

Original article

Continuous Ion Exchange Separation of Uranium from Copper Sulphate Solution

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Abstract

This experiment demonstrates the technique of radiochemical separation of uranium from copper using ion-exchange resin. A mixture of uranyl sulphate and copper sulphate solutions was passed through an ion-exchange column packed with Amberlite resin (IRA-420, chloride form), where uranium was retained on the resin as the complex anion $[\text{UO}_2(\text{SO}_4)_3]^{4-}$. Copper was first eluted with distilled water, followed by elution of the retained uranium complex using ammonium nitrate solution. All collected aliquots were measured by gamma-ray spectroscopy under low-background counting conditions, with the system calibrated against a ^{152}Eu source. The results confirm successful separation of uranium from copper, with an average uranium recovery of 83.8% achieved upon elution with ammonium nitrate. The uranium daughter product, ^{234}Th , passed through the column to a greater extent than uranium itself, indicating that separation between uranium and its own decay products was less complete than the uranium-copper separation.

Keywords. Ion-exchange Resin, Uranium-copper Separation, Radiochemical Separation, Amberlite IRA-420, Gamma-ray Spectroscopy.

Introduction

In various hydrometallurgical processes, aqueous solutions contain significant concentrations of dissolved metal ions such as cobalt, gold, uranium, and copper. This experiment focuses on methods and apparatus used for the treatment of metal-containing solutions. More specifically, it investigates a continuous ion-exchange technique that is particularly effective for the selective concentration and removal of metal ions from aqueous solutions, either for resource recovery or contamination control purposes [1].

Advancements in chemical precipitation and ion-exchange techniques are currently being developed in national laboratories to improve separation efficiency and operational performance [2]. Ion exchange is a well-established chemical separation method that utilizes columns packed with ion-exchange resins. These resins are widely applied in separation, purification, and decontamination processes. They contain mobile ions that can be exchanged with ions present in the surrounding solution as it flows through the resin bed.

Uranium can be extracted from two principal types of leaching systems: acid leaching and carbonate leaching. In carbonate leach solutions, uranium predominantly exists as the anionic complex $[\text{UO}_2(\text{CO}_3)_3]^{4-}$ [1]. In contrast, in sulfuric acid leach solutions, uranium may be present either as the uranyl cation UO_2^{2+} or as negatively charged sulfate complexes such as $[\text{UO}_2(\text{SO}_4)_2]^{2-}$ and $[\text{UO}_2(\text{SO}_4)_3]^{4-}$ [2], [3]. The chemical form of uranium in solution strongly influences its behavior during ion-exchange processes, as the charge and stability of these complexes determine their affinity toward anion or cation exchange resins [4], [5].

Ion exchange (IX) is a reversible process involving the transfer of ions between a solution and a solid insoluble medium [4]. Ion-exchange resins operate through functional groups permanently attached to a polymeric matrix; as a counter-ion solution passes through the resin, ions of opposite charge bind electrostatically to these groups until equilibrium is established, with ions of higher valency and larger ionic size generally exhibiting stronger affinity [4], [5]. In this experiment, the resin used was Amberlite IRA-420 (Cl⁻), a strongly basic anion-exchange resin containing chloride ions as exchangeable counter-ions [4-6].

This paper describes a continuous ion-exchange procedure for separating uranium from copper in a mixed sulphate solution, and for assessing the extent to which uranium can be separated from its own decay daughter, ^{234}Th , under the same conditions.

Methods**Materials**

A 10% w/v uranyl sulphate solution and a 10% w/v copper sulphate solution were used as the feed solutions, with ammonium nitrate solution used as the eluting agent. The stationary phase was Amberlite IRA-420 anion-exchange resin (chloride form), packed into a glass ion-exchange column. Gamma-ray spectrometric analysis was performed using a detector calibrated against a ^{152}Eu standard source.

Ion-Exchange Reaction

Uranium was retained on the resin as the complex sulphate anion, according to the reaction:

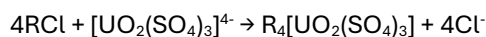


Figure 1. Ion-exchange resin beads.

