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AN OPTICAL SENSOR FOR ON-LINE ANALYSIS OF PHOSPHATE MINERALS

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AN OPTICAL SENSOR FOR ON-LINE ANALYSIS OF PHOSPHATE MINERALS

Final Report
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PERSPECTIVE

Patrick Zhang, Research Director

On-line analysis and process automation are momentous tools for achieving optimal industry efficiency. The rewards of a good on-stream analysis technique coupled with modern process control include improved recovery of P_2O_5 , reduced chemical consumption and higher grade of the final concentrate. As the phosphate reserves switch from high grade ores to low grade and more difficult-to-process ores, improvement in plant efficiency and capacity is even more attractive. Unfortunately, unlike in many other industries process control in phosphate beneficiation plants is still very primitive even though the industry ages over a century. This is mainly attributed to the following difficulties related to chemical analysis of the plant slurry: (1) multi-elements analysis is required for good process control, including P_2O_5 , CaO, SiO_2 , and MgO, (2) the slurry is a heterogeneous system with about 30% solids and (3) the major elements to be analyzed, phosphorus and calcium, are low in atomic number.

Searching for a accurate and rapid on-stream phosphate assay technique has been one of the major issues on FIPR's agenda since its inception. In May 1, 1991, FIPR Board of Directors approved the project entitled "An Optical Sensor for On-Line Analysis of Phosphate Minerals".

In this investigation, two sensor have been considered. The first approach involves the use of an optical fiber sensor to determine the relative reflected intensity of white light incident on a phosphate sample. The relatively reflected intensity is inversely related to the BPL content. In the second method, a UV light source was used to create photoluminescence in a phosphate sample. The intensity of the photoluminescence is directly related to the BPL content.

Results of this study indicate that both approaches are capable of providing an indication of BPL content. However, the photoluminescence technique is limited by the low intensity of the resulting fluorescence output, and by the difficulty in differentiating the fluorescence output from the normal reflectance emanating from the sample. The optical fiber sensor gave consistent results, but with $\pm 30\%$ relative error. However, the investigators believed that the error could be reduced substantially by increasing the number of samples included in the calibration curve and by calibrating the sensor for a single stream within a single plant.

As is pointed out above, BPL determination alone is not enough for optimal process control. Before being made possible to analyze silica, magnesium, calcium and even iron with certain accuracy, the optical sensor will find limited use in the phosphate industry.

ABSTRACT

A study has been conducted to investigate methods for rapid determination of BPL content in phosphate ores using optical sensing technology. In this investigation, two approaches have been considered. The first approach involves the use of an optical fiber sensor to determine the relative reflected intensity of white light incident on a phosphate sample. The relative reflected intensity is inversely related to the BPL content. The second approach involves the use of a U.V. light source to create photoluminescence in a phosphate sample. The intensity of the photoluminescence is directly related to the BPL content.

Results of this study indicate that both approaches are capable of providing an indication of BPL content; however, the photoluminescence technique is limited by the low intensity of the resulting fluorescence output, and by the difficulty in differentiating the fluorescence output from the normal reflectance emanating from the sample. Furthermore, the photoluminescence technique is not able to eliminate the effect of phosphate color variation on the resulting calibration curve. The reflected light technique is also affected by variations in the phosphate mineral color; however, a procedure for normalizing the data has been developed which greatly reduces this effect.

A calibration curve has been developed using samples from IMC's Kingsford, Four Corners and Noralyn operations. A statistical analysis of this curve indicates that the reflected light technique is capable of analyzing a typical amine concentrate or tailing sample within approximately $\pm 30\%$ relative error for a 90% prediction band (i.e., the analysis of 9 samples out of 10 will fall within $\pm 30\%$ of the true assay). Included within this relative error is the error associated with the chemical assays used as standards. A detailed analysis of these assays indicates that in some cases this error can be as high as 25%. Previous experience with sensor calibration indicates that the relative error of the optical sensor can be greatly reduced by including additional points in the calibration curve and by generating separate calibration curves for concentrate and tailing. In fact, a video-based off-line analyzer, which has been extensively calibrated and tested on site, is currently being used at the Texasgulf phosphate operation in North Carolina to analyze amine concentrate samples within $\pm 1\%$ relative error.

Preliminary tests on slurry samples using the reflected light technique indicate that the accuracy of wet analysis is comparable to that of dry analysis. However, since these tests were conducted using a stationary sample, further work is needed to determine how the sensor will respond when the slurry is in motion.

Based on the results obtained from this study, it appears that future work should be focused on two areas. In the long term, the reflected light technique shows sufficient promise to warrant further development. Future work in this area should concentrate on adapting the sensor to slurry applications and testing it in an on-line configuration. In the short term, the normalization procedure developed in the present work indicates that the video-based sensor developed for North Carolina phosphate could be adapted for Florida phosphate. The current success of this sensor in North Carolina warrants the installation and testing of this device in Florida.

1. INTRODUCTION

The switch in phosphate reserves from high-quality ores to more difficult reserves has resulted in increased pressure for improvements in plant efficiency and capacity. One of the most effective methods of improving plant performance is the use of process control. Unfortunately, the phosphate industry lacks the appropriate on-line instrumentation to implement assay-based control schemes. Specifically, a need exists for a suitable means of providing on-line analysis of BPL content in process streams around the flotation circuit.

The primary objective of this project was to develop an optically-based sensor for phosphate analysis. The sensor was designed, using fiber optics technology, to discriminate between the dark phosphate minerals and the light gangue minerals as a means of providing a phosphate assay. Two sensing approaches, photoluminescence and reflected light, were explored as a means of providing an on-line measure of BPL content independent of the color variations normally associated with the Florida ores.

The work was carried out as a one-year project with a six-month extension covering the period from May 1, 1991 to October 31, 1992. The project was originally divided into three tasks including: Task 1 - Sensor Development, Task 2 - Sampling System Development and Task 3 - On-Line Testing. Task 1 was to precede Tasks 2 and 3 until the sensor had been demonstrated to give satisfactory response to variations in BPL content irrespective of the variations in color observed from reflected white light. Approximately 9 months into the project it was decided that work in Task 1 needed to be explored further. Thus, a change of scope was agreed upon by FIPR and VPI&SU to concentrate all remaining efforts on sensor development and to leave on-line testing to a future project should the sensor development efforts prove successful.

2. SENSOR DEVELOPMENT

2.1 Sample Acquisition

Samples for testing and calibrating the various optical sensing methods were provided by Dr. R. L. Wiegel of IMC. The first set of samples was received in June 1991 and consisted of fine feed, amine concentrate and fine tailings from three different plants. In order of increasing sample brightness these plants were Kingsford, Four Corners and Noralyn. A second set of samples was provided by Dr. Wiegel in September 1992. This set included a fourth plant, the Hopewell plant, which had material slightly brighter than the Noralyn plant. In this case, a given set of feed, concentrate and tailing samples were collected at approximately the same time in order to test a data normalization procedure that had been developed. Both sets of samples along with their assays are listed in Tables 1 and 2.

Table 1. June 1991 Sample Set.

Sample	Date	Percent BPL	Percent Insol.
Kingsford Fine Feed	5-17-91	12.51	79.94
	5-29-91	9.00	86.07
	5-30-91	9.51	83.62
Amine Conc.	5-17-91	72.35	3.54
	5-31-91	73.65	1.95
	5-31-91	73.74	2.22
Fine Tailing	5-17-91	1.34	96.24
	5-29-91	1.82	95.69
	5-30-91	1.74	94.20
Four Corners Fine Feed	5-20-91	6.22	90.21
	5-23-91	5.69	89.39
	5-28-91	6.28	89.57
Amine Conc.	5-20-91	69.48	4.89
	5-23-91	72.39	3.20
	5-28-91	71.28	2.93
Fine Tailing	5-20-91	1.39	97.00
	5-23-91	1.59	95.29
	5-28-91	1.17	96.19
Noralyne Fine Feed	5-9-91	17.97	73.73
	5-13-91	14.91	76.01
	---	13.25	79.47
Amine Conc.	5-9-91	71.61	3.18
	5-13-91	73.16	1.96
	5-15-91	73.63	3.45
Fine Tailing	5-9-91	1.96	95.78
	5-15-91	1.18	94.39
	6-1-91	0.81	95.14

Table 2. September 1992 Sample Set.

Sample	Date	Percent BPL	Percent Insol.
Kingsford Fine Feed	8-12-92	9.52	---
	8-13-92	9.14	---
	8-14-92	8.77	---
	8-15-92	9.63	---
	8-16-92	11.56	---
	8-17-92	11.32	---
	8-18-92	11.36	---
Amine Conc.	8-12-92	73.61	3.68
	8-13-92	74.44	2.80
	8-14-92	74.28	3.18
	8-15-92	73.18	4.54
	8-16-92	73.36	4.04
	8-17-92	73.71	4.21
	8-18-92	74.28	3.68
Fine Tailing	8-12-92	1.82	---
	8-13-92	2.06	---
	8-14-92	2.26	---
	8-15-92	3.42	---
	8-16-92	2.84	---
	8-17-92	2.34	---
	8-18-92	2.42	---

Table 2. (Continued)

Sample	Date	Percent BPL	Percent Insol.
Four Corners Fine Feed	8-12-92	8.16	---
	8-13-92	8.42	---
	8-14-92	7.14	---
	8-16-92	7.23	---
	8-17-92	10.09	---
	8-19-92	8.12	---
Amine Conc.	8-12-92	69.32	5.46
	8-13-92	71.50	3.38
	8-14-92	70.22	4.08
	8-16-92	69.38	4.32
	8-17-92	69.87	5.26
	8-19-92	70.94	4.23
Fine Tailing	8-12-92	1.46	---
	8-13-92	1.40	---
	8-14-92	1.76	---
	8-16-92	5.12	---
	8-17-92	1.96	---
	8-19-92	2.47	---

Table 2. (Continued)

Sample	Date	Percent BPL	Percent Insol.
Noralyn Fine Feed	8-10-92	13.76	---
	8-11-92	16.38	---
	8-12-92	15.18	---
	8-13-92	14.54	---
	8-14-92	14.72	---
	8-15-92	14.17	---
	8-16-92	15.87	---
Amine Conc.	8-10-92	72.26	2.61
	8-11-92	72.42	3.32
	8-12-92	72.68	3.76
	8-13-92	74.02	3.82
	8-14-92	72.94	3.78
	8-15-92	71.64	4.28
	8-16-92	72.05	4.51
Fine Tailing	8-10-92	1.54	---
	8-11-92	1.58	---
	8-12-92	2.87	---
	8-13-92	2.22	---
	8-14-92	1.89	---
	8-15-92	1.48	---
	8-16-92	2.56	---

Table 2. (Continued)

Sample	Date	Percent BPL	Percent Insol.
Hopewell Fine Feed	8-10-92	15.50	---
	8-11-92	24.29	---
	8-12-92	23.96	---
	8-13-92	21.44	---
	8-14-92	24.38	---
	8-15-92	22.68	---
	8-16-92	22.28	---
Amine Conc.	8-10-92	75.52	4.14
	8-11-92	75.65	4.22
	8-12-92	77.20	3.61
	8-13-92	76.06	2.94
	8-14-92	76.44	3.48
	8-15-92	77.02	3.56
	8-16-92	76.69	3.66
Fine Tailing	8-10-92	4.09	---
	8-11-92	7.05	---
	8-12-92	5.76	---
	8-13-92	5.79	---
	8-14-92	5.15	---
	8-15-92	5.22	---
	8-16-92	3.67	---

2.2 Reflected Light Analysis

2.2.1 Experimental Apparatus

Reflected light analysis of the June 1991 sample set was originally carried out using the experimental set-up shown in Figure 1. As shown, a white light source is injected into a lead-in fiber bundle. The light out of the bundle is incident to a sample cell filled with a given phosphate sample. The reflection from the sample is collected by a second lead-out fiber bundle and propagates to a photodetector. A reference optical channel is used to increase stability and repeatability of the measurement. The sensor output is given by the ratio of the intensity of the signal channel to the reference channel at the photodetector. A microcomputer is used to obtain the voltage levels from the photodetectors and to average these measurements 100 times to smooth out variations and fluctuations in the readings. Each different color or gray level of the sample corresponds to a different intensity of reflected light from the sample, and hence, a different output ratio from the sensor. The BPL content can be extracted from the output ratio of the sensor provided a definite relationship exists between reflected intensity and BPL content.

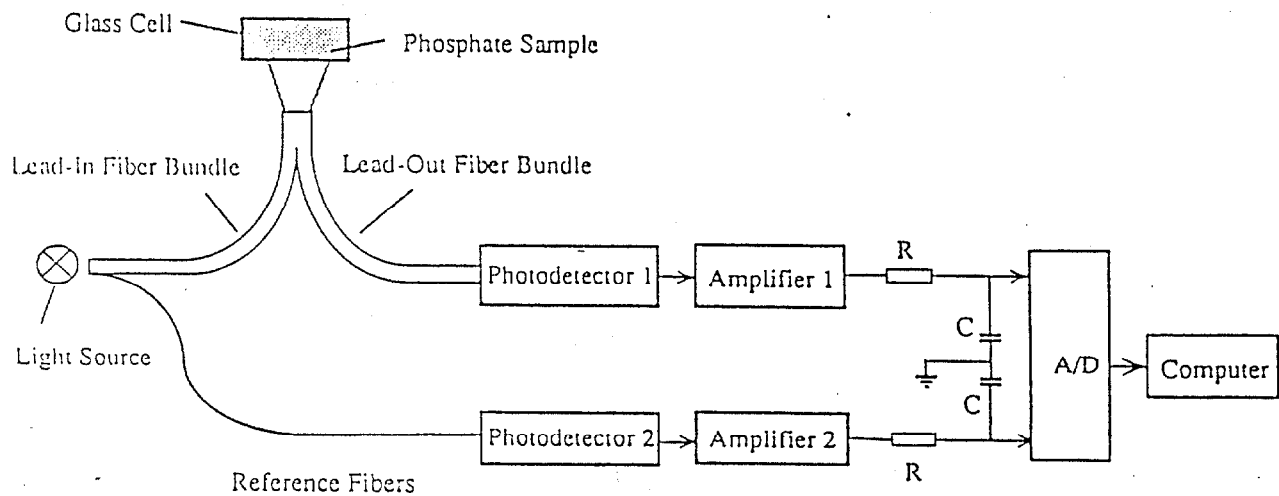


Figure 1. Schematic of the reflected light experimental set-up.

2.2.2 Preliminary Testing

Results of the measurements obtained for the June 1991 sample set are shown in Figure 2. As shown, a correlation clearly exists between the BPL content of the sample and the relative reflected intensity of the incident light. In fact, the samples from the Kingsford and Four Corners plants appear to fit the same calibration curve with the exception of one or two points. On the other hand, the much lighter-colored Noralyn samples lie along a separate calibration curve and also exhibit more scatter. It is interesting to note that the calibration curves exhibit an exponential form as evident from the linear plots obtained when the data is plotted on a semi-log scale (Figure 3).

