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Separation of Plutonium, Uranium, and
Neodymium in Mixed Uranium-Plutonium Fuels
for Burnup Measurement

by

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IMPROVED TWO-COLUMN ION EXCHANGE SEPARATION OF PLUTONIUM, URANIUM, AND NEODYMIUM IN MIXED URANIUM-PLUTONIUM FUELS FOR BURNUP MEASUREMENT

by

S. F. Marsh, M. R. Ortiz,
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ABSTRACT

Fractions of plutonium, uranium, and neodymium are separated for the isotopic-dilution, mass-spectrometric determination of burnup in mixed uranium-plutonium fuels. The separation uses two sequential ion exchange columns operated at room temperature. The major procedural operations are fuming of the sample with HF and HClO_4 ; transferring with 12M HCl to an anion exchange column; converting the effluent containing neodymium, other fission products, and americium to a nitrate medium; isolating neodymium by chromatographic elution on a second anion exchange column with a $\text{HNO}_3\text{-CH}_3\text{OH}$ elutriant; and eluting plutonium from the first column with 0.1M HCl-12M HCl followed by elution of uranium with 0.1M HCl. This procedure is an improvement of one published previously. The effects of variables upon the operations of this improved method are discussed with emphasis on ion exchange resins and column operation.

I. INTRODUCTION

In the fission process a portion of the initial nuclear fuel converts to lighter elements with the release of energy. The extent of this conversion is known in the nuclear energy field as burnup, defined here as

Burnup = Atom percent fission

$$= (100) \frac{\text{Number of fissions}}{\text{Initial number of heavy atoms}} .$$

Burnup is a fundamental measurement essential to both advanced fuel development studies and the evaluation of power reactor operation.

The most reliable technique for measuring burnup is based on destructive analyses for a selected fission

product and the residual heavy atoms in a representative sample of the irradiated fuel. This technique is applicable to fuels of all compositions at all burnup levels; has small error contribution from uncertainties of cross-section data, even for fast reactor spectra; and does not require the analysis of an archive, unirradiated fuel sample.¹ Desirable properties of a fission-product burnup monitor are: (1) an accurately known fission-yield value that is essentially constant for different fissioning nuclides and changes little with neutron energy, (2) insensitivity to production or depletion from parasitic neutron captures such that the number of atoms is directly proportional to the number of fissions, and (3) no migration, relative to uranium and plutonium, within the fuel.

The rare earths, which comprise about one-half of the heavy fission-product peak, most effectively satisfy these criteria. Individual and selected sums of stable

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neodymium isotopes are especially useful as burnup monitors for various mixed uranium-plutonium fuels now being developed and proposed for fast breeder reactors. About two years ago, we developed a sequential separation procedure that provided individual fractions of plutonium, uranium, and neodymium designed for burnup determination by the technique of isotope-dilution mass spectrometry.² This procedure was characterized by using two ion exchange columns as contrasted to as many as five major operations of previous procedures.³⁻⁵ The purpose of this report is to describe recent improvements in the procedure as it currently is used.

II. RECOMMENDED EXPERIMENTAL PROCEDURE

The operations of this procedure, as outlined in Fig. 1, are discussed in sequence. Details, such as reagent preparation and volumes of ion exchange elutriants, are given in Sec. III.

A. Fuming with Hydrofluoric and Perchloric Acids

This step of the original procedure has been retained without significant change. The sample is added to a Kynar (or Teflon) vial that contains a measured quantity of a triple spike (usually ^{242}Pu , ^{233}U , and ^{150}Nd) mixture. Hydrofluoric acid and HClO_4 are added, and the mixture is fumed to dryness. This treatment dissociates polymeric plutonium species and oxidizes plutonium to Pu^{6+} , thereby giving chemical identity to the sample plutonium isotopes and the spike ^{242}Pu . Glass vials are not used in order to avoid contamination from leachable natural uranium and neodymium.

