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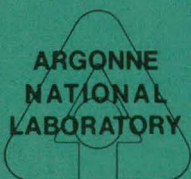
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**THE SEPARATION OF RUBIDIUM FROM
IRRADIATED ALUMINUM-ENCAPSULATED URANIUM**

by

**E. P. Horwitz, F. J. Schmitz,
and D. J. Rokop**

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Chemistry Division

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January 1982

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ABSTRACT

A procedure was developed for separating rubidium from irradiated aluminum encapsulated uranium. The separations procedure produces a final ultra-high purity RbCl product for subsequent high performance mass spectrometric analysis. The procedure involves first removing most of the macro-components and fission products by strong base anion exchange using, first, concentrated HCl, then oxalic acid media and second, selectively separating rubidium from alkaline-earth ions and other alkali-metal ions, including cesium, using Bio-Rex[®]-40 cation-exchange resin. The resultant RbCl is then put through a final vacuum sublimation step. Ultra-pure reagents and specially clean glassware are used throughout the procedure to minimize contamination by naturally-occurring rubidium.

INTRODUCTION

This report describes a procedure for the separation of rubidium from dissolved irradiated uranium. The general processing approach is to remove most of the macro-components and fission products by anion exchange using, first, concentrated hydrochloric acid, and then oxalic acid media, and to selectively separate rubidium from alkaline earths and other alkali metals using Bio-Rex[®]-40 cation-exchange resin. The resultant rubidium chloride is then put through a final vacuum sublimation step. The objective of the separations scheme is to obtain a final RbCl product (starting with $\sim 1-5 \times 10^{15}$ atoms of Rb) of such high purity that a steadily increasing rubidium ion current is obtained when aliquots (of the final product) are heated on filaments for mass spectrometric analysis.

The separation procedure was developed using one-to-five volume percent (v/o) aliquots of a mock-up solution containing 3.04 grams (0.113 moles) of aluminum, 0.899 grams (3.78×10^{-3} moles) of uranium, and 7.05 grams (0.0351 moles) of mercury per 100 ml of 4 M HNO₃. The components of this solution are representative of dissolved irradiated uranium samples encapsulated in aluminum described in reference 1. The aliquots of the mock-up solution were spiked with tracer concentrations of fission products and/or various isotopic mixtures of rubidium.

The high-purity reagents and ion-exchange resins used in the separation procedure are listed in Appendixes A and B. Chromatographic column sizes and packing methods, special cleaning procedures for glassware, and special reagent purifications are also described in Appendixes A and B. A description of the equipment used for vacuum sublimation of RbCl is given in Appendix C.

Chemical Procedure

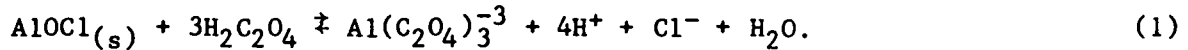
The chemical processing procedure is divided into the following three steps:

1. Removal of major gross constituents from the rubidium by anion exchange in HCl and oxalate media.
2. Selective chromatographic separation of rubidium from other alkali metals by cation exchange using Bio-Rex[®]-40 resin.
3. Final ultra-purification of rubidium from microgram quantities of organic and inorganic impurities by vacuum sublimation.

1A. A one-to-five v/o aliquot of the mock-up solution is converted from nitrate to chloride salts by evaporation to dryness twice in 6 M HCl, using an acid-leached and -annealed beaker. (See Appendix A.) The residue is then dissolved in ~2-4 ml of 8 M HCl containing 10 v/o of 8 M HCl saturated in Cl₂. The Cl₂ oxidizes any Pu(III) to (IV) and Hg(I) to (II). Rubidium, together with the other alkali metal ions, is then separated from a large number of elements by conventional anion exchange in 8 M HCl using a disposable column packed with AG1-X8, 100-200 mesh resin. Under these conditions, U(VI) and Pu(IV), as well as most of the elements in families VA through VIIIA, IB, and IIB, are absorbed on the resin as their chlorometallic complexes and/or oxygenated anions.² [Cr(III), Mn(II), Co(II), and Ni(II) are exceptions.] The alkali, alkaline earth, aluminum(III), lanthanide(III), and transplutonium(III) ions are not absorbed on the column.

