# Recent developments in the instrumentation and automation of uranium-processing plants

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#### SYNOPSIS

The paper discusses work at the National Institute for Metallurgy on the evaluation of measuring techniques for the automatic control of free acid in leaching, of the ratio of ferric to ferrous ion in leaching, of pH in liquid-liquid extraction, and of uranium in loaded solvent. Commercially available instruments, and the modifications that have been made to adapt them to South African uranium plants, are also described.

#### SAMEVATTING

Die verhandeling gaan oor die werk by die Nasionale Instituut vir Metallurgie in verband met die evaluering van meettegnieke vir die outomatiese beheer van vrye suur by loging, die verhouding van verri- tot ferro-ione by loging, die pH by vloeistof-vloeistofekstraksie, en uraan in 'n belaste oplosmiddel. Die instrumente wat in die handel verkrybaar is, en die wysigings wat aangebring is om hulle by Suid-Afrikaanse uraanaanlegginge aan te pas, word ook beskryf.

### **INTRODUCTION**

Until recently uranium-processing plants have been poorly instrumented. A possible explanation is that suitable and reliable instruments were not available for the measurement of many parameters (e.g., acid concentration, ratio of ferrous to ferric ion, pH of organic solutions, and uranium content in solvents), and the instruments that were available were not sufficiently robust to withstand the harsh environment normally encountered in these plants.

Although there are sound economic reasons for the replacement of labour by instruments and for the optimization of uranium extraction, no serious attempt has been made to evaluate present-day technology. Generally, the tendency has been for plant instrumentation to follow the instrumentation in previous plants, even though this was often illconceived and remained operative only for an initial period. Furthermore, few technicians and engineers in the South African mining and metallurgical industry are capable of implementing new ideas in instrumentation and, thus, of improving the performance of manually-operated plants and removing the barrier of distrust towards instrumentation shown by most plant personnel. In addition, many commercial firms supply an instrument

without any explanations of the application, engineering, installation, and commissioning, and the user has then to make the 'best of it' from his limited experience. Very seldom, therefore, is there a feed-back of useful information to the manufacturers, and the instruments are not usually tailor-made.

Because of this unsatisfactory state of affairs in plant instrumentation, the National Institute for Metallurgy (NIM) decided to evaluate certain commercially available instrumentation that could be used in uranium-processing plants, and attempt improvements further development where these were considered desirable. NIM is fortunate in including among its staff metallurgists, chemical engineers, and instrument engineers, and a frequent exchange of expertise was possible in this project. In addition, the investigation was not confined to laboratory experiments but included plant trials in two uraniumprocessing plants, with the cooperation of both management and personnel.

This paper briefly describes some of the work that was done in this field during 1971 and 1972.

### LEACHING PLANT

Leaching can be defined as the process by which the valuable constituents of an ore are dissolved with suitable reagents. Prior to 1957, uranium-containing ore in South Africa was treated first for the re-

covery of gold by the conventional cyanide process and then for the recovery of uranium1. However, the 'reverse leach' is now favoured, i.e., the uranium is leached before the gold, which, it is claimed, improves the recovery of gold. Uranium can be dissolved by a number of acids, but sulphuric acid, which is the most economical, is normally used. This acid is an effective solvent for UO<sub>2</sub>, and the uranium, which is present in the ore in various forms, is therefore oxidized before extraction by a suitable oxidizing reagent. The most common oxidizer used is manganese dioxide. The stages in the leaching process at which the different reagents are added varies from mine to mine.

The effects of these two reagents on the extraction of uranium are shown in Figs. 1 (for manganese dioxide) and 2 (for acid). Both figures show an initial steep rise in extraction as the reagents are added. Above an extraction of 90 per cent, the curves flatten considerably, and, at an extraction of about 93 per cent, they indicate that the extraction will not be improved by further additions of reagents.

