

## DEPARTMENT OF MINES AND TECHNICAL SURVEYS MINES BRANCH

## INSTRUMENTATION IN THE CYANIDATION PROCESS

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EXTRACTION METALLURGY DIVISION

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# Instrumentation in the Cyanidation Process

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#### ABSTRACT

It is believed that many of the problems encountered in the operation of gold cyanidation plants are difficult to resolve due to the lack of sufficient and reliable information regarding the variations and actual values of operating variables at any given time. The application of instruments to monitor the more important variables in the cyanidation process should result in more efficient plant operation and a better understanding of the process. The use of pH and conductivity sensing devices for alkalinity measurements, a monitor to determine the dissolved oxygen content of plant solutions, a continuous cyanide titrator and sonic control of grinding circuits are discussed. Although the discussion is oriented toward the cyanide process, much of the paper is applicable to other ore treatment operations.

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#### Introduction

I T is known that in Canada today the process industries in general are making increasing use of instrumentation and automatic control. That the mineral industries are taking part in this development is indicated by statistics supplied by the Industrial Instruments Manufacturing Association (1), which show that 2 per cent of the new investment in machinery and equipment by the Canadian metals and ceramics industries in 1964 was spent on instrumentation as compared with 2.6 per cent for all industries. Whether this expenditure is sufficient or not is open to argument, but it is safe to say that few, if any, of the Canadian cyanidation plants, most of which were built many years ago, have spent even this much on instrumentation.

As the current practice of cyanidation in Canada apparently is lagging behind the trend toward the application of modern instrumentation and control techniques, it seems reasonable that cyanide plant operators should give some consideration to the following questions:

- 1. Is there evidence that instrumentation and automatic control applied to the cyanidation process would improve the operation, and, if so, to what variables should it be applied?
- 2. Are instruments available to monitor the important variables, and what difficulties can be encountered in using them?
- 3. Would the application of instrumentation improve the process to the extent that the cost of the instruments involved would be justified?

Because of the lack of practical experience with the use of instruments in the cyanidation treatment of gold ores, it is not possible to answer these questions positively at the present time. However, we now have a fund of information that can assist the gold mill W. A. Gow

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operator in assessing the value of applying instrumentation to his particular operation.

The extraction of gold from an ore by an alkalinecyanide solution is influenced by: the mineralogical composition of the ore; the size distribution of the ground ore; the cyanide, lime and oxygen concentrations in the plant solutions; the concentration of contaminants in the plant solutions (which is related to all the aforementioned variables); and the contact time during agitation(2). Of these variables, those that can be controlled by the operator involve the grind and reagent concentrations, including the dissolved oxygen content of the solutions.

There is ample evidence, from plant and laboratory experience, that for any given ore there is an optimum combination of grind and cyanide, lime and oxygen concentration that will produce the best "cost plus tails" results, with the agitation time available at the plant, and that relatively small deviations from these optimum conditions can affect the plant's efficiency appreciably. It is, therefore, a major objective of the cyanide plant operator to determine the values of these four variables that are best suited to his ore, and then to maintain them as closely as possible. Reliable data gathering and control procedures will allow him to meet this objective; thus, the question arises as to whether the present practice of controlling the important variables serves this purpose.

That the control methods presently used in Canadian cyanidation mills (which depend on manual measurement and adjustment of the control variables by the plant personnel) do not provide the type of control required for efficient operation is shown in Figure 1. This illustrates, for several randomly picked Canadian cyanidation plants, how lime and cyanide concentrations varied with time at key control points, and also how the grind varied with time at one plant. It can be seen that at every one of these plants at least one of these important variables deviated 20 per cent or more from the average value over both short and long periods. There are no data shown in Figure 1 for oxygen concentrations in the plant solutions, because the complexity of the manual procedure for analyzing dissolved oxygen discourages the manual measurement of this variable.

To sum up, it is apparent that the proficiency of manual plant control techniques leaves considerable room for improvement in maintaining control variables within close tolerances. Because of this, it is certainly worth considering the use of instrumentation and automatic adjustment to obtain closer control with a resulting improvement in plant efficiency. Regarding the variables to be automatically controlled, it is suggested that first consideration be given to those that influence the gold extraction: grind, and lime, cyanide and oxygen concentrations (2).