A 15 cm x 0.7 cm i.d. bed is required for a 5 v/o aliquot whereas a 10 cm x 0.7 cm i.d. column bed is used for smaller size aliquots. The first three-to-four free column volumes (FCV) of eluate contain the entire alkali metal ion fraction in addition to all of the aluminum. After collecting the rubidium fraction, the entire column is discarded.

1B. The rubidium fraction (contained in ~6-8 ml of 8 M HCl) is evaporated completely to dryness under an I.R. lamp. The resultant white residue, which is a predominantly aluminum oxychloride, is dissolved in 0.2 M H₂C₂O₄. A minimum of 15-20 ml of 0.2 M H₂C₂O₄ solution is required for dissolution of the aluminum oxychloride for each 1 v/o aliquot of mock-up solution. Dissolution of the aluminum oxychloride is facilitated by stirring and heating under an I.R. lamp. The oxalate anion complexes aluminum according to the following equation:



Rubidium, together with the other alkali-metal ions, is then separated from the tris-oxalatoaluminum(III) complex by anion exchange in 0.2 M $\text{H}_2\text{C}_2\text{O}_4$ using a column packed with AG1-X8, 100-200 mesh resin. Under these conditions, Al(III), Ti(IV), Zr(IV), V(V), Mo(VI), Fe(III) and U(VI) are absorbed on the resin as oxalato complexes.³ The alkali, alkaline earths, lanthanide(III), and transplutonium(III) ions are not absorbed on this column and are completely eluted in the oxalic-acid eluate plus one FCV of 0.2 M $\text{H}_2\text{C}_2\text{O}_4$ rinse. The feed solution is usually made 0.1 M in H_2O_2 to aid in solubilizing any gross quantities of V(V), Ti(IV), and Mo(VI). Approximately 6 ml of bed volume of anion-exchange resin is required for each 1 v/o aliquot of mock-up solution (0.2 ml of bed volume/mg of Al). As much as 60-70% of the column capacity can be consumed by the tris-oxalatoaluminum(III) complex and still achieve a decontamination factor of aluminum from alkali metals of 10^5 .

The anion-oxalate column can be reused by stripping the oxalato-metal complexes listed above using the following elution sequence: 5 FCV's H_2O , 10 FCV's 6 M HCl, and 10 FCV's of 0.1 M HCl. Aluminum is eluted in the 6 M HCl fraction.

Rubidium is then removed from the oxalic-acid eluate by absorbing it on a column packed with AG50-X8, 100-200 mesh resin on the H^+ cycle in H_2O . Approximately 1 ml of bed volume per 7-8 ml of oxalic-acid eluate is used to ensure complete absorption of the rubidium. The pH of the oxalic-acid solution (see Eq. 1) also has a pronounced influence on the absorption of Rb^{+1} by the cation-exchange resin and should not be below 1.0. If no less than 15-20 ml of 0.2 M $\text{H}_2\text{C}_2\text{O}_4$ solution is used to dissolve 30 mg of Al, then the pH of the oxalic-acid eluate from the anion-oxalate column will be in a safe range, i.e., >1.0. After absorption of rubidium, together with other alkali metal, alkaline earth, lanthanide(III), and transplutonium(III) ions, by the cation-exchange resin, the column is eluted with 5-10 FCV's of H_2O (thoroughly rinsing the reservoir) and 7 FCV's of 4 M HCl. The 4 M HCl completely elutes the alkali metals (Rb) ions and most of the alkaline earths ions. The lanthanide(III) and transplutonium(III) ions remain largely on the column. Any aluminum present in the oxalic-acid load solution does not absorb on the cation column due to the stability of the tris-oxalato complex. Thus, an additional decontamination (probably 10^3) of aluminum from rubidium is obtained on this column.

The cation column can be reused by stripping the lanthanide(III) and transplutonium(III) ions with 15-20 FCV's of 6 M HCl followed by H_2O elution to pH = 4.

