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**RADIOCHEMICAL PURIFICATION PROCEDURES
FOR THE ELEMENTS**

RELEASED FOR ANNOUNCEMENT
IN NUCLEAR SCIENCE ABSTRACTS

Livermore, California

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UNIVERSITY OF CALIFORNIA
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Livermore, California
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Compiled by Manfred Lindner

July 13, 1965

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FOREWORD

The forerunner of this report, UCRL-4377, was compiled in 1954 principally for internal distribution and use. However, over the years an unexpected number of requests was received for copies of the compilation from many laboratories in both this country and abroad. In the meantime the number of radiochemical separation schemes developed at this laboratory had increased considerably. In view of the apparent general interest in such procedures it was felt that a compilation of the latest methods (as of January 1965) now in use at the Lawrence Radiation Laboratory (LRL), Livermore would be worthwhile.

The format adopted in the present compilation follows that used in UCRL-4377 as closely as possible. There is generally one procedure for each element, but in a number of cases several procedures have been included when the technique, the time involved, or the purification were sufficiently different as to serve different possible needs for different occasions. UCRL-4377 contained procedures for 22 elements. The present report contains radiochemical separations for 48 elements. The rare earths have been considered as one element although the procedure describes the separations in sufficient detail to allow for their individual isolation. Procedures for only five elements have been carried over essentially unchanged from the previous compilation.

During the decade since UCRL-4377 was first published, the Radiochemistry Division at the Livermore Site has carried out a large number of research problems — both fundamental and applied. As a result, many new or original separation schemes have been developed, and many of the older ones have been revised or replaced. Procedures in use at this laboratory but devised elsewhere are not included in this report.

Much of the basis for this compilation is the vast field of inorganic literature which has come to fruition largely in the past two or three decades. The same standards of workability have been sought as in UCRL-4377; procedures have been tested and retested. Schemes involving unpredictable or erratic behavior have been avoided. In short, the procedures are reliable, although the introduction of independent "variations" (i. e., if not trivial ones) by the browsing experimenter can be expected to lead to difficulties. As in any procedure, poor chemical yields may result without at least limited trial experience. It is therefore recommended that the user become familiar with the laboratory execution of a procedure before it is applied to a crucial sample.

Finally, it should be pointed out that this treatment is in no way meant to serve as a survey of the radiochemistry of the elements. The reader pursuing this field is referred to the recent exhaustive treatment "Radiochemistry of the Elements," a monograph series published under the auspices of the National Academy of Sciences in cooperation with the United States Atomic Energy Commission.

The compiler is indebted to the many individual experimenters responsible for development of the chemical separations and for their cooperation and consultation in the preparation of the collection. Particular thanks are also due Mrs. Shauna Ness for the typing and proofreading of the material contained herein.

RADIOCHEMICAL PURIFICATION PROCEDURES
FOR THE ELEMENTS

Compiled by Manfred Lindner

Lawrence Radiation Laboratory, University of California
Livermore, California

July 13, 1965

ABSTRACT

Radiochemical procedures, developed by various experimenters at the Lawrence Radiation Laboratory in Livermore, are described for the purification of 48 individual elements from complex mixtures of radioactive species, preponderantly fission products. The degree of purification and the time required to perform the separations are given.

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BERYLLIUM

G. M. Iddings

Purification: From a 6-day-old solution containing 8×10^{14} fissions and $> 10^9$ beta counts per minute, a sample of Be^7 was obtained which showed a beta activity of < 2 counts per minute.

Yield: About 65%.

Separation Time: ~ 6 hours exclusive of ignition.