B. Anion Exchange Separation of Plutonium, Uranium, and Neodymium

The major change in this step is substituting a macroporous resin, AGMP-1, 50-100 mesh, for the conventional

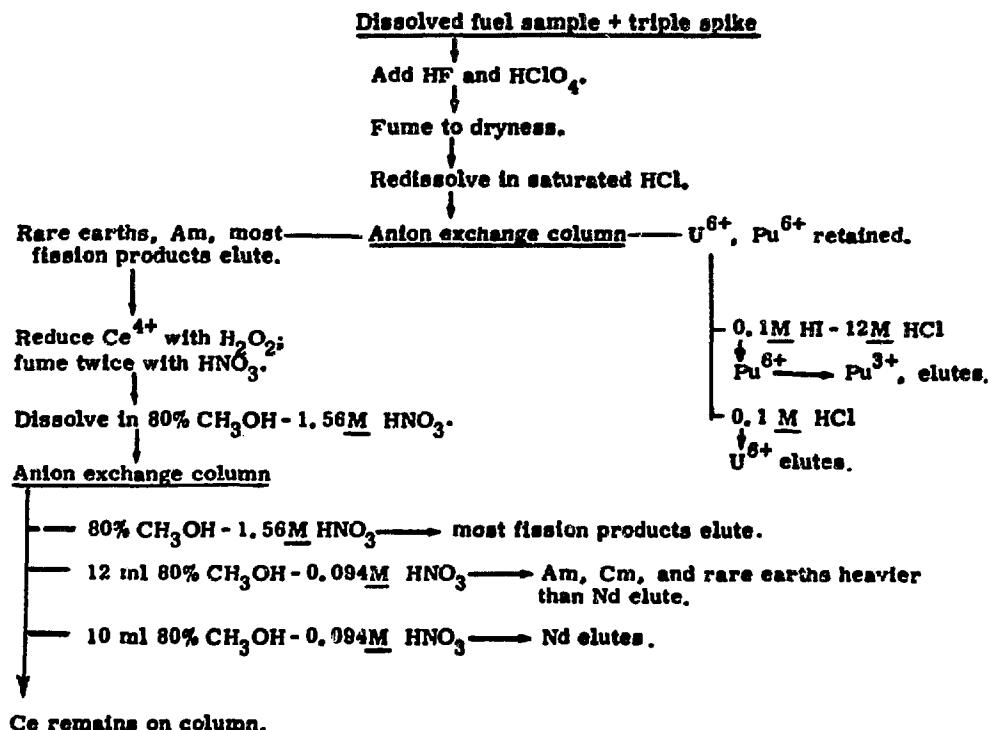


Fig. 1.
Outline of procedure.

ion exchange resin, AGI-X10, 50-100 mesh, used in the original procedure. A polyethylene ion exchange column is used instead of the former custom-fabricated glass column. It is easily made by simply cutting off the top of an inexpensive, commercially available polyethylene dropper. The low cost (about \$0.05 per dropper) of the polyethylene ion exchange column permits one-time usage, thereby avoiding potential cross-contamination from reusing glass columns. Volumes of the elutriants were changed proportionally for the dimensions of the new column.

The residue resulting from the HF-HClO₄ fuming is dissolved in saturated HCl and transferred to the ion exchange column. This reagent is prepared by passing HCl gas into quartz-distilled HCl using polyethylene ware. Analytical reagent grade HCl is not recommended because it contains variable amounts of natural uranium and neodymium.

As shown in Fig. 1, Pu⁶⁺ and U⁶⁺ sorb on the anion exchange resin from strong HCl, whereas neodymium, other rare earths, americium, and most fission products do not. The sorption is stronger for Pu⁶⁺ than for Pu⁴⁺, with the distribution coefficient (K_d) exceeding 1000 from 12M HCl for both valence states.⁶

After flushing the nonsorbed ions from the column with saturated HCl, plutonium is eluted with a 0.1M Hf-saturated HCl mixture. Because the reduction to the nonsorbed Pu³⁺ is not rapid, wait 10 min after the bulk of the plutonium is eluted to permit essentially complete reduction. The remaining plutonium then is eluted, again with 0.1M Hf-saturated HCl. Uranium is still retained as U⁶⁺, which then is eluted with 0.1M HCl.

A typical elution sequence, using the AGMP-1 resin and the conditions detailed in Sec. II, is presented as Fig. 2. The separation factors for americium and uranium, relative to plutonium, and for plutonium, relative to uranium, are essentially the same as obtained for the previously used AGI-X10 resin. These values are 3×10^2 , 1×10^5 , and 1×10^3 , respectively.