The average extraction obtained in South Africa is about 85 per cent, and the uranium in the tailings is therefore worth a considerable sum, especially when the large throughput in the South African mining industry<sup>3</sup> is taken into consideration. Manual control based on manual analytical techniques, al-

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though relatively speedy, cannot result in improved extraction unless a constant watch is kept on the process. The uranium content of the ore is not the only process variable, but considerable flow variations are introduced through process demands. There therefore seems to be an ideal case for the improvement of extraction by continuous measuring and control apparatus that can react immediately to process changes.

Control of the ratio of incoming ore to reagents is a possible approach to the problem, but, as far as is known, no attempt has been made to implement such a system. An alternative approach is the measurement of oxidation potentials (from which the ratio of ferrous to ferric ions can be determined) and of conductivity (from which the residual acid can be calculated). Both measurements have been made instrumentally, but, mainly because of the fragile equipment employed, no lasting success has been obtained.

The normal procedure in a uranium-leaching plant is as follows. Sulphuric acid is fed continuously

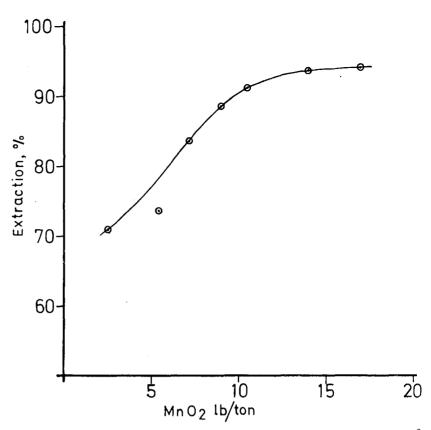


Fig. I—The effect of manganese dioxide on leaching efficiency (after Pinkney²)

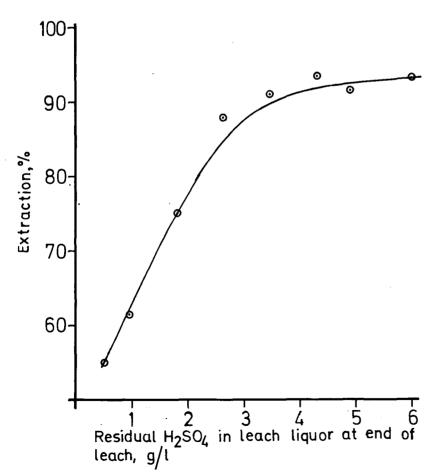


Fig. 2—The effect of residual acid on leaching efficiency (at optimum additions of manganese) (after Pinkney<sup>2</sup>)

into the first pachuca through a pneumatically operated valve, the amount being measured by a rotameter. Manganese dioxide (pyrolusite) is made up as a slurry and is fed continuously from a pneumatically adjustable splitter box, the excess being returned to the slurry make-up system. The incoming pulp is measured with a 'tonnage box' in which the total flow is diverted, and the time required to fill the box is measured. The specific gravity of the pulp and the time required give a reasonably accurate measurement of the dry tonnage throughput. However, magnetic flowmeters and radio-isotope density meters are rapidly replacing these out-dated throughput-measuring techniques. Temperature, which also plays a part in the period of leaching, is maintained manually between 40 and 50 °C by steam heating. Again, attempts are being made to control the temperature automatically.

## ELECTRODELESS CONDUCTIVITY MEASUREMENTS FOR THE CONTROL OF ACID

Since 1960, when Eichholz and Bettens<sup>4</sup> published a paper describing an electrodeless conductivity probe, several papers<sup>5-8</sup> have been published on the development of

electrodeless conductivity meters, and others<sup>9-11</sup> on the industrial application of such meters. Improved extractions and lower acid consumptions were claimed<sup>9</sup>, and the measurement of conductivities at very high acid concentrations (up to 90 per cent sulphuric acid) was said<sup>10</sup> to be possible.

