TOPICAL REPORT WIN-27
DEVELOPMENT OF AN EFFICIENT ELUANT FOR REMOVAL OF MOLYBDENUM
FROM ANION EXCHANGE RESINS

By

Kenneth P. Quinlan and Robert J. Barry

AUG 1 1956

Kenneth P. Quinlan
Kenneth P. Quinlan

Robert J. Barry
Robert J. Barry

Approved by:

A. M. Ross

A. M. Ross
Chemical Department

S. S. Breitenstein
S. S. Breitenstein
Technical Director

Contract AT(49-6)-924

National Lead Company, Inc.
Raw Materials Development Laboratory
Holton Street
Winchester, Massachusetts

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THE DEVELOPMENT OF AN EFFICIENT ELUANT FOR THE REMOVAL
OF MOLYBDENUM FROM ANION EXCHANGE RESINS

ABSTRACT

The poisoning of anion exchange resins by molybdenum adsorption has been briefly studied. Although no satisfactory method was found to prevent the adsorption of molybdenum from pregnant leach liquors, an effective eluant for molybdenum was developed which is superior to the 5-10% sodium hydroxide solutions previously used for this purpose. It was found that the presence of small quantities of a good displacing ion such as chloride or nitrate in dilute solutions of sodium or ammonium hydroxide greatly increases the speed and efficiency of molybdenum removal. Investigations also revealed that by the addition of 0.5 per cent sodium or ammonium hydroxide, RIP barren eluate bleed can be used effectively as an eluant for molybdenum.

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INTRODUCTION

When acid leach liquors obtained from uranium ores containing molybdenum are contacted with an anion exchange resin, both uranium and molybdenum are adsorbed. The adsorbed molybdenum is not removed in the normal uranium elution process, and when the uranium-barren resin is recycled, the resin sites already occupied by molybdenum are unavailable for uranium adsorption. In succeeding cycles, more and more sites become occupied by molybdenum, and the resin capacity for uranium is seriously lowered. This rapid accumulation of an adsorbed species is known as resin "poisoning." The many disadvantages of such a situation are self-evident.

Recent studies (1, 2) have emphasized the seriousness of molybdenum poisoning, and an investigation appeared desirable to find a method to alleviate this problem. This paper presents the results of this investigation.

SUMMARY

An investigation was carried out to find a method for alleviating the problem of resin poisoning by acid leach liquors containing molybdenum. The investigation included the following: a study of the nature of molybdenum adsorption, the possibility of complexing the molybdenum prior to adsorption, the effects of different variables such as pH and sulfate concentration on the adsorption of molybdenum from leach liquors, the applicability of a scavenger system to rid the leach liquor of molybdenum, and the removal of molybdenum from resin after uranium elution.

Observations of the adsorption of molybdenum from leach liquors and the effect of such variables as pH and sulfate concentration on molybdenum adsorption offered no immediate solution to the problem. Studies of a scavenger system showed promise of removing the molybdenum from leach liquors by adsorption on Amberlite IRA-400 resin previously loaded with uranium. Scavenger studies with Amberlite XE-123 indicated that this resin, saturated with uranium, could not be made to adsorb molybdenum adequately at reasonable retention times.

A solution to the molybdenum poisoning problem was obtained by the utilization of aqueous sodium or ammonium hydroxide containing either nitrate or chloride as eluants for molybdenum. It was found that solutions containing as little as 0.5 per cent caustic or ammonia and as little as 0.1 molar chloride or nitrate will effect virtually complete molybdenum removal from Amberlite XE-123 within ten bed volumes. Increasing the concentrations of hydroxide and displacing ion increases the rate of molybdenum elution. No significant difference between the elution characteristics of nitrate and chloride is observed.

The rate of molybdenum elution may be increased by pretreatment of the loaded resin with nitric acid. The use of acidified nitrate solutions for uranium elution would therefore be expected to increase the subsequent rate of molybdenum removal by alkaline nitrate or chloride solutions.

The possibility of using the barren eluate bleed from RIP systems as an eluant for the removal of molybdenum

by the addition of sodium and ammonium hydroxide was investigated. It was found that synthetic bleed solutions become highly effective molybdenum eluants on addition of sodium or ammonium hydroxide. Virtually complete removal of molybdenum was observed with five bed volumes of bleed solution containing 0.5 per cent sodium or ammonium hydroxide.