Chemists from several laboratories informed us of difficulties they encountered with the separation using batches of AGI-X10 resin different from that we used. Our investigation of AG-1 resins of varying mesh size and cross-linkage (Sec. II.D) revealed that the particle-size distribution and elution characteristics varied among resin lots claimed to be identical. Macroporous resin has been used successfully to overcome variable separation efficiencies obtained with different batches of conventional gel-type anion exchange resin.⁷ Reported advantages of macroporous resin included higher loading per unit of resin, elution in a smaller volume, and higher decontamination factors. Of the various resins we evaluated, an available lot (Bio-Rad lot number 9881) of AGMP-1,

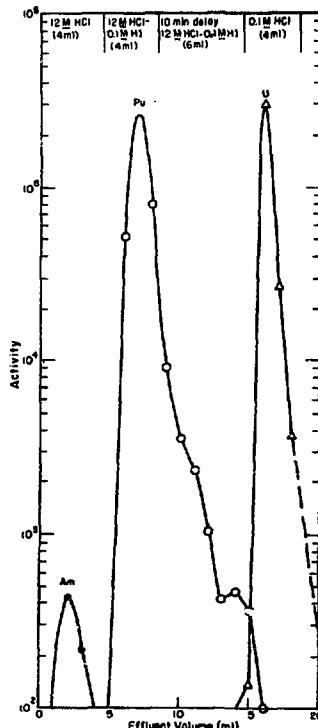


Fig. 2.
Separation of americium, plutonium, and uranium on first anion exchange resin column.

50-100 mesh, macroporous resin provided the cleanest separation of plutonium and uranium. This resin, used for routine samples in our laboratory, is recommended.

C. Treatment of First-Column Eluate for Chromatographic Separation of Neodymium

This step of the original procedure has not been changed. The neodymium-containing eluate is evaporated, fumed with concentrated HNO₃, and then fumed with dilute HNO₃ plus H₂O₂. This removes chloride and reduces fission-product cerium, partially oxidized to Ce⁴⁺ in the earlier fuming with HClO₄, to Ce³⁺. A chloride-free system is necessary for reproducible chromatographic separation for neodymium. The elution position of Ce⁴⁺ overlaps that of neodymium, whereas the position of Ce³⁺ does not.

D. Chromatographic Separation of Neodymium

The major change in this step is substituting AGMP-1, 200-400 mesh, anion exchange resin for the previously used, AG1-X2, 200-400 mesh resin. Again the inexpensive polyethylene column is substituted for the previously used glass column.

Rare earths form only weak nitrate complexes in aqueous HNO_3 . Complex formation is enhanced at higher nitrate concentrations, attainable by adding nitrate salts, by adding a miscible organic solvent, or by adding both.⁸⁻¹² The chromatographic separation of neodymium from other fission products including adjacent rare earths, used in this procedure, is based on anion exchange of nitrate complexes in a $\text{HNO}_3\text{-CH}_3\text{OH}$ medium.³

The neodymium-containing residue is transferred with a high concentration $\text{HNO}_3\text{-CH}_3\text{OH}$ loading solution to an AGMP-1 anion exchange column. The high nitrate concentration provides for the sorption of all rare earths in a narrow band at the top of the resin bed. The individual rare earths then are eluted chromatographically with a weaker $\text{HNO}_3\text{-CH}_3\text{OH}$ solution.

In the original procedure elution conditions had been established to give a well-separated neodymium fraction for a certain lot of AG1-X2, 200-400 mesh resin. These conditions were used for about 18 months, until this lot of resin was exhausted. During these 18 months, chemists from several other laboratories reported unsatisfactory separations using the recommended conditions. Our need to change to another resin lot coupled with other laboratories' reported difficulties prompted an investigation of the among-lot differences of ion exchange resins.

Microscopic examination of resin lots labeled identical in type, cross-linkage, and mesh size showed significant differences in absolute particle size and in particle-size distribution. Because particle size affects flow-rate and bead-diffusion kinetics which, in turn, affect ion exchange characteristics, different lots of identically labeled 1-X2 resins were size graded by aqueous settling. The rare earth elution patterns obtained for different lots of equal grade-size resins still differed greatly. This indicated that other factors, probably associated with the resin composition, differed among lots. From the practical standpoint, the different results emphasized the need to establish experimentally the elution conditions for each resin lot.

Several size-graded lots of AG1-X2, 200-400 mesh resin were evaluated for the chromatographic separation using variable eluting solution compositions. For these studies, radiotracers $^{152,154}\text{Eu}$, ^{149}Pm , ^{147}Nd , and ^{144}Ce served to establish the elution positions and separation factors. For usefulness in the intended burnup determination application, a neodymium fraction should be

obtained with about 90% neodymium recovery, less than 0.1% cerium, and reasonably low levels of other elements. With the previously used eluting agent of 0.008M $\text{HNO}_3\text{-90\% CH}_3\text{OH}$, satisfactory chromatographic separation of neodymium was not obtained with any of the AG1-X2, 200-400 mesh, size-graded lots evaluated. Variable eluting reagent compositions then were tested with none giving a satisfactory separation. Furthermore, the resin columns contained a substantial portion of the added ^{147}Nd activity, even after completing the chromatographic separation, when the ^{147}Nd activity in the effluent had become negligible. The activity in the resin columns, believed due to irreversible diffusion of the liquid phase into the resin particles, probably increases with increasing organic solvent content. For these reasons a macroporous resin, AGMP-1, was evaluated.