2. The 4 M HCl fraction containing the RbCl and other alkali-metal chlorides is evaporated to dryness under a heat lamp and the resultant residue (which is very minute at this stage) is dissolved in ~1-2 ml of H_2O . The alkali-metal fraction (in H_2O) is then loaded onto a small calibrated Bio-Rex®-40

cation-exchange resin column with a bed size of 5 cm x 0.054 cm² and a FCV of 0.135 ml. Since the K_d for Rb^{+1} absorption from H_2O is $>10^3$, a large number of H_2O rinses of the beaker used for the evaporation may be carried out without any loss of Rb .⁴ After loading the column, the alkali metal ions are separated by eluting with 0.75 M HCl at 50°C. The rubidium fraction is collected in a 1 ml polypropylene centrifuge tube. A typical separation of Na(band I), Rb(band II) and Cs(band III) is shown in Fig. 1. The rubidium fraction cut points are located between 6 and 10 FCV's. Except for a small cross-contamination of potassium ions, which elutes between Na and Rb, the rubidium fraction is essentially free from other alkali metal ions. The decontamination from Na and Cs is $>10^3$ and $>10^5$, respectively. Calcium(II), aluminum(III), lanthanide(III), and transplutonium(III) ions elute well behind the rubidium band, especially the latter two groups of elements. However, a small microgram quantity of yellow resinous material bleeds off the Bio-Rex[®]-40 column during elution. This organic material leaves a residue on evaporation which reduces the emission of rubidium ions during the mass spectrometric analysis of the final sample. Once the rubidium band is eluted from the column (10 FCV's), the cesium can be quickly eluted using ~4 FCV's of 4 M HCl.

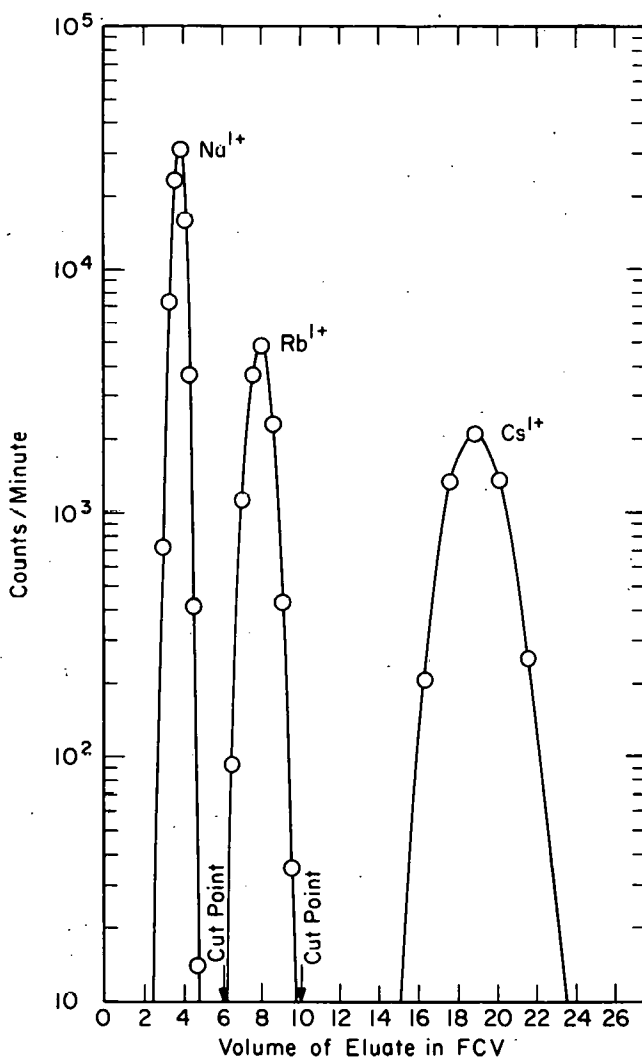


Fig. 1

Separation of Na^{+1} (band I), Rb^{+1} (band II), and Cs^{+1} (band III) Using Bio-Rex[®]-40 Cation-Exchange Resin (40-50 μ m). Elutrient 0.75 M HCl, 50°C, and column bed size 5 cm x 0.054 cm². FCV = 134 μ l. ANL Neg. No. 122-1482.

The Bio-Rex[®]-40 column can be reused by stripping with ~10 FCV's of 4 M HCl followed by 10 FCV's of H₂O to a pH = 4. Once the column is calibrated, at least five column runs can be made before another calibration is necessary.