PRELIMINARY INVESTIGATIONS

Adsorption of Molybdenum and Uranium from Leach Liquors

A sulfuric acid leach liquor from Dakota lignite ore, selected because of its relatively high molybdenum content, was passed through an ion exchange column containing 20 ml of wet-settled Amberlite IRA-400 resin in order to study the loadings of molybdenum and uranium simultaneously. To completely saturate the resin with uranium, 120 bed volumes of leach solution were required. At uranium saturation, the molybdenum loading was 7.5 grams per liter of wet-settled resin. The molybdenum, however, did not reach saturation even at 150 bed volumes, indicating that the resin, although saturated with uranium, was still capable of adsorbing molybdenum, which was found to be slowly displacing the uranium from the resin. This build-up of molybdenum on continued cycles demonstrates the serious nature of this poisoning problem. Since mild reduction of acid leach liquors containing molybdenum results in the formation of "molybdenum blue," a compound containing molybdenum (V) and (VI), it is apparent that the molybdenum in acid leach liquors occurs solely in the hexavalent form. Consequently, all studies were limited to hexavalent molybdenum.

Effect of pH on Molybdenum Adsorption

In order to study the effect of pH on molybdenum adsorption, both distribution and loading data were obtained. Saturation curves were obtained for three synthetic leach solutions, using 20 ml of wet-settled IRA-400 and a retention time of four minutes. The three solutions, each containing 0.75 gram molybdenum per liter as ammonium molybdate, were prepared and adjusted to pH values of 3.0, 6.7, and 11.0, respectively, by the addition of either sulfuric acid or sodium hydroxide. The neutral and alkaline solutions resulted in respective loadings of 79.7 and 77.1 grams molybdenum per liter of wet-settled resin. However, the passage of more than 120 bed volumes of the acid solution through the column failed to completely saturate the resin. The loading at shut-down was 113 grams per liter of wet-settled resin.

The distribution coefficients were determined by equilibrating weighed amounts of Amberlite IRA-400 with solutions containing 0.5 gram molybdenum per liter, at several pH levels between 0 and 12. The results of this study are shown graphically in Figure 1. In Figure 1, a maximum is reached at about pH 3.5, with a sharp decline thereafter. Since studies at constant sulfate concentration also displayed this maximum, it is highly unlikely that the maximum is attributable to competition between the sulfate ion and molybdenum ion. The decline in distribution coefficients at pH values lower than 3.5 may be attributable to steric effects, since the polymolybdates increase in size with decreasing pH.

It is a well-established fact that molybdenum forms polymolybdates in acid solution. Throughout the alkaline range, however, molybdenum is reported to exist simply as MoO_4^{2-} (3). Thus, the constant loadings and distribution coefficients (within experimental error) for the neutral and alkaline solutions reported above, as well as the much different values in the acid region, would be expected.

Effect of Sulfate Concentration on the Adsorption of Molybdenum and Uranium

The effect of sulfate concentration on the adsorption of molybdenum and uranium was studied in the hope that an optimum sulfate concentration could be found at which the molybdenum adsorption would be at a minimum and the uranium adsorption would be at a relatively high value. However, in the range of sulfate concentration from 10 to 80 grams per liter, no such point was found.

As illustrated in Figure 2, the uranium distribution coefficient decreased at approximately the same rate as the molybdenum distribution coefficient did, with increasing sulfate concentration. This phase of the investigation was consequently discontinued.

Scavenger System for Removal of Molybdenum from Leach Liquors

During an investigation of the adsorption characteristics of molybdenum on Amberlite IRA-400 resin, it was observed that at normal retention times (four to six minutes), molybdenum continued to be adsorbed strongly after the resin was saturated with uranium. In fact, the molybdenum was found to be slowly displacing the uranium from the resin. Since it had previously been observed that unusually high molybdenum loadings could be obtained with this resin, the possibility of employing uranium-saturated IRA-400 resin as a scavenger for molybdenum appeared worthy of evaluation.