Figure 1.-Plant Conditions Observed with Plants under Manual Control.

#### **Measurement of Lime Concentration**

The first problem in the instrumentation and automatic control of a process is, of course, that of obtaining an instrument that will measure, or indicate in some way, the value of the variable under consideration. The instrument chosen must be compatible with the process characteristics, it must provide an accurate and reliable indication of the variable being measured, and it must give trouble-free performance. It is important, therefore, when considering the application of instrumentation, to understand in a general way how the available instruments operate, and the conditions that might affect their efficient operation. If instrumentation is applied without this understanding the results may be disappointing, and further experiments in the field of instrumentation and control will be discouraged. In this paper, therefore, the discussion of the instruments now available for measuring and indicating lime, cyanide and oxygen contents in plant solutions and for controlling grinding circuits will include some explanation of the operating principles involved and comments on their limitations.

The lime concentration of a cyanide plant solution is simply a means of expressing the alkalinity of the

solution, a property which can be expressed equally well by the solution's pH value. Consequently, an obvious means of measuring the lime concentration is by the use of pH electrodes (3). Two electrodes are used together-the glass electrode and the reference electrode. As shown in Figure 2, the glass electrode consists of a wire coated with silver chloride and immersed in a buffered silver chloride solution within a glass envelope; the reference electrode consists of a silver wire coated with silver chloride and immersed in a saturated potassium chloride solution. The glass electrode has a pH-sensitive glass membrane tip. When this assembly is immersed in a solution, a potential proportional to the pH is developed across the glass membrane. The reference electrode produces a stable potential against which the changing potential of the pH electrode is measured. The necessary liquid junction between the reference electrode and the sample stream is effected by means of an orifice-type liquid junction. The glass electrode is temperature sensitive, but this effect can be controlled by a temperature compensator.

One point that must be kept in mind when considering pH measuring instruments is the logarithmic relationship between pH and hydrogen ion concentration. Figure 2 shows the pH plotted against lime concentration; it can be seen that the pH value of the solution is more sensitive to changes in lime concentration at low lime concentrations than at high concentrations (2).

With this brief review on pH and the method of measurement, it is possible to comment on the applicability of the pH electrodes to the measurement of the alkalinity of cyanide plant solutions. The obvious advantage of using pH electrodes is that the property

of the solution of interest is measured directly. On the other hand, as shown in Figure 2, at lime concentrations of over 1.0 lb./ton, a relatively large increase in lime concentration is required to produce a small increase in pH; consequently, at the higher lime concentrations, pH electrodes may not provide a sufficiently precise reading to allow the alkalinity to be controlled within the tolerable limits. Also important is the fact that the connection between the two electrodes through the test solution is controlled by the contacts of the electrodes within the solution. If either of these contacts becomes fouled (owing to the precipitation of salts from the plant solutions), the electrodes can be rendered inoperative. It is common in gold cyanidation plants to have calcium carbonate or calcium sulphate precipitating out of solution; in such plants, there would probably be difficulty in attempting to use pH equipment. Also, the electrodes must be handled carefully, as the glass membranes are fragile.

From these remarks, it may be concluded that pH electrodes can be used successfully for controlling the alkalinity or lime concentration of cyanidation plant solutions where the lime concentration is below about 0.75 lb. CaO/ton, and where there is little tendency for the precipitation of salts. 100

Another instrument that can be used to provide a measure of the lime concentration in cvanidation plant solutions is the electrodeless conductivity cell\* (4). Figure 3 is a sketch of a cell of this type with which studies have been conducted at the Mines Branch, The cell consists of two toroidal coils, one having 12 turns of wire around it, the other 5000 turns. The coils are mounted in a cylindrical container made of stainless steel and filled with silicone rubber to insulate the coils from each other. The container has a 0.5-inch bore through the center, the wall of which is made of an insulating material, in



\*An electrodeless conductivity cell is manufactured by Industrial Instruments Inc., New Jersey, and marketed by Lisle Instrument Systems Ltd., Toronto.