During 1970, NIM bought a commercially available electrodeless conductivity meter for evaluation in South African extraction plants. The results<sup>12</sup> were not convincing, and NIM developed its own design, which is aimed at making the conductivity measurements specific for acid and at reducing the effect of slurry temperature and density. The prototype was tested13, and four more such units having the required robustness were manufactured locally and tested during 1971. The performance of these instruments was excellent, and it is envisaged that similar instruments will be used in all major extraction plants. As a result of the report<sup>13</sup> on the prototype, an overseas manufacturer asked NIM to evaluate their latest design of electrodeless conductivity meter for local conditions. The resulting measurements, compared with those obtained with the previously practised manual methods, are encouraging.

### Principle of Operation of the Electrodeless Conductivity Meter

The measuring probe of an electrodeless system consists of two toroidal coils spaced on a nonconducting tube. There is no direct electrical coupling between the coils unless the probe is immersed in an electrolyte. The liquid inside the tube and outside the protective cover of the toroidal coils forms a 'liquid loop'. One toroidal coil,

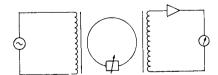


Fig. 3—Basic measuring system of the electrodeless conductivity meter

which is fed with a high-frequency current of constant amplitude, acts as the primary coil of a transformer. The 'liquid loop' becomes the secondary single-turn coil, in which an alternating current is magnetically induced. The secondary current in the liquid varies in direct proportion to the conductance of the liquid. As the second toroidal coil is also fitted around the tube, the current in the 'liquid loop' generates a current in this coil. Thus the 'liquid loop' becomes a primary winding to the second coil. The alternating current from the second coil is amplified, and either metered direct or used in a feed-back null-balance system (see Figs. 3 and 6).

### The Probe

The probe (Fig. 4) consists of an inner and an outer plastic cylinder between which the ferrite cores containing the toroidal coils are fitted. Each coil is electrostatically screened. A thermistor, used for temperature compensation, is attached to the inner tube. A wire loop, used for calibration, is threaded through the probe along the inner plastic cylinder, and the remaining space between the inner and outer cylinders is filled with an appropriate potting cement. A plastic pipe, which is used as a protective cover for the leads to and from the coils and for the mechanical support of

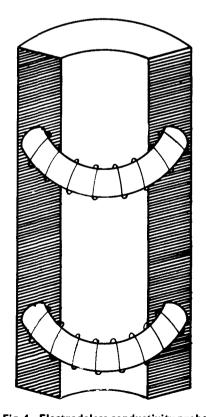


Fig. 4—Electrodeless conductivity probe

the probe, is attached to the measuring head.

The probe, when not immersed in an electrolyte, can be calibrated direct in terms of resistance by the connection of a variable resistance to the wire loop threaded through the probe. After calibration, the wire loop is disconnected and the probe is immersed in the electrolyte so that the central tube is vertical. With the large volume of electrolyte surrounding the outer case, the cell constant becomes almost wholly dependent on the dimensions of the inner tube. For extreme conditions of wear, the probe casing is rubber-coated.

### Electronics for the NIM System

As long interconnecting cables are generally necessary on a plant, the electronic system (Fig. 5) is subdivided into a probe-head unit and a main unit, the latter being situated in the control room.

The probe-head unit comprises a multivibrator oscillator and buffer amplifier feeding the transmitter coil, and a combined source follower and impedance converter connected to the receiving coil. Because of its amplitude stability, the multivibrator was chosen in preference to a sine-wave oscillator. The working frequency is between 15 and 20 kHz.

In the main unit, the signal from the impedance converter is amplified at a gain that precludes saturation of subsequent stages. This gain is fixed at the time of installation and depends on the maximum conductance encountered. The next stage is a linear rectifier giving a d.c. output of 0 to 6 V without distortions. This signal is fed to a stage that enables a d.c. back-off signal to be applied. This back-off signal is controllable from the front panel as the 'Level' setting. This stage also provides temperature compensation, probe thermistor being connected in its feed-back network. A variablegain stage follows, controlled by the front panel 'Range' setting, where the range of the instrument is preset. The last stage processes the signal so that the output is compatible with the current requirements of an electropneumatic converter.