A 20-ml column of IRA-400 was saturated with uranium from a synthetic leach liquor. This column was then backwashed for cleaning and classification, and Dakota Lignite leach liquor containing 0.07 gram Mo per liter was passed downward through it at a retention time of four minutes. Analysis of the effluent disclosed that an average of 80 per cent of the molybdenum present was adsorbed for 55 bed volumes, with the effluent containing approximately 1.5 grams U_3O_8 per liter and 0.014 gram Mo per liter. At this point, the supply of feed became exhausted, and the test was continued with another batch of Dakota Lignite leach liquor, containing 0.87 gram U_3O_8 per liter and 0.62 gram Mo per liter. The concentration of molybdenum in this liquor was unusually high, since

strenuous leaching conditions had been employed in its production. The column briefly continued to adsorb 80 per cent of the molybdenum, but this gradually dropped to 52 per cent as the throughput was continued to 135 bed volumes. The final effluent was found to contain approximately 1 gram U_3O_8 per liter and 0.29 gram Mo per liter.

Although the extent to which this system may profitably be used with liquors containing realistic concentrations of molybdenum was not conclusively demonstrated, the results of the cursory evaluation described above appear encouraging, and it is reasonably safe to assume that this system would continue to adsorb molybdenum satisfactorily for a considerably larger number of bed volumes with a leach liquor containing more normal concentrations of molybdenum.

Amberlite XE-123 resin was also briefly evaluated as a scavenger for molybdenum, for possible application to RIP systems. A 20-ml bed volume of uranium-saturated XE-123 was used with Dakota lignite leach liquor assaying 0.62 gram molybdenum and 0.98 gram U_3O_8 per liter. Despite the fact that the retention time was increased to six minutes for the larger size resin beads, after the passage of six bed volumes, the effluent was found to contain 0.53 gram molybdenum per liter, or 85 per cent of the molybdenum originally present.

Since XE-123 and IRA-400 have the same active chemical groups, and differ only in structure, they both would be expected to display the same favorable scavenging characteristics toward molybdenum. It would appear, however, that greatly increased retention times may be necessary to permit adequate diffusion and more satisfactory absorption of molybdenum.

ELUANT EVALUATION

Elution of Molybdenum with Sodium Hydroxide Solutions Containing Nitrate or Chloride

In order to determine the effect of the presence of small amounts of good displacing ions such as chloride or nitrate in caustic molybdenum eluants, preliminary studies were conducted with Amberlite IRA-400 previously loaded with pure molybdate solutions. Retention time was ten minutes, with 20-ml bed volumes. A 0.2M NO_3^- solution containing 6 per cent NaOH removed 75 per cent of the molybdenum present on the resin in five bed volumes, while a 10 per cent NaOH solution containing no nitrate removed only 69 per cent in 30 bed volumes. These results clearly indicated that the presence of nitrate ion in the regenerating agent increases both the rate and the efficiency of molybdenum removal. Since similar results were obtained with the addition of chloride ion, a program was initiated to determine optimum concentrations of caustic and displacing ion.

Additional elution tests were therefore conducted with 20-ml bed volumes of Amberlite XE-123 resin, previously loaded with approximately 25 grams Mo per liter. The eluants were 6 per cent sodium hydroxide solutions containing varying amounts of sodium chloride or sodium nitrate. The amounts of molybdenum removed by each eluant were calculated from the molybdenum content of the eluant and the molybdenum content of the resin after elution. The results of these tests, presented in Table I, show that the presence of chloride or nitrate in the caustic greatly increases the rate and efficiency of molybdenum removal, the chloride ion being slightly more effective than the nitrate ion. Virtually complete molybdenum removal was realized with all concentrations of additive studied, as low as 0.1M, with only the rate being increased by increasing the concentrations.

Since elutions conducted with neutral chloride solutions removed only negligible amounts of molybdenum, it can be concluded that the presence of some caustic is necessary in the molybdenum eluant. However, preliminary tests indicated qualitatively that considerably less than 6 per cent caustic could be used effectively in the eluant, and consequently another series of elution tests was performed with varying

concentrations of sodium hydroxide in 0.3M chloride or nitrate solutions. As in previous tests, 20-ml bed volumes of Amberlite XE-123 were used previously loaded with approximately 25 grams Mo per liter of wet-settled resin. From the results of these elutions, presented in Table II, it is apparent that concentrations of sodium hydroxide as low as 0.5 per cent will remove molybdenum efficiently, with the only apparent advantage of higher concentrations being increased rate of removal.