1. To an acid solution of mixed activities, add 10 mg Be, about 5 mg of yttrium, and ~5 mg of Zr carriers.
2. Make the solution ammoniacal. Centrifuge and wash precipitate with water. Discard the supernate and wash.
3. Add 10 ml 5 M KOH and stir with platinum wire stirrer. Digest for not more than 5 minutes in a hot-water bath. Centrifuge and retain supernatant in clean cone. Repeat leach with another 10 ml 5 M KOH. Combine KOH supernates. Discard residual mixed $\text{Y}(\text{OH})_3$ and $\text{Zr}(\text{OH})_4$ precipitate.
4. To the KOH solution add ~5 mg Te^{+4} carrier. Add ~10 ml of 1 M tetrasodium ethylenediamine tetraacetate (Na_4EDTA) in pH ~ 12 NaOH solution (see NOTE 1). Heat in a hot-water bath for ~3 minutes, then cool to room temperature. Divide the sample between two 40 ml centrifuge cones. Add enough (~15 ml) of a saturated solution of NH_4Cl to each portion to lower the pH to 9-10. Centrifuge and wash $\text{Be}(\text{OH})_2$ precipitate twice with water and recombine Be samples into one cone. Discard supernates.
5. Dissolve $\text{Be}(\text{OH})_2$ in 2 or 3 drops of conc. HCl and add ~15 ml of a buffer solution of 1 M Na_2SO_3 and 1 M NaHSO_3 (~pH 6). Check pH.
6. Transfer solutions to a 60-ml, cylindrical, open-top, separatory funnel (with stem detached immediately below the stopcock).
7. Add 30 ml of 0.40 M TTA* in benzene, and extract the Be for 10 minutes by rapid stirring with a motor-driven glass rod stirrer (paddle).
8. Wash organic phase with ~15 ml of 1 N NaOH for 10 seconds. Immediately drain aqueous layer and repeat 1 N NaOH wash for 10 seconds. Immediately drain aqueous layer and proceed to next step (see NOTE 2).
9. Wash organic phase with ~15 ml of 8 N HNO_3 for 5 minutes (see NOTE 3).
10. Wash organic phase four times with ~15 ml of conc. HCl, allowing 5 minutes for each wash (see NOTE 4).
11. Back-extract Be from organic phase with ~15 ml of 2 parts conc. formic acid and 1 part conc. HCl for 15 minutes. Repeat back-extraction and combine aqueous layers.
12. Pass the strongly acid solutions through a Dowex 1 anion exchange column (6 mm i. d. $\times$ 7 cm in length) preconditioned with conc. HCl. Collect effluent in a 125-ml Erlenmeyer flask. (Be does not adsorb on the resin.)

13. Cautiously add conc. NH_4OH (cooling Erlenmeyer flask under the tap if necessary) until $\text{pH} \geq 9$. Wait ~10 minutes for the hydroxide to precipitate completely. Centrifuge and wash the precipitate twice with water. Discard supernates.
14. Add 10 ml of 3 N KOH to the precipitate. Add Br_2 dropwise until solution becomes yellow. Stir (see NOTE 5). Add a saturated solution of Na_2S dropwise until solution becomes colorless, then add 5 drops excess (see NOTE 6).
15. Add 10 ml of a saturated solution of NH_4Cl . Centrifuge precipitate and wash with water. Discard supernates.
16. Add ~20 ml of 0.1 N HCl and ~5 mg Fe^{+++} carrier. Warm to ~60°C in water bath.
17. Add 1.5 ml of 5% 8-quinolinol in 2 N HAc and 5-10 ml of 2 N NH_4Ac to bring solution to ~pH 5. Check pH. Let stand for 5 minutes in 60°C bath, then cool to room temperature. Filter through 12.5-cm diam No. 42 Whatman paper. Discard precipitate. (Separation - from Be - of Al, Pa, Zr, Nb and others.)
18. Make filtrate basic with NH_4OH . Filter through 11.0-cm diam No. 42 Whatman paper. Wash with 1% solution of NH_4Ac .
19. Ignite filter paper and contents to BeO for one hour at 1000°C in a No. 0 Coors porcelain crucible.
20. Transfer to tared aluminum hat and weigh rapidly (BeO is somewhat hygroscopic). Add a few drops of "Duco" cement (diluted ten-fold with ethyl acetate), "Zapon," or similar lacquer agent. Count 477 keV gamma ray in scintillation counter and check for impurities by beta counting (~1% of the gamma radiation is detected by a typical gas-filled proportional or Geiger counter).

NOTE 1: Na_4EDTA in basic solution will complex macro amounts of aluminum (and many other elements) and will provide a separation of Al from Be when the $\text{Be}(\text{OH})_2$ is precipitated from this solution.

NOTE 2: 1 N NaOH will wash out the "free" TTA* from the organic layer. These washes are done rapidly to minimize any loss of Be.

NOTE 3: Te^{+4} is slowly reduced to Te metal by the sulfite, and is carried along at the interface of the benzene-aqueous mixture. A 5-minute wash with 8 N HNO_3 will oxidize finely divided Te metal to Te^{+4} , placing it in the acid layer. This oxidation is not very efficient unless the two phases are churned together rapidly.

NOTE 4: Because the two NaOH washes remove at least 99% of the "free" TTA, Np^{+4} , Zr, and Pa should be removed rather completely with the conc. HCl acid washes. The time required for these washes depends on how well the phases are mixed. The chemical procedure should not be interrupted until all of the conc. HCl washes are completed.