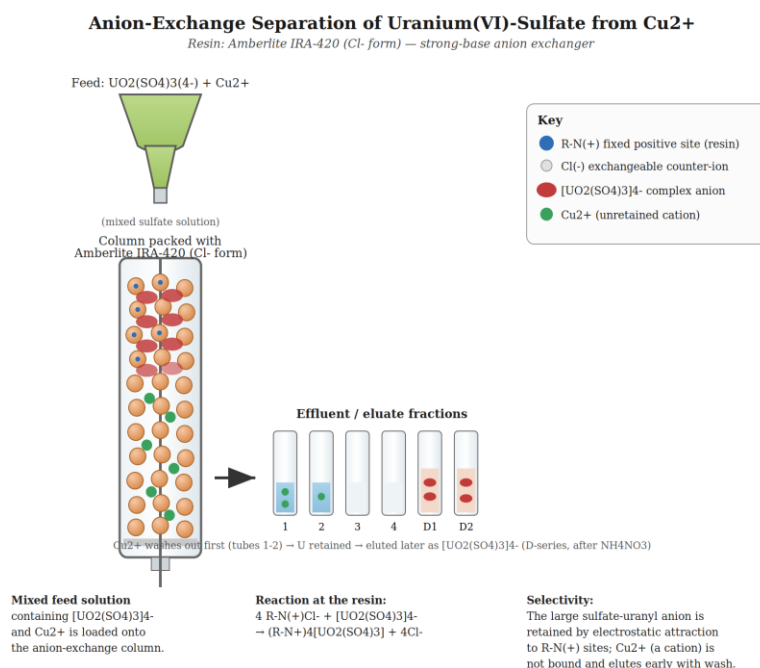


Figure 2. Anion-exchange separation of the uranium-sulfate complex from Cu²⁺ on Amberlite IRA-420 (Cl⁻ form).

Figure 2. Ion-exchange separation process.

Experimental Procedure

1. Calibration of the gamma-ray spectrometer using a europium (Eu) standard source.
2. Background spectrum measurement of uranyl sulfate for 300 seconds under low-background conditions.
3. Preparation of a mixed solution by combining uranyl sulfate (20 mL, 10% w/v) and copper sulfate (80 mL, 10% w/v) to produce a green solution in which uranium is present as the complex anion $[\text{UO}_2(\text{SO}_4)_3]^{4-}$.
4. Wash the column with 60 mL of distilled water.
5. Collection of the distilled-water wash in three 20 mL aliquots, labeled A1, A2, and A3.
6. Passage of the prepared mixed solution through the ion-exchange column at a controlled flow rate of 5-10 mL/min.
7. Collection of the column effluent in 20 mL aliquots, labeled sequentially B1 to B5.
8. Washing of the column with distilled water, collecting fractions C1-C6.
9. Testing of the final wash fraction (C6) for residual copper using dilute ammonia solution.
10. Additional washing (60 mL of water) until complete removal of copper ions.
11. Elution of the retained uranium using ammonium nitrate solution, collecting eluate fractions D1-D9.
12. Gamma-ray spectrometric analysis of all collected aliquots under low-background counting conditions.

Results

For system calibration, a ^{152}Eu source was used to establish the energy of each peak. (Table 1) lists the four main gamma-ray peaks used to compare the aliquots against the original uranyl sulphate solution.

Table 1. Gamma-ray energy peaks in the original uranyl sulphate solution.

γ -rays peak (keV)	Counts (in 300s)
234Th (62.97 keV)	1353 $\pm$ 4.01%
234Th (92.38 keV)	5885 $\pm$ 1.98%
235U (143.7 keV)	260 $\pm$ 10.91%
235U (185.57 keV)	1175 $\pm$ 3.85%

Table 2. Counts on each aliquot and their associated errors.