Effect of Reduced Molybdenum on Resin

Several samples of resin poisoned by molybdenum in actual use of the Grand Junction Pilot Plant were available for testing with alkaline chloride or nitrate eluants. Since most of these samples bore the green color characteristic of resins containing reduced molybdenum, they were used to study the relative ease with which reduced molybdenum is removed by the eluants under investigation.

Amberlite XE-123 resin was loaded from a pure solution until it contained 25 grams of molybdenum per liter. The resin column then was exposed to direct sunlight to catalyze the reduction of the molybdenum on it, as evidenced by the appearance of a green color throughout the entire column. A 25-ml column of this resin was eluted along with similar columns of resin poisoned in actual use, to afford a comparison between the two. These resins were eluted as described above with alkaline chloride and alkaline nitrate solutions. There was no significant difference observed in the behavior of the resin poisoned in actual use and the resin loaded from a pure molybdenum solution. The results of this test were compared to the test described in the previous section where the resin exhibited no green color. The comparison is shown in Table III.

The data indicate no significant difference in the rate of molybdenum elution between the reduced species and the molybdenum in its higher oxidation state, although there was a slight difference in the effectiveness of the elution. In all the systems studied, the effectiveness of elution was slightly greater with the oxidized molybdenum, one or two per cent more molybdenum usually being removed.

Nitric Acid Pretreatment of Resin Containing Reduced Molybdenum

Because of the slight difference in elution efficiency observed between the reduced and oxidized molybdenum, the effect of oxidizing the reduced molybdenum on the resin with nitric acid prior to elution with the eluants being studied was investigated. Two types of resins were used. One was the green-colored Amberlite XE-123 which was used in the preceding section, and the other was Permutit SK resin, containing 11.2 grams Mo per liter which had gone through 17 cycles in the CCD-column section at the Grand Junction Pilot Plant. The reduced molybdenum on the resin was oxidized by the passage of 1.0M nitric acid through 20-ml of wet-settled resin until the green color was no longer evident on the resin. The resin was then washed with H₂O until the effluent was neutral to pH paper. The molybdenum was eluted with 4% NaOH containing 0.3M nitrate. The data are shown in Table IV.

The results show that the rate of molybdenum elution is increased by pretreatment with nitric acid. The pretreatment showed little if any effect on the ultimate per cent of molybdenum removed. From these results, it would appear that the use of acidified nitrate solutions commonly employed for uranium elution, would increase the subsequent rate of molybdenum removal upon treatment with NaOH containing chloride or nitrate.

Use of Uranium-Barren Eluate Bleed for Molybdenum Elution

Since the barren eluate bleed from RIP operation contains nitrate, several tests were performed in which synthetic barren eluate bleed was made basic with small amounts of sodium hydroxide. Because of the promising results of these preliminary tests, two series of elutions were performed with synthetic barren eluate bleeds containing 0.7M NO₃⁻ and 90 grams SO₄⁼ per liter. In one series varying concentrations of sodium hydroxide were added, while in the other, ammonium hydroxide was used. The results of these elutions are presented in Table V. It is apparent from these results that both systems are satisfactory for molybdenum elution, and it would appear that an efficient eluate for molybdenum removal could be prepared by the addition of small amounts of sodium or ammonium hydroxide to RIP barren eluate bleed.

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Table I

Elution of Molybdenum from Amberlite XE-123 with 6%
NaOH Containing Varying Amounts of Chloride and Nitrate