NOTE 5: BrO^- in alkaline solution immediately oxidizes Te^0 and Te^{+4} to the +6 state and Sb^{+3} to Sb^{+5} .

NOTE 6: An excess of Na_2S solution at this point will ensure that Mo, Sn, Sb, Te, and W will remain in solution even when the pH is lowered in Step 15 in which Be is reprecipitated.

* Thenoyltrifluoroacetone.

PHOSPHORUS

K. V. Marsh

- Purification: 5×10^{10} atoms of P^{32} from a 7-day-old solution of 10^{14} fissions counted through 14 half-lives showed no evidence of contamination by longer-lived foreign radionuclides. If the entire procedure is used, decontamination from arsenic is better than a factor of 10^7 .
- Yield: About 60%.
- Separation Time: 4-5 hours for duplicate samples.
- Reagents: Dowex AG-1X8, 50-100 mesh, anion exchange resin; magnesia mixture (50 g $MgCl_2 \cdot 6 H_2O$, 100 g NH_4Cl , 3 to 5 drops conc. HCl , 500 ml water); Zr carrier (15 mg/ml Zr(IV) as $ZrOCl_2$ in 1-2 M HCl).
- Remarks: To obtain good separation from arsenic, the As is reduced by I^- and S^{2-} to the +3 state early in the procedure and never reoxidized. Zr carrier as the nitrate salt or in nitric acid solution should not be used to precipitate zirconium phosphate after Step 1. Each of the As_2S_3 precipitations and the conc. HCl anion column gives a decontamination factor from arsenic of about 300. If extreme decontamination from arsenic is not required, time may be saved by eliminating the column and merely filtering the As_2S_3 in Step 7. In fact, a very short procedure which gives good decontamination from most fission products results from eliminating Steps 4 and 8, inclusive.
1. To the sample in a 40-ml lusteroid or other HF -impervious centrifuge tube, add 1.0 ml of standardized phosphorus carrier (about 5 mg P/ml as $(NH_4)_2 HPO_4$ in water) and 15-20 ml of conc. HNO_3 . Heat the solution on a steam bath and add 45 mg of Zr carrier. Continue heating until the precipitate coagulates (~15 minutes), centrifuge, and discard the supernate (NOTE 1).
 2. Wash the precipitate with a mixture of ~20 ml of 6 N HCl , 5 mg of As(V) (added as $Na_2 HAsO_4$ in water), and 20 mg of I^- (added as NaI in water). Discard the wash.
 3. Dissolve the precipitate in 0.1 ml of conc. HF , add 10 ml conc. HCl , 5 mg As(V), 20 mg I^- , 1 drop of 10% aerosol, and saturate with H_2S for ~1 minute. Centrifuge and filter the supernate through a Whatman No. 40 paper in a plastic funnel into a clean lusteroid tube. Wash the As_2S_3 with 5-10 ml of 6 N HCl and pour through filter. Wash the filter paper with 5-10 ml of 6 N HCl and discard the filter.
 4. To the combined filtrates add 50 mg of Al^{+3} (as $AlCl_3$ either solid or in water solution) and 10 mg of Zr(IV)(chloride). Heat in hot-water bath about 20 minutes, centrifuge, and wash precipitate with 15 ml of water containing about 5 mg of Zr carrier. If the wash is not well-centrifuged, serious (but not obvious) losses of the zirconium phosphate may occur due to peptization of the precipitate.