3. The rubidium fraction (~5-6 x 10² μl in volume and 0.75 M in HCl) is given a final chromatographic purification and concentration using a mini-cation-exchange column 2 cm x 2 mm i.d. (0.06 ml bed volume and 0.03 ml FCV) in size and packed with 17-25 μm diameter AG50-X8 resin on the hydrogen cycle in H₂O. After loading the rubidium fraction from above, the column is washed with 5 FCV's of H₂O. The Rb⁺¹ ions are then stripped from the column into a 1 ml polypropylene centrifuge tube using 3-4 FCV's of 4 M HCl. At this point in the process, the overall chemical yield of rubidium is ~99%.

The rubidium fraction (~90 μl in volume) is transferred in 30 μl portions into a small platinum dish (~50 μl in volume and 1 cm in diameter) using a Rainin P200 digital Pipetman[®] and a polypropylene disposable pipet and evaporated to dryness under an I.R. lamp. A very small residue is usually visible in the platinum dish. The platinum dish is then placed on a pedestal in the vacuum sublimation apparatus and covered with a 1 ml quartz beaker. (See Appendix C.) After closing the apparatus and connecting it to a vacuum manifold, the pressure is reduced to 10⁻² - 10⁻³ mm Hg. The apparatus is closed off at the pressure stopcock, removed from the vacuum manifold, and placed inside the copper coils of an induction heater. The platinum dish is then heated to redness for approximated 1-2 seconds, three successive times. Over 99.5% of the RbCl is sublimed from the platinum dish. Approximately 91-92% of the total RbCl originally deposited in the platinum dish is collected in the 1 ml quartz beaker. A small residue is still present in the platinum dish after sublimation; however, the quartz beaker is perfectly clean and shows no visible sign of any residue. One hundred microliters of 2 M HCl is added to the quartz beaker to dissolve the sublimed RbCl. Two microliter aliquots of this sample are dried on a tantalum filament for mass spectrometric analysis.

RESULTS

The separation scheme described above gives an ultra-pure RbCl product which consistently gives a steadily increasing rubidium ion current when heated on filaments for mass spectrometric analysis. Filament loadings are normally in the range of 1-5 x 10¹³ atoms; however, as little as 2 x 10¹² atoms of rubidium have also produced an increasing rubidium ion current on the mass spectrometer.

The overall yield of rubidium is ~90% with the major loss occurring during the vacuum sublimation step. Improved recovery of rubidium during sublimation can probably be achieved by cooling the bottom of the inverted quartz beaker. However, this modification would require a more involved sublimation apparatus. The vacuum sublimation step as described is very simple and fairly rapid. The

sublimation step can be eliminated (and still achieve good emission behavior of Rb) by starting the chemistry with 10^{16} atoms (or more) of rubidium, but still loading $\sim 2 \times 10^{13}$ atoms of Rb on the filament. In this case, the ratio of rubidium atoms to contaminant atoms is sufficiently high for analytical conditions.

Natural rubidium samples which have gone through the chemistry have given the same isotopic composition (within the experimental mass spectrometric error) as the starting material. However, when rubidium samples containing high 87/85 ratios are put through the chemistry, the level of natural rubidium in reagents and glassware, as well as that introduced adventitiously, becomes important. Rubidium blank determinations measured by isotopic dilution analysis are in the range of 5×10^{12} - 10^{13} atoms of natural rubidium. Efforts are being made to lower this level still further. Because of the level of natural rubidium found in the blank, 4-5 v/o aliquots containing $4-5 \times 10^{15}$ atoms of fission-product rubidium have been put through the chemistry. In this case, the isotopic composition agrees (within experimental error) with that of the starting material.

APPENDIX A

Reagents and Glassware

Water - Ultra-pure water was obtained from a Milli-Q2 system water purifier (Millipore Corp., Bedford, Massachusetts). This water has a specific resistance at 25°C of 18 megohm-cm and contains no more than ~25 ppb of total dissolved inorganics and less than 10 ppb of heavy metals. The ultra-pure water was used for the preparation of all reagents and for rinsing all leached glassware.

Glassware - All glassware, such as beakers (Pyrex and quartz), centrifuge tubes, transfer pipets, and ion-exchange columns were cleaned by soaking in hot 6 M HCl for several hours, rinsing in ultra-pure water, and drying in an oven at 110°C. The glassware (except quartz) which was used after the oxalate anion column was cleaned as described above and then wrapped in aluminum foil and annealed at 500°C. This procedure makes the glass surface more resistant to subsequent leaching.

