank. Effluent is piped from the two effluent streams into the shed. A three way valve controls which stream is sampled at any given time. A small sample is taken from the effluent stream for COD analysis. The surplus is piped to a balance tank. The contents of the balance tank are returned to the flume from where the sample originated (controlled by a three way valve). There is an ultra sonic flow transmitter on each of the two effluent stream flumes. These transmitters given instantaneous flow readings. The valves and pumps are controlled by a PLC program. The COD readings are then manipulated, together with flow readings to produce a lactose loss figure. The loss figures are reported as key performance indicators for each shift. The operator interface shows the status of the valves, pump and a COD reading, instantaneous losses, flow rates, previous hour's flow and loss and the previous day's figures.

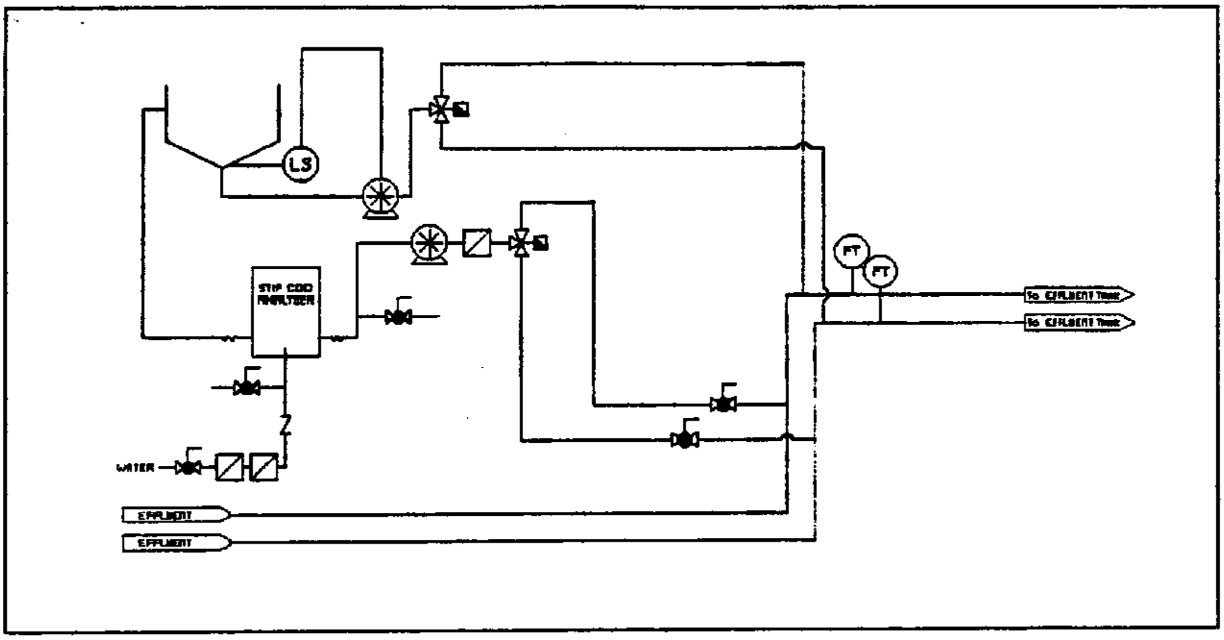

Figure 4 - Simplified Piping and Instrument Diagram of system set up

Several problems were experienced with the installation of the unit - these delayed the integration of the instrument into the loss monitoring system. These were mainly due to the instrument being the only unit in the country and everyone was operating with limited experience. The problems included instrument operation, commissioning and installation of the ancillary equipment. All of these problems have now been successfully resolved.

The unit is calibrated following a set procedure against laboratory COD values. An effluent sample is run through the analyser and then tested (for COD) in a laboratory. The sample is then diluted, run through the analyser again and also tested in laboratory. An internal calibration setting is then altered to adjust the instrument COD readings to match the laboratory COD results. It is recommended that the instrument is calibrated 6 monthly.

7. Limitations

We did experience response time problems early on in the project but these have now been resolved with the alteration of some of the parameters installed during commissioning. Response time problems could have arisen as measurements were required from two different effluent streams. It was known however, that the two streams generally had similar COD readings and only in an "event" circumstance would the two streams would differ radically in COD. As the process is continuous it takes time for the instrument to adjust to a step change in the COD concentration. Pumps must speed up or slow down while maintaining the difference in ozone concentration at the two probes.

The instrument is affected by other components in LNZ effluent streams. Anything that has a COD will be measured, therefore, extensive data gathering was required to correlate the COD value to the lactose percentage over various ranges and levels of contaminants. From this data it was possible to reduce the impact of the additional components on the loss rate. Trials were carried out to measure the affect of CIP solutions on the instrument COD readings. It was found that strong nitric acid solutions lowered the reported COD of a solution. A strong caustic solution initially elevated the reported COD reading but then settled to a more expected reading. The impact of the additional components in the effluent streams has been assessed and is now considered to be within acceptable limits.

8. Performance Data

Information from the loss monitoring system is available in various flexible formats. The data is used for loss investigations and as performance indicators for regular review by the production staff. On a monthly basis, the data is incorporated into the Production Management Reports where it is used as part of the overall review of the company's performance.

Typical outputs from the system are shown in Figures 5 and 6. To identify the cause of the various peaks, investigations are carried out by checking process log books, valve positions and process records. In these examples the large peaks where identified as:

Peak Number	Cause
1	Water boil of an evaporator and CIP of a Crystalliser
2	CIP of a Crystalliser
3	Process tank emptied and cleaned
4	Evaporator water and acid CIP.
5	Emptying of a Crystalliser, screen bowl centrifuges and pre-drier for CIP
6	Evaporator full clean, evaporator onto product
7	Evaporator water boil
8	Product transfer from evaporator

Figures 5 and 6 show an excellent correlation between the COD readings and losses for those events. This type of investigation can lead to a reduction in losses by optimising process parameters. As a result of the information provided by the COD analyser, several drain times have been altered to reduce the amount of product lost. In addition, errors in valve programming have been identified and remedied from the information provided by the COD analyser. The implementation of the new loss monitoring system has helped reduce losses at LNZ. Further work is being done to quantify the savings and pay back on the system.

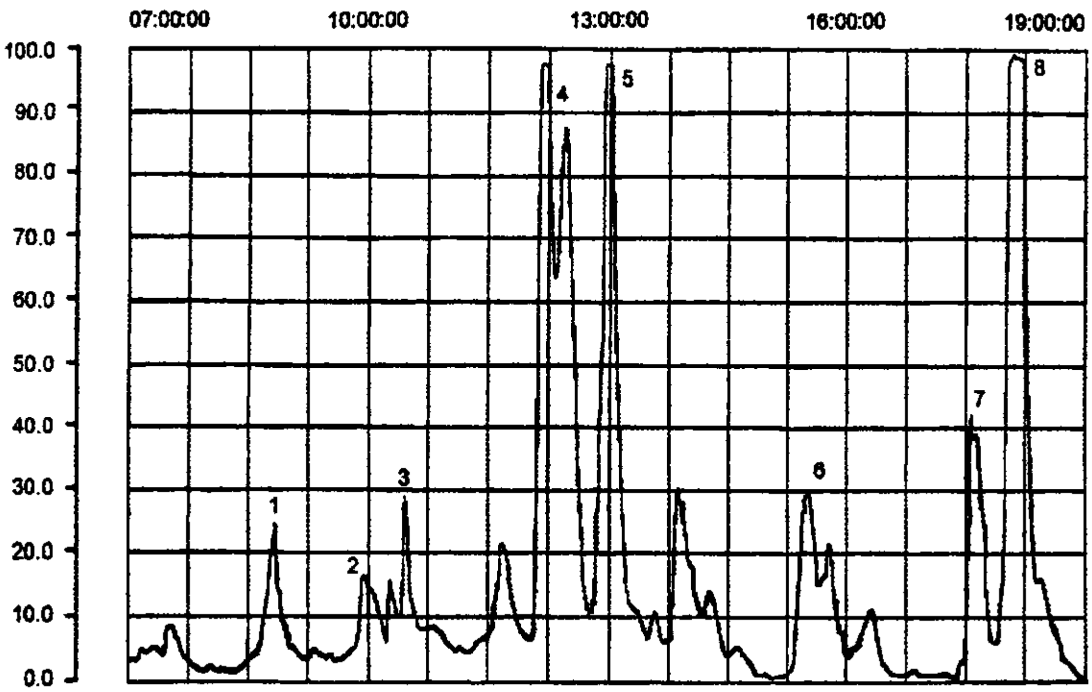

Figure 5 - COD Readings over a 12 hour period

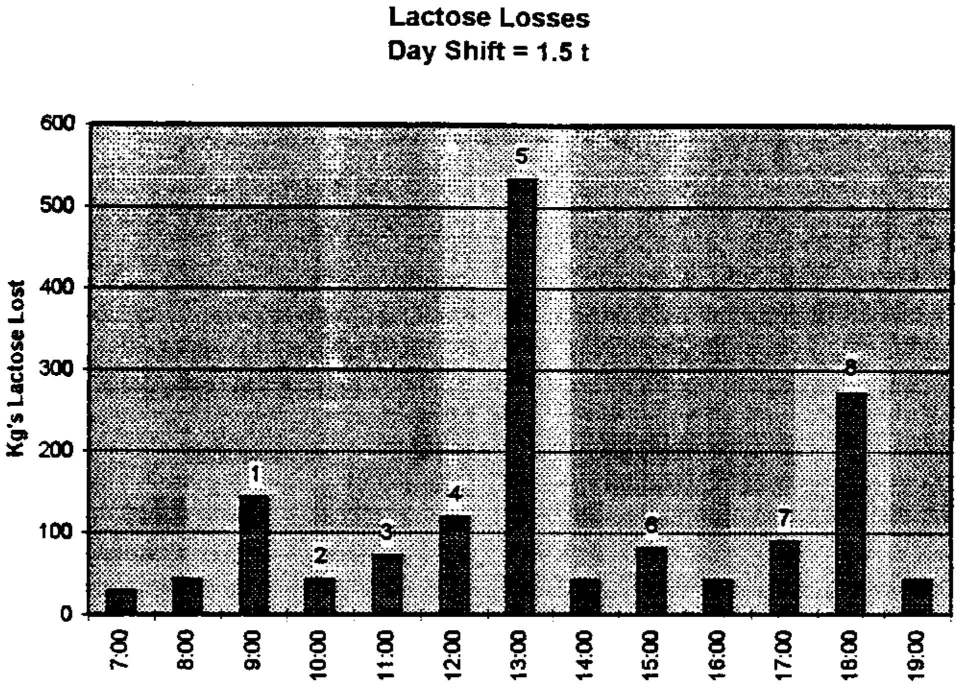

Figure 6 - Lactose Losses for a 12 hour period

9. Review

Primary Goals

I. *The system is highly capable of quickly detecting lactose losses*

II. *The degree of accuracy of the quantifying of the losses has yet to be fully verified and will be validated during the up coming season. However, there are no indications that the quantified losses will not be reasonably accurate.*

The systems requirements for the monitoring system were met as follows:

- *The STIP COD Analyser can measure lactose content by measuring the COD of a sample.*
- *The STIP COD Analyser is affected by other components of the effluent stream but within acceptable limits.*
- *Feed back is only lagged by three minutes plus the transport time of the effluent from the source to the main tank. This is well within acceptable limits.*
- *Continuous logging and trending of the readings has made it possible to investigate any abnormal incidents.*
- *Maintenance is very easy to carryout with a step through menu instruction. Maintenance of the instrument takes less than half an hour a week with an additional half an hour every second week. The instrument reliability is adequate. There are several critical alarms that will shut the instrument down (and therefore the loss monitoring system). On average there will be a critical alarm once per week. The cause of the malfunctions is being worked on so that their incidence can be reduced. The instrument does have the capability of being connected via a RS232 port, this can be used to notify the user that there has been a critical alarm. LNZ uses a 4-20mA signal to capture the information from the instrument. This information only pertains to the COD reading and so there is no notification that a critical alarm has been triggered, at this time.*
- *During normal operation the supervision of the instrument is limited to the maintenance required. However, while the problem of critical alarm shutdown continues, an additional 5 - 10 minutes/day is required to ensure the instrument is operational.*

10. Conclusion

The decision to purchase this instrument has met all of the critical parameters identified. Although some operational issues still require attention it is providing reliable data on lactose losses from the process.