5. Add 10 ml of 2 N NaOH to the precipitate, stir thoroughly, and heat in water bath. Centrifuge and retain the supernate in a clean cone. Wash the precipitate with 10 ml of water and add washings to supernate. Discard the precipitate.
6. Add 10 ml of magnesia mixture to the solution. Add conc. HCl until all the $\text{Mg}(\text{OH})_2$ dissolves. Stir the mixture vigorously and add carbonate-free conc. NH_4OH dropwise until a precipitate of $\text{MgNH}_4\text{PO}_4 \cdot 6 \text{H}_2\text{O}$ just begins to form. Continue stirring 10-15 seconds and add 5 ml of conc. NH_4OH . Stir and allow the mixture to stand about 15 minutes. Centrifuge and discard the supernate.
7. Dissolve the precipitate in 5 ml of conc. HCl, add 5 mg of As(V), 20 mg of I^- , 1 drop of aerosol, and saturate with H_2S for about one minute. Add another 5 ml of conc. HCl and centrifuge.
8. Pour the supernate through a column of Dowex AG-1X8 ion exchange resin (50-100 mesh, chloride form) which has been previously washed with conc. HCl (column dimensions 6-mm diam by 10-12 cm). Retain the effluent in a clean cone, wash the As_2S_3 precipitate with 5-10 ml of conc. HCl, centrifuge, and pour washings through the column. Wash the column with 10 ml of conc. HCl and discard the column, collecting the column washes in the same cone with the original effluent.
9. Add 50 mg of Zr carrier to the column effluent and heat in water bath about 15 minutes. Centrifuge and wash the precipitate with 10 ml of water. Be sure to centrifuge the wash thoroughly but do not add any acid or acid salts to promote coagulation in the wash. Discard supernatant and wash.
10. Add 1.0 ml of 2.0 N NaOH to the precipitate, stir thoroughly, and heat in the water bath 10 minutes. Dilute to 10 ml with water, stir, centrifuge, retain the supernate in a clean tube and wash the precipitate with 10 ml of water. Combine the wash with the leach solution, discard the precipitates, and dilute the combined solutions to 40 ml (NOTE 2). Pour the solution through a column of Dowex AG-1X8, 50-100 mesh, chloride form, which has previously been washed Cl^- -free with water (column dimensions 6-mm diam by 10-12 cm). Wash the column with 15 ml of 0.10 N NaOH, and discard effluent and wash.
11. Elute the $\text{PO}_4^{=}$ into a clean cone with 10 ml of 1 N NaOH. Discard the column (NOTE 3).
12. Add 10 ml of magnesia mixture and precipitate $\text{MgNH}_4\text{PO}_4 \cdot 6 \text{H}_2\text{O}$ as in Step 6.
13. Filter the precipitate onto a weighed No. 42 Whatman filter circle, 7/8-in. diam, using a ground-off Hirsch funnel and filter chimney. Wash the precipitate with small portions of 1:20 NH_4OH followed by 95% ethanol. Draw air through the precipitate for a few seconds. Allow the precipitate to stand near the balance for about 30 minutes. Weigh, mount, and count.

- NOTE 1: If a large amount of $\text{SO}_4^{=}$ is present, the precipitation of zirconium phosphate is incomplete. The complete precipitation of zirconium phosphate is slow, and if time is available the yield can be improved somewhat by allowing the mixture to cool and stand several hours.
- NOTE 2: For complete leaching of the zirconium phosphate the NaOH concentration must be at least 2 N; however, for complete absorption of the $\text{PO}_4^{=}$ on the column the NaOH concentration must be less than 0.1 N.
- NOTE 3: Instead of 1 N NaOH, 0.1 N HCl or even 0.1 N NaCl may be used to elute the $\text{PO}_4^{=}$.

SULFUR

J. A. Miskel

Decontamination: 4×10^{10} atoms of S^{35} isolated from a 4-month-old solution of 10^{14} fissions showed no detectable contamination by foreign radionuclides when the decay was followed through one half-life; a beta-ray absorption curve showed no detectable contamination over a factor of at least 500.

Yield: 50-70%.

Separation Time: About 2-1/2 hours.

1. To the active solution in dilute acid (not sulfuric), add 5 mg $SO_4^{=}$ carrier. Neutralize with NH_4OH , then make slightly acid with HCl. Heat the solution, add 10 mg Ba^{++} and digest the $BaSO_4$ precipitate. Centrifuge, discard supernatant liquid, and wash precipitate once with dilute HCl.
2. Dry the $BaSO_4$ in an oven, then transfer the precipitate to a platinum boat.
3. Place the boat with sample into a quartz furnace tube and place in a horizontal tube furnace. Pass H_2 saturated with HCl over the sample and heat furnace to 900-1000°C (see NOTE 1). Pass the effluent gas through an ammoniacal solution of Cu^{++} , dispersing gas beneath the surface of the solution with a sintered glass disc (see NOTE 2).
4. Filter CuS into a tared filter paper, dry, weigh, and mount for β counting.

NOTE 1: The H_2 can be saturated with HCl by dispersing it beneath the surface of an aqueous solution (6 M) of HCl with a sintered glass dish.

NOTE 2: The hydrogen emerging from the Cu^{++} solution should be burned to prevent accumulation of hydrogen. However, the air in the system should first be flushed with hydrogen before burning the exit gas, so as to avoid an explosive mixture.