Electronics with Null-Balance System

The null-balance system is illustrated in Fig. 6. The signal from the

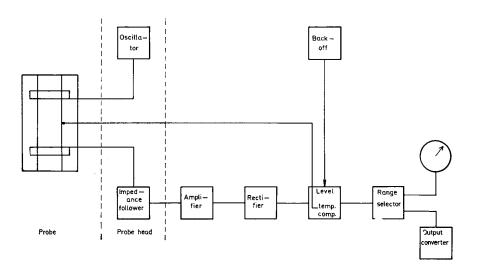


Fig. 5—NIM electrodeless system

receiver coil is amplified, phase detected, and passed to a servo-motor system that drives a slide wire. The slide wire is fed with a set fraction of the energizing a.c. signal to the transmitter coil. The wiper arm is connected, via a buffer amplifier and thermistor network, to a compensating coil wound alongside but opposite to the receiver coil so that, at the appropriate balance point, the output approaches zero. A pointer coupled to the wiper arm indicates the conductance. A slave slide-wire transmits a signal suitable for an electropneumatic converter.

The range of the instrument can be preset by adjustment of the relative fraction of supply signal that is to be fed to the main slide-wire. Temperature compensation is provided by the thermistor.

Generally, this system has improved freedom from drifts in frequency and amplitude because it is phase locked and the input amplifier works at a constant low level of signal. However, mechanical wear on the slide-wires may be high owing to the constant oscillation round the same point. This oscillation results from the passage of bubbles through the probe, which affects the conductivity reading. One commercially available instrument based on a null-balance system is the Beckman RIS5 meter.

### Influence of Temperature

The conductance of an electrolyte rises with temperature, principally

as the result of an increase in the mobility of the constituent ions. For sulphuric acid, the temperature coefficient of conductance is about 1,2 per cent per degree Celsius. The ferrite cores are affected by temperature: the magnification factor (Q) of the coil is reduced as the temperature increases—an opposite but smaller effect than the change due to the temperature change in the electrolyte.

In both previously described conductivity systems (NIM system and null-balance system), temperature compensation is applied by the use of a thermistor embedded in the probe head. In the NIM system, the thermistor is included in the negative feed-back system of one of the stages so that the gain of this stage is proportionally reduced as temperature increases. The null-balance system has its thermistor included in the feed to the compensation coil,

which achieves the same end results.

The compensation factor can be adjusted in the NIM system according to the results of tests in solutions of heated electrolytes in which the probe is immersed. The Beckman RIS5 does not have variable temperature compensation.

Practical tests on the temperature coefficients of actual slurries present difficulties in that conductance is a dynamic feature, the reaction proceeding during tests and the conductance being modified correspondingly.

### Effect of other Ions

Unlike pH measurements, the conductivity-measuring system is not specific for the H<sup>+</sup> ion but responds to any free ion. However, the mobility of the H<sup>+</sup> ion is far greater than that of the other ions in solution, and the concentration of these interfering ions in local leaching plants is small and relatively constant.

In both the conductivity-measuring systems described, it is possible for the conductivity signal, which is affected by interfering ions, to be backed off so that the instrument can be scaled direct in grams per litre of acid.

### Influence of Pulp Density

Non-conducting solid particles inside the inner cylinder of the probe have a marked influence on the cell response and hence on the range of measurement selected when the probe is to be used in a pulp instead of in a solution. NIM found that the range should be increased about  $2\frac{1}{2}$  times for a pulp of 60 per cent solids, compared with the range for a

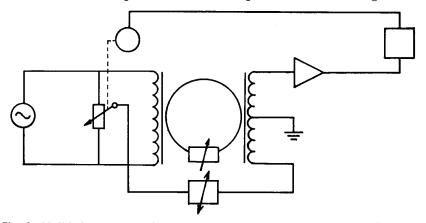


Fig. 6—Null-balance measuring system of the electrodeless conductivity meter

solution. However, because the relative density of the slurry in local leaching plants is kept constant (to within 2 per cent), further compensation is not required once the instrument has been calibrated with its probe immersed in pulp. A greater hazard is the occasional build-up of deposits within the tube, but this effect is minimized by the vertical orientation of the probe and by routine cleaning. Bubbles passing through the probe cause an oscillation of the conductance readings. Suitable time constants inserted in the measuring systems help to minimize this effect.

