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THE USE OF RADIOACTIVE TRACERS IN ION EXCHANGE STUDIES

by

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EXTRACTION METALLURGY DIVISION



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A. J. Gilmore*

SUMMARY

In exploratory studies of the adsorption of the aurocyanide $[Au(CN)_2]$ anion from an alkaline sodium cyanide solution by Rohm and Haas Amberlite IRA-400 strong base resin, the gold concentration in the effluent was followed with the aid of a radioactive tracer. Using the gold isotope, Au^{198} , as the tracer, a method was devised that provided a continuous indication of the gold content of the effluent, and a rapid, satisfactorily accurate method for analysing the effluent for gold. The method would be equally useful in any ion exchange study when suitable radioactive isotopes of the ions of interest are available.

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INTRODUCTION

This study was made in connection with the possible use of ion exchange techniques in the cyanide process for recovering gold from its ores. In this exploratory study, bench scale ion exchange equipment was used for the absorption of the aurocyanide anion by a strong base quaternary ammonium exchanger. The capacity of the equipment was such that about 17 bed volumes of effluent were required for radiometric and chemical analysis of the effluent for gold content. A large sample volume in proportion to the resin volume is generally considered to be unsatisfactory. The magnitude of any sudden change in the analysis of the effluent during the time taken to collect the sample would not be detected. Previous investigators⁽¹⁾ had reported that rapid and unexpected changes occurred in the adsorption of the aurocyanide anion by strong base resins. Consequently, it was important that a procedure be used that would allow these changes to be observed.

The method used involved the addition of radioactive gold to the ion exchange feed solution. By this means, the gold content in the effluent could be followed continuously by means of a radioactivity monitor and a continuous chart recorder. This report describes the apparatus and procedure used where Au ¹⁹⁸ with a half-life of 2.7 days was the source of radioactivity. Some results illustrate the usefulness of this tracer technique on ion exchange studies.

Test Apparatus

The equipment for this adsorption study was arranged as shown in Figure 1. Three, 3 inches by 0.5 inch I.D. polyethylene, columns were each half-filled with 3 mls of Rohmand Haas Amberlite IRA 400 resin in the chloride form. The influent solution was fed by gravity from a head tank. The connecting tubing was either glass or tygon.

The radiation detectors were standard photomultiplier assemblies with sodium iodide phosphors. The detectors were built by Mines Branch staff, but similar detectors may be obtained commercially from the Harshaw Chemical Company of Cleveland, Ohio.

A coil of glass tubing wound four times around the detector head exposed about 20 ml of radioactive effluent to the detector. The detector output was amplified and counted by a Tracer Inc. Precision Ratemeter (Model SC-34A). A continuous recording of the ratemeter output was made on a Phillips strip Chart Recorder (Model PR 2216U).

This arrangement provided the means for continuously observing the





progress of the gold adsorption by the resin. As a protective measure the resin columns were shielded with 2 in. of lead. The radiation detectors were similarly shielded to minimize atmospheric radiations.

The influent solution was prepared by adding 0.91 g. of gold as potassium aurocyanide and 0.015 g. of radioactive gold foil to 13 litres of an industrial gold mill solution. The radioactive foil, radiated to about 60 millicurries, was dissolved in 500 ml. of a sodium cyanide solution containing 0.75 g. NaCN/1. When the foil dissolved, this solution was added to the main volume of the influent solution. The radioactive gold comprised less than 1.5 % of the total gold in the influent solution.

The chemical analysis of the influent solution is given in Table 1. The gold concentration of 2.1 oz/ton in this solution was used so that the resin would be loaded before the radioactive gold isotope Au^{198} , with its short half-life, decayed to a level that was too low for detection.

TABLE 1

Chemical Analysis of the Influent Solution

- 2.1 oz/tonAu - <0.002 oz/tonAg NaCN -0.2 g/10.73 g/1CaO -CNS 0.058 g/1_ 0.013 g/lCu - <0.0004 g/1Ni

Test Procedure

The gold-bearing cyanide solution containing the radioactive gold tracer was fed by gravity to the columns of resin. The initial flow was through the first two columns in series. When breakthrough occurred at the lead column, the third column was connected into the series. This third column was used mainly as a trap to prevent radiation being discharged from the shielded columns. The average flow rate of 1.85 ml./min, through the 3 ml of resin, is equivalent to a retention time of 0.65 min.

At irregular intervals, 50 ml samples of effluent were collected in 2 oz. glass vials 7.5 cm. high by 4.5 cm. in diameter. The effluent was sampled when significant changes in the radioactivity were indicated by the strip chart recorder reading. One 50 ml. sample of the feed solution, diluted to 1/100 by volume, was placed in a bottle of the same geometry as those used for the effluent samples. Each time the radioactivity of the

effluent solution was measured by a beta-gamma equilibrium $counter^{(2)}$, the diluted feed solution was also measured. Assuming atmospheric radiation to be constant, the ratio of effluent radioactivity to feed radioactivity is proportional to the ratio of effluent gold analysis to influent gold analysis. The gold analysis of the effluent could then be calculated as:

$\frac{\text{Radioactivity of effluent}}{\text{Radioactivity of feed x 100}} \text{ x gold analysis of feed}$

The influent and effluent samples were then chemically analyzed for gold after being stored for 10 to 12 days. This storage period was to allow the radioactivity to decay to a safe level before it was subjected to chemical analysis. The results of these chemical analyses were compared with those obtained previously by radiometric measurement.