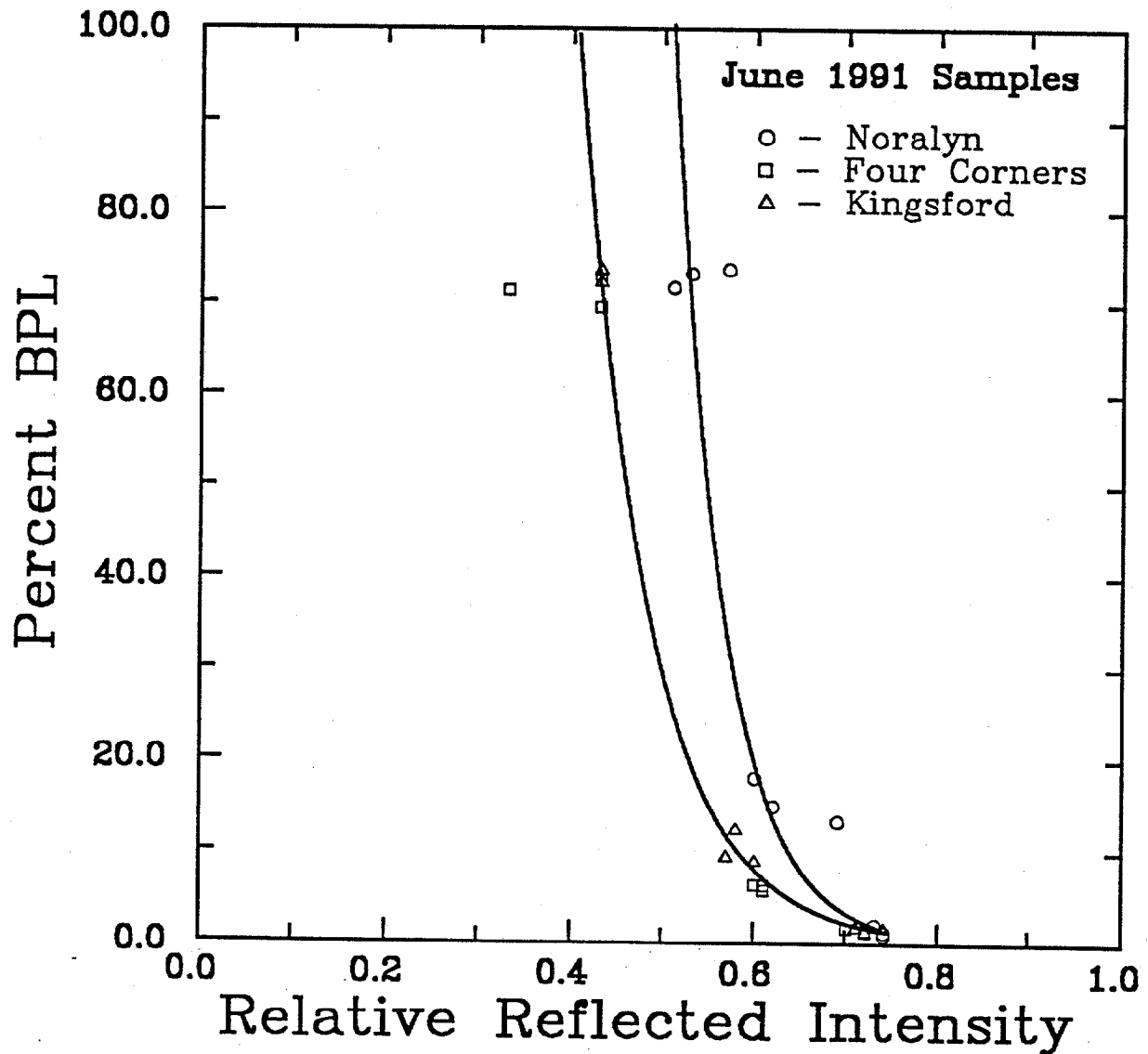


Figure 2. BPL calibration curve for the June 1991 sample set obtained using the reflected light sensor.

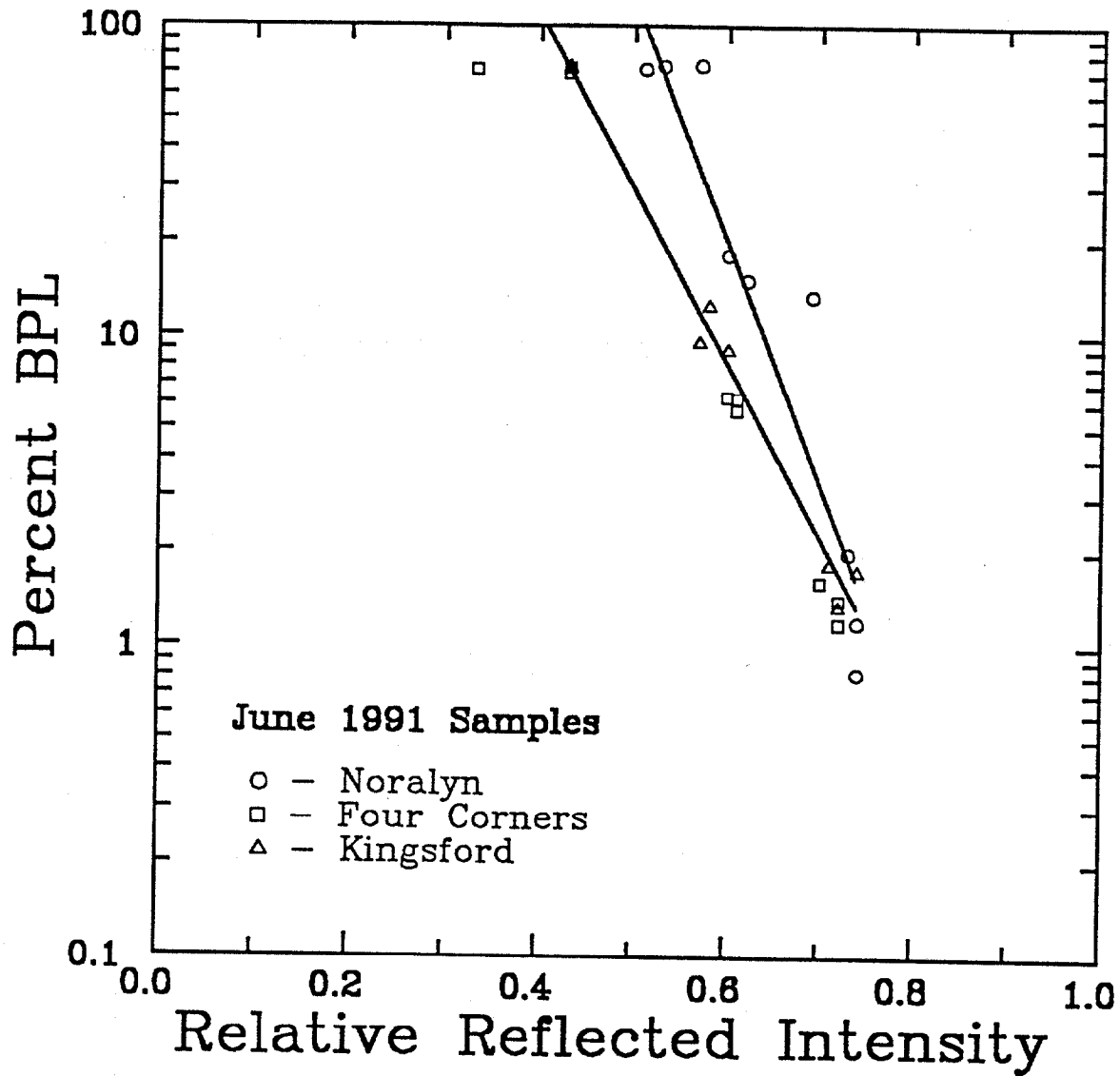


Figure 3. BPL calibration curve (exponential form) for the June 1991 sample set obtained using the reflected light sensor.

According to the results shown in Figures 2 and 3, the color of the phosphate mineral at a given plant appears to be relatively constant over a two-week period. This means that the data from each plant can be represented by a single calibration curve. Furthermore, the phosphate mineral color at the Kingsford and Four Corners plants is sufficiently similar that the data from both plants can be plotted on the same calibration curve. Thus, these preliminary results indicate that a simple reflected light sensor may have potential as an on-line BPL analyzer. On the other hand, the Noralyn sample requires a separate calibration curve and may be subject to a greater color variation than the samples from the other two plants as indicated by the increased data scatter.

2.2.3 Data Normalization

In an attempt to reduce or eliminate all color variation, both within a plant and from plant-to-plant, a data normalization scheme has been devised in which all values are normalized relative to the feed. This procedure yields the terms “normalized BPL content” and “normalized reflected intensity” where normalized BPL content is given by:

$$\text{Normalized BPL Content} = \frac{\%BPL}{\%BPL_{Feed}} \quad (1)$$

and normalized reflected intensity by:

$$\text{Normalized Reflected Intensity} = \frac{\text{Relative Reflected Intensity}}{\text{Relative Reflected Intensity}_{Feed}} \quad (2)$$

Using this procedure, the data from the June 1991 data set were normalized and replotted as shown in Figure 4 and Table 3. In this case, only those samples of feed, concentrate and tailing which were collected on the same day were used in the analysis. As shown, all of the data, with the exception of one or two points, fall along the same line indicating the success of the normalization procedure.

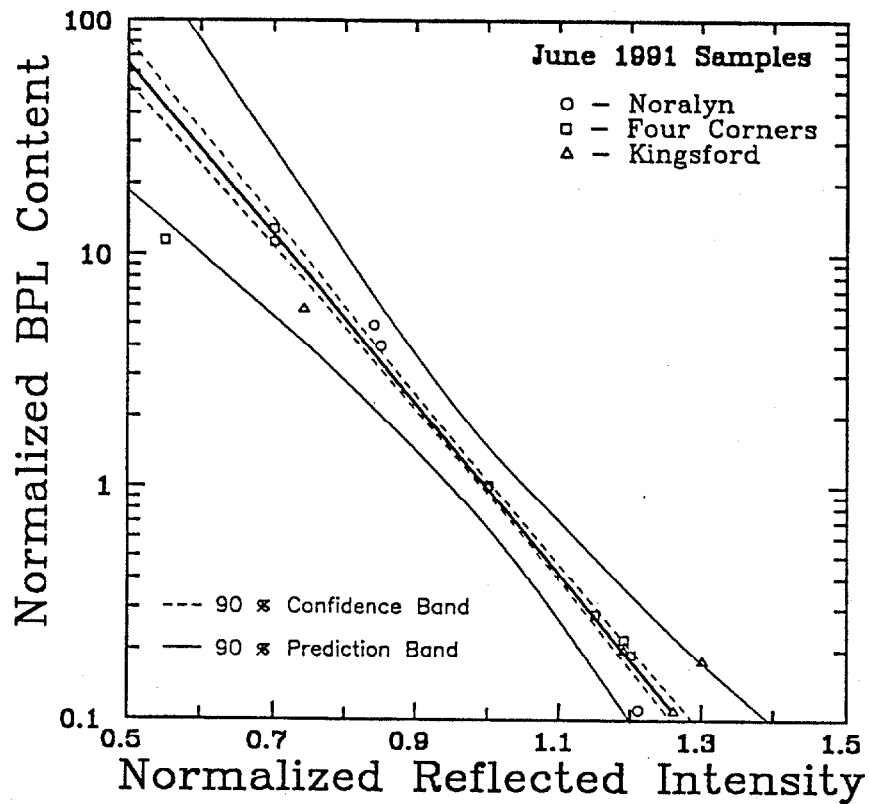


Figure 4. Normalized calibration curve for the June 1991 sample set obtained using the reflected light sensor.

Table 3. Reflected Light Analysis of Phosphate Ore (June 1991 Sample Set).

Sample	Date	Percent BPL	Normalized BPL Content	Relative Reflected Intensity	Normalized Reflected Intensity
Four Corners Fine Feed	5-20-91	6.22	1.00	0.61	1.00
	5-23-91	5.69	1.00	0.61	1.00
	5-28-91	6.28	1.00	0.60	1.00
Amine Conc.	5-20-91	69.48	11.17	0.43	0.70
	5-23-91	72.39	12.72	0.43	0.70
	5-28-91	71.28	11.35	0.33	0.55
Fine Tailing	5-20-91	1.39	0.22	0.72	1.19
	5-23-91	1.59	0.28	0.70	1.15
	5-28-91	1.17	0.19	0.72	1.20
Kingsford Fine Feed	5-17-91	12.51	1.00	0.58	1.00
	5-29-91	9.00	1.00	0.60	1.00
	5-30-91	9.51	1.00	0.57	1.00
Amine Conc.	5-17-91	72.35	5.78	0.43	0.74
	5-31-91	73.65	—	0.43	—
	5-31-91	73.74	—	0.43	—
Fine Tailing	5-17-91	1.34	0.11	0.72	1.26
	5-29-91	1.82	0.20	0.71	1.19
	5-30-91	1.74	0.18	0.74	1.30
Noralyln Fine Feed	5-09-91	17.97	1.00	0.60	1.00
	5-13-91	14.91	1.00	0.62	1.00
	—	13.25	—	0.69	—
Amine Conc.	5-09-91	71.61	3.98	0.51	0.85
	5-13-91	73.16	4.91	0.53	0.84
	5-15-91	73.63	—	0.57	—
Fine Tailing	5-09-91	1.96	0.11	0.73	1.21
	5-15-91	1.18	—	0.74	—
	6-01-91	0.81	—	0.74	—

Also shown in Figure 4 are the 90% confidence band and the 90% prediction band. The 90% confidence band indicates (within a 90% probability) the region where the best-fit line to a given data set exists. Based on this definition, it is clear that the calibration curve shown in Figure 4 is defined quite accurately. The 90% prediction band, on the other hand, indicates (within a 90% probability) the region where any single predicted value exists. In other words, if the normalized reflectance of a given sample was determined to be 0.8, the sensor would produce a normalized BPL reading of approximately 5 based on the calibration curve. However, 90% of the time, the true assay of the sample could range anywhere from 2 to 10. Thus, the 90% prediction band has the most meaning in terms of establishing the accuracy of the sensor.

There are two factors which control the spread of the 90% prediction band. These include (i) the scatter in the data, caused by such things as color variation, sample bias, chemical analysis error, etc., and (ii) the number of data points used in the calculation. The wide prediction band shown in Figure 4 for the June 1991 sample set is largely a result of the small sample size used in the calculation. It is expected that the spread of the prediction band can be greatly reduced by increasing the number of samples used in the calculation. This concept will be illustrated in the following section.

2.2.4 Statistical Analysis

Based on the preliminary success with the data normalization procedure, a second sample set was obtained from IMC in September 1992. In this case, an effort was made to collect feed, concentrate and tailing samples at approximately the same time for the specific purpose of statistically analyzing the normalization procedure. Once again, samples were collected from the Noralyn, Four Corners and Kingsford plants. An additional sample set from the Hopewell plant was also obtained; however, due to its light color, adjustments to the experimental set-up were required. Since these adjustments would make it difficult to compare the September 1992 samples with the June 1991 samples, it was decided to exclude the Hopewell samples from the analysis. Some Noralyn samples that were too light in color were also excluded from the analysis for the same reason. The results of the analyses obtained for the remaining samples are illustrated in Figure 5 and Table 4.

As shown the prediction band for the September 1992 sample set is much tighter than that shown previously for the June 1991 set. This is largely due to the increased number of data points included in the September 1992 set. At the same time, the confidence bands for the two sample sets are nearly identical and indicate a high degree of confidence in the lines used to fit each set of data. Furthermore, the equation representing the September 1992 calibration curve is nearly identical to that for the June 1991 curve (Table-5) indicating that either the phosphate color did not change appreciably during this period or the normalization procedure effectively eliminated any variations caused by changes in phosphate color. The same conclusions are true for the individual sets of data obtained for each plant as shown in Figures 6 - 8. Although the best fit calibration is slightly different for each plant, the samples obtained in June 1991 and in September 1992 seem to follow the same trend for each plant. This result tends to suggest that individual calibrations obtained for each plant may be a more appropriate means of calibrating the sensor.