HCl Solutions - All hydrochloric acid solutions were prepared using Ultrex® grade acids (J. T. Baker Chemical Co., Phillipsburg, New Jersey) and ultra-pure water. The resultant acid solutions were stored in acid-leached polyethylene bottles.

Oxalic Acid - The oxalic-acid solution was prepared from recrystallized analytical-grade oxalic acid and ultra-pure water. Isotopic dilution analysis of this oxalic-acid solution showed $\sim 1 \times 10^{13}$ atoms of natural Rb/100 ml of 0.2 M solution. This rubidium content can be reduced to an insignificant level by eluting the oxalic-acid solution through an ion-exchange column packed with AG50-X8 resin on the hydrogen cycle. The cation column is first preconditioned with 10 FCV's of high purity 6 M HCl, 10 FCV's of H₂O, and 5 FCV's of 0.2 M H₂C₂O₄. The following oxalic-acid eluate should be collected directly in the beaker containing the aluminum oxychloride to be dissolved. This procedure avoids the problem of storage of the ultra-pure oxalic acid. The ion-exchange column can be stored in 0.2 M H₂C₂O₄. At least 100 FCV's of 0.2 M H₂C₂O₄ can be eluted through the cation column before it is necessary to re-precondition the column as described above.

APPENDIX B

Ion-Exchange Columns

All ion-exchange resins were purchased from Bio-Rad Laboratories, Richmond, Calif. Columns were packed by forcing an aqueous slurry (usually 0.1 M HCl) of the resin into the column by applying nitrogen pressure at the ball joint of the column reservoir.

Anion-Exchange Columns, AG1-X8, 100-200 Mesh - The HCl-anion-exchange column was packed in a 0.7 cm x 10 cm or 0.7 cm x 15 cm Bio-Rad disposable column made from polypropylene and Pyrex glass. This column was eluted with ultra-pure H₂O until a colorless eluate was obtained after standing overnight in the H₂O, and then preconditioned with 10 FCV's of 8 M HCl.

The oxalic-acid anion-exchange column was made from 1.35 cm i.d. Pyrex glass tubing and was fitted at the top with a reservoir containing a ball joint and at the bottom with a coarse glass frit. This column was also preconditioned with H₂O as described above, followed by 10 FCV's 0.1 M HCl, 5 FCV's of H₂O and 6 FCV's of 0.2 M H₂C₂O₄.

Cation-Exchange Column, AG50-X8, 100-200 Mesh - The large cation-exchange column was made from 1 cm i.d. Pyrex glass tubing and was fitted at the top with a reservoir containing a ball joint and at the bottom with a coarse glass frit. This column was also extensively washed with H₂O as described above, followed by 10 FCV's of 6 M HCl and 10 FCV's of H₂O until a pH of 4 was attained.

Mini-Cation-Exchange Column, AG50-X8 (17-25 μ diameter) - The mini-column was made from a 2 mm i.d. microbore (MB-2-150) Laboratory Data Control (LDC) type column by sealing a small reservoir-ball joint to a 3 cm length of tubing. The LDC outlet fittings and Teflon-bed support were used for the bottom of the column. The stainless steel porous frit in the LDC Teflon bed support was replaced with a piece of polypropylene wool. A 1 μ m Nuclepore membrane filter was then placed on top of the Teflon bed support to prevent small particles of resin from passing into the eluate.⁵ This column design gives very small column dead space below the packed bed (~2 μ l for the mini-column). Preconditioning of the mini-column was identical to that of the large cation column.

Cation-Exchange Column, Bio-Rex[®]-40 - The Bio-Rex[®]-40 column was made from a 3 mm i.d. microbore (MB-3-150) Laboratory Data Control column as described above. The LDC outlet fittings and bed support described above were also used for the bottom of the Bio-Rex[®]-40 column. A 40-50 μ m particle size fraction of Bio-Rex[®]-40 resin was separated from the 200-400 mesh commercial material by elutriation using a 0-20 ml/min Beckman Model 746 metering pump. Prior to separating the desired particle size fraction, the Bio-Rex[®]-40 resin was washed extensively by elutriation to remove the "fines." The column was slurry packed in 4 M HCl with pressure and preconditioned using 10 FCV's of 4 M HCl and 10 FCV's of H₂O until a pH of 4-5 is reached. Carrier-free ⁸³Rb (83d) was used to calibrate the column, although the Rb⁺¹ consistently peaked at 7.5-8.0 FCV's on three separate packed columns, when loaded from H₂O and eluted with 0.75 M HCl at 50°C.