### Plant Operation and Calibration

Many of the undesirable effects, such as density and interfering ions, can be circumvented by the use of twin probes in differential mode. i.e., one in the slurry before the addition of acid and one after the addition. However, this procedure was found to be unnecessary because plant conditions remain relatively stable. Static calibration of the probe and meter was found to be unsuitable. In the pachucas, acid concentration is in a dynamic relationship with the pulp and, when samples are removed, the pulp acidity varies inversely with time.

It was found best for the calibration to be done in situ by direct comparison with the routine acid titrations. The correlation thus found can be simulated electrically, i.e., the test-loop resistance is varied to give equivalent readings. Thereafter the instrument gain is varied for any suitable range by reference to these equivalent resistance readings. A typical calibration plot is shown in Fig. 7.

NIM has used conductivity meters of various designs for the last twelve months in plant control. Correlation between the metered free acid and the acid indicated by titrations is very good indeed. For instance, in a plant in which the free acid ranged from 0 to 6 g/l, titration and meter indication agreed to within 0,1 to 0,2 g/l. On automatic control, the free-acid content could be maintained at  $3,5 \pm 0,2$  g/l.

In another plant, where a different meter and control circuit were used and a different ore was leached, the

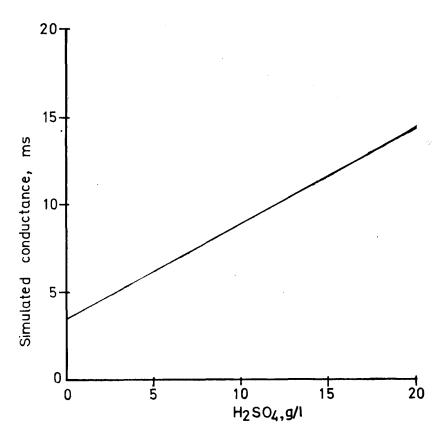


Fig. 7-Relationship between conductance and acid concentration in slurry

range selected for free acid was 3 to 15 g/l. Titration and meter indication agreed to within 0,5 g/l. On automatic control, the free-acid content could be maintained at  $6.8 \pm 0.4$  g/l. Before automatic control was employed, an operator trying to maintain stable conditions managed to stay only within 2 to 10 g/l. Reference to Fig. 2 shows that the extraction must have been very low when the free acid was 2 g/l and that a considerable amount of acid must have been wasted when the concentration of free acid was 10 g/l. Such a high concentration of free acid would require the addition of extra lime to the effluent-treatment plant.

### REDOX METERS FOR THE CONTROL OF OXIDANT

It is difficult to find the reasons for the failure of redox-potential measurements in the local industry. It has been claimed that the platinum electrode is poisoned by the sulphides present and that the reference electrode is too fragile for use in pulps. For harsh environments, NIM developed a robust

probe holder and inserted a LAZA-RAN reference electrode. This electrode, which became available only in 1970, is made from a solid polymer composite and is therefore ideally suited to harsh plant environments. In addition, NIM made a plasticencased platinum electrode. Poisoning of this electrode has not been noticed and it is assumed that the self-cleaning action resulting from the abrasive pulp has prevented any poisoning.

### Principle of Operation

A platinum electrode immersed in a solution of ferric and ferrous ions assumes a half-cell potential  $E_p$ , gaining or losing electrons according to the state of the redox reaction

$$Fe^{2+\leftarrow}Fe^{3+}+\epsilon$$
.