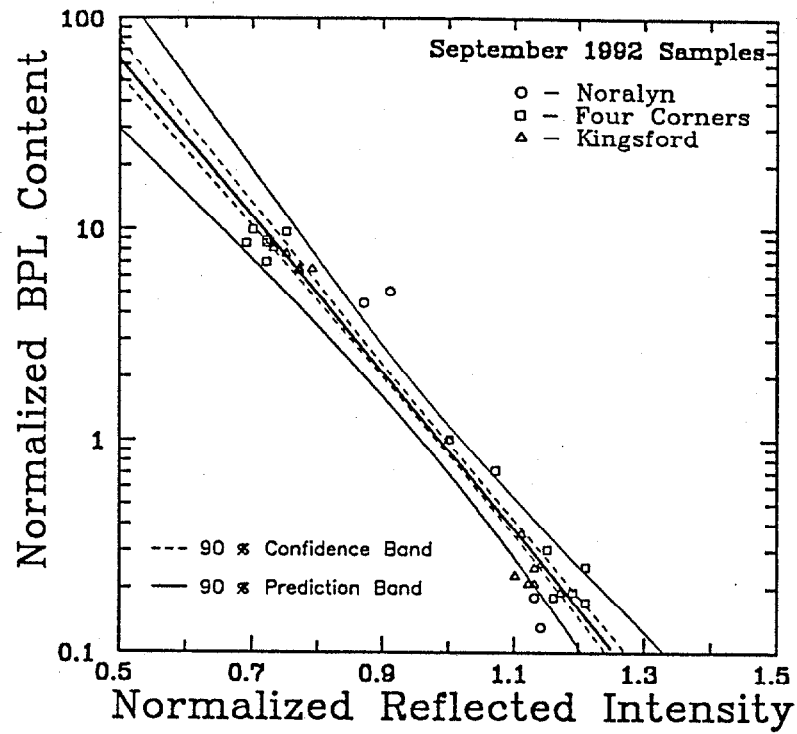


Figure 5. Normalized calibration curve for the September 1992 sample set obtained using the reflected light sensor.

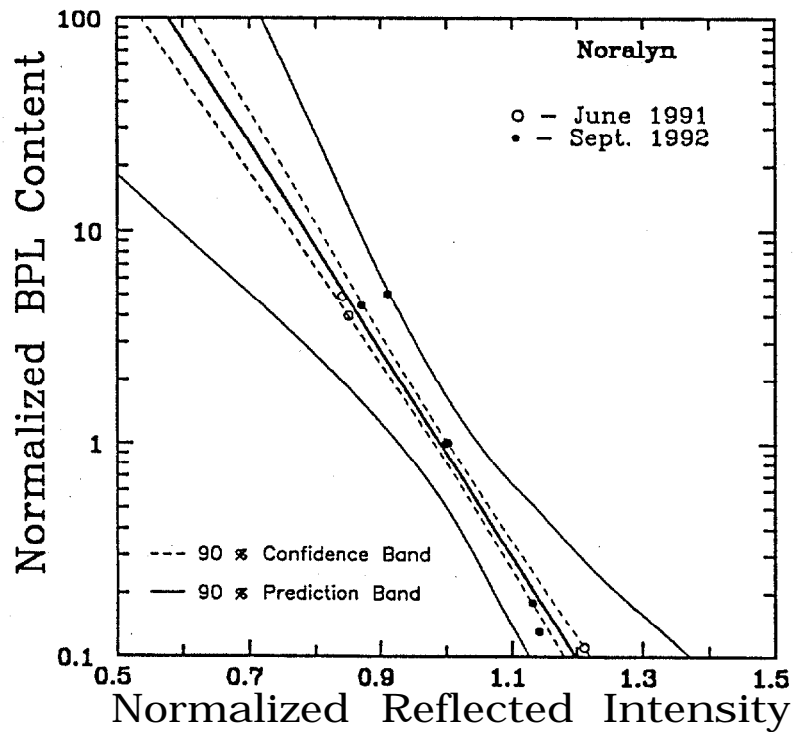


Figure 6. Normalized calibration curve for the Noralyn plant.

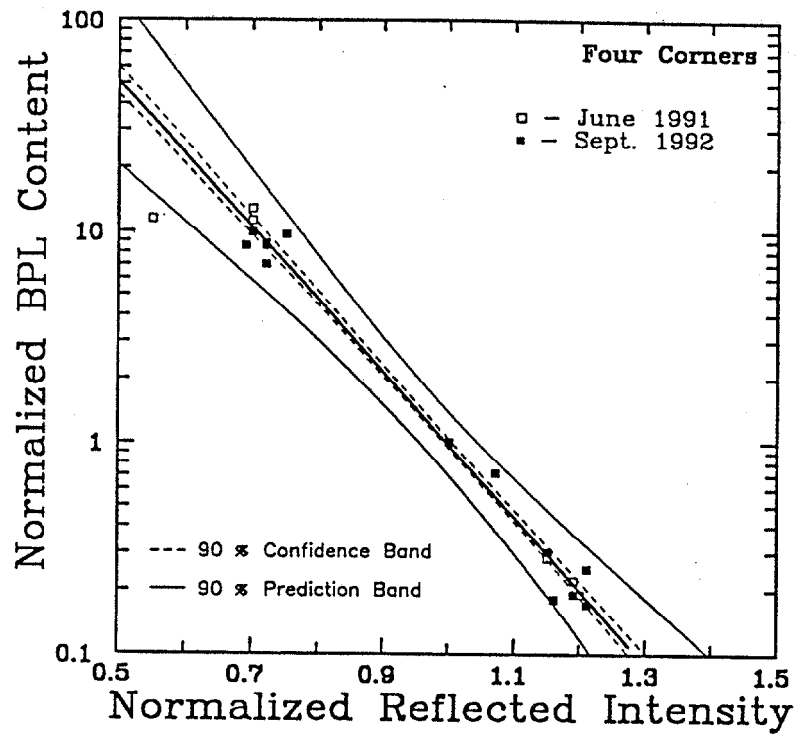


Figure 7. Normalized calibration curve for the Four Corners plant.

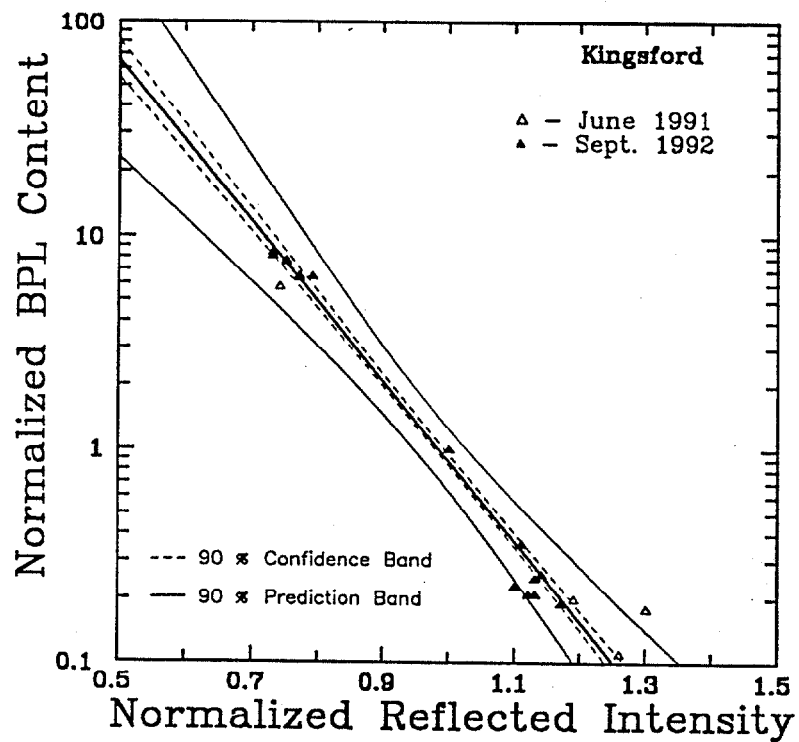


Figure 8. Normalized calibration curve for the Kingsford plant.

Table 4. Reflected Light Analysis of Phosphate Ore (September 1992 Sample Set).

Sample	Date	Percent BPL	Normalized BPL Content	Relative Reflected Intensity	Normalized Reflected Intensity	Percent Variation in Chemical Assay
Noralya Fine Feed	8-12-92	16.38	1.00	0.87	1.00	1.4
	8-14-92	14.54	1.00	0.88	1.00	1.5
Amine Conc.	8-12-92	72.68	4.44	0.76	0.87	1.8
	8-14-92	72.94	5.02	0.80	0.91	1.7
Fine Tailing	8-12-92	2.87	0.18	1.00	1.15	7.1
	8-14-92	1.89	0.13	0.99	1.13	2.7
Four Corners Fine Feed	8-12-92	8.16	1.00	0.84	1.00	0.4
	8-13-92	8.42	1.00	0.83	1.00	1.1
	8-14-92	7.14	1.00	0.82	1.00	0.4
	8-16-92	7.23	1.00	0.84	1.00	1.5
	8-17-92	10.09	1.00	0.84	1.00	3.2
	8-19-92	8.12	1.00	0.83	1.00	7.1
Amine Conc.	8-12-92	69.32	8.50	0.61	0.72	1.1
	8-13-92	71.50	8.49	0.58	0.69	1.4
	8-14-92	70.22	9.83	0.58	0.70	0.5
	8-16-92	69.38	9.60	0.63	0.75	1.6
	8-17-92	69.87	6.92	0.60	0.72	3.5
	8-19-92	70.94	8.74	0.60	0.72	8.0
Fine Tailing	8-12-92	1.46	0.18	0.98	1.16	4.5
	8-13-92	1.40	0.17	1.00	1.21	3.9
	8-14-92	1.76	0.25	1.00	1.22	10.6
	8-16-92	5.12	0.71	0.91	1.08	3.5
	8-17-92	1.96	0.19	0.99	1.19	13.7
	8-19-92	2.47	0.30	0.96	1.15	18.5

Table 4. (Continued)

Sample	Date	Percent BPL	Normalized BPL Content	Relative Reflected Intensity	Normalized Reflected Intensity	Percent Variation in Chemical Assay
Kingsford Fine Feed	8-12-92	9.52	1.00	0.86	1.00	5.1
	8-13-92	9.14	1.00	0.87	1.00	2.3
	8-14-92	8.77	1.00	0.87	1.00	5.9
	8-15-92	9.63	1.00	0.88	1.00	5.8
	8-16-92	11.56	1.00	0.89	1.00	2.9
	8-17-92	11.32	1.00	0.89	1.00	2.9
	8-18-92	11.36	1.00	0.89	1.00	2.7
Amine Conc.	8-12-92	73.60	7.73	0.64	0.74	5.8
	8-13-92	74.44	8.14	0.62	0.72	2.9
	8-14-92	74.28	8.47	0.64	0.73	6.7
	8-15-92	73.18	7.60	0.66	0.75	6.3
	8-16-92	73.36	6.35	0.68	0.77	3.3
	8-17-92	73.70	6.51	0.70	0.79	3.5
	8-18-92	74.28	6.54	0.69	0.77	2.8
Fine Tailing	8-12-92	1.82	0.19	1.00	1.17	25.9
	8-13-92	2.06	0.23	0.95	1.10	21.4
	8-14-92	2.26	0.26	0.99	1.14	15.5
	8-15-92	3.42	0.36	0.97	1.10	15.5
	8-16-92	2.84	0.25	0.99	1.12	8.2
	8-17-92	2.34	0.21	0.99	1.12	8.8
	8-18-92	2.42	0.21	1.00	1.13	9.9

Table 5. Calibration Equations.

Data Set	Equation
All Samples	$Y_{BPL} = 4400 \exp(-8.5X_R)$
June 1991	$Y_{BPL} = 4400 \exp(-8.4X_R)$
September 1992	$Y_{BPL} = 4500 \exp(-8.5X_R)$
Noralyn	$Y_{BPL} = 62000 \exp(-11X_R)$
Four Corners	$Y_{BPL} = 2700 \exp(-7.9X_R)$
Kingsford	$Y_{BPL} = 4900 \exp(-8.6X_R)$
Kingsford (Wet)	$Y_{BPL} = 3600 \exp(-8.0X_R)$

Note: Y_{BPL} - Normalize BPL Content

X_R - Normalized Reflected Intensity

When both sets of data are plotted together, as shown in Figure 9, the prediction band is further reduced due to the increased number of data points. With the current set of data, this produces the smallest prediction band. For example, if one assumes a normalized BPL content of 7 as a typical concentrate value and 0.25 as a typical tailing assay, the accuracy of the measurement can be quantified based on the prediction band as shown in Table 6. Based on this information, the relative error of the measurement ranges from approximately 30-40%. While this number may seem high, it should be noted that the calibration is based on the chemical assays provided by IMC. Any error in the chemical assay is reflected in the calibration error of the optical sensor. Since IMC provided duplicate assays for the samples used in this investigation, it is possible to get a rough approximation of the amount of error associated with the chemical assays. As shown in Table 4, some of the assays contain as much as 20-25% error while the average error for all samples is approximately 6%. Therefore, the error contribution of the optical sensor may actually be closer to 20-30%.

Based on previous work with a video-based sensor developed for North Carolina phosphate, it appears that the relative error reported in the present work could be greatly reduced by increasing the data base used for calibration and by developing separate calibration curves for individual streams within each plant. Because of the large number of samples which must be included in such a calibration, this type of work should be done on-site. However, the potential benefits are worth the effort when one considers that the video-based system currently in use at Texasgulf is capable of determining P_2O_5 content and CaO/P_2O_5 ratio in amine concentrate samples within $\pm 1\%$ relative error. A recent paper on this sensor is included in the Appendix of this report.

26.5 Summary

A reflected-light-based optical sensor has been developed which is capable of analyzing the multi-colored phosphate ores found in Florida and which shows potential as a possible on-line system for phosphate analysis. A data normalization procedure developed in the present project provides a convenient solution to the color variation problem associated with Florida phosphate while any long term variations in phosphate color can be accommodated by periodically updating the calibration curve through appropriate software. The overall calibration curve obtained in this work indicates that the sensor is capable of analyzing relative BPL content in amine concentrate and fine tailing streams with a relative error of 20-30% when the error introduced by chemical analysis is taken into account. Furthermore, it appears that the accuracy of the optical sensor can be improved by incorporating additional sample points and by calibrating the sensor for a single stream in a single plant rather than using one overall calibration.