CALCIUM

M. Lindner

Purification: In an atom/fission ratio of 3×10^{-5} , Ca^{45} was isolated from a 30-day-old fission mixture and decay followed through one half-life with no evidence of either shorter- or longer-lived impurities. No gamma radiation could be detected in the samples.

Yield: 30-50%.

Separation Time: About 5 hours for four samples.

1. To an acid solution containing the activity, add 10 mg of Ca^{++} carrier (if necessary) several mg of Fe^{+3} and bring to a boil for several minutes to expel CO_2 .
2. Either pass in NH_3 gas until the solution is basic to phenolphthalein, or add carbonate-free NH_4OH to the same point. Centrifuge and discard precipitate. Add several more mg Fe^{+3} , also discarding the resultant precipitate.
3. To the supernate, add a sufficient excess of Na_2CO_3 solution that further addition brings about no further white precipitate. Centrifuge and wash precipitate with water.
4. Dissolve precipitate in 10 ml of 2 N HCl . Add about 1 mg each of Te^{+4} , Sb^{+3} , Bi^{+3} , and Cu^{++} . Saturate the solution with H_2S , centrifuge, and discard precipitate.
5. Make solution basic with NH_4OH , and add Na_2CO_3 solution until precipitation is complete.
6. Centrifuge, wash precipitate, and dissolve in minimum volume of HCl .
7. Fume to near-dryness and take up in about 10 ml water.
8. Add about 1 ml of Dowex-50 cation exchange resin (200-400 mesh) and equilibrate for about 10 minutes. Pipet resin onto a column of Dowex-50, 200-400 mesh, about 10 in. long and about 1/4 in. i. d.
9. Add an amount of 1.2 N ammonium lactate solution sufficient to elute the calcium fraction, but neither the strontium nor barium fractions. This must be pre-determined for the particular resin batch and column dimensions, (e. g., by collecting eluate in Na_2CO_3 solution and noting precipitate size) but once so done, the given volumes will consistently give satisfactory separation. As a rough guide, about 15-20 ml will probably remove most or all of the Ca (Sr should not begin to elute until approximately 40 or 50 ml pass through).
10. Collect the eluate in a 40-ml centrifuge cone. Add 2-ml saturated ammonium oxalate solution. Centrifuge and wash precipitate.
11. Dissolve precipitate in HNO_3 . Add sufficient KBrO_3 to ensure destruction of the oxalate and heat for 10 minutes on a hot-water bath.
12. Repeat Steps 5 through 10. Wash the final Ca oxalate precipitate once with water, once with alcohol, and mount.

SCANDIUM

G. M. Iddings

Purification: From a 2-week-old solution containing 1.1×10^{16} fissions, a final sample was obtained which had a beta activity of about 50 c/m (with ~35% geometry) and which was not further identified.

Yield: ~65%.

Separation Time: ~6 hours.

1. To 25 ml of solution of mixed activities in 6 M HCl in a 40-ml glass centrifuge cone, add 10 mg of scandium carrier, ~10 mg of Y, ~5 mg of Te^{+4} and ~2 mg of Zr. Add 2 ml of 6% H_2SO_3 and 1 ml of 1 M H_3PO_4 . Stir and heat on a hot-water bath for 5 minutes. Cool to room temperature. Centrifuge and discard the precipitate. To the supernatant add ~2 mg Zr and 2 mg Te^{+4} and heat, repeating the Zr phosphate-Te metal scavenge. Discard the precipitate. Pour the supernatant into a 50-ml lusteroid tube or other HF-impervious tube.
2. Add conc. NH_4OH in excess. Centrifuge and discard the supernatant. Wash the precipitate twice with ~15 ml of water to remove salts (especially Na^+). Centrifuge and discard the wash solutions.
3. Dissolve the precipitate in a few drops of 6 M HNO_3 , and add 5 ml of water. Add an equal volume of Na-free 6 M NH_4F , making the final solution ~3 M in NH_4F and pH 4-5. Heat in a hot-water bath for ~3 minutes. Cool and centrifuge. Pour the supernatant into a new 50-ml HF-impervious centrifuge tube. Add 10 ml of Na-free 3 M NH_4F to the precipitate. Heat for 3 minutes. Cool and centrifuge, combining this supernatant with the previous one. Wash the YF_3 with ~5 ml of water. Centrifuge and combine supernatants. Discard the YF_3 .
4. To the combined supernates add 1 ml of sat. KBrO_3 solution and heat in a hot-water bath for ~5 minutes. Add ~10 ml conc. HNO_3 (the pH should be ~0.3 or less). Heat in a hot-water bath for ~5 minutes. Cool to room temperature. Centrifuge and discard the supernatant. Wash the precipitate with ~5 ml of water. Centrifuge and discard wash.
5. Dissolve the ScF_3 with 1 ml of 5% H_3BO_3 . Mix thoroughly and heat in hot-water bath for ~3 minutes. Add 2 ml conc. HNO_3 and transfer the solution to a 40-ml glass cone using 2 ml 6 M HClO_4 as a rinse. Heat carefully to strong fumes of HClO_4 . Dilute to ~20 ml with water and add an excess of conc. NH_4OH . Centrifuge and discard the supernatant. Wash the precipitate once with ~30 ml of water.
6. Dissolve the $\text{Sc}(\text{OH})_3$ in ~20 ml of 0.1 M HCl (pH should be 1.5-2.0). Transfer the solution to a 60-ml cylindrical separatory funnel. Add ~30 ml of 0.40 M TTA* in benzene and stir vigorously for ~3 minutes. Drain and discard aqueous layer, washing the organic layer four times with ~20 ml portions of 0.05 M HCl for ~3 minutes each time.