The effects that varying CH_3OH and HNO_3 levels have upon the elution characteristics of neodymium, using AGMP-1, resin, 200-400 mesh, (Bio-Rad lot number 10627) are presented in Fig. 3. An eluting solution of 0.094M $\text{HNO}_3\text{-80\% CH}_3\text{OH}$ (prepared as described in Sec. II) was selected. Once again, it is emphasized that

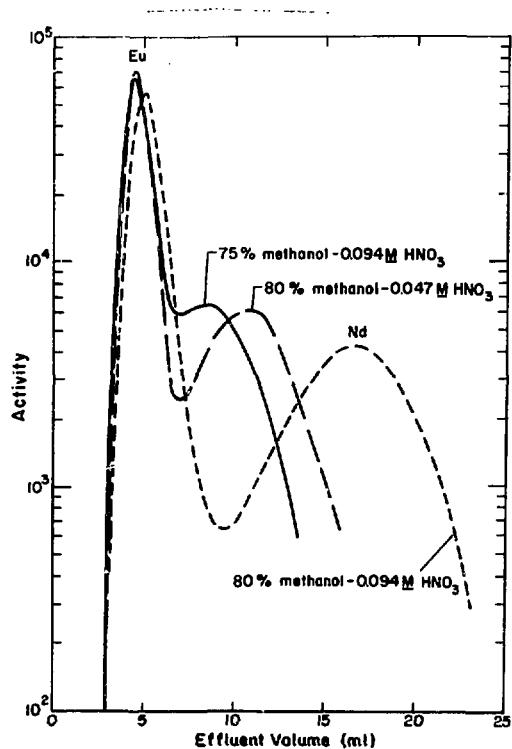


Fig. 3.
Effect of varying CH_3OH and HNO_3 concentration of the eluting solution on the chromatographic separation of neodymium.

this solution is applicable to this particular resin lot and that experiments will be necessary to establish conditions for other resin lots.

The chromatographic separation of rare earths is one of the most demanding separations for inorganic ions. The most critical factors affecting the separation are the aqueous-CH₃OH composition and the flow rate. To maintain a consistent aqueous-CH₃OH composition throughout the column operation, the CH₃OH content of the solution used for loading the neodymium-containing sample onto the column was decreased to 80% from the previously used 90%. The HNO₃ concentration was increased to 1.56M from 0.8M to ensure initial sorption of the nitrate complexes in a narrow band at the top of the column. The columns are operated in a transparent plastic enclosure containing an open container of the eluting reagent. This maintains an atmosphere saturated with a vapor identical to that above the columns, minimizing evaporation of the eluting reagent and consequent composition change. To maintain a constant flow of the eluting solution under a constant head pressure, a simple apparatus was devised. The eluting solution is placed in a 30-ml polyethylene bottle which is inverted into the column reservoir. The design ensures that the volume of liquid flowing from the bottle to the reservoir exactly equals that flowing from the column. An added benefit of using an automated flow is that the analyst is free for other tasks during the two approximately 2-h periods of column elution.

III. DETAILS OF RECOMMENDED PROCEDURE

A. Ion Exchange Columns and Resins

1. **Column and Flow-Rate Bottle Preparation.** Use commercial, disposable polyethylene droppers, 6-mm-i.d., 60-mm-long, with a 2-ml reservoir. (Suitable droppers are obtainable from the Nalge Co., Rochester, N.Y. 14625, article No. 6219.) Cut off the top of the dropper to form the reservoir, and place a small wad of quartz wool in the tip.

The polyethylene column reservoir may be enlarged and the circumference made round, if desired, by inserting a 1-dram vial into the reservoir, heating with a hot air gun, and then cooling the plastic reservoir with H₂O to retain its altered configuration.

Plastic 30-ml dropping bottles, to be used for automatic column operation, are modified by cutting the delivery tip to a length of about 15 mm. It may be necessary to enlarge the tip opening (with a heated glass rod) to obtain proper flow characteristics when these bottles are inverted into the second column reservoirs.