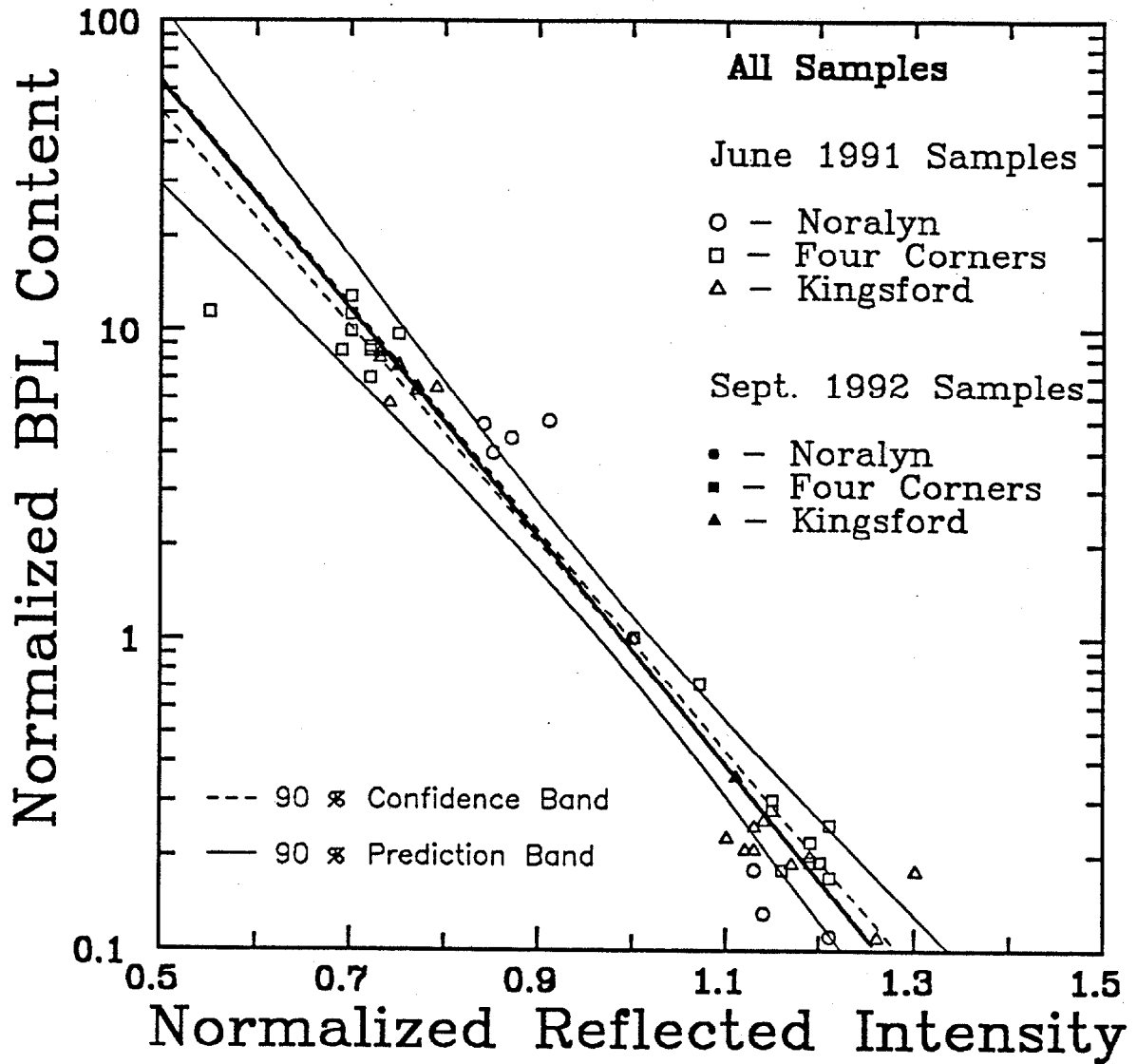


Figure 9. Normalized calibration curve for the overall sample set obtained using the reflected light sensor.

Table 6. Accuracy of the Reflected Light Analysis System.

Stream	Absolute Error	Relative Error
Concentrate ($Y_{BPL} = 7$)	+2.9, -2.1	+41%, -30%
Tailing ($Y_{BPL} = 0.25$)	+0.09, -0.07	+36%, -28%

2.3 Photoluminescence Analysis

Many phosphate minerals are known to fluoresce under ultraviolet light. This fluorescence largely results from trace amounts of rare earths present in these minerals. Since these trace elements vary with the BPL content of the ore, it was felt that the amount of fluorescence emitted by a given sample should be proportional to the BPL content irrespective of the sample color. Thus, a series of tests were conducted to explore the use of photoluminescence as a means of optically measuring BPL content in phosphate ores.

2.3.1 Preliminary Testing

The experimental set-up for photoluminescence testing is shown in Figure 10. As shown, the output of a light source is incident to a phosphate sample. A portion of the reflected light and emitted fluorescence is focused and sent to an Optical Spectrum Analyzer (OSA). The OSA scans the wavelength range from 400 to 1600 nm looking for peaks in the output indicative of fluorescence.

In the initial test work, a number of light sources were used including white light, a deuterium UV lamp and a high-powered Argon laser. No fluorescence was detected with any of these systems.

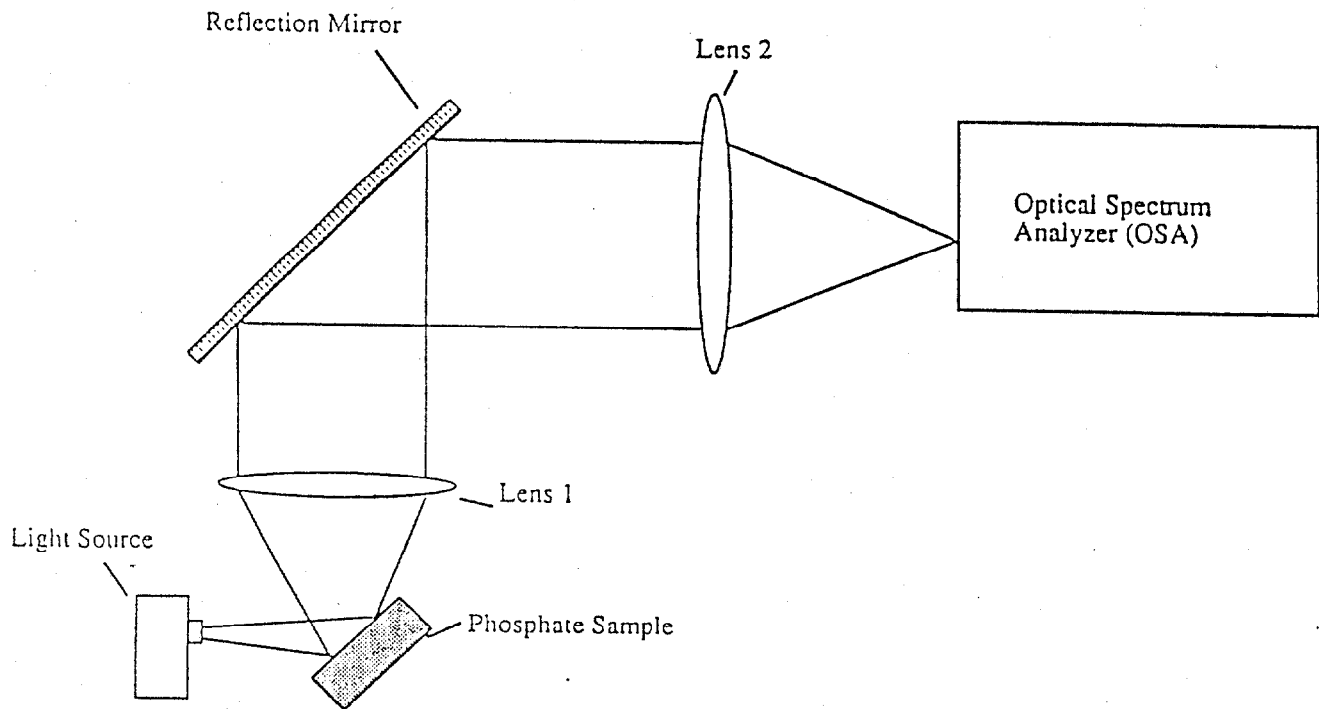


Figure 10. Schematic of experimental set-up for photoluminescence studies.

2.3.2 Laser-Raman Spectroscopy

In order to obtain a sensitivity which is high enough to observe possible fluorescence emitted from a phosphate mineral sample, an ISA Model U1000 Raman Micro-Probe, with a sensitivity of opto-electronic detection of less than 50 photons per second, was employed. Figure 11 shows a schematic of this Raman probe system. An Argon laser with an output optical power of 8 watts was used as the light source in these experiments. The incident laser beam is focused down on the test specimen with a focal spot dimension of approximately 50 μm . A lens with a large numerical aperture is used to focus the reflected light from the test specimen into a double monochromator. A photomultiplier with a sensitivity on the order of 10 photons per second is placed at the output slit of the monochromator. The output signal from the photomultiplier is fed to a lock-in amplifier to further improve the signal-to-noise ratio of the system. To ensure a high sensitivity, the bandwidth of the opto-electronic detection system is set very small resulting in a total wavelength scan time of 30 minutes. This scan is controlled by a microcomputer.

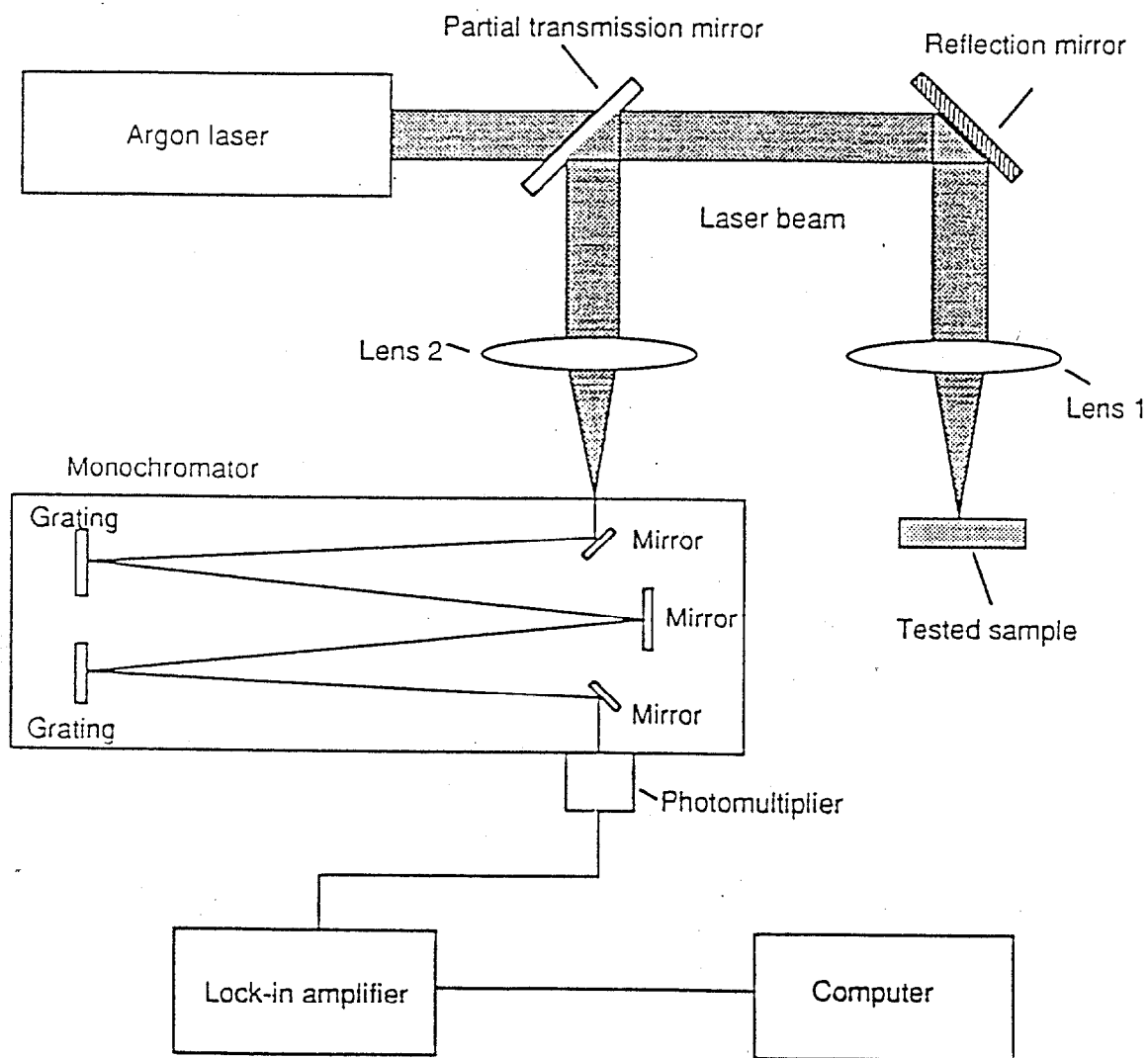


Figure 11. Schematic of the Laser-Raman Probe system.

Samples of fine feed, amine concentrate and fine tailing from the June 1991 sample set were analyzed using the Laser-Raman system. The optical output generated by this system is shown in Figures 12-14 for each of the three plants studied. In these figures, the abscissa represents the change in wave number relative to the wave number of the incident Argon laser while the ordinate gives the optical power level, expressed in photons per second, received by the photomultiplier. As shown, the Laser-Raman output generally increases in magnitude as the BPL content of the sample increases although there is considerable scatter in the data. This is in contrast to the reflected light output which was seen to decrease as BPL content increased. Thus, it is believed that at least part of the output from the Laser-Raman system is related to fluorescence of the phosphate minerals present in the sample. On the other hand, the sharp peak in the output at a specific wavelength, usually characteristic of fluorescence, is not present, and the broad shape of the output signal seems to indicate that reflected light is also present in this signal.

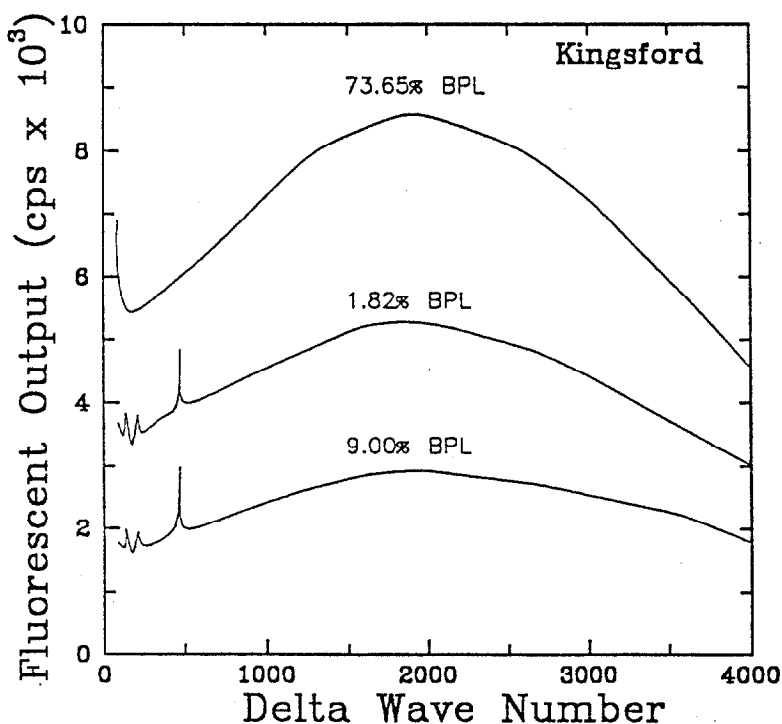


Figure 12. Laser-Raman output for Kingsford samples.