Aliquot No.	234Th (62.97 keV)	234Th (92.38 keV)	235U (143.7 keV)	235U (185.57 keV)
A1	56 $\pm$ 14.39	213 $\pm$ 7.38	35 $\pm$ 19.05	197 $\pm$ 7.39
A2	32 $\pm$ 19.05	57 $\pm$ 15.28	35 $\pm$ 17.85	114 $\pm$ 9.75
A3	2 $\pm$ 93.99	16 $\pm$ 27.44	6 $\pm$ 46.65	30 $\pm$ 18.54
B1	14 $\pm$ 29.28	44 $\pm$ 15.85	9 $\pm$ 39.82	27 $\pm$ 19.57
B2	17 $\pm$ 28.52	181 $\pm$ 7.92	5 $\pm$ 52.69	17 $\pm$ 24.91
B3	27 $\pm$ 22.62	31 $\pm$ 34.85	3 $\pm$ 93.13	8 $\pm$ 47.21
B4	74 $\pm$ 12.85	4 $\pm$ 268.48	3 $\pm$ 88.60	2 $\pm$ 162.26
B5	39 $\pm$ 29.52	0	4 $\pm$ 75	5 $\pm$ 70.29
C1	32 $\pm$ 31.35	0	0	6 $\pm$ 48.54
C2	30 $\pm$ 25.49	0	0	0
C3	56 $\pm$ 15.54	0	4 $\pm$ 60.81	2 $\pm$ 99.41
C4	76 $\pm$ 12.88	303 $\pm$ 6.04	0	3 $\pm$ 135.26
C5	63 $\pm$ 14.30	209 $\pm$ 7.58	2 $\pm$ 204	7 $\pm$ 45.63
C6	0	15 $\pm$ 36.38	2 $\pm$ 108.37	1 $\pm$ 177.9
C7	48 $\pm$ 15.63	139 $\pm$ 9.41	3 $\pm$ 125.80	12 $\pm$ 31.57
C8	17 $\pm$ 44.83	192 $\pm$ 8.05	1 $\pm$ 25	14 $\pm$ 26.73
C9	18 $\pm$ 28.42	80 $\pm$ 11.73	4 $\pm$ 94.29	9 $\pm$ 37.52
D1	134 $\pm$ 10.24	540 $\pm$ 4.66	16 $\pm$ 33.06	45 $\pm$ 16.34
D2	180 $\pm$ 8.62	634 $\pm$ 4.34	39 $\pm$ 20.23	133 $\pm$ 9.14
D3	136 $\pm$ 9.46	448 $\pm$ 5.91	38 $\pm$ 20.68	1 $\pm$ 100.00
D4	90 $\pm$ 12.07	378 $\pm$ 5.91	44 $\pm$ 16.40	167 $\pm$ 8.15
D5	147 $\pm$ 9.77	576 $\pm$ 4.68	24 $\pm$ 33.09	170 $\pm$ 8.00
D6	88 $\pm$ 12.33	275 $\pm$ 6.82	42 $\pm$ 19.23	152 $\pm$ 8.50
D7	59 $\pm$ 14.56	148 $\pm$ 9.92	18 $\pm$ 36.95	132 $\pm$ 8.96
D8	31 $\pm$ 22.56	128 $\pm$ 9.91	11 $\pm$ 51.36	78 $\pm$ 12.19
D9	6 $\pm$ 78.09	37 $\pm$ 18.15	5 $\pm$ 73.95	21 $\pm$ 23.50

Table 2 lists the four main gamma-ray peaks for all aliquots. The aliquot C6 was mixed with dilute ammonia because a small amount of copper solution remained in that fraction; this was done to test for residual copper and accounts for the variation seen between aliquots.

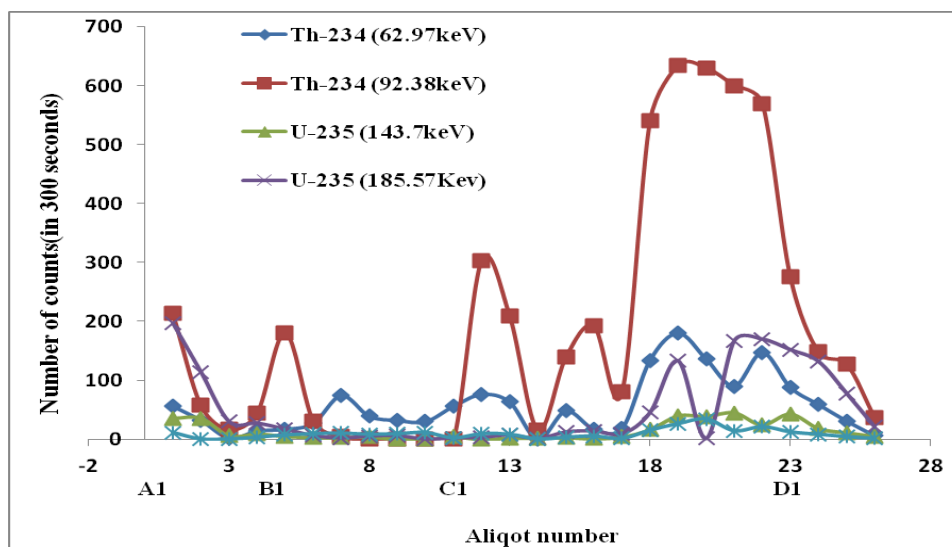
**Figure 3. Aliquot gamma-ray activity during the ion-exchange separation of uranium.**

Figure 3 provides a qualitative visual comparison of the relative abundances of ^{235}U and ^{234}Th across the aliquots, and shows the markedly higher concentration of ^{234}Th passing through the column relative to ^{235}U .

Dilution and Recovery Calculations

Combining 20 mL of uranyl sulphate (10% w/v) with 80 mL of copper sulphate (10% w/v) diluted the uranyl sulphate to an outset concentration of 2% w/v. When 200 mL of ammonium nitrate was subsequently passed through the column, the average concentration of uranium in the eluate would be 1% if all of the retained uranium complex anion were recovered in that volume, i.e. the uranium would be diluted rather than concentrated by the elution step.

