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THE PREVENTION OF COBALTICYANIDE
POISONING OF ION EXCHANGE RESINS
USED ON SOUTH AFRICAN GOLD ORE
RESIDUE LEACH LIQUORS

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The Prevention of Cobalticyanide Poisoning
of Ion Exchange Resins Used on South
African Gold Ore Residue Leach Liquors

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A B S T R A C T

As a result of the cobalticyanide fouling of the quaternary anion exchange resins with respect to uranium capacity, a study has been made on the use of a weak base anion exchange resin for a scavenger. It has been shown that Amberlite XE-111₄ can function as a scavenger in that it adsorbs cobalticyanide preferentially to uranium and that it can be efficiently eluted with caustic.

The Prevention of Cobalticyanide Poisoning
of Ion Exchange Resins Used on South
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Introduction

The presence of cobalticyanide in the pregnant liquor solutions at West Rand, South Africa has resulted in a decrease in uranium capacity of the ion exchange beds. Attempts to remove this poisoning from Amberlite IRA-400 by the usual regenerants have not been successful (RMO-2521). At present, the only alternative is the removal of this contaminant prior to passage of the pregnant liquor through the ion exchange beds.

Three possible methods have been proposed for this purpose. These are as follows:

- (1) Water re-pulping of ore to wash out cobalticyanide and other cyanides prior to acid leaching of ore.
- (2) Chemical precipitation of cobalticyanide with some heavy metal.
- (3) Use of a weak base scavenger prior to adsorption on Amberlite IRA-400 resin.

Of these three methods, the first was considered practical in some cases but would probably not solve the problem entirely. The second was considered costly but not too effective. The third was considered to be practical especially when used in conjunction with the first.

A study was made of the possibility of removing the cobalticyanide using an ion exchange column as a scavenger. The resins tested were Amberlite IR-4B, XE-76, XE-114, fresh Amberlite IRA-400, and fouled Amberlite IRA-400. The procedure followed was to load columns of resin with synthetic solutions containing cobalticyanide with a background of magnesium sulfate to simulate the conditions of the pregnant liquors used at West Rand.

Cobalticyanide Breakthrough Capacities of Various Anion Exchangers

Columns of 20 - 25 ml. of fresh resin beds were prepared in 50 ml. burettes and each treated with 500 ml. of 5% sulfuric acid to convert to the sulfate form. The columns were then rinsed with water to a pH 2 - 3. A column of fouled resin was placed in a burette and rinsed with water.

A synthetic liquor solution was prepared containing:

0.615 mg. Co/l. as $K_3Co(CN)_6$
30 g. $SO_4^{=}$ /l. as $MgSO_4$
pH = 2.5 adjusted with sulfuric acid.

This solution was passed through the columns at a 1 gal/ft.³/min. rate. Results of these experiments are in figure 1 and table 1.

Table 1

<u>Resin</u>	<u>Capacity to breakthrough (0.004 g/l)</u>	<u>Bed Volumes</u>
IR-4B	1.3 mg. Co/ml resin	4.0
XE-76	5.9 mg. Co/ml resin	9.6
XE-114	8.5 mg. Co/ml resin	13.8
IRA-400 (fresh)	18.8 mg. Co/ml resin	30.5
IRA-400 (fouled)	0 mg. Co/ml resin	0

Table 1 and Figure 1 indicate that XE-114 would be the most promising scavenger of the resins studied. Amberlite IRA-400 (fresh) would not be satisfactory, at present, because no economically feasible regenerant is known. The IRA-400 (fouled) was obtained from the Set IV resin columns of West Rand after several months (50 cycles) of operation. The sample was taken 18 inches from the filter. This column immediately leaked cobalt which eliminates the use of this bed for a scavenger.

The column containing XE-114 was eluted with 10% sodium hydroxide. Twenty-three bed volumes of caustic removed 95% the adsorbed cobalt. 4.6 bed volumes of elutriant removed 86% of the cobalt (Fig. 2). Because of the high capacity of this resin for cobalticyanide, its ease of cobalt elution, and its generally satisfactory physical properties, XE-114 was chosen for further study.

Properties of XE-114

XE-114 is a new, commercially developed product of Rohm and Haas Company. Its capacity and physical properties are as follows:

Moisture	-	63.1%
Total Capacity	-	9.0 me/gm.
Total Capacity	-	2.5 me/ml.
Density	-	43.8 lbs/ft. ³
Effective size	-	0.44 mm.

This resin is more basic than Amberlites IR-4B and IR-45 but less so than Amberlite IRA-400.

Use of XE-114 as a Scavenger for Cobalticyanide

To demonstrate the use of XE-114 as a scavenger for cobalticyanide the following study was made:

Two columns of IRA-400 and one column of XE-114 were prepared in their sulfate form with 5% sulfuric acid. One column of IRA-400 was loaded directly with a synthetic pregnant liquor. The other column of IRA-400 was placed in series with the column of XE-114 with the influent solution first passing through the XE-114.

The pregnant liquor solutions had the following composition:

0.58 g. U/l. as UO_2SO_4
0.058 g. Co/l. as $\text{K}_3\text{Co}(\text{CN})_6$
30 g. $\text{SO}_4^{=}$ /l. as MgSO_4
pH = 1.7 adjusted with sulfuric acid
Flow rate = 1 gal/ft.³/min.

This solution was passed through the columns until the IRA-400 beds were saturated with uranium (Table 2, Figure 3). The effluents were analyzed for both uranium and cobalt.

Table 2

	<u>XE-114</u>	<u>IRA-400a</u>	<u>IRA-400b</u>
Amount of resin	27.1 ml.	23.7 ml.	24.1 ml.
Breakthrough capacity	16 mg. U/ml resin	49 mg. U/ml resin	48 mg. U/ml resin
Saturation capacity	31 mg. U/ml resin	60 mg. U/ml resin	52 mg. U/ml resin
Total Cobalt leakage	3.8 mg. Co/ml resin	0	0
Total Cobalt adsorbed	7.0 mg. Co/ml resin	4.4 mg. Co/ml resin	8.2 mg. Co/ml resin

a - with scavenger

b - without scavenger

Table 2 indicates that the bed with scavenger did have a higher uranium capacity than the bed without scavenger. Figures 3 and 4 indicates that in each case the uranium effluent concentration exceeded the uranium influent concentration. This would mean that the cobalticyanide is adsorbed more readily than the uranyl sulfate complex. No cobalt was detected in the effluents from the IRA-400 columns.

The three columns of loaded resin were eluted with a $N/3 \text{ HNO}_3 - 2N/3 \text{ NH}_4\text{NO}_3$ solution, the effluent being analyzed for uranium and cobalt (Fig. 5 and 6). The uranium was not removed as readily from XE-114 as from IRA-400. No cobalt was removed from the IRA-400 column with scavenger. Approximately 4 mg. Co (2% of total adsorbed) was removed from the IRA-400 column without scavenger.

The cobalt removal from XE-114 was slower than that experienced with the 10% sodium hydroxide treatment (Figure 2). The remaining cobalt was eluted with a 5% sodium carbonate solution. (Figure 7). This eluting agent was more effective than the nitric acid - ammonium nitrate system but less effective than the caustic treatment.

The IRA-400 was removed from the burettes, washed, and dried. The resin was ignited and analyzed for cobalt electrolytically (Table 3).

Table 3

	<u>IRA-400 (With scavenger)</u>	<u>IRA-400 (without scavenger)</u>
% residue on ignition	3.20 %	3.84 %
% cobalt as Co_3O_4	2.42 %	3.52 %
% non-cobalt ash	0.78 %	0.32 %

The resin from the column with scavenger was definitely less fouled than the one without scavenger.

Analysis of Cobalticyanide

The method of analysis for cobalt was that reported by Affsprung, Barnes, and Potratz in Anal. Chem. 23, 1680 (1951). An aliquot of solution containing 20 - 600 μ of cobalt was treated with 5 ml. of concentrated nitric acid and evaporated to dryness. The residue was taken up with 2-3 ml of concentrated sulfuric acid and heated to fumes of SO_3 for 5 minutes. The solution was cooled, diluted to 10 ml. with water and the acidity adjusted to pH of 2-6 with aqueous ammonia. Four ml. of 50% ammonium thiocyanate was added followed by sufficient solid ammonium bifluoride to decolorize any red ferric thiocyanate complex. Twelve drops of a 2% solution of tetraphenylarsonium chloride was added and the resultant blue compound extracted with 8 ml. of chloroform. This extraction was repeated with additional tetraphenylarsonium chloride added until the aqueous phase became colorless. The chloroform extractions were diluted to 25 ml. and the cobalt determined colorimetrically at 620 $m\mu$.

Conclusions

Use of a column of XE-111 μ as a scavenger indicates that this resin will decrease the amount of cobalt poisoning of IRA-400 beds. The use of such a system may eliminate this difficulty entirely since the amount of cobalt present in West Rand liquors (1-6 ppm vs. 58 ppm used in this study) would not saturate the XE-111 μ in one cycle as was found here.

Sufficient evidence has been accumulated to show that the use of XE-111 μ will adsorb the trace quantities of cobalticyanide present in South African leach liquor preferentially to the uranium and that the resin can be eluted with sodium hydroxide or sodium carbonate.

A study of the effect of flow rates, pregnant liquor composition, pH, etc. on uranium capacity is now being studied in the laboratory and will be reported shortly. In addition, a cycle dependent upon a carbonate elution is also in progress.

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Figure 6
Elution of Cobalt from KE-111
with N/3 HNO_3 - 2N/3 NH_4NO_3