The magnitude of this potential is derived from a modified form of the Nernst equation

$$E_p = E_o + k \log \left( \frac{\text{Fe}^{3+}}{\text{Fe}^{2+}} \right)$$

where  $E_o$  is the standard electrode potential and k is a factor dependent

on temperature (absolute). (At a leaching temperature of  $55\,^{\circ}$ C,  $k=65\,\text{mV}$ ). The emf from the complete cell is as follows:

 $E_c = E_p + E_r + E_j$ , where  $E_r$  is the reference potential and  $E_j$  represents the liquid junction potentials. A logarithmic plot of concentration ratios

$$\left(\frac{\mathrm{Fe^{3+}}}{\mathrm{Fe^{2+}}}\right)$$
 versus  $E_c$ 

gives a straight line whose slope (K)is 65 mV per decade at a temperature of 55°C. Where the oxidized and reduced ions are equal in concentration (i.e., log 1=0), the line intercepts the y axis at a potential  $E_{co}$ . This potential varies with the concentration of other ion species present, e.g.,  $H^+$ ,  $SO_4^{2-}$ , and  $PO_4^{3-}$ . However, under controlled conditions of conductivity and in a slurry of reasonably constant make-up, the potential  $E_c$  provides a useful guide to the ratio of ferric and ferrous ions and can be used for the control of this ratio at a fixed level.

### The Probe

The probe head (Fig. 8) is constructed of polypropylene and contains three chambers accommodating a platinum electrode, a reference electrode, and a stainless-steel ground connector.

The platinum electrode is in the form of a capsule for ease of replacement. The body of the capsule, fabricated from polypropylene, carries a sealing ridge and is coneshaped at one end. The platinum element is thermally sealed into the cone so that the tip protrudes at the apex. This shape was found to minimize the problems resulting from coating of the electrode by deposits of calcium sulphate.

The LAZARAN reference electrode<sup>14</sup>, a silver-silver chloride type, is totally encased in polymer, having at its tip a large liquid junction area constructed of porous polymer. This electrode is strong enough to withstand the vigorous pulp agitation in the pachucas and does not require an external reservoir of potassium chloride.

The electrodes and ground connection are sealed into the head chambers of the probe by screwed glands and O-rings. The probe head is supported by a 2 m pipe, which

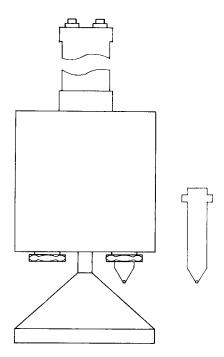


Fig. 8-The emf probe

also carries the shielded connecting cable. The whole system is slightly air-pressurized to preclude any leaks.

### The Electronics

The measuring unit consists of twin FET source followers of highinput impedance whose outputs are combined in a differential stage. The resulting signal is processed by further stages, where back-off voltage is added and amplification applied to produce an output compatible with that of an electropneumatic converter. Thus a change in input potential of 400 to 600 mV results in a full-scale change of output (4 to 20 mA or 10 to 50 mV).

The differential system avoids common-mode interference by stray alternating currents, and the high-input impedance on both electrodes gives some immunity against variations in input resistance as the electrodes become contaminated. The measuring unit in use is a Beckman type 941 pH-analyser, modified by inclusion of a preset gain control and a noise filter at the input.

### Plant Performance

During plant trials extending over a period of eight months, the redox electrode system has worked successfully. The recorded potentials were compared with the concentrations of ferric and ferrous ion as analysed chemically at corresponding times, and a useful correlation (Fig. 9) was

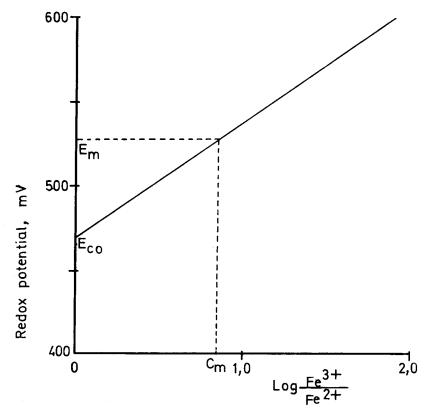


Fig. 9—Platinum LAZARAN probe — potentials versus ferric/ferrous concentrations

found to exist according to the Nernst equation, despite point scatter at the higher levels of ferric ion. As the variations of  $\rm H^+$  and  $\rm SO_4^{2-}$  decreased with conductivity control, so this scatter lessened. A slope of 66 mV per decade of ferricto-ferrous ion ratio was found, agreeing well with the predicted value.