The percentage uranium concentration in each aliquot was calculated by comparing its gamma-ray peak counts with those of the original uranyl sulphate solution. For the ^{235}U peak at 143.7 keV (original count 260 ± 10.91 in 300 s) in aliquot A1 (35 ± 19.05 counts in 300 s):

Percentage uranium concentration (U_1) = $(35 \pm 19.05 / 260 \pm 10.91) \times 100 = 13.46\% \pm 2.95\%$

For the second uranium peak (U_2 , original count 1175 ± 3.85 in 300 s; aliquot A1 count 197 ± 7.39):

Percentage uranium concentration (U_2) = $(197 \pm 7.39 / 1175 \pm 3.85) \times 100 = 16.77\% \pm 1.40\%$

The percentage recovery of uranium was then calculated by comparing the total counts across all D aliquots (the ammonium nitrate eluate fractions) with the original solution:

Percentage recovery (U_1) = $(237 \pm 274.35 / 260 \pm 10.91) \times 100 = 91.15\% \pm 25.14\%$

Percentage recovery (U_2) = $(899 \pm 164.18 / 1175 \pm 3.85) \times 100 = 76.51\% \pm 42.64\%$

Average percentage recovery of uranium = $(91.15\% + 76.51\%) / 2 = 83.8\% \pm 33.9\%$

Discussion

The results demonstrate that the Amberlite IRA-420 anion-exchange resin selectively retained the anionic uranium-sulphate complex $[\text{UO}_2(\text{SO}_4)_3]^{4-}$ while allowing copper to pass through the column largely unretained, consistent with the resin's selectivity for anionic species over the cationic Cu^{2+} ion. The average uranium recovery of 83.8% upon elution with ammonium nitrate confirms that the bulk of the retained uranium complex anion could be recovered from the resin, though the relatively large uncertainty ($\pm 33.9\%$) reflects the low count rates and correspondingly large statistical errors in several of the D-series aliquots.

A notable finding is that the uranium daughter product ^{234}Th passed through the column to a substantially greater extent than uranium itself. This indicates that, while the separation of uranium from copper was achieved effectively, the separation of uranium from its own decay daughters was less complete, since ^{234}Th does not form the same stable anionic sulphate complex and is therefore not retained by the anion-exchange resin in the same way. This distinction is important for applications where isolating uranium from its decay products, rather than from other metals, is the primary objective.

A limitation of the present study is that copper removal was confirmed qualitatively rather than quantitatively. Residual copper was tested only on the final wash fraction (C6) using dilute ammonia solution, following the original laboratory protocol [7]; quantitative copper concentration measurements (e.g., by atomic absorption or ICP-OES spectroscopy) were not performed across the A-, B-, and C-series fractions. Quantitative copper monitoring across all effluent and wash fractions is recommended in future work to more rigorously confirm the extent and timing of copper breakthrough.

Conclusion

This experiment demonstrated a continuous ion-exchange method for separating uranium from copper, in which a mixed feed solution was passed through an Amberlite IRA-420 resin bed. Most of the uranium was retained on the resin as the complex anion $[\text{UO}_2(\text{SO}_4)_3]^{4-}$, while copper passed through largely unretained. The retained uranium was subsequently flushed from the resin using ammonium nitrate solution, with an average recovery of 83.8%.

The separation of uranium from copper was achieved successfully; however, separation of uranium from its own decay daughter, ^{234}Th , was less complete, with ^{234}Th passing through the column to a much greater extent than uranium itself. Overall, the experiment relied on comparing the gamma-ray spectra of the different solutions and aimed to characterise the separation behaviour of both the target metals and the uranium decay daughters.

Acknowledgments

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Conflicts of Interest

The authors declare no conflicts of interest.

References

1. Helfferich F. Ion Exchange. New York (NY): McGraw-Hill; 1962.
2. Kunin R. Ion Exchange Resins. 2nd ed. New York (NY): Wiley; 1958.
3. Calmon C, Kressman TRE, editors. Ion Exchangers in Organic and Biochemistry. New York (NY): Interscience Publishers; 1957.

4. Harris DC. Quantitative Chemical Analysis. 9th ed. New York (NY): W. H. Freeman and Company; 2016.
5. Fritz JS, Schenk GH. Quantitative Analytical Chemistry. 5th ed. Boston (MA): Allyn and Bacon; 1987.
6. SAMCO. What Is Ion Exchange Resin and How Does It Work? [Internet]. 2017 [cited 2021 Oct 6]. Available from: <https://www.samcotech.com/ion-exchange-resin-work-process>
7. University of Surrey. Laboratory script, REP 5 – Ion Exchange Separation of Uranium from Copper. Surrey (UK): University of Surrey; 2010 Sep.
8. Dardel F, Arden TV. Ion Exchangers. In: Ullmann's Encyclopedia of Industrial Chemistry. Weinheim (Germany): Wiley-VCH; 2008.
9. Aryal S. Ion Exchange Chromatography [Internet]. Microbe Notes; 2018 [cited 2021 Oct 3]. Available from: <https://microbenotes.com/ion-exchange-chromatography/>