Flow rates of 0.1 ± 0.02 ml/min are suitable. The combination of tip length of these bottles and reservoir height of the columns determines the height of the liquid head in the reservoir column operation.

2. **First Column Resin.** Transfer a water slurry of AGMP-1, 50-100 mesh, chloride-form resin to a polyethylene column until the settled height is just below the reservoir. Pass 6 ml of saturated HCl through the column. Keep wet with saturated HCl until use. (Do not prepare a column until a sample is ready.)

3. **Second Column Resin.** Prepare equilibrated resin and store as follows. Fill a 30-mm-diam, 120-mm-high glass column with a distilled water slurry of AGMP-1, 200-400 mesh, chloride-form resin. Pass 160 ml of 8M HNO₃, then 160 ml of loading solution (1.56M HNO₃ - 80% CH₃OH; see Sec. III.B, Reagents) through the column. Verify that the resin is free of chloride by adding AgNO₃ to the final effluent solution; no AgCl should form. Transfer the equilibrated resin with loading solution to a storage bottle until ready to use.

When a sample is ready, transfer equilibrated AGMP-1, 200-400 mesh resin to a polyethylene column until the settled height is just below the reservoir. Keep wet with the loading solution until use.

B. Reagents

1. **Hydrochloric Acid (Saturated, 0.1M).** Using polyethylene apparatus, saturate quartz-distilled HCl with HCl gas. Cooling in an ice bath is necessary because the reaction is exothermic. Verify that the HCl is at least 12M by titrating with standard NaOH solution. Prepare 0.1M HCl by diluting 1 ml of the saturated HCl to 120 ml with quartz-distilled water. Store the saturated HCl in a quartz bottle and the 0.1M HCl in a polyethylene bottle.

2. **Hydriodic Acid-Hydrochloric Acid Mixture (0.1M HI - 12M HCl).** Dilute 1 ml of distilled HI to 55 ml with saturated HCl. Prepare this reagent fresh before use. The HI is distilled, preferably in a quartz apparatus, to remove H₃PO₄, the holding reductant in AR grade HI. Store the distilled HI in flame-sealed quartz ampoules or in plastic containers under an inert atmosphere.

3. **Hydrofluoric Acid (1M).** Dilute 3 ml of concentrated HF to 91 ml with quartz-distilled water and store in a polyethylene dropping bottle.

4. **Nitric Acid (15.6M, 1M).** Use quartz-distilled HNO₃ for the 15.6M reagent. Mix 10 ml of quartz-distilled

HNO_3 and 146 ml of quartz-distilled water for the 1*M* reagent. Store the 15.6*M* HNO_3 in a quartz bottle and the 1*M* HNO_3 in a polyethylene bottle.

5. Perchloric Acid (1*M*). Mix 8 ml of concentrated (12*M*) HClO_4 and 88 ml of quartz-distilled water, and store in a polyethylene bottle.

6. Hydrogen Peroxide (30%). Refrigerate this commercial reagent when not in use.

7. Loading Solution (1.56*M* HNO_3 in 80% CH_3OH). Prepare 100 ml of 7.8*M* HNO_3 by diluting 50 ml of quartz-distilled HNO_3 to 100 ml with distilled water. Prepare the loading solution by diluting 20 ml of this 7.8*M* HNO_3 to 100 ml with AR grade anhydrous CH_3OH . The CH_3OH must be free of organic impurities such as aldehydes. A sniff-test usually is adequate to verify purity. Store the 7.8*M* HNO_3 and the loading solution in quartz or preleached, borosilicate glass bottles with tight-fitting stoppers.

8. Eluting Solution (0.094*M* HNO_3 in 80% CH_3OH). Prepare 100 ml of 0.47*M* HNO_3 by diluting 3.00 ml of quartz-distilled HNO_3 to 100 ml with distilled water in a 100-ml volumetric flask. Store this reagent in a quartz or polyethylene bottle with a tight-fitting stopper.

Prepare the eluting solution just before use by pipetting 20.0 ml of the 0.47*M* HNO_3 into a 100-ml volumetric flask and diluting to the mark with AR grade anhydrous CH_3OH . The CH_3OH must be free of organic impurities as described above.

C. Sample Analysis

1. Add the sample to a Teflon or Kynar tube containing the mixed ^{233}U - ^{242}Pu - ^{150}Nd spike.

2. Add 2 drops of 1*M* HF and 10 drops of 1*M* HClO_4 .

• The HF serves to dissociate polymerized species of plutonium.