7. Stir organic layer for 2 minutes with ~20 ml of 2 M HCl. Allow layers to settle a few minutes before draining aqueous layer into a clean cone.
8. To the aqueous layer add an excess of NH_4OH . Centrifuge and discard the supernatant. Wash the precipitate with ~30 ml of water. Centrifuge and discard the wash.
9. Dissolve the $\text{Sc}(\text{OH})_3$ with ~8 ml of conc. HCl. Pass the solution (~10 ml total volume) through a Dowex-1 anion exchange resin column (7 cm in length $\times$ 6-mm i. d., 50-100 mesh) and collect effluent. Wash the resin column with ~4 ml of 12 M HCl, combining both effluents.
10. Carefully add an excess of conc. NH_4OH to the combined effluents. Cool, centrifuge and discard supernatant. Wash the precipitate with ~30 ml of water. Centrifuge and discard wash.
11. Dissolve precipitate with 3 drops of conc. HCl. Add ~15 ml of saturated NH_4SCN solution. Final solution should have pH $\geq$ 0.8. Transfer the solution to a 60-ml cylindrical separatory funnel. Add ~25 ml of ethyl n-butyl ketone (3-heptanone) (NOTE 1). Stir the layers vigorously for 2 minutes using a motor-driven glass stirring-rod paddle. Drain and discard the aqueous layer. Wash the organic layer three times with ~15 ml of saturated NH_4SCN solution for ~1 minute per wash. Discard the aqueous layers.
12. Stir the ketone layer twice with 7-ml portions of 6 M HCl for 1 minute each time. Discard the organic layer.
13. To the combined aqueous layers add an excess of NH_4OH . Dilute to ~40 ml with water. Cool to room temperature, centrifuge and discard the supernatant. Wash the $\text{Sc}(\text{OH})_3$ with ~30 ml of water. Centrifuge and discard wash.
14. Dissolve the precipitate with 0.5 ml of conc. HCl. Add ~30 ml of 1 M ammonium tartrate and add conc. NH_4OH dropwise until pH 7-8 (Bromothymol Blue turns from yellow to blue); then add 4 drops excess. Centrifuge and discard any foreign material.
15. Heat in a boiling water bath for ~20 minutes. Cool to room temperature. Centrifuge and discard the supernatant. Wash the scandium ammonium tartrate with ~20 ml of water and filter through 7-cm No. 42 Whatman paper.
16. Ignite to Sc_2O_3 in a furnace for ~20 minutes at 800°C in a No. 00 Coors porcelain crucible. Weigh, mount, and count.

NOTE 1: Ethyl n-butyl ketone is more specific for Sc than is hexone (methyl isobutyl ketone), another ketone commonly used for extraction of inorganic ions. Therefore, this extraction with the washes gives a better separation from thorium and the rare earths.

* Thenoyltrifluoroacetone.

VANADIUM

G. M. Iddings

Purification: From a 7-day-old solution containing $\sim 1.3 \times 10^{15}$ fissions, a sample was obtained which had a beta activity of ~ 10 c/m with $\sim 30\%$ geometry.

Yield: $\sim 50\%$.

Separation Time: About 8 hours.

1. To 20 ml of a solution of mixed activities in ~ 2 M HCl in a 40-ml glass centrifuge cone, add 5 mg of V^{+4} , ~ 10 mg of Y, ~ 2 mg of Be, ~ 2 mg Ce^{+3} . