

INDUSTRIAL TRIAL OF MINTEK'S XANTHOPROBE AT THE ELAND PLATINUM CONCENTRATOR

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Abstract

The Xanthoprobe is an automated online instrument developed by Mintek for the measurement of residual xanthate concentration directly from flotation concentrate cells. Xanthate is a commonly used collector in flotation processes where its residual concentration has an impact on the recovery of metal sulphides. Xanthoprobe has been installed at the Eland Platinum PGM concentrator since March 2010 to establish the long-term robustness and measurement accuracy of the instrument in an industrial environment.

Several measurement techniques were initially investigated during the development of the Xanthoprobe, namely, direct electrode measurement, chemical titration and UV spectrophotometry. Electrode measurement proved to be too slow and was heavily affected by membrane blockages. The titration method is chemically intensive and would not be ideal for an automated instrument. Ultraviolet (UV) spectrophotometry showed the best results and continues to be the most promising of the techniques investigated.

The measurement sequence is entirely automated and is based on Mintek's Cynoprobe online cyanide analyser. The filtration system consists of a primary filter, followed by a secondary filter for sample clarification. Once filtered, the clear sample is measured by a UV spectrophotometer at a wavelength of 301 nm.

Validation tests show that Xanthoprobe measurements of industrial samples correlate well with manually measured samples. To better understand the dynamics of xanthate, samples were taken from cells along the primary and secondary rougher circuits. There were differences in the free xanthate concentrations throughout the primary and secondary circuits. The lower and higher concentrations respectively were attributed to fast and slow floating sulphides. Results from the long term testing show that measurements from the Xanthoprobe correlate with changes in sodium isobutyl xanthate (SIBX) dosage made by the plant.

The industrial test campaign has demonstrated that the instrument can measure free xanthate concentrations in an industrial plant and has great potential to broaden the horizon of flotation control and optimisation. However, further development is continuing, to optimise certain aspects of the instrument to improve operation.

Keywords: automated online measuring instrument, residual xanthate, flotation pulps, PGM, spectrophotometry

1 Introduction

In mineral concentration by flotation, reagents are added in order to manipulate the pulp chemistry and enhance the differences in mineral surface hydrophobicity which facilitates separation. Flotation is complex with many interactive sub-processes occurring in the pulp and froth phases and determining accurate reagent dosage rates is a challenge. Metallurgists need to holistically determine reagent dosing rates based on the mineralogy of feed tonnage of the ore being processed, as well as on the grades and recoveries required. To aid in reagent monitoring on industrial plants, continuous offline reagent analyses can be conducted through extensive laboratory tests. Xanthate can be measured in the laboratory through the use of UV spectrophotometry, with examples of this dating back to work done by Pomianowski & Leja (1963). Measurement methodology was further improved by Jones & Woodcock (1973 and 1979). However, laboratory analyses of manual slurry samples has not found widespread acceptance given the amount of time they take to perform, and the difficulties of performing such work in an industrial environment.

Online measurement instruments have the ability to provide plant metallurgists with valuable feedback on the current state of essential processes, such as reagent dosing, for use in control strategies on a metallurgical concentration plant. Continuous monitoring of reagents, xanthate in particular, for control has been studied as early as 1956 by Bushell and Malnarich. However, the challenges in taking the relatively simple laboratory technique for measuring xanthate, and using it in an industrial application, have limited the development of an online instrument.

The most significant progress in online xanthate measurement was the xanthate monitor developed by Hao *et al* (2008). The xanthate monitoring system effectively measured xanthate concentrations under laboratory conditions when floating sulphide nickel ore containing pentlandite. The xanthate monitor was made up of an online filter, a peristaltic pump, a UV spectrophotometer and a computer to record and show the spectrophotometer outputs. Apart from this, there has been no further literature on the performance of a similar device in an industrial setting.

Based on the fact that xanthate concentration is a key factor in mineral flotation and that there are no commercialised real-time xanthate measurement devices available on the market, Mintek developed the Xanthoprobe. This device is able to provide online industrial concentration readings of free xanthate ions using UV spectrophotometry. The Xanthoprobe is ultimately intended to be used in a reagent control strategy through online measurement which is then linked to reagent dosing.

In this paper, investigations into the Xanthoprobe's ability to accurately and robustly measure online industrial samples were carried out through industrial testing. The investigations were also intended to establish the benefits that could be obtained through a regular measurement of free xanthate concentration.

1.1 Previous Findings

Based on previous studies, Mintek (Lalla *et al.*, 2009) carried out extensive laboratory test work to determine the viability of developing an online xanthate measuring instrument. The fundamental issues identified included the measurement limitations imposed by Beer's Law, interferences by other chemical substances found in flotation slurries and the filtration methods necessary to produce a clear solution. The following key conclusions were drawn:

- The method proposed to measure xanthate ions in solution is UV Spectrophotometry. The concentration of the species under investigation can then be determined by Beer's Law (Pomianowski & Leja, 1963; Jones & Woodcock 1973; Jones & Woodcock, 1979). Due to Beer's Law limitations, the free xanthate ion can only be accurately measured at low concentrations using spectrophotometry. Mintek has employed an efficient method of overcoming this limitation.
- Industrial xanthate concentration measurement is difficult due to the complex chemical environment of flotation circuits. Jones & Woodcock (1973 & 1979) performed extensive studies on interfering chemical species at the xanthate measurement wavelength. The Xanthoprobe incorporates methods to account for these interferences.
- Two-stage filtration is essential to produce a clear solution which is required for spectrophotometry.
- The spectrophotometer's ability to accurately measure industrial xanthate solutions was validated in the laboratory. The test procedure involved the addition of incremental, known amounts of xanthate to an industrial sample. The spectrophotometer was able to successfully detect the changes.

1.2 Current Status

The Xanthoprobe is a fully automated modular instrument consisting of a multistage filtration system, sampling mechanics, a graphical user interface and controller electronics. The filtration system consists of a primary filter (stainless steel filter probe) which is immersed into the flotation pulp stream, a pump to draw samples and a secondary filter for sample clarification. The prototype instrument was automated using Mintek's StarCS control platform on a PC while future industrial versions will be automated using a PLC.

By means of a UV spectrophotometer, the Xanthoprobe measures free xanthate concentrations in a clear sample at a wavelength of 301 nm approximately every 35-40 minutes. Considerable research and development effort has been employed to include methods in the instrument which prevent cross-contamination of samples.

After successful laboratory testing, the Xanthoprobe was installed on the Xstrata Eland Platinum concentrator in order to run validation, robustness and benefits analyses. The instrument is being tested in the platinum industry and once successful further development can be done to move on to other industries such as base metals concentration. In addition, it might be possible to measure other reagent chemicals creating further opportunities for the Xanthoprobe.

1.3 Value to End User

A simple feasibility analysis demonstrates the benefits which could be provided by the Xanthoprobe. Two situations are considered, a low-benefit scenario and a high benefit scenario. The low-benefit scenario represents a small plant with a low ore feed rate, in the order of 15,000 ton per day and a low xanthate dosage rate of 100 g/t. The high-benefit scenario considers a high feed-rate plant processing 144,000 tons per day and using a xanthate dosage rate of 300 g/t.

It is assumed that the Xanthoprobe provides a total saving of 10% xanthate per ton of feed ore. Using a xanthate price of R10,000 per ton (\$1100-\$1800, alibaba.com), under the low-benefit scenario the annual savings would be in the order of R 550,000. The large-scale plant could expect savings of over R 15,8 million per year.

Using a similar calculation, a medium scale plant (72,000 tons per day, dosing at 200g/t) would save in the order of R 5,3 million per year.

These scenarios do not consider the added benefits of a stable flotation chemical environment, and the resultant improved metallurgical performance.

2 Experimental

The Xanthoprobe was installed at the discharge end of the secondary rougher circuit. It was protected by a custom-built roof structure. The primary filter was immersed in the tailings overflow tank.

Various tests were performed in order to determine whether the instrument is able to measure industrial samples, to better understand xanthate in flotation, and the instrument's effectiveness at long term xanthate concentration measurements. All manual samples were measured with a UV spectrophotometer, after being individually clarified using a 0.45 µm laboratory syringe filter.

2.1 Validation Testing Procedure

Before any long term or benefit investigations could be performed, a simple test was carried out to determine whether the onsite readings were valid. The automated Xanthoprobe concentration measurements were compared to manually measured spectrophotometer concentrations. Each test consisted of taking 5 triplicate manual samples from the tailings overflow tank every time the Xanthoprobe drew a sample (approximately every hour). Samples were taken in 500 ml plastic bottles and kept on ice to prevent xanthate decomposition, for later laboratory analysis. This test was performed on 2010/04/08 and 2010/08/03.

2.2 Circuit Sampling Procedure

To better understand the behaviour of xanthate in the flotation circuits, samples were taken from cells along the primary and secondary rougher circuits. A number of measurements were carried out along Eland Platinum's primary and secondary rougher circuits. Triplicate samples were taken from various cells marked by the red dots in Figure 1. Samples were kept on ice in 500 ml plastic bottles, for later measurement.

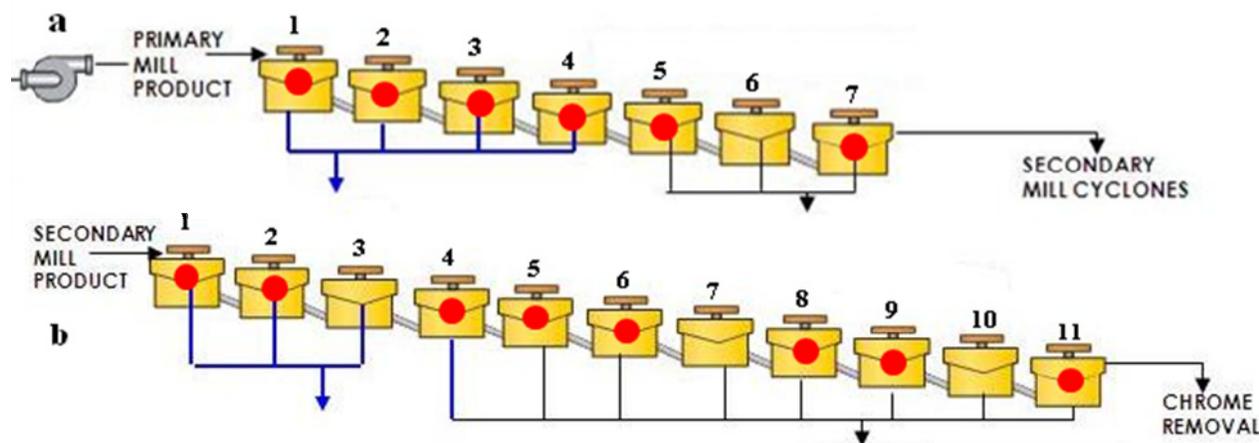


Figure 1. The primary (a) and secondary (b) rougher circuits.

2.3 Improvements and Testing on an Industrial Site

The purpose of this phase was two-fold. A considerable portion of this phase was intended to improve the instrument under industrial conditions. Considerable effort was spent on pinpointing hardware and software issues and resolving those issues. During this period the sampling and filtering system was improved, the software was upgraded and a number of other aspects were modified and enhanced.

Uninterrupted data was needed to effectively evaluate the Xanthoprobe's measuring capabilities and a portion of the industrial testing phase was used to gather test data. Sampling from the tailings stream of cell 11, Xanthoprobe measurement data was collected between 2010/10/10 and 2010/10/22. This period had minimal interruptions and plant incidents affecting continuous sampling.

3 Results and Discussion

Note that all the experimental results have been normalised (using the same factor) in order to keep proprietary data confidential. The units ng/t will be used to indicate normalised g/t. This represents the normalised amount of xanthate per ton of feed.

3.1 Validation Testing

Table 1 shows the concentration readings in ng/t and the error in % for validation tests conducted over two periods during the industrial trial.

Table 1. Xanthoprobe validation results.

Test 1: 2010/04/08	Sample #	Manual	Xanthoprobe	% Error
	1	*0.50 ±0.02	0.44	20.00
	2	0.51 ±0.02	0.47	11.32
	3	0.54 ±0.03	0.46	16.36
	4	0.53 ±0.02	0.50	1.96
	5	0.53 ±0.02	0.50	10.71
Test 2: 2010/08/03	Sample #	Manual	Xanthoprobe	% Error
	1	0.55 ±0.02	0.47	6.00
	2	0.53 ±0.01	0.48	5.88
	3	0.55 ±0.01	0.50	9.09
	4	0.51 ±0.01	0.51	3.77
	5	0.56 ±0.01	0.47	11.32

* ± indicates the standard deviation above and below the average of the triplicate concentration measurements

The first validation test occurred in April 2010, and showed reasonable correlation with manual sampling. The average error was 12.07%, with a standard deviation of 6.8.

After this test the instrument was improved, with modifications mainly to the sampling and filtration system. A second test, conducted in August 2010, showed that the modifications improved the consistency of the readings resulting in a decrease in the average error (to 7.21%) as well as lowering the standard deviation to 2.98.

3.2 Circuit Sampling

SIBX (Sodium isobutyl xanthate) is dosed in the primary rougher surge tank (14.98 ng/t), primary rougher cell 4 (2.45 ng/t), in the secondary rougher surge tank (9.46 ng/t) and secondary rougher cell 5 (2.37 ng/t). These are indicated in

Figure 2 below.

The Xanthoprobe instrument provides a concentration measurement, in units of grams of free xanthate per ton of water. In keeping with units familiar to industry, the free xanthate concentration measurements were converted to grams of xanthate per ton ore (g/t) based on the percentage solids and the feed tonnage. To protect confidential information, these were then normalised, as represented by the unit ng/t.

Note that no samples were taken from cell 6 in the primary roughers and cells 3, 7 and 10 in the secondary roughers.

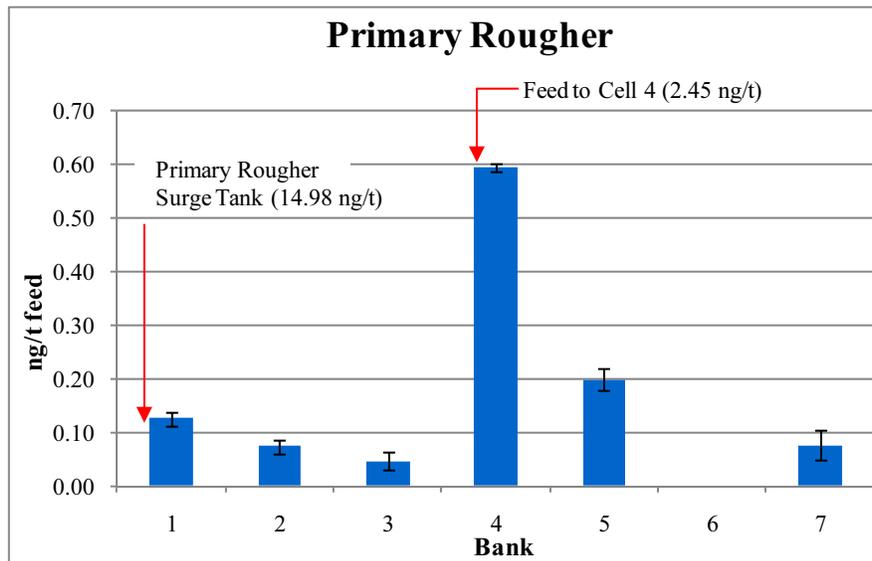


Figure 2. Normalised free xanthate concentrations in the primary rougher circuit (with error bars showing standard deviation of the samples).

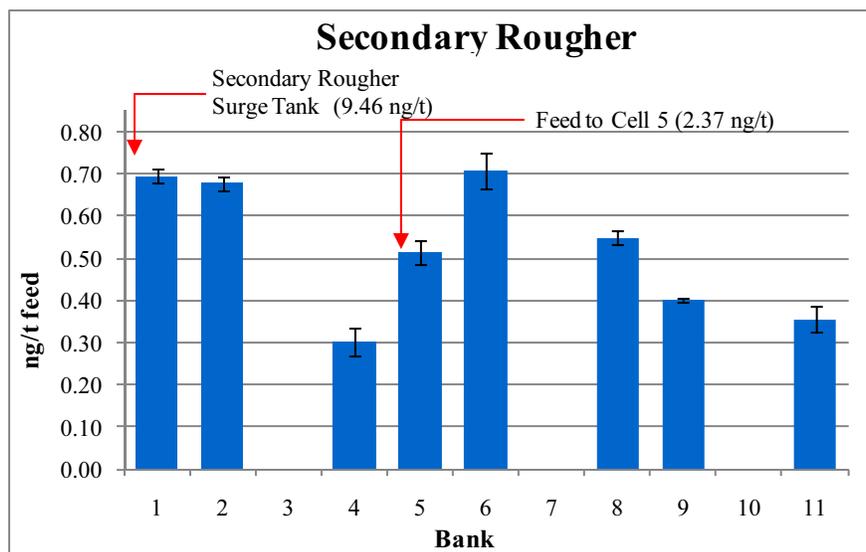


Figure 3. Normalised free xanthate concentrations in the secondary rougher circuit (with error bars showing standard deviation of the samples).

Primary rougher cell 1 had a free xanthate concentration of 0.12 ng/t and showed a gradual decrease in cells 2 and 3. After dosing, cell 4 had a concentration of 0.6 ng/t while cell 5 showed a sudden drop in concentration to 0.2 ng/t.

On the other hand, secondary rougher cell 1 had a concentration of 0.69 ng/t with no significant decrease in cell 2. After dosing, cell 5 measured 0.52 ng/t with cell 6 showing a considerable increase in concentration to 0.7 ng/t. The concentrations decreased along the remainder of the circuit with the final cell having a concentration of 0.35 ng/t. The concentration readings represent the free xanthate in solution, which is available for further attachment to mineral particles.

Referring to Figure 2 and Figure 3, the concentration readings showed interesting behaviour:

- Firstly, the free xanthate concentrations measured in cells 1-3 of the primary roughers were much lower than those measured in the secondary rougher cells 1-4. It is important to note that there is a higher SIBX dosage rate in the primary rougher surge tank (14.98 ng/t) than in the secondary surge tank (9.46 ng/t).
- In addition, the xanthate concentrations in the last few cells of the primary roughers were much lower compared to those in the secondary circuit with similar SIBX dosage rates in cell 4 and 5 of the primary and secondary circuits respectively.
- Most notably, there was a sudden drop in free xanthate in cell 5 after dosage in cell 4 in the primary roughers, while in the secondary roughers, an increase in concentration is observed in cell 6 after dosing in cell 5.

The primary roughers would predominantly contain a high percentage of fast floating sulphides and this can lead to an increase in collector adsorption. This explains the reduced free xanthate concentration readings in cells 1-3 as well as the sudden drop in cell 5 after SIBX addition (Figure 2). The slower floating sulphides would be found in the secondary circuit resulting in slower SIBX absorption. This would justify the higher free xanthate readings in the secondary roughers.

However, the increase in concentration from cell 5 to cell 6 in Figure 3 may be attributed to SIBX being short circuited out of cell 5 and into cell 6. There is no evidence that this is also occurring in the primary rougher due to the fast SIBX absorption, but it cannot be ruled out. This may have negative implications on the process operation.

3.3 Improvements and Testing on an Industrial Site

Figure 4 shows uninterrupted residual xanthate concentration measurements of the secondary rougher final tails taken by the Xanthoprobe as well as the secondary circuit SIBX dosage rates between 2010/10/10 and 2010/10/22.

Up to the 17th October, the SIBX dosage rate in cell 5 was unchanged at ± 1.9 ng/t while the Xanthoprobe steadily read ± 0.28 ng/t. An increase in SIBX dosage after the 17th resulted in a sharp increase in free xanthate concentration readings. Between the 19th and 21st the SIBX dosage rate and Xanthoprobe readings stayed unchanged at ± 2.8 ng/t and approximately ± 0.42 ng/t respectively. Thereafter, the Xanthoprobe responded to a further increase in SIBX dosage rate from 2.7 to 3.2 ng/t.

These results show that the Xanthoprobe measurement is strongly affected by changes in cell five's SIBX dosage rates - more so than the secondary rougher surge tank dosing. This is consistent with the down-the-bank measurements, which showed that there was minimal free xanthate available in cell 4, after the surge-tank dosing (Figure 4).

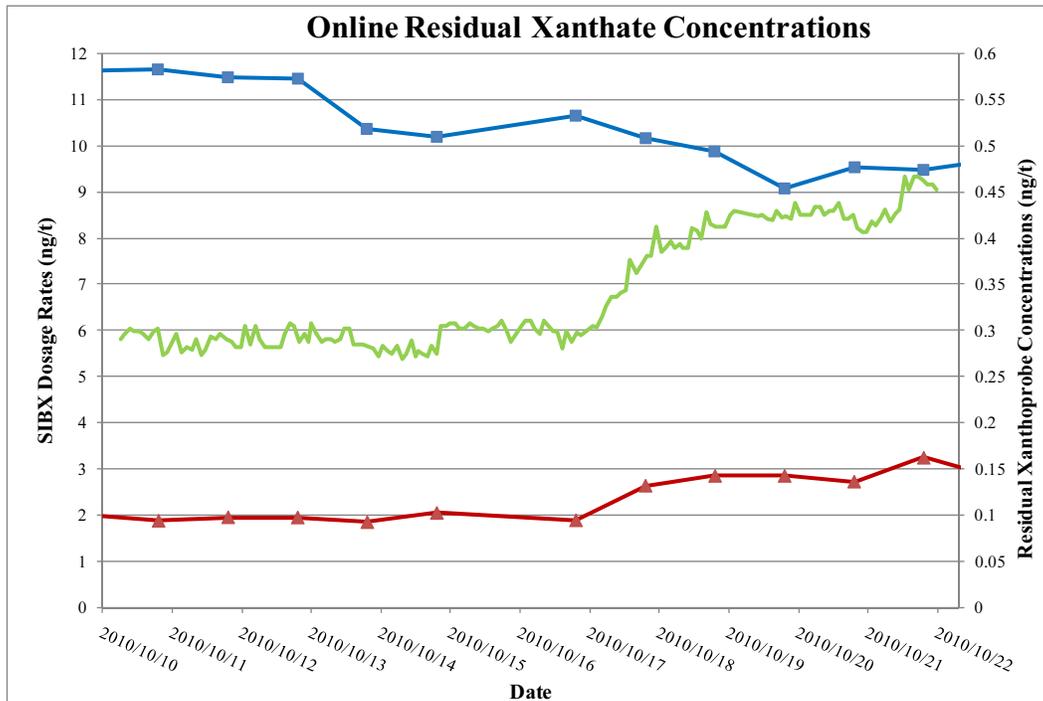


Figure 4. Normalised Xanthoprobe concentration readings (—, secondary axis), SIBX dosage rates in the secondary rougher surge tank (■, primary axis) and in cell 5 (▲, primary axis) in ng/t.

The ability of the Xanthoprobe to track the SIBX dosing in cell 5 can be used to control and monitor xanthate dosage rates. Through further long term testing, correlations can be drawn between optimum SIBX dosage rates and the free xanthate concentrations. In addition, the residual xanthate concentrations can also indicate whether the plant is over- or under dosing and this can lead to cost saving. An additional measurement in cell 4 would also provide useful information on the required dosage rate to the surge tank. As previously described, a plant which processes 72,000 ton of ore per day could save R5.3 million if they reduce their xanthate wastage by 10%.

4 Conclusion

The industrial trial of the instrument at Eland Platinum has greatly assisted in identifying existing design flaws and verifying the Xanthoprobe's ability to provide accurate results. Successful validation of onsite performance was carried out, which showed that modifications to the instrument improved the accuracy and consistency of online measurements.

The results obtained from the circuit sampling gave interesting insight into xanthate behaviour in a flotation circuit. The primary rougher section had lower free xanthate concentrations due to the presence of faster floating minerals, while the secondary circuit showed a slower uptake rate of xanthate. An interesting finding was that some of the xanthate short circuited out of cell 5 into cell 6 in the secondary roughers. This could have negative process and metallurgical implications and should be taken into account in terms of metallurgical optimisation.

The Xanthoprobe measurement tracked the secondary rougher cell 5 SIBX dosage rates. These measurements should be suitable for incorporation into an advanced process control strategy. In addition, based on these measurements, the Xanthoprobe can be used to prevent xanthate overdosing and to ensure a stable chemical environment for flotation.

The industrial trial has helped to identify hardware and software issues. Further long term analysis needs to be done for each ore type before satisfactory conclusions can be drawn on the potential for xanthate control. Nevertheless, the ability of the Xanthoprobe to measure online residual xanthate concentrations has broadened the future possibilities for online reagent control and optimisation, as well as cost saving.

5 Acknowledgements

This paper is published with the approval of Mintek.

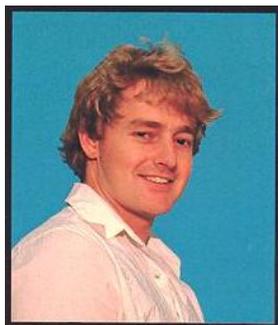
The authors gratefully thank Eland Platinum for granting Mintek permission to install and test the Xanthoprobe onsite as well as for their accommodating cooperation throughout the industrial trial.

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Julian Knight graduated from the University of Stellenbosch with a BEng Chemical Engineering degree in 2008. Since then he has been employed as a process control engineer in the Measurement and Control Division at Mintek. Julian's responsibilities include both commercial and R&D aspects. He has been involved in the implementation and maintenance of flotation process control solutions to clients in the PGM and BM industries in South Africa and Zimbabwe. His major R&D work over the past 3 years has been centred on the development of Mintek's Xanthoprobe. He has successfully developed the product to the industrial prototype stage, and is currently leading the project through the final development stages.