Figure 2.-

(a) Graph of pH vs. Lime Concentration. Figure 3.—Sketch of Conductivity Cell for Electrodeless
 (b) Sketches of pH electrodes. Conductivity Meter.

this case, Teflon. The cell, which is 3.5 inches in diameter by 3 inches high, is supported by a long tube that also serves to shield the leads to the toroidal coils from moisture. In use, the cell is immersed in the solution or pulp to be tested and a sinusoidal voltage of 1000 cps is applied to the primary coil. The secondary coil will then have a sinusoidal voltage induced in it that is proportional to the conductivity of the liquid.

Unlike its pH value, the conductivity of a lime solution is related to the lime concentration of the solution by what is essentially a straight line function. Consequently, the sensitivity of the conductivity cell is constant across the range of lime concentrations of interest to the gold plant operator, and will, therefore, produce a reading of equal precision for all lime concentrations. The main disadvantage of using conductivity as a measure of the lime concentration is that the conductivity cell reads the total conductivity of the solution, which includes the conductivity contributed by the cyanide and various other compounds in solution, as well as that contributed by the lime. The fact that the hydroxyl ion is stronger electrolytically, by a factor of up to three, than the other ions present helps to compensate for this. Two other characteristics of the conductivity cell that should be kept in mind when using it in cyanidation plants are that the cell is temperature-sensitive and that the measured conductivity of the pulp will depend on the solids content. However, conductivity equipment can be obtained with either manual or automatic temperature compensation, and it is unlikely that the pulp density will vary sufficiently to introduce a serious error.

For best results when using the conductivity cell to provide an indication of lime concentration in gold plant slurries, the concentrations of sodium cyanide and of other ions, such as sulphate and thiocyanate, should not vary significantly, nor should the liquidsolid ratio. These are not so important if the lime concentration is being controlled at a high value, in





which case the lime will contribute to such a large proportion of the total conductivity that the effect of changes in the concentration of the other ions will be negligible.

Figure 4 shows the improvement obtained in an operating gold plant in the control of lime concentration in the overflow from a classifier (operating in closed circuit with a ball mill regrinding flotation concentrate) when the control was effected automatically, using an electrodeless conductivity cell(5). The cell was immersed in the classifier overflow and the signal from the cell automatically regulated the rate at which lime was added to the regrind mill feed. The results obtained using manual control (shown by the solid line) represented a range in pH value of from 11.0 to 12.0 and the individual hourly readings over a 12-hour period deviated as much as 80 per cent from the average reading. With automatic control (broken line), the pH value was kept between 11.2 and 11.3, representing deviations from the average of 15 per cent. The automatic control functioned



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Figure 6.—Photograph of the Continuous Cyanide Titrator.

well in spite of the fact that the conductivity cell was monitoring a pulp in which both the cyanide concentration and the pulp density were varying appreciably. It was estimated that the automatic control improved the plant efficiency to such an extent that the control equipment would pay for itself in from six to nine months.

#### Measurement of Cyanide Concentration

A possible method for measuring the cyanide concentration involves an automatic potentiometric titration of the plant solution. Figure 5 is a schematic presentation of an instrument, recently developed by Mr. J. C. Ingles of the Mines Branch, that can be used for this titration (6). Essentially, the instrument consists of two precision metering pumps, one to meter the solution under test and the other to meter the titrating solution, which is 0.01 M sodium argentocyanide, 0.02 M sodium hydroxide and 0.05 M ammonia. The pumps are adjusted to provide the flow ratio suited to the solution under test. Both solutions are pumped to a common mixing container from which they flow by gravity to the measuring section, and then by gravity to was'e. The E.M.F. developed across the



Figure 7.-Chart from the Continuous Cyanide Titrator.

two electrodes in the measuring section is proportional to the free cyanide concentration in the solution under test and can be used to operate a recorder or controller. *Figure 6* is a photograph showing the assembled unit.

The precision and reproducibility that can be obtained with this instrument are shown in Figures 7 and 8. The chart in Figure 7 was obtained by recording the electrode E.M.F. values when measuring pure cyanide solutions of different concentrations over a 24-hour period. Figure 8 is similar to Figure 7 except that plant solutions were used. It is apparent from these charts that small changes in cyanide concentration result in large changes in E.M.F. values, and that good reproducibility is obtained on switching from one cyanide concentration to another and back again.