Since only one ratemeter and one strip chart recorder were available, the output from only one detector could be recorded at a time. Manual switching of the ratemeter and recorder from detector to detector was required to follow the adsorption characteristics of the first and second columns of resin.

RESULTS AND DISCUSSION

Table 2 shows the gold analysis of the effluent as determined by both the radiometric and chemical methods for samples taken from the first and second columns of resin. The analyses obtained by the two methods are in reasonably good agreement. The radiometric analyses have a tendency to be higher than the chemical analyses, but the order of magnitude of the difference is not great. The results are good considering that the experimental error in analysing small volumes of low-grade gold-bearing solutions by most methods is relatively high. In addition, the apparent accuracy of the radiometric method coupled with its speed makes it an attractive control technique for use in a resin column study. A radiometric analysis was done in five minutes compared to the two or three days necessary for a chemical analysis.

Figure 2 shows plots of the gold analyses of the effluent samples from the first two columns as the gold adsorption proceeds. The radioactivity level of the effluent from the first column, as indicated by the chart recorder reading, is also shown. The chemical analysis and the recorder readings indicate a normal adsorption, up to about 1000 bed volumes. At 1030 bed volumes, the curve, based on the chemical analyses, shows a slight discontinuity. Because of the relatively large sample volumes required for chemical analysis, this discontinuity is smoothed out to such a degree that it is almost unnoticeable. On the other hand, the magnitude and characteristics of the discontinuity is clearly shown by the graph of the recorder readings. It

TABLE 2

Comparison of Gold Concentration in Radioactive Effluent Solutions by Β, γ Equilibrium Counter and Chemical Analysis

Effluent	Resin Column		B, γ Equilibrium	Chemical Analysis
Bed Volumes	Sampled		Counter Analysis	oz Au/Ton Soln
(1 bed vol = 3 mls)	1	2	oz Au/Ton Soln	
0 (Feed)			2.1	2.1
175	x		0.00036	<0.001
525	x		0.00063	<0.001
622	x		0.0025	<0.002
812	x		0.0084	0.007
9 3 8	x		0.0116	0.007
1032	x		0.025	0.018
1048	x		0.061	0.042
1138	x		0.084	0.060
1385	x		1.4	1.6
1582	x		2.3	2.4
1598		х	0.0044	0.003
1682	x		2.96	2.9
1698		x	0.011	0.009
1818		x	0.0074	0.007
1862		x	0.029	0.026
1878		x	0.048	0.040
1895		x	0.048	0.037
1912	ł	x	0.061	0.049
1952	x	1	2.96	3.0

is beyond the scope of this paper to discuss the reasons for such discontinuities in the adsorption behaviour of gold by anion resins, but other investigators⁽¹⁾ have observed similar discontinuities during gold adsorption. The important point here is that the radioactive tracer technique resulted in the prompt detection of the phenomenon and provided a complete record of its characteristic.

At the peak of the loading curve, the effluent concentration was greater than the influent concentration due to self elution, and could not be followed by the recorder. The radioactivity of the effluent solutions during this latter part of the loading was so high that the monitor and recorder pen were driven off-scale. This experience indicates the importance of adding the amount of radioactive tracer required to suit the capabilities of the monitoring and recording equipment.



Figure 2. Typical Loading Curve for Gold

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The graph of the effluent analyses from the second resin column showed the loading characteristics to be similar to that of the first column of resin. However, it was only possible to record the loading behaviour of the first column during the critical break through, due to the manual switching of the ratemeter and recorder.

CONCLUSIONS

The use of radioactive Au¹⁹⁸ as a tracer in gold-bearing cyanide solutions is a valuable aid in obtaining relevant data in exploratory ion exchange studies. The technique provides a method of rapid gold analysis, and a means of continuously observing and recording the adsorption characteristics.

The radioactive tracer technique, used to advantage in gold ion exchange studies, could be applied with equal success to other ion exchange work where suitable radioactive isotopes of the metal under study are available.

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REFERENCES

1. J. Davison, F.O. Read, F.D.L. Noakes and T.V. Arden, "Ion Exchange for Gold Recovery", Transactions of the Institution of Mining and Metallurgy. Vol. 70, Part 5, p. 247-290, (1960-61).

2. G.G. Eichholz, J.W. Hilborn and C. McMahon, "The Determination of Uranium and Thorium in Ores", Department of Mines and Technical Surveys, Mines Branch, Radioactivity Division Topical Report TR-99/52, August 1952.

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