3. Evaporate just to dryness, using a heated copper block and a hot-air jet.

• Do not start the hot-air jet until the solution has refluxed for 30 min, and do not bake the residue or it may not dissolve completely in the next operation.

• Fuming HClO_4 serves to oxidize plutonium to Pu^{6+} .

4. Add 0.5 ml of saturated HCl, and agitate the tube to dissolve the residue.

5. Prepare a first ion exchange column as previously described.

• Collect the 6 ml or more of saturated HCl effluent in a waste receptacle.

6. Place a Teflon or Kynar tube, labeled with the sample designation plus "Nd eluate," under the column.

7. Transfer the dissolved residue from Step 4 to the column with a new polyethylene dropper.

8. Add 0.5 ml of saturated HCl to the sample tube as a rinse, then transfer it to the column with the polyethylene dropper used in Step 7.

• Discard the dropper and sample tube after the rinse is transferred to the column.

• To ensure that the residue was dissolved and transferred to the column, gamma count the empty tube relative to the column with a portable meter.

9. Pass 4 ml of saturated HCl through the column in 1-ml increments using a new polyethylene dropper.

• Rinse the reservoir walls with each 1-ml increment, waiting between increments for the reservoir to empty.

• Neodymium, other fission products, and americium are eluted.

10. Evaporate the neodymium effluent to dryness in a copper block using *only* the hot-air jet until the volume has been reduced by at least 20%; then the copper block also may be heated.

• The saturated HCl will outgas too vigorously if initially heated from below.

• The next operation for the neodymium fraction is Step 17.

11. Place a clean Teflon or Kynar tube, labeled with the sample designation plus "Pu fraction," under the first column.

12. Pass 4 ml of 0.1*M* HI-saturated HCl through the column using a new polyethylene dropper; then wait 10 min for the complete reduction to Pu^{3+} before proceeding to Step 13.

• Plutonium reduces to Pu^{3+} , which elutes.

• Reserve this tube for the mass spectrometric determination of plutonium.

13. Place a waste receptacle under the column; then pass 6 ml of 0.1*M* HI-saturated HCl through the column.

• Plutonium tailings elute and are discarded.

14. Pass 0.5 ml of 0.1*M* HCl through the column using a new polyethylene dropper.

• Rinse the reservoir walls when adding this reagent.

• This completes the elution of plutonium with no significant elution of uranium.

15. Place a clean Teflon or Kynar tube, labeled with the sample designation plus "U fraction," under the column.

16. Pass 4 ml of 0.1M HCl through the column using the dropper from Step 4; then discard the column.
 - Uranium is eluted.
 - Reserve the "U fraction" tube for the mass spectrometric determination of uranium.
17. Add 0.5 ml of 15.6M HNO₃ to the tube containing the dried neodymium effluent from Step 10, and evaporate to dryness.
 - Chloride is volatilized.
18. Add 2 drops of 30% H₂O₂ and 1 ml of 1M HNO₃, and again evaporate to dryness.
 - Ce⁴⁺ reduces to Ce³⁺.
19. Add 0.5 ml of loading solution, and agitate the tube to dissolve the residue.
20. Prepare a second ion exchange column as previously described.
 - The following operations are done in the plastic enclosure containing an open vessel of eluting solution.
21. Place a waste receptacle under the column.
22. Transfer the dissolved residue from Step 19 to the column with a new polyethylene dropper.
23. Add another 0.5 ml of loading solution to the tube as a rinse; then transfer it to the column with the polyethylene dropper used in Step 22.
24. Pass another 2 ml of loading solution through the column.
 - Most fission products other than rare earths are eluted.
25. Pass 12 ml of eluting solution through the column.
 - Using a pipet, rinse the reservoir walls by adding two 1-ml portions of the eluting solution. Wait for the reservoir to empty before adding the second 1-ml portion.
 - Deliver the next 10 ml of eluting solution from a polyethylene bottle inverted into the column reservoir.
 - When not removing solution, keep the volumetric flask containing the eluting solution stoppered so that evaporation does not change the aqueous-methanol ratio.
 - Americium and rare earths heavier than neodymium are eluted.
26. Place a clean Teflon or Kynar container, labeled with the sample designation plus "Nd fraction," under the column.
27. Pass 10 ml of eluting solution through the column from the polyethylene bottle inverted in the reservoir.
 - Neodymium is eluted.
 - Reserve the "Nd fraction" container for the mass spectrometric determination of neodymium.

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