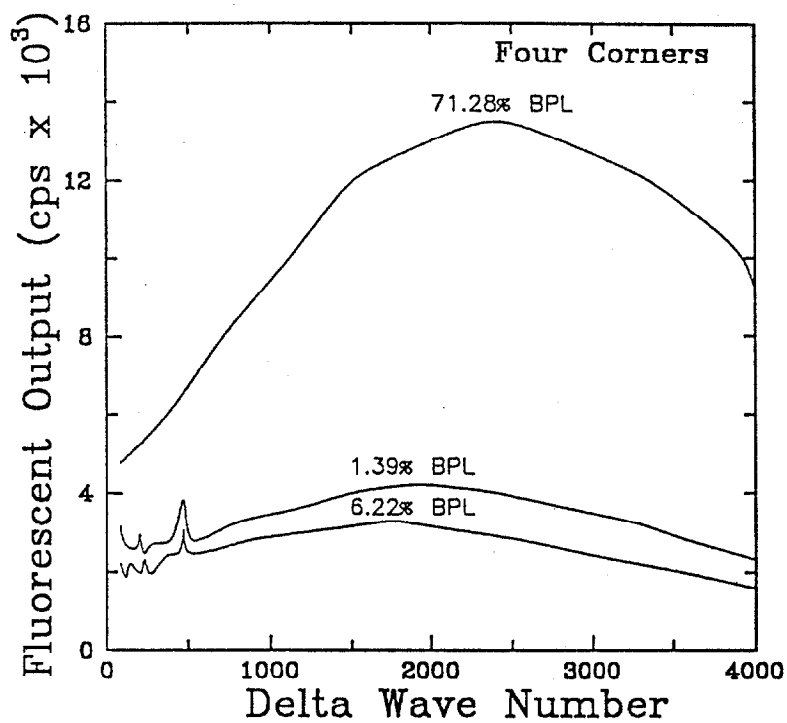


Figure 13. Laser-Raman output for Four Corners samples.

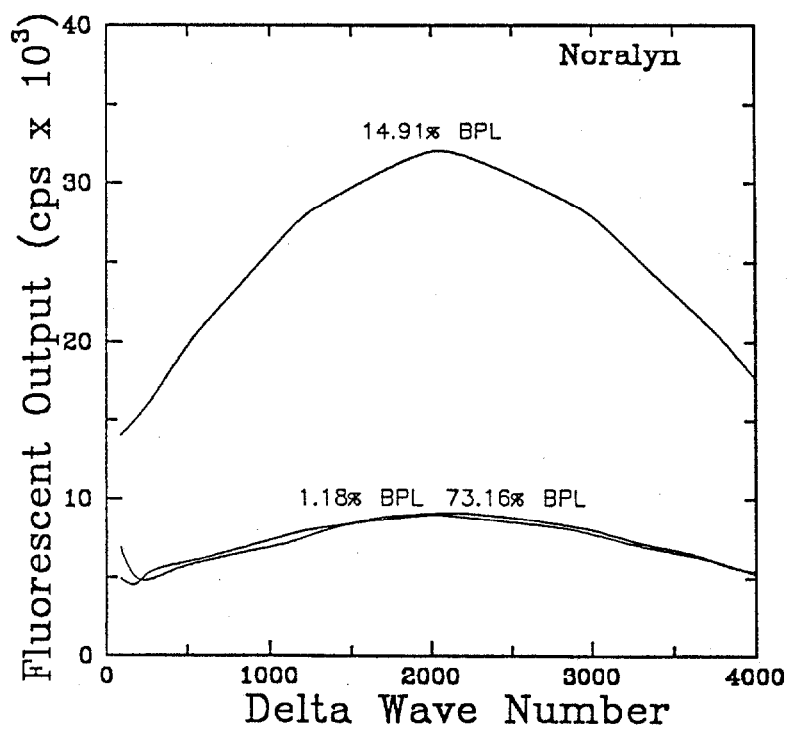


Figure 14. Laser-Raman output for Noralyn samples.

Figure 15 shows BPL content of the sample plotted as a function of the peak intensity level of the Laser-Raman output. Once again, the data tend to indicate that there is a general trend for the peak intensity level to increase as BPL content increases. In fact, if one eliminates the feed and tailings sample from the Noralyn plant (i.e, two point out of nine), it appears that those samples with BPL content less than 10% give a fluorescent output between 2000 and 6000 cps, while those samples with a BPL content greater than 70% give a fluorescent output between 8000 and 13,000 cps. It should be noted, however, that there is considerable scatter in the data, and the relationship between BPL content and fluorescent output does not appear to be independent of sample color. Part of the reason for the data scatter is the small focal spot size of the light source (approximately 50 μm) relative to the size of the particles (approximately 100 μm). This means that an individual particle has great influence over the result of the analysis. In order to minimize this size effect, three samples of Four Corners material were ground to reduce the particle size to approximately 10 μm . These samples were then analyzed and the results plotted as shown in Figure 16. As shown, the scatter is now reduced to the point where fluorescent output continuously increases with BPL content in an exponential manner.

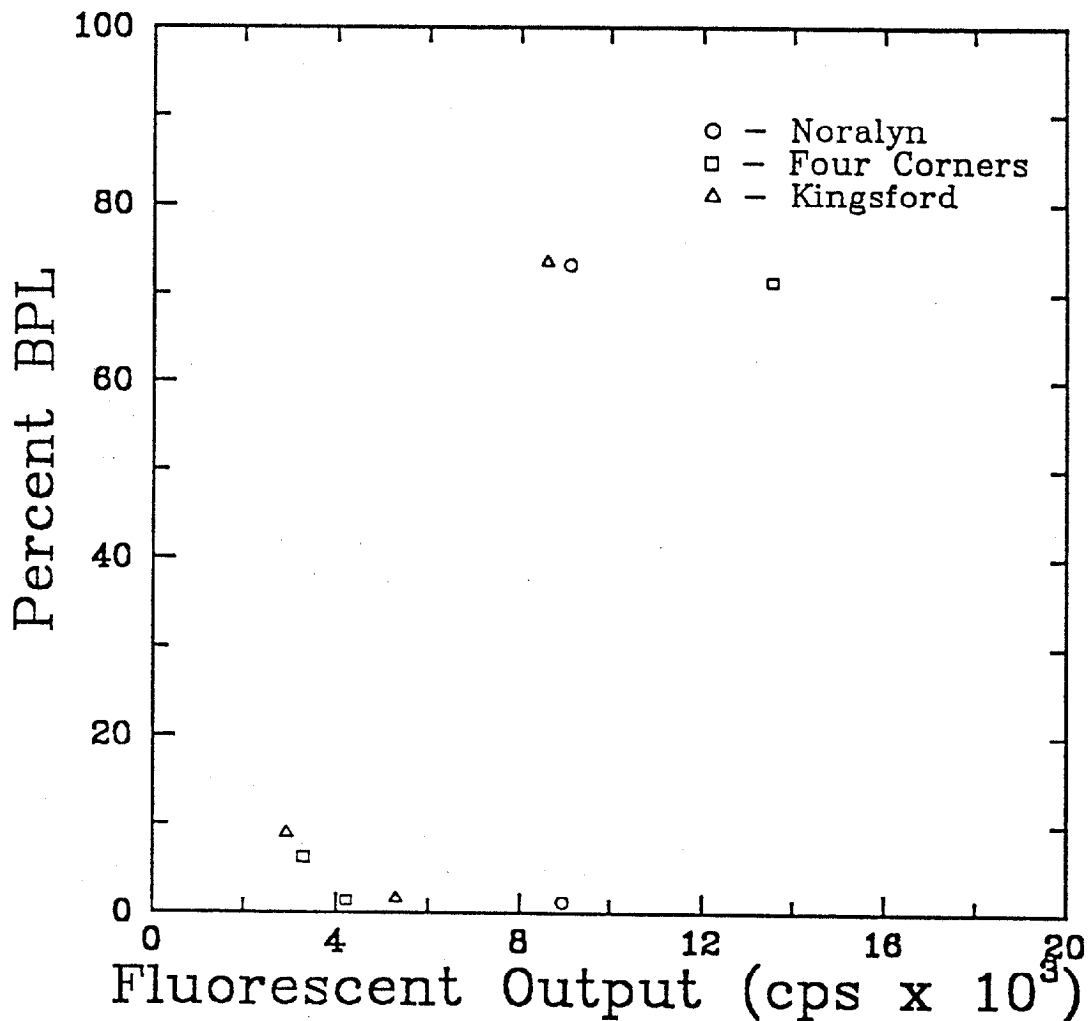


Figure 15. BPL calibration curve obtained using Laser-Raman device.

Based on this encouraging result, a detailed test program was conducted using three samples from each of the Kingsford, Four Corners and Noralyn sample groups. In this study, all samples were finely ground so that more particles would lie within the beam. Furthermore, the focusing lens was removed from the Laser-Raman device increasing the beam size to 1.3 mm. Finally, each sample was analyzed three times at different sample positions and the values averaged in order to provide a representative measurement. The results of this work are shown in Table 7 and Figure 17.

As shown, even with the expanded beam size, there is significant variation in the fluorescence power measured at various locations across the surface of a given sample. However, when the average fluorescence power is plotted, as shown in Figure 17, an exponential trend similar to that observed for the earlier analysis of the Four Corners sample is observed for the Kingsford and the Noralyn samples. Unfortunately, the new analysis of the Four Corners sample resulted in significant scatter in the data, casting doubt on the reproducibility of this technique. Perhaps the most discouraging finding of this work is the fact that the photoluminescence approach does not seem to eliminate the color effect since there are three distinct calibration curves representing each of the three plants.

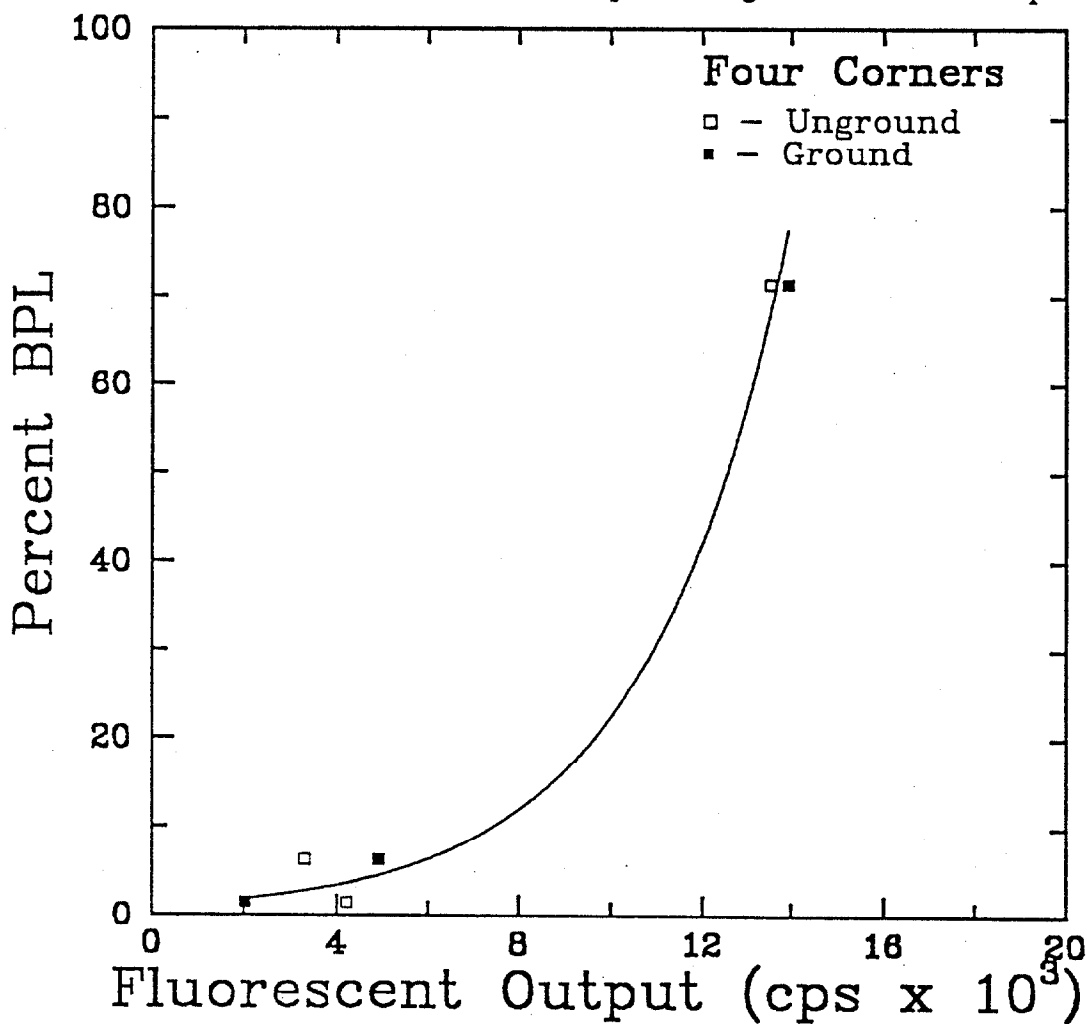


Figure 16. BPL calibration curve for Four Corners sample (ground and unground) obtained using Laser-Raman device.

Table 7. Photoluminescence Analysis of Phosphate Ore.

Sample	BPL Content	Fluorescence Power (photon/sec)	Avg. Fluorescence Power (photon/sec)
Four Corners Amine Conc. 5/23/91	72.39	3186 4040 3342	3523
Four Corners Amine Conc. 5/28/91	71.28	2135 2352 1981	2156
Four Corners Fine Feed 5/20/91	6.22	2042 2170 2107	2106
Four Corners Fine Tails 5/20/91	1.39	2047 2962 2047	2352
Kingsford Amine Conc. 5/31/91	73.65	5084 5442 5290	5272
Kingsford Fine Feed 5/29/91	9.00	3342 3470 2546	3119
Kingsford Fine Tails 5/29/91	1.82	2277 2104 1676	2019
Noralyn Amine Conc. 5/13/91	73.16	7564 7354 6492	7137
Noralyn Fine Feed 5/13/91	14.91	5730 6055 5545	5777
Noralyn Fine Tails 5/15/91	1.18	3009 2275 3516	2933

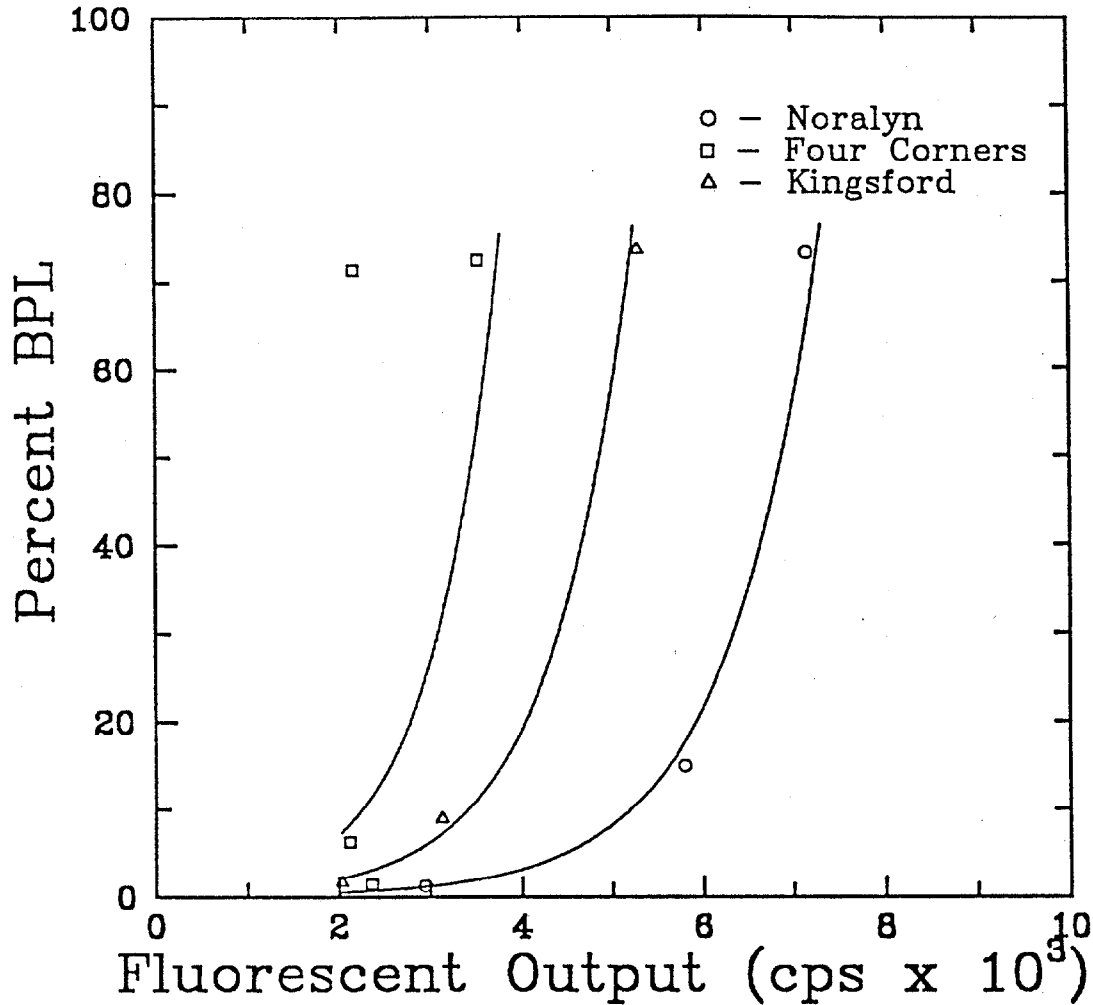


Figure 17. BPL calibration curves for photoluminescence-based sensor.