APPENDIX C

Vacuum Sublimation Apparatus and Platinum Dish Fabrication

A drawing of the apparatus used for the vacuum sublimation of RbCl is shown in Fig. 2. The arrangement of the platinum dish and cover beaker on the pedestal is also shown in Fig. 2.

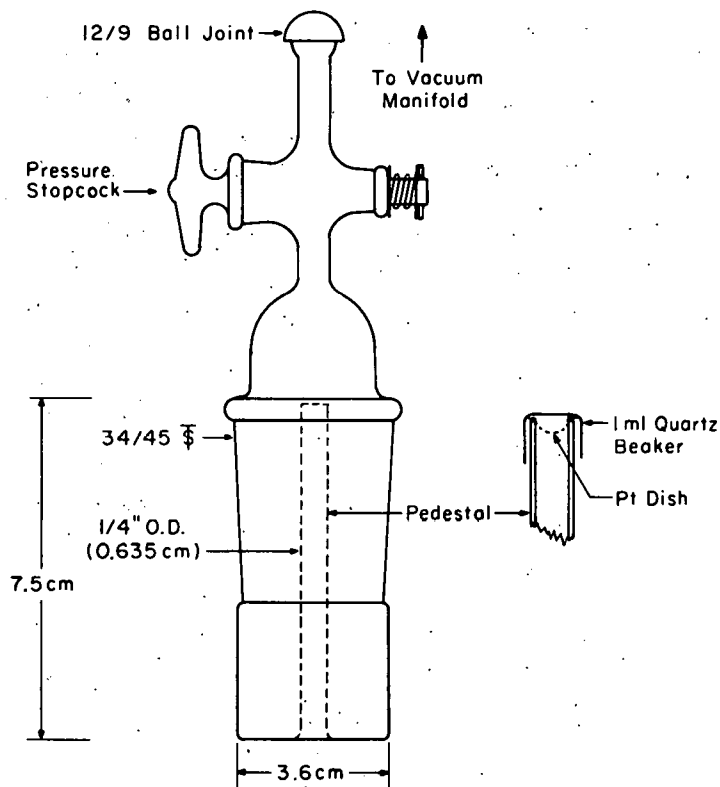


Fig. 2

Apparatus for Vacuum Sublimation of RbCl. ANL Neg. No. 122-1481.

Platinum dishes were fabricated by placing a 15/16 inch diameter platinum disc in a 15/16 inch diameter opening (1/4 inch deep) in the bottom section of a brass die. The platinum disc fits tightly in the opening and is centered directly over a dish-shaped depression 1/4 inch in diameter and 1/8 inch deep. The top piece of the die, which fits in the 15/16 inch opening of the bottom section, contains a centered 3/8 inch diameter hole fitted with a rod. A die shaped platinum dish was produced by placing a 3/8 inch stainless steel ball bearing in the hole in the top of the die, replacing the rod, and then giving the rod a tap with a small hammer. Excess platinum was trimmed from the dish with a scissors.

Platinum dishes were cleaned by leaching in boiling 6 M HCl for several hours, and rinsing thoroughly with ultra-high purity H₂O.

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REFERENCES

1. A. H. Jaffey, E. P. Steinberg, J. E. Gindler, J. Gray, Jr., E. P. Horwitz, J. P. Hughes, and F. J. Schmitz, *Branching Fraction in the Radioactive Decay of ^{85m}Kr* , Argonne National Laboratory Report ANL-79-107 (Feb 1980).
2. K. A. Kraus and F. Nelson, *Proc. First Intern. Conf. Peaceful Uses At. Energy*, Geneva, Switzerland, Vol. 7, 113 (1956).
3. F. W. E. Strelow, C. N. Liebenberg, and F. Von S. Toerien, *Anal. Chim. Acta* 47, 251 (1969).
4. F. W. E. Strelow, C. N. Liebenberg, and F. Von S. Toerien, *Anal. Chim. Acta* 43, 465 (1968).
5. E. P. Horwitz, C. A. A. Bloomquist, and W. H. Delphin, *J. Chromatog. Sci.* 15, 41 (1977).

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