Because of the necessity of closely regulating the flow rates, the pumps required in the cyanide titrator are not suitable for pumping slurries; consequently, the instrument can only be used on clear, filtered solutions. It is essential, then, when measuring and controlling the cyanide concentration of a slurry using this titrator, to provide a means for obtaining a sample of the clear solution and delivering it to the instrument. This is not a difficult problem, and one

simple procedure, now under study at the Mines Branch, is shown in Figure 9. A sample stream is taken from the vessel containing the slurry, the cyanide concentration of which is being controlled. This stream is pumped back into the vessel or on to the next step in the process. In the discharge line from the pump, a porous plastic filter is installed on which a pressure of 6 or 7 psi is maintained by means of a throttle valve on the discharge side of the filter. When the pump is operating, some clear solution is forced through the filter while most of the slurry flows through the inside of the filter, thus providing a self-cleaning action. On a laboratory set-up, a slurry containing 30 per cent solids was pumped through a 1-in. line in which a filter 6 inches long and 0.5 inch in diameter was installed. With the filter under 6 psi pre sure, 10 ml/min. of clear solution was obtained over a period of two weeks. This is sufficient flow for the cyanide titrator, which requires a maximum of 5 ml/min.; when the filter capacity drops below the minimum desirable level, a new filter can be substituted in a matter of minutes. The cost of the porous plastic filter is under two dollars. This arrangement is still under development, but it appears to be promising.



Figure 8.-Chart from the Continuous Cyanide Titrator.





Pulp Flow

Figure 9.—Arrangement for Obtaining Clear Solution from a Pulp for the Cyanide Titrator.

#### Measurement of Dissolved Oxygen Concentration

Although there are several instruments available for measuring the dissolved oxygen concentration in pulps and solutions, the one which has been under test at the Mines Branch is a Beckman Model 764 dissolved oxygen monitor. The electrode for this unit shown schematically in Figure 10, consists of a gold cathode, mounted in an epoxy casing, and an anode of coiled silver wire. A potassium chloride gel is the



Figure 10 .- Sketch of the Oxygen Monitor Electrode Assembly.

electrolyte providing the electrical connection between the cathode and anode. The diffusion of oxygen to the cathode is controlled by a Teflon membrane, and the whole system is isolated from the solution or slurry under test by a gas-permeable silicone rubber membrane. An external voltage of 0.8 volt is applied between the anode and cathode. When the electrode assembly is immersed in the stream under test, oxygen in the stream diffuses through the membrane and is reduced at the cathode. This causes a current to be developed proportional to the dissolved oxygen content of the stream.

Test work at the Mines Branch has shown that this instrument will operate with reliability and accuracy in a vigorously agitated slurry, containing 50 per cent solids, for a period of three weeks before the electrode requires maintenance and the silicone rubber diaphragm requires replacement. When using this oxygen monitor in conjunction with an on-off controller, it was found that control to within  $\pm 2$  per cent was obtained. Although the monitor has not been tested in plant operation as yet, there is no apparent reason why it will not give satisfactory performance in a gold plant.

When locating the electrode assembly in a plant, it should be placed in such a way that the sample stream flows past the electrode at a speed of at least 3 ft. per second. Also, the electrode should not be exposed to a stream containing air bubbles, as the oxygen in the bubbles will cause erroneous measurements.

#### **Control** of Grind

In grinding the ore for cyanidation, the objective should be to produce at all times the grind most suitable for the ore characteristics. The particles must be small enough to ensure that the gold is exposed to the dissolving action of the cyanide solution, and at the same time overgrinding should be avoided (as overgrinding is detrimental to the efficient mechanical

and chemical operation of the plant(2)). This means then, that a well-controlled grinding unit should consistently produce a ground product having essentially the same size distribution. It would appear, at first glance, that this could only be done if an instrument was available that would automatically provide a continuous or frequent size analysis of the ground product and would adjust the rate of feed to the unit according to the analysis. An instrument such as this is not available at present, but an alternative approach, that of using some operating variable of the grinding unit that depends on the size distribution of the ground product and that can be continuously measured, may offer a solution to the problem. The sound emanating from the grinding mill may be such a variable.

The use of the sound of a grinding mill to control the feed rate is not new. There are many such installations in the cement and iron ore industries. In principle, the sonic controller measures the intensity of a narrow frequency range of the sound from the mill, the frequency range chosen being specific for the mill. An increase in the sound intensity above the control point causes the feed rate to be increased and vice-versa. Although this type of control has operated successfully in certain applications, it is not clear what causes the sound to vary and consequently it is not clear what characteristic of the grind is, in fact, being controlled.