2.3.3 Summary

Test work conducted using a photoluminescence technique to determine BPL content in phosphate samples shows that, in general, there is an increase in the peak optical power from a Laser-Raman Spectrometer as the BPL content of the phosphate sample increases. Unfortunately, the sensing technique is subject to significant data scatter due to the small size of the incident light beam relative to the particle size, and due to the fact that the Laser-Raman output contains both fluorescent and reflected light. The particle size effect can be minimized to some extent by making repeated measurements on a sample or by reducing the particle size. In an on-line, application the former would probably be more practical. The combination of both fluorescence and reflectance in the output from the Laser-Raman device; however, makes it impossible to determine a BPL assay which is totally independent of the sample color. Thus, the photoluminescence technique does not appear to be a viable alternative for on-line BPL analysis.

2.4 Wet Analysis

A preliminary set of tests were conducted to determine if the reflected-light analysis technique could be adapted for wet analysis. This is a prime concern if the technique is to be used for on-line analysis. Therefore, a sample cell was constructed in which a slurry could either be suspended by a stirrer or allowed to settle against a glass measurement port. In this initial work, only the latter case was studied. The resulting calibration curve for the September 1992 Kingsford sample is shown in Figure 18. As shown, an excellent correlation is obtained between the relative BPL content of the sample and the normalized reflected intensity for the stationary wet material.

Ultimately, it would be useful to design an optical sensor that could be used on moving slurries. At present, however, the effect of variation in percent solids is unknown. It is expected that changes in percent solids will affect the reflectivity of the slurry in a manner independent of the BPL content. If this is the case, it may be necessary to incorporate an on-line density sensor to provide a pulp density correction for the BPL analysis. It may also be possible to design a sampling system which could provide a sample at a relatively constant percent solids. In any case, it appears worthwhile to pursue further development of an on-line slurry analysis system using the reflected light technique.

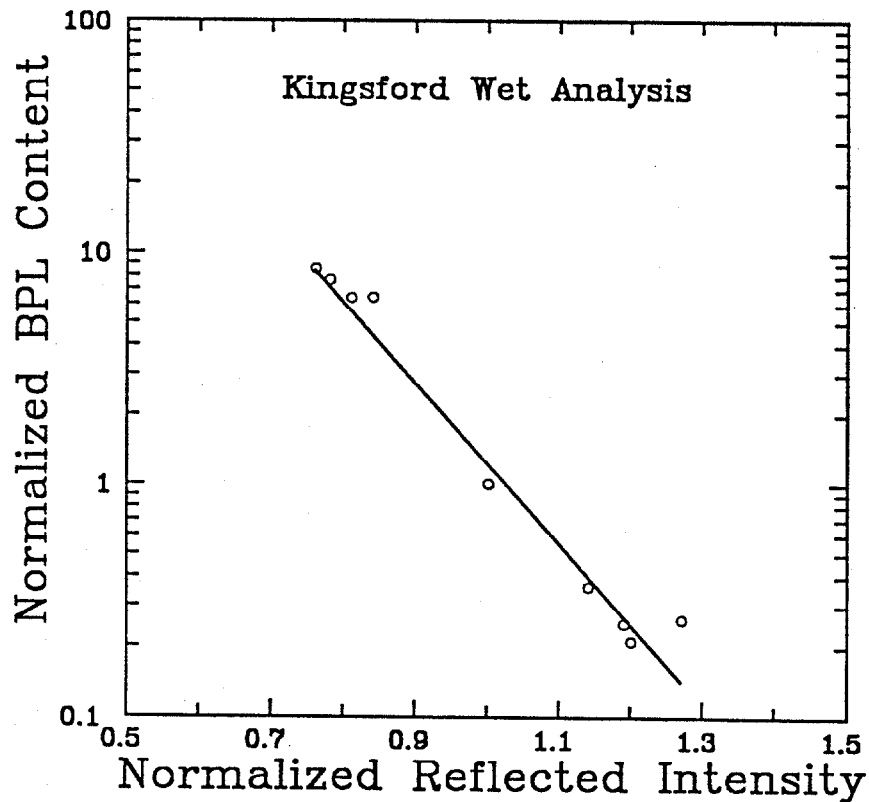


Figure 18. Calibration curve for wet analysis of the September 1992 Kingsford sample using the reflected light sensor.

3. SUMMARY AND CONCLUSIONS

A study has been conducted to investigate optical methods for rapid determination of BPL content in samples of fine flotation feed, concentrate and tailings. Two optical approaches for sensing BPL content have been considered in this work, reflected light and photoluminescence. Based on the results obtained, the following conclusions can be made.

1. Both sensing techniques provide an optical output which can be related to BPL content.
2. The photoluminescence technique is limited by the low intensity of the fluorescence output and by the difficulty in differentiating fluorescence from reflectance. Thus, the technique is not independent of phosphate color.
3. The reflected light technique in conjunction with a data normalization procedure can be used to successfully predict BPL content over a wide range of phosphate color variation.
4. A calibration curve based on samples obtained from three different IMC plants indicates that BPL content can be predicted with approximately $\pm 30\%$ relative error using the reflected light approach. This figure includes an average chemical assay variation of approximately $\pm 6\%$ and individual chemical assays varying by as much as $\pm 25\%$.
5. Although the relative error is high, the data obtained in this work and previous experience indicate that the error could be reduced substantially by increasing the number of samples included in the calibration curve and by calibrating the sensor for a single stream within a single plant.
6. Preliminary results obtained using the reflected light technique on stationary wet samples indicate an excellent correlation between BPL content and reflected intensity. Thus, the initial groundwork has been laid for adapting the reflected light technique to on-line analysis.

4. RECOMMENDATIONS FOR FUTURE WORK

Based on the results obtained in this project and in other work by the PI's on optical sensing of phosphate, the following recommendations are made.

1. **Establish a second research project to adapt the reflected-light technique to on-line slurry analysis.** The present research project has shown that the reflected-light approach in conjunction with a data normalization procedure can be used to determine BPL content in Florida phosphate samples irrespective of the color variations in the phosphate mineral. A new project should now be initiated to adapt this system to slurry analysis and determine the effects of such things as percent solids, slurry flow rate, etc. on the BPL analysis. Such test work should be confined to laboratory and pilot-scale testing to provide well controlled conditions.
2. **Establish a third research project to install and calibrate the optical analyzer at a Florida phosphate operation.** Assuming No. 1 above is successful, the optical analyzer should be tested on-site as the final stage in turning the instrument over to the industry.
3. **Install and calibrate the current video-based phosphate analyzer developed for Texasgulf (see Appendix) at a Florida phosphate operation.** Based on the fact that the present project has shown that reflected light can be used on Florida phosphate, it is proposed to test the video-based system at a Florida plant. Such a system could be ready for calibration on-site in a matter of months.

APPENDIX

Phosphate Analysis by Optical Image Processing:
Sensor Development and In-Plant Testing

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Chapter 20

PHOSPHATE ANALYSIS BY OPTICAL IMAGE PROCESSING: SENSOR DEVELOPMENT AND IN-PLANT TESTING

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ABSTRACT

A rapid, bench-top analyzer has been developed to determine P_2O_5 content and CaO/P_2O_5 ratio in phosphate ores. The analyzer utilizes a portable computer equipped with a frame grabber to measure the reflective intensity (gray level) of phosphate samples placed in front of a television camera. P_2O_5 content is related to the average gray level of the sample, while CaO/P_2O_5 ratio is determined from the amount of bright material present. The sensor is currently being used to analyze flotation concentrate samples at Texasgulf's phosphate operation near Aurora, North Carolina. Sensor development along with results from laboratory and in-plant testing are discussed.

INTRODUCTION

The phosphate industry in the Southeastern U.S. is well known as a major force in the world mineral picture with nearly one-third of the world phosphate production emanating from the states of North Carolina and Florida. Based on current reserve projections, this trend is not expected to change for many years to come (McHardy, 1983; Lawver, McClintock and Snow, 1978).

Phosphate reserves in North Carolina are concentrated primarily along the Atlantic Coastal Plain. They differ from Florida reserves in that they are generally deeper; however, the uniformity of the deposit tends to make extraction more efficient (Davis, Llewellyn and Sullivan, 1986). Furthermore, the reagent dosages required to upgrade these reserves by froth flotation are usually less than required for Florida phosphate, while the phosphate lost during fine clay removal is about one-tenth of that associated with the Florida ores. On the other hand, North Carolina phosphate concentrates generally contain 30 - 31% P_2O_5 as compared to the average Florida grade of around 33%. North Carolina concentrates also have a slightly higher CaO/P_2O_5 ratio (calcium ratio), requiring additional sulfuric acid in the phosphoric acid production step (Redeker, 1966).

As a result of the inherently lower final concentrate grade and higher sulfuric acid consumption associated with the North Carolina phosphate, accurate process control is essential for maintaining product quality. Process control systems have been in place in the mineral processing industry for nearly 30 years, and the benefits of such systems are well documented. Process control systems for the phosphate industry have been proposed (Davis, Jordan and Stanley, 1990); however, the phosphate industry lacks appropriate instrumentation to implement such schemes. Specifically, a need exists for a suitable means of providing rapid analysis of P_2O_5 grade and calcium ratio in process streams around the flotation circuit. At present, phosphate grades are either determined in a qualitative manner by inspection (i.e., through the difference in color between the dark phosphate minerals and the light gangue minerals), or in a quantitative manner by wet chemical analysis. In the latter case, up to four hours of digestion and analysis may be required to generate an assay. Some common on-line analysis techniques such as X-ray fluorescence and neutron activation have been tested on phosphate ores, but they are generally inaccurate on materials of such low atomic number as calcium and phosphate. On-line nuclear magnetic resonance (NMR) has shown some promise in determining P_2O_5 content in slurry streams; however, this technique is still unable to provide information on the calcium ratio (King and Cooper, 1990).

The purpose of this paper is to describe the development and testing of an optical sensor for determining P_2O_5 content and calcium ratio in phosphate ores. The sensor utilizes image processing technology to provide rapid, off-line analyses of dry flotation concentrate samples. Details of the operating principles, equipment configuration, and testing and calibration are discussed.

BACKGROUND

Phosphate Processing

Preparation of Flotation Feed. Run of mine ore is hydraulically transported to the concentrator by two matrix pumping systems. Each pumping system provides the concentrator with up to 300 kg/s (1,200 tph) of ore matrix assaying approximately 14% P_2O_5 . Processing begins at the washer building with two consecutive stages of screening to remove the coarse oversize (reject) material. The first screening stage is comprised of three scalping screens which reject all material coarser than 25 mm (1 in). The undersize from each scalping screen reports to five second stage screens where a separation is made at 1.7 mm (10 mesh). The plus 1.7 mm (10 mesh) and plus 25 mm (1 in) rejects are combined and conveyed to a stockpile. The typical assay of the coarse rejects is 18 to 22% P_2O_5 with a CaO/P_2O_5 ratio of 1.9 to 2.4.

A series of four clay removal steps are performed in the feed preparation circuit prior to anionic flotation. The minus 1.7 mm (10 mesh) ore is combined and blended in one agitated mix tank prior to the first stage of clay removal. Eight pumps are then used to feed four separate clay removal circuits. In each of the four circuits the first stage of clay removal is comprised of six 0.6 m (24 in) hydrocyclones. Overflows are discarded as clay fines and underflows report to a static surge tank. Ore from the surge tank is cycloned in a single 0.9 m (36 in) cyclone with the overflow again discarded. The underflow then reports to a set of high attrition scrubbers and to an agitated storage tank. The ore is pumped from the agitated storage tank to four 0.5 m (20 in) cyclones. Overflows are again discarded and underflows report to a pair of double screw classifiers. The classifiers are the last step in the clay removal process prior to fatty acid flotation. The classifier sand becomes fatty acid flotation feed while the overflows are discarded as clay. The feed to the fatty acid flotation circuits is approximately 17% P_2O_5 and is essentially plus 74 μ m (200 mesh). The combined P_2O_5 content in the feed preparation fine clay stream is 3 to 4 percent and is primarily caused by tramp oversize losses from the hydrocyclones.

Fatty Acid Flotation. The fatty acid (anionic) flotation circuit is comprised of four individual circuits. The ore is treated with reagents in a series of four conditioners with pre-saponified anionic tall oil and #2 fuel oil prior to flotation. The phosphate values are floated as fatty acid concentrate and the sand tailing is discarded for use in land reclamation. The fatty acid concentrate (28% P_2O_5) is de-oiled with acid waste water and sulfuric acid through a series of four scrubbers. This fatty acid concentrate is the feed source for all phosphoric acid production within the plant site.

Amine Flotation. De-oiled fatty acid concentrate from two of the four circuits is pumped to two separate amine (cationic) flotation circuits. The concentrate is classified with a pair of hydrocyclones and a single spiral classifier in two successive steps to remove the clay fines generated during fatty acid flotation and de-oiling. Amine acetate and #2 fuel oil are added in the flotation cells to remove the silica by differential flotation. The resulting amine concentrate is sampled and analyzed with the optical analyzer for both P_2O_5 content and calcium ratio. Amine concentrate specifications require a P_2O_5 content greater than 30.20% and a calcium ratio less than 1.620. The optical analyzer is used routinely to bring the amine concentrate assays into specification prior to depositing the concentrate in an open air stockpile.

Optical Sensing

long before automatic analysis techniques or even chemical analysis techniques were developed, the human eye was used as the primary means of determining mineral composition, grade, particle size, etc., in mineral processing operations. Even today, it is not uncommon for a skilled operator to control a flotation circuit on the basis of froth color. On the other hand, optical instrumentation has only been in use in the mineral processing industry since the early 1970's.

One of the earliest forms of optical instrumentation was the optical ore sorter. In an optical ore sorter, laser light is reflected off the surface of coarse ore particles as they fall off the end of a belt. A rough estimate of the grade of these particles is determined from the intensity of the reflected light, and gangue particles are rejected by an air blast from a compressed air source. These devices typically operate on 30-150 mm particles (Wills, 1988).

More recently, Nelson and Oblad (1989) described a device for monitoring the ash content in a coal flotation refuse stream by means of reflected light. In this device, laser light is transmitted along a fiber optic cable to a canister which receives refuse slurry from a bypass line out of a refuse discharge box. Backscattered light from the refuse slurry is transmitted to a photoconductor and the signal is taken to be proportional to the ash content of the stream. An ultrasonic transducer is used to prevent deposits from forming on the end of the fiber optic cable.

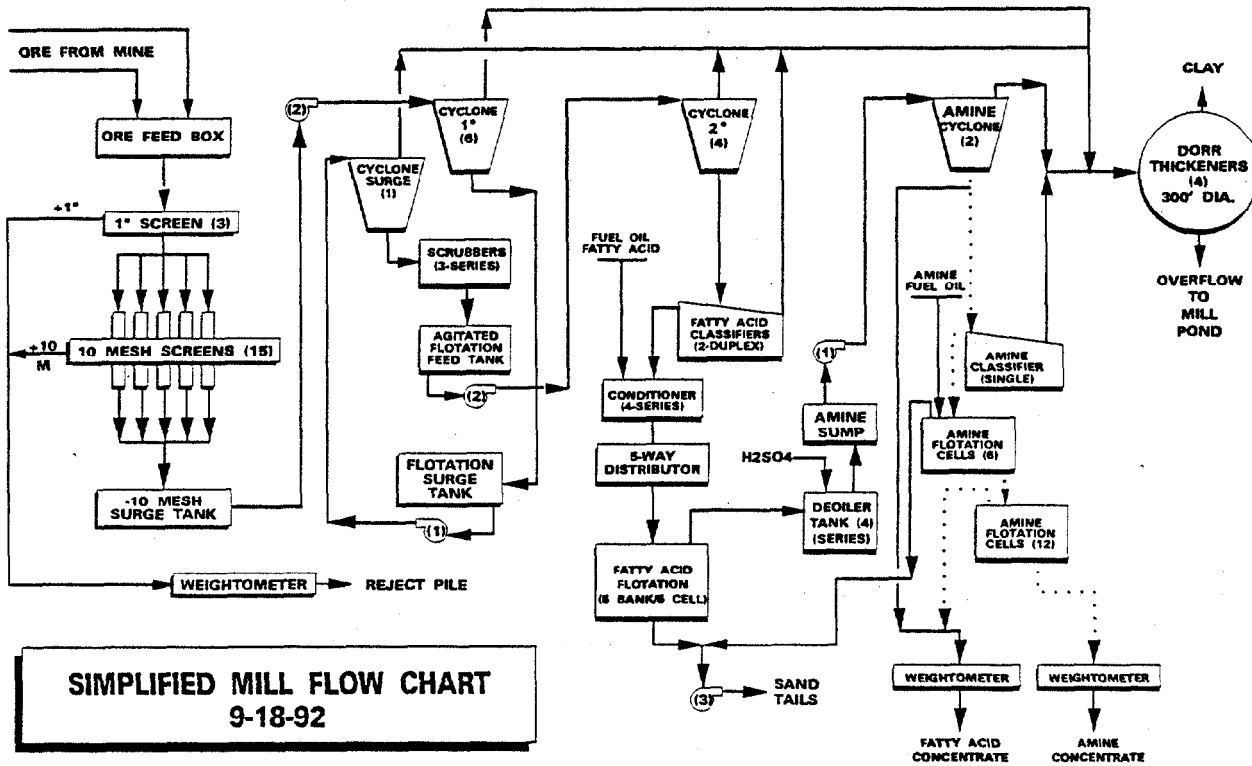


Figure 1. Texasgulf's phosphate flotation flowsheet.

In another recent publication, Gebhardt and Ahn (1992) described the use of a commercially-available color sensor as a means of monitoring froth color. The device was tested on froth products containing chalcopyrite, pyrite and molybdenite, and froth color was correlated to froth composition. Although the work was preliminary, the device appeared to show potential as an on-line analysis tool.

Image analysis systems are another form of optical instrumentation that can be used to determine quantitative information from images acquired via some form of video input source. Under image analysis, an image is presented to the system, digitized and stored in a computer. During the digitization process, the various shades of gray that comprise the image are given values ranging from 0 to 255 where 0 indicates black and 255 indicates white. These shades of gray are known as gray levels. An entire image may cover a wide range of gray levels and the frequency of occurrence of each of these gray levels can be seen in a gray level histogram. The gray levels can be manipulated in a variety of ways to provide quantitative information about the image.

In the late 1970's and throughout the 1980's, image analysis systems became commonly used in mineral processing as a means of characterizing particle mineralogy, size, shape, texture and degree of liberation. Recently, O'Kane et al. (1990) described preliminary test work using an image analysis system to determine the grade of a phosphate tailing. In this work, individual grains of a phosphate tailing sample were placed on a red background and the grains were photographed. The photograph was then digitized and the image was segmented into apatite, quartz and background using an image analysis system. Assays of each component were determined from the area of each phase present. Although the analysis of individual mineral grains may not be practical in a plant environment, this application demonstrates how image analysis could be used to determine the assay of a phosphate ore.

The original work leading to the development of the sensor described in this paper was carried out in 1987. Unlike the procedure used by O'Kane et al., no attempt was made to separate individual mineral grains. Samples of flotation concentrate, feed and tailing were obtained from

Texasgulf and mixed together in various quantities to provide a range of P_2O_5 grades for analysis. The mixtures were spread out on a white piece of paper and placed in front of a television camera. An appropriate gray level was chosen to separate the dark phosphate minerals from the light gangue minerals and the image was segmented at this gray level. From this segmented image, the area percent of the dark phase contained in the overall image was determined and plotted versus the actual P_2O_5 content of the sample. The resulting plot is shown in Figure 2. As shown, an excellent correlation was obtained between the area percent of the dark phase and the P_2O_5 content of the mixtures. Based on this result, a project was set up in 1991 between VPI&SU and Texasgulf to develop a bench-top unit for rapid analysis of phosphate ores. The following is a description of the sensor development.

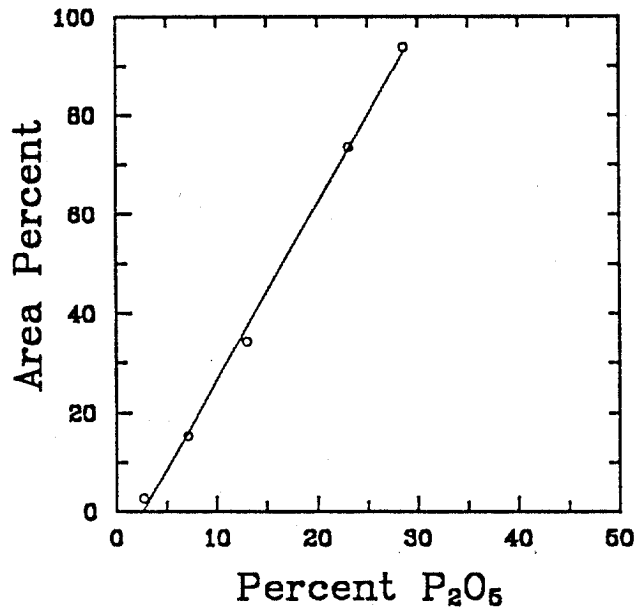


Figure 2. Relationship between area percent of dark phase and P_2O_5 content for dry mixtures of phosphate flotation feed, concentrate and tailing.

SENSOR DEVELOPMENT

Equipment

The various components which make up the optical phosphate analyzer are shown schematically in Figure 3. These components consist of a 25 Mhz personal computer with frame grabber, a black-and-white CCD television camera, a color video monitor, a fiber optic illuminator and a 23 x 23 x 18 cm (9 x 9 x 7 in) gray PVC sample box. As shown in the overall schematic (Figure 3a), the television camera is connected to the video input port of the frame grabber while the color monitor is attached to the video output port. The light which provides constant illumination for the sample is controlled by the fiber optic illuminator which is powered through a signal conditioner. The signal conditioner is used to ensure that line voltage fluctuations do not cause fluctuations in the illumination of the sample. Uniform sample illumination is provided by a ring light mounted around the lens of the television camera as shown in the detailed schematic (Figure 3b). The intensity of the incident light is monitored by a photocell mounted in the back wall of the sample box and connected to a digital multimeter. The setting on the fiber optic illuminator is adjusted until a prearranged light intensity is achieved as determined from the digital multimeter. Adjustments are made to the light intensity as needed to maintain constant illumination of the sample.

Measurements are made by placing a dry sample of flotation concentrate in a 7 cm (2.75 in) diameter watch glass. The watch glass is placed under the television camera and the sample box door is closed. The light setting is adjusted as needed and the image is digitized. The resulting gray level information is processed by the computer and the P_2O_5 content and calcium ratio are displayed on the computer screen.

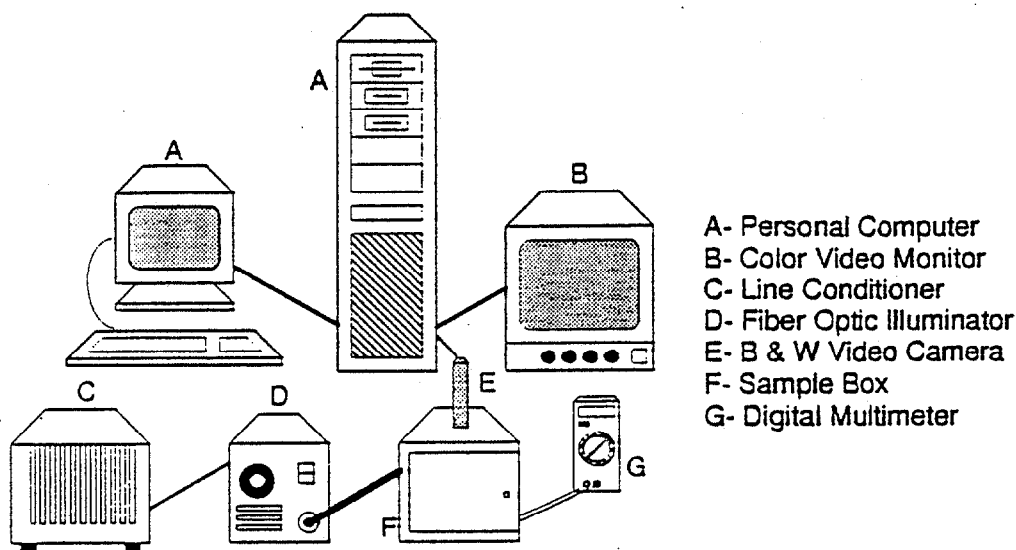


Figure 3a. Overall schematic.

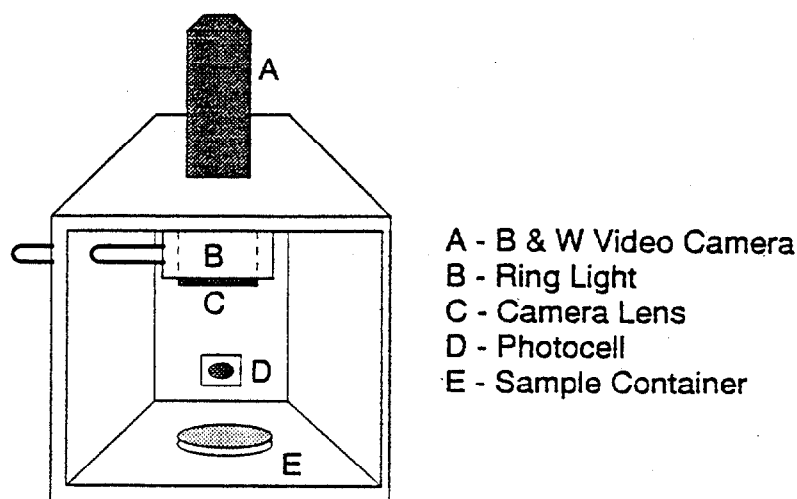


Figure 3b. Detailed schematic of sample box.

Figure 3. Schematic diagrams of optical phosphate analyzer.

Software

Processing of the images collected by the television camera is carried out using the OPTIMAS software package marketed by BioScan Inc (1991). This package runs under the Windows environment and provides a complete system for image measurement and analysis. Image analysis can be carried out interactively using built-in functions or batch applications can be written using the C-language-capabilities of the system. The latter feature was used in this exercise to provide a user-friendly application for use by a plant operator with no knowledge of image processing.

The software developed for the optical phosphate analyzer is illustrated in Figure 4. The system is set up such that the computer immediately comes up with the screen shown in Figure 4a. After the operator clicks on O.K., a prompt appears requesting the stream to be analyzed (Figure 4b). This information is used by the program to locate the appropriate calibration data. The operator is then asked to insert a sample into the sample box and the analysis is carried out. If the operator chooses, several samples may be analyzed and the results averaged to reduce sample bias. Alternatively, the P_2O_5 content and calcium ratio from a single sample can be displayed. Finally, the assay information is displayed (Figures 4c) and an option is provided which allows the operator to log this information with an appropriate label. The entire program runs in under 15 seconds per sample or in under 2 minutes for an average of five or six samples.

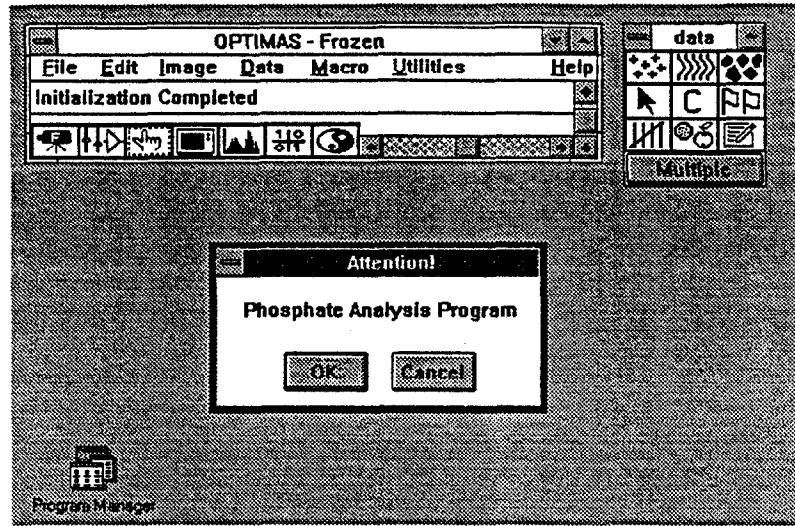


Figure 4a. Opening screen.

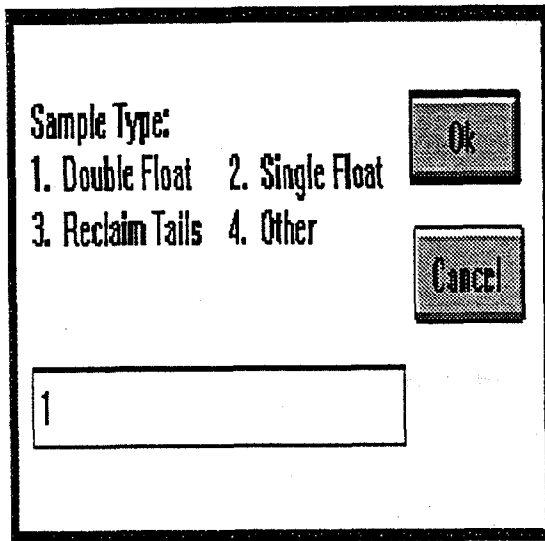


Figure 4b. Sample Type.

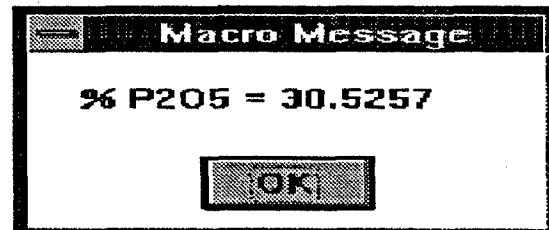


Figure 4c. Assays.

Figure 4. Phosphate analysis program examples.

Calibration and Operation

The optical phosphate analyzer makes its measurements in terms of gray level. These measurements must be converted to P_2O_5 content and calcium ratio in order to provide useful information to the plant operators. The basis for carrying out this conversion is illustrated in Figure 5 which shows the typical gray level distributions obtained for four different samples; The samples, in order of increasing content, consist of amine tailing, flotation feed, fatty acid concentrate and amine concentrate. As shown, the gray level distributions become narrower and shift to lower gray levels as the P_2O_5 content increases. This is due to the increasing amount of dark phosphate mineral present in the sample and the reduction of light-colored calcite and quartz. It is also interesting to note that there is a small peak in the gray level distribution which occurs at a gray level around 250 (see the inset in Figure 5). This peak corresponds to the nearly white calcite particles which appear in each sample. As shown, the peak is highest for the amine tailing, slightly lower for the flotation feed and lowest for the fatty acid concentrate. The calcite peak is found to increase slightly for the amine concentrate as quartz is rejected from this material and the calcite content increases.

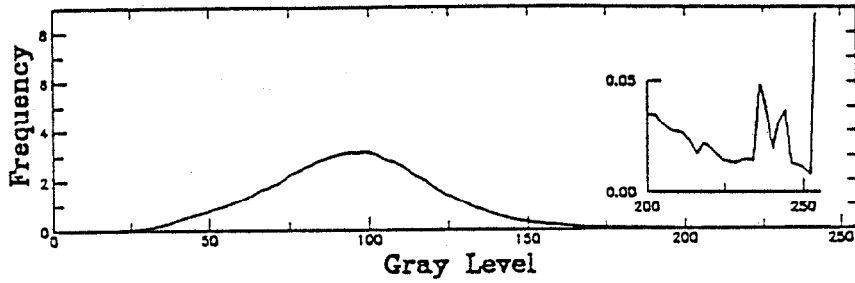


Figure 5a. Amine tailing.

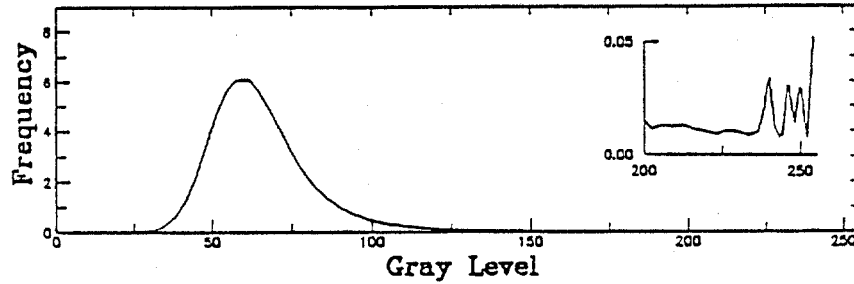


Figure 5b. Flotation feed.

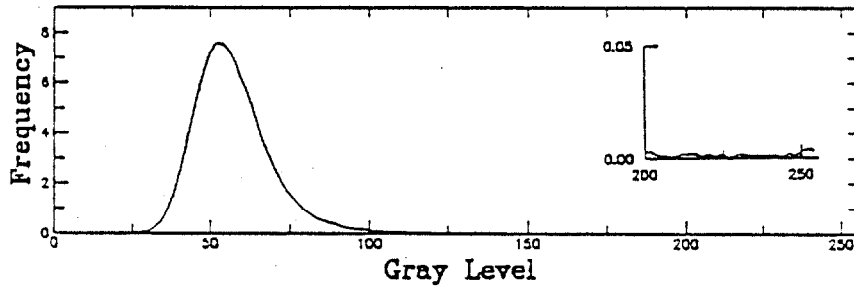


Figure 5c. Fatty acid concentrate.

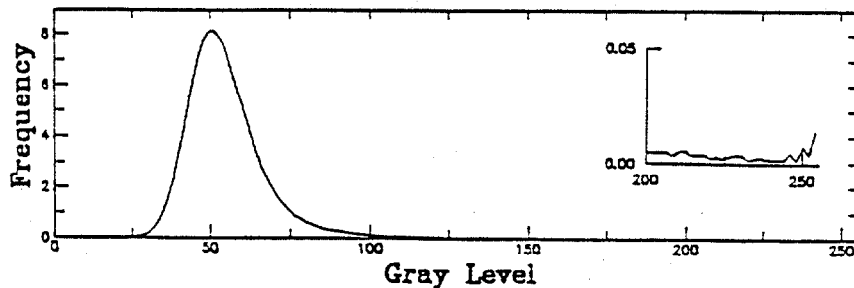


Figure 5d. Amine concentrate.

Figure 5. Gray level distributions for various phosphate samples.

Based on the information obtained from the gray level distributions, it was decided to correlate P_2O_5 content to mean gray level and calcium ratio to the area percent of the image with gray level greater than 210 (i.e., the area under the calcite peak). The correlation between mean gray level and P_2O_5 content for the entire range of P_2O_5 values is shown in Figure 6. As shown, the expected trend in the data is evident over the entire range of P_2O_5 values. However, unlike the relationship shown in Figure 2, the trend is not perfectly linear. This finding is very important since it illustrates the fact that sensor calibration curves developed from mixtures may not be accurate if the mixture composition does not occur as part of the normal processing of the ore.

As a result of the information shown in Figure 6, it was determined that a single calibration curve for all streams would not be sufficient to provide the necessary accuracy. Therefore,

individual calibration curves were developed for each stream. Due to the fact that fatty acid and amine concentrates were of primary interest, the major effort was focused on tuning the sensor for these two streams. Typical calibration curves obtained for these streams are shown in Figures 7 and 8 along with their 90% confidence intervals. As shown, the calibration curve for the amine concentrate has a tighter confidence interval and a slightly steeper slope than that for the fatty acid concentrate indicating that it is easier to distinguish small changes in P_2O_5 content with greater accuracy for the amine concentrate. On the other hand, the accuracy on the fatty acid concentrate is quite good considering the range of assays over which the data were collected.

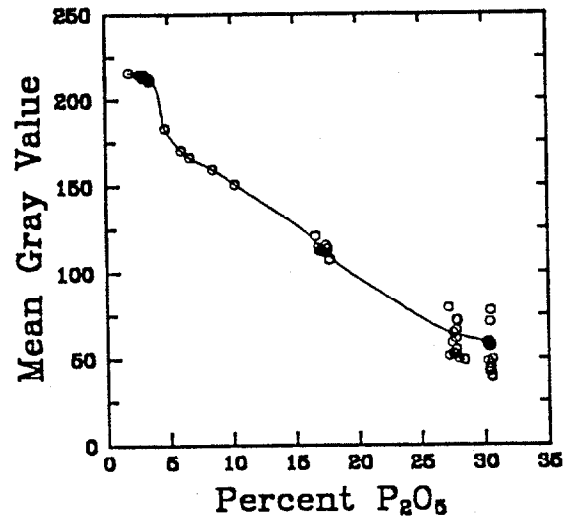


Figure 6. Relationship between mean gray level and P_2O_5 content for Texasgulf phosphate samples.

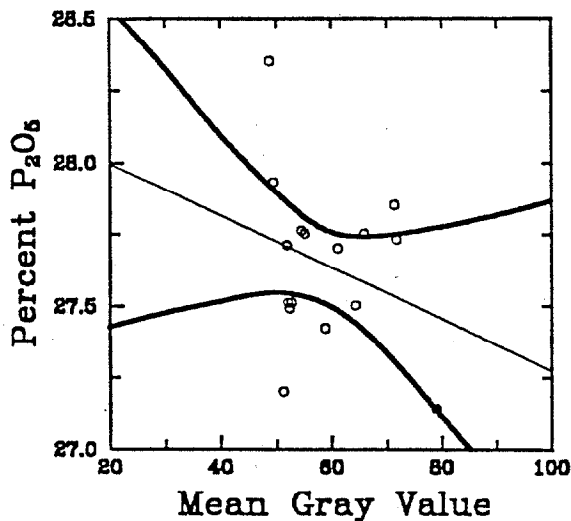


Figure 7. Fatty acid concentrate calibration curve.

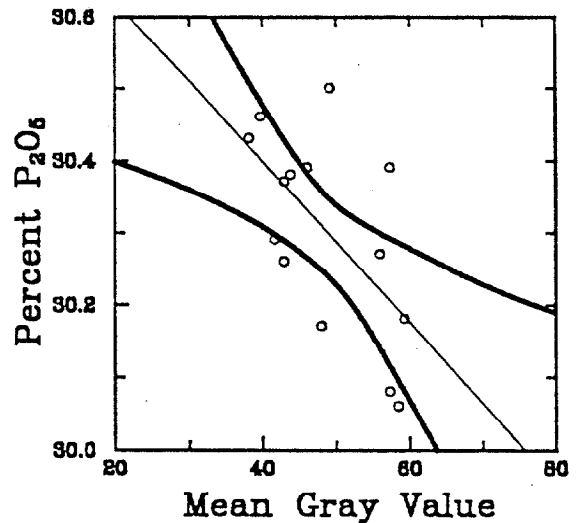


Figure 8. Amine concentrate calibration curve.

In the case of the calcium ratio, a detailed sampling and analysis study indicated that a single calibration curve could be used for both fatty acid and amine concentrates. An example of this calibration curve is shown in Figure 9. Although the data exhibit considerable scatter, a linear trend is clearly evident. Considering the expanded calcium ratio scale used in this plot, the accuracy of the analysis is quite good and well within the range desired by Texasgulf.

All calibration curves were entered into the phosphate analysis program using a cubic spline algorithm. Although linear relationships were used for all calibrations, the spline algorithm provided additional flexibility if a non-linear relationship were ever required. A final summary of the accuracy of the optical phosphate analyzer is shown in Table 1.

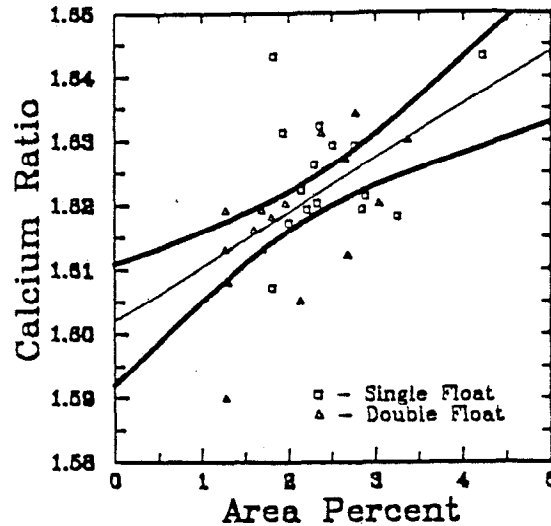


Figure 9. Calcium ratio calibration curve.

Table 1. Accuracy chart for optical phosphate analyzer.

Stream	Assay (Based on 90% confidence interval)
Fatty Acid Concentrate (P_2O_5)	$27.6 \pm 0.14 \%$
Amine Concentrate (P_2O_5)	$30.3 \pm 0.05 \%$
Amine or Fatty Acid Concentrate (Ca Ratio)	1.62 ± 0.003

IN-PLANT TESTING

The optical analyzer was introduced to the concentrator in December 1991. Prior to this time, control of P_2O_5 grade and calcium ratio for the amine concentrate was accomplished by sending samples at four hour intervals to the quality lab. Wet chemical analyses were conducted on these samples using colorimetric methods to determine P_2O_5 and CaO content. The assays were then used to compute the corresponding calcium ratio. Total turnaround time was typically two to four hours. This time was eventually reduced to 20 minutes with the introduction of the optical analyzer.

The software package developed by VPI&SU allowed flexibility in assaying by providing methods for both wet and dry analysis. Wet samples of flotation concentrate were prepared by vacuum filtration to create a filter cake. The filter cake was then packed into special sample cups for optical analysis. The wet method was preferred by flotation operators since it required only 10 minutes to complete. Unfortunately, assays obtained using the optical analyzer and the quality lab in January 1992 demonstrated that there were significant discrepancies between the two methods. Sample preparation for the optical analyzer was found to be highly variable in terms of moisture content and compactness. Thus, the wet method was abandoned in February 1992 in favor of the dry method. Dry analysis samples are prepared by collecting approximately 450 g (1 lb) of wet concentrate. The sample is dried in a microwave oven and poured into four to six laboratory watch glasses. Drying time is determined by wet sample weight and typically requires eight to ten minutes. Although analysis time was doubled by conversion to the dry method, accuracy was improved to the point where the flotation operators were beginning to rely on the analysis information.

Throughout the months of March and April, the optical analyzer calibration was steadily improved by correlating optical assays to those obtained from the quality lab. Assay discrepancies were also improved through procedural changes in sampling, sample preparation and duplicate analyses of control samples by the quality lab. In May 1992, the optical analyzer was considered reliable and became the primary method for measuring calcium content in the amine concentrate. Comparative analyses of the calcium ratios obtained from the analyzer and the quality lab indicated a deviation between the two of 0.014. This difference was reduced to 0.009 in June and 0.006 in July.

Calibration of the optical analyzer for P_2O_5 determinations has been more difficult because of color variations in the ore. Phosphate staining of the silica and quartz in the amine concentrate is suspected to be the major cause of color variations. These stains darken the silica and confound the assay determination. Comparative analyses of P_2O_5 assays obtained from the optical analyzer and the quality lab indicated a deviation between the two of 0.29% in May. In June the difference was 0.32% and in July 0.19% as the ore matrix and staining characteristics changed from month to month. On-line calibration software is being developed to reduce this problem.

SUMMARY AND CONCLUSIONS

1. An optical phosphate analyzer has been developed for providing rapid, off-line analysis of P_2O_5 content and calcium ratio in process streams around a phosphate flotation circuit.
2. The analyzer is based on a simple image processing system built around a personal computer.
3. A customized, Windows-based software package has been developed to provide a user-friendly environment for the plant operators.
4. The system is currently calibrated for analyzing fatty acid and amine concentrates although the capabilities exist to calibrate for other streams,
5. The sensor is currently being used on a routine basis at the Texasgulf phosphate facility near Aurora, North Carolina. The accuracy of the device is comparable to wet chemical analysis.

ACKNOWLEDGEMENTS

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