A redox potential  $(E_m)$  of 530 mV was found to represent an approximate ratio  $(C_m)$  of ferric to ferrous ion of 7 to 1. This potential was used as the control setting from which the amount of oxidant (manganese dioxide) to be added to the pachuca was determined. The control loop comprised a two-term pneumatic controller and a pneumatically driven splitter box that apportioned a slurry of the oxidant to the pachuca or recycle, as dictated by the control signal. Under controlled conditions, routine chemical analyses of the ferric and ferrous ion concentrations showed a marked increase in stability over the previous manual methods. Contamination of the cone electrode was very small, a run of three weeks causing the electrode to deviate in its output by only 1 per cent. Before automatic control was employed, the ratio of ferric to ferrous ion could vary as much as 1 to 1 and 20 to 1, and a ratio of 7 to 1 was seldom obtained. These extreme values meant either that the extraction was very low or the wastage very high.

### LIQUID-LIQUID EXTRACTION PLANT

After the pulp has been filtered and most solids have therefore been removed from the uranium-containing (pregnant) solution, the pregnant solution enters the liquid-liquid extraction plant. Most extraction plants at present use the Purlex process, which includes the following steps: uranium is extracted into an organic solvent in the extraction section, the impurities are removed in the scrubbing section, the uranium is back-extracted into an aqueous medium in the stripping section and is then precipitated with ammonia, and, after thickening and filtering, the uranium slurry is sent to a central processing plant. The process of extraction, scrubbing, and stripping is automatically controlled. Two fields in which instrumentation has so far failed, namely pH control of organic solutions and the determination of the uranium in the loaded solvent, are reported below.

Ammonium hydroxide is used for the adjustment of pH and is prepared by the continuous metering of the deionized water flowing into a make-up tank and by adjustment of the ratio of the ammonia gas flowing into this tank to the water flow. The measured values for the flow of ammonia gas are compensated for temperature and pressure. A new method for the preparation of ammonium hydroxide, at present being developed at NIM, promises to effect considerable reductions in the instrumentation used.

### pH MEASUREMENT

pH is generally monitored in the aqueous phase as it flows through the mixer-settlers of the extraction plant, although process requirements are optimized in respect of time lags and reagent control when pH is measured in the organic continuous phase. The measurement of pH in the aqueous emulsion discontinuous present in this phase involves difficulties because a high impedance is presented to both the glass and reference electrodes. Furthermore, the solvent tends to block the pores at the tip of the reference electrode, causing discontinuity of the liquid junction. Standard pH systems cater only for single high-impedance input on the glass electrode because the reference junction normally has a relatively low impedance (Zrl) to an aqueous solution, and common-mode interference, with respect of ground, is of little consequence. However, in the discontinuous aqueous emulsion Zrl is large, and common-mode effects are pronounced. A system using a separate reference-channel source follower largely overcomes this effect, the input impedance of the reference channel Zir being much higher than  $Zrl^{15}$ . The other difficulty, that of pore blockage, is overcome by the use of the LAZA-RAN 'oil resist' electrode.