Cumulative Per Cent Molybdenum Removed by Bed Volumes

Eluant	1	2	3	4	5	6	7	8	9	10
6%NaOH	28.6	57.8	67.6	73.0	76.7	79.5	81.7	83.6	85.0	86.3
6%NaOH - 0.1M Cl ⁻	25.9	51.7	60.9	68.7	76.7	84.2	89.9	94.2	97.1	98.6
6%NaOH - 0.3M Cl ⁻	32.1	80.5	96.9	98.6	98.7	98.8	98.9	98.9	98.9	99.0
6%NaOH - 0.5M Cl ⁻	28.3	68.2	93.6	98.3	99.3	99.6	99.7	99.7	99.8	99.8
6%NaOH - 0.75M Cl ⁻	34.2	84.3	96.6	99.0	99.6	99.7	99.8	99.8	99.8	99.8
6%NaOH - 1.0M Cl ⁻	41.5	92.1	97.6	99.2	99.6	99.7	99.8	99.8	99.8	99.8
6%NaOH - 1.5M Cl ⁻	45.2	94.5	98.7	99.6	99.8	99.8	99.9	99.9	99.9	99.9
6%NaOH - 0.1M NO ₃ ⁻	32.3	58.6	64.8	70.7	75.7	79.8	84.4	89.2	94.3	99.8
6%NaOH - 0.3M NO ₃ ⁻	36.6	61.7	77.6	93.2	99.0	99.7	99.8	99.8	99.8	99.8
6%NaOH - 0.5M NO ₃ ⁻	44.1	73.4	92.7	97.5	98.6	99.0	99.2	99.3	99.3	99.3
6%NaOH - 0.75M NO ₃ ⁻	49.1	92.3	97.2	98.9	99.3	99.4	99.5	99.6	99.6	99.6
6%NaOH - 1.0M NO ₃ ⁻	46.4	91.2	98.1	99.3	99.5	99.6	99.6	99.7	99.7	99.7
6%NaOH - 1.5M NO ₃ ⁻	56.6	95.7	98.2	99.1	99.3	99.4	99.5	99.6	99.6	99.6

Table II

Elution of Molybdenum from Amberlite XE-123 with Varying
Concentrations of NaOH Containing Constant Amounts of Cl⁻ or NO₃⁻

<u>Cumulative Per Cent Molybdenum Removed by Bed Volumes</u>										
<u>Eluant</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>
0.5%NaOH - 0.3M NO ₃	0.02	0.38	24.7	66.0	94.9	98.4	99.8	99.0	99.0	99.0
1.0%NaOH - 0.3M NO ₃	0.01	5.5	36.0	69.1	95.3	98.7	99.2	99.3	99.3	99.3
2.0%NaOH - 0.3M NO ₃	1.6	26.9	48.9	79.7	97.2	99.0	99.3	99.3	99.3	99.3
3.0%NaOH - 0.3M NO ₃	10.0	36.4	51.9	81.8	97.5	98.9	99.2	99.2	99.3	99.3
4.0%NaOH - 0.3M NO ₃	14.1	38.1	55.7	84.2	98.1	99.5	99.7	99.8	99.8	99.8
6.0%NaOH - 0.3M NO ₃	36.6	61.7	77.6	93.2	99.0	99.7	99.8	99.8	99.8	99.8
0.5%NaOH - 0.3M Cl ⁻	0.02	2.4	24.7	59.9	79.8	90.8	97.1	97.4	98.6	99.2
1.0%NaOH - 0.3M Cl ⁻	0.44	17.2	45.5	72.1	87.4	94.7	97.6	99.3	98.8	99.5
2.0%NaOH - 0.3M Cl ⁻	0.20	26.1	53.1	78.9	92.2	97.0	98.7	99.5	99.3	99.5
3.0%NaOH - 0.3M Cl ⁻	9.0	36.1	62.3	85.1	94.9	97.9	99.0	99.5	99.4	99.5
4.0%NaOH - 0.3M Cl ⁻	14.6	39.2	64.8	88.1	97.0	99.2	99.7	99.8	99.8	99.8
6.0%NaOH - 0.3M Cl ⁻	32.1	80.4	96.4	98.6	98.7	98.8	98.9	98.9	98.9	99.0

Table III

Effect of Reduced Molybdenum Present on Amberlite XE-123
on the Elution with NaOH Containing Cl⁻ or NO₃⁻

Eluant	Color of Resin	Cumulative Per Cent Molybdenum Removed by Bed Volumes									
		1	2	3	4	5	6	7	8	9	10
1%NaOH - 0.3M NO ₃ ⁻	green	0.01	3.2	34.9	65.9	90.3	92.9	93.2	93.3	93.3	93.4
	normal	0.01	5.5	36.0	69.1	95.3	98.7	99.2	99.3	99.3	99.3
2%NaOH - 0.3M NO ₃ ⁻	green	1.5	27.4	48.4	77.9	96.8	98.4	98.6	98.7	98.7	98.8
	normal	1.6	26.9	48.9	79.7	97.2	99.0	99.3	99.3	99.3	99.3
4%NaOH - 0.3M NO ₃ ⁻	green	16.2	41.4	63.6	88.7	97.7	98.5	98.6	98.7	98.7	98.7
	normal	14.1	38.1	55.7	84.2	98.1	99.5	99.7	99.8	99.8	99.8
2%NaOH - 0.3M Cl ⁻	green	0.1	27.0	54.0	79.6	92.0	96.7	98.1	98.6	98.9	98.9
	normal	0.2	26.1	53.1	78.9	92.0	97.0	98.7	99.3	99.5	99.5
4%NaOH - 0.3M Cl ⁻	green	13.0	40.7	66.6	87.0	94.8	97.6	98.3	98.5	98.6	98.6
	normal	14.6	39.2	64.8	88.1	97.0	99.2	99.7	99.8	99.8	99.8