Some recent work at the Mines Branch has indicated that the intensity of certain frequencies in the sound from a ball mill depends on the size distribution of the ground particles in the mill. Figure 11 shows the intensities of audio frequencies in the range of 500 to 4,000 cycles per second from a 2-ft. by 8inch Hardinge conical mill when it was grinding quartz at rates of 50, 100 and 150 lbs./hr. In all three runs, with the exception of the feed rate, all the grinding variables such as pulp density, ball load, feed size distribution and mill speed were similar. It was also determined that the weight of pulp in the mill while the mill was operating was only 3 per cent higher when the feed rate was 150 lbs./hr, than it was at 50 lbs./hr. The size distribution of the ground product, however, was 82 per cent minus 200 mesh at 50 lbs./hr., 68 per cent minus 200 mesh at 100 lbs./hr. and 55 per cent minus 200 mesh at 150 lbs./ hr. As the feed rate was increased, the sound intensity particularly at a frequency of 1,600 cps, decreased markedly. On the basis of these results, there is certainly a suggestion that the only grinding condition that varied sufficiently to produce such pronounced changes in the sound intensity was the size distribution of the ground product. Of course, these early results do not establish a correlation between the sound of an operating ball mill and the size distribution of the ground product, but there is an indication that sonic control of a grinding circuit would result in the production of a uniform grind.

Further light will be shed on the efficiency of the sonic control of grinding operations on a gold ore by the operation at East Malartic Mines Limited at Norrie, Quebec, where sonic controls were recently installed. In this installation, the sounds from the primary rod mill and the two secondary ball mills are used to control the feed rate to the rod mill. An automatic weighing instrument on the feed belt to the rod mill also controls the rate at which water is added to the rod mill so that the liquid-solid ratio to the mill is kept constant. Although comparative operating data before and after the controls were installed are





not available yet, the mill staff is satisfied with the control system and is convinced that it will benefit the efficiency of the operation. It is expected that when the results are available they will show that the instrumentation and the control have resulted in increased throughput and a more uniform grind.

#### Discussion

So far, it has been shown that manual control of the important operating variables in a gold cyanidation plant is unsatisfactory, and that instruments for the automatic measuring and control of these variables are available. The final consideration is whether instrumentation and automatic control would improve the operation sufficiently to pay for the cost of the equipment. As mentioned before, the results of the field test of the conductivity cell to control the lime in a gold mill indicated that the improvement in operation was sufficient to pay for the control equipment in six to nine months. The staff of East Malartic Mines Limited estimate that the automatic controls on the grinding circuit will pay for themselves in a year or less.

The combined cost of a conductivity cell or pH meter, a cyanide titrator and an oxygen monitor (along with the related control equipment), plus a sonic controller for the grinding circuit, would probably be between \$20,000 and \$25,000. If this equipment were installed in a 1000-tpd gold mill where the heads had a value of \$10.00 per ton and reagent costs were \$0.15/ ton, the cost of the instruments would be recovered in less than two years if their use resulted in an increase in extraction of 0.25 per cent and a decrease in reagent cost of 10 per cent. This is, in fact, a rather modest improvement and it is not unreasonable to expect an improvement of at least this order to result from the use of instrumentation and automatic control of the variables related to the chemistry of the cyanide process.

There is, of course, more to the successful application of instrumentation and automatic control than simply obtaining a suitable device for measuring the variables of interest. A control system has, infact, four major components: the sensing device, the controller,

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the control element (usually a pneumatic valve or a variable-speed motor) and the process itself. In this paper, the sensing device is the only component that has been discussed, but a good control system is the result of design based on the characteristics of all the elements of the system(7). However, if a suitable sensing device is available, the other problems in the design of the whole system can usually be solved with the help of someone experienced in control application, as all control systems, with the exception of the sensing device, are basically similar in design.

This paper has described sensing devices for measuring and controlling four variables of the cyanide process, all of them related to the chemistry of cyanidation. There are, of course, other operating variables that lend themselves to automatic control-pulp density and flow rates for example(8)-but the variables that affect the chemistry of the cyanidation process are probably the most important ones to consider and should receive first attention.

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