The pH-control system has been in operation for eight months, and the results are encouraging. A change of 5 pH units, between pH values of 2 and 7, produces a 10 to 50 mA output signal from the pH-system. which is coupled direct to a threeterm electronic controller. The control signal (10 to 50 mA) is fed through an electropneumatic converter to the reagent control valve. An effective differential of +0.1 pH unit in the control limit has been maintained. Buffer checks showed only minimal drifts (0,1 pH unit) during this period, although an increase in the time constant of the electrode system was noted. This was found to be due to a coating of ammonia diuranate on the glass electrode. The electrodes washed in dilute acid (0.1 N hydrochloric or nitric acid), and the fast response time was restored. Comparison measurements of pH in the organic continuous phase were made in the laboratory by the measurement of the aqueous fraction after the organic and aqueous components had been allowed to separate out and the organic phase had been decanted. Laboratory measurements showed that the new pH system reduced average pH deviations by a factor of 3.

Comparative results showed a constant difference of 0,3 pH unit between the manual and on-line measurements, but the correlation was otherwise excellent. Expected interference from stray liquid-junction potentials was comparatively unimportant.

Control in the aqueous phase introduces a time lag, which has undesirable side-effects such as the formation of crud. The latter is minimized by control of the pH in the organic phase.

### MEASUREMENT OF URANIUM IN SOLUTION

As previously mentioned, the uranium is extracted into an organic solvent. For reasons of economy, the solvent should be loaded with uranium at an optimum level, which seems to be about 4 g of  $\rm U_3O_8$  per litre. As there were no suitable analysers for uranium in a solvent solution, the South African Atomic Energy Board set about evaluating the use of radio-isotope measurements and found that a non-dispersive X-ray-fluorescence analyser would be capable of measuring the uranium content. NIM then set about

building such an analyser that would be robust enough for the plant environment. The analyser<sup>16</sup> incorporates a cobalt-57 radio-isotope, which emits 122 keV X-ray radiation. This radiation excites the uranium, which then emits the characteristic fluorescence at 98,4 keV. The fluorescence is monitored by a solid-state high-resolution Xray detector. The detector crystal is lithium-drifted germanium and has to be maintained at extremely low temperatures, which are obtained from a copper rod that is in contact with the crystal and is immersed in a dewar of liquid nitrogen. The radio-isotope is mounted in a lead collar that surrounds the crystal, and it emits gamma rays upwards into the sample flowcell, which is placed on top of the lead collar. By selection of appropriate physical and electronic conditions, it is possible for the relation between signal and uranium concentration to be made linear up to at least 5 g of U<sub>3</sub>O<sub>8</sub> per litre. With this analyser an accuracy of better than 5 per cent of the indicated uranium content was obtained. However, as capital and maintenance costs were excessive, new methods for the determination of uranium were investigated at NIM.

The best alternative technique was found to be based on X-ray absorption<sup>17</sup>. The 60 keV radiation from an americium radio-isotope is more heavily absorbed by uranium than by the kerosene matrices of a Purlex solvent. In this instrument the sample flowcell is placed between the radio-isotope and the detector. The radio-isotope is mounted in a lead enclosure that has a small hole through which radiation is collimated and emitted. The radiation passes through the sample and is partly absorbed, and the remaining radiation is detected by a sodium iodide scintillator and photomultiplier, the pulses obtained being amplified and counted. A ratemeter converts these pulses into a continuous analogue signal, which is then predominantly a function of the uranium in the solvent. The analyser in its present form is accurate to within 0.2 g of the indicated uranium content in the range from 2 to 4 g of  $\rm U_3O_8$  per litre. The radio-isotope has a half-life of 458 years, and, as the remaining equipment is based on the solid-state technique, very little maintenance is required. Further tests to improve the accuracy are still being done.

### **CONCLUSION**

Although considerable advances have been made in measuring and control techniques for South African uranium plants, work is continuing on the testing of these techniques and the development of new ones. It is hoped that other uranium-processing plants in South Africa will apply the latest developments in instrumentation so that they, too, can benefit from the improvements obtained.

The measuring techniques discussed will undoubtedly find application in other metal-extraction processes. Experimental investigations are to be undertaken to evaluate the use in copper- and zinc-extraction plants of conductivity meters, redox meters, and radio-isotope X-ray analysers similar to those discussed in this paper.

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