Table IV

Effect of Nitric Acid Prior to Molybdenum Elution

with 4% NaOH Containing 0.3M NO₃⁻

		<u>Cumulative Per Cent Molybdenum Removed by Bed Volumes</u>									
Resin	Treatment	1	2	3	4	5	6	7	8	9	10
Amberlite XE-123	12 B.V. of 1.0M HNO ₃ prior to elution	36.0	84.9	96.7	97.4	97.6	97.8	98.0	98.0	98.0	98.0
Amberlite XE-123	Eluted directly	16.2	41.4	63.6	88.7	97.7	98.5	98.6	98.7	98.7	98.7
Permutit SK	15 B.V. of 1.0M HNO ₃ prior to elution	19.4	71.7	93.4	96.5	97.2	97.7	98.0	98.3	98.5	98.7
Permutit SK	Eluted directly	0.01	53.2	88.1	94.8	96.1	96.7	96.9	97.2	97.4	97.5

Table V

Elution of Molybdenum from Amberlite XE-123 with Varying
Concentrations of NaOH Containing 0.7M NO₃⁻(NaNO₃) and 90 g SO₄/1(NH₄)₂SO₄

<u>Cumulative Per Cent Molybdenum Removed by Bed Volumes</u>											
<u>Eluant</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	
0.5%NaOH - 0.7M NO ₃ - 90 g SO ₄ /1	4.2	29.8	72.4	95.4	97.0	97.2	97.5	97.7	97.8	97.8	
1.0%NaOH - 0.7M NO ₃ - 90 g SO ₄ /1	15.7	62.6	94.9	96.7	97.5	97.8	98.1	98.2	98.3	98.4	
2.0%NaOH - 0.7M NO ₃ - 90 g SO ₄ /1	17.2	74.2	95.5	97.6	98.3	98.3	98.4	98.5	98.6	98.7	
4.0%NaOH - 0.7M NO ₃ - 90 g SO ₄ /1	23.6	77.4	96.4	98.1	98.6	98.7	98.9	98.9	99.0	99.0	
6.0%NaOH - 0.7M NO ₃ - 90 g SO ₄ /1	25.7	75.5	95.8	98.9	98.6	98.8	99.0	99.1	99.2	99.2	
0.5%NH ₄ OH - 0.7M NO ₃ - 90 g SO ₄ /1	1.9	21.9	58.1	91.1	97.1	97.8	98.0	98.2	98.3	98.4	
1.0%NH ₄ OH - 0.7M NO ₃ - 90 g SO ₄ /1	15.6	58.8	95.9	97.6	98.4	98.6	98.8	98.9	98.9	99.0	
2.0%NH ₄ OH - 0.7M NO ₃ - 90 g SO ₄ /1	19.5	82.2	96.9	98.3	98.8	98.9	99.0	99.1	99.2	99.2	
3.0%NH ₄ OH - 0.7M NO ₃ - 90 g SO ₄ /1	33.1	81.4	97.1	98.5	98.9	99.1	99.2	99.3	99.3	99.4	
4.0%NH ₄ OH - 0.7M NO ₃ - 90 g SO ₄ /1	36.8	83.7	97.3	98.6	99.0	99.2	99.3	99.4	99.4	99.4	
6.0%NH ₄ OH - 0.7M NO ₃ - 90 g SO ₄ /1	40.5	85.6	97.9	98.9	99.2	99.3	99.4	99.4	99.4	99.4	

Figure I
Molybdenum Distribution on IRA-400 As A Function of pH

Figure II
Distribution Coefficients for Molybdenum and Uranium
as a Function of the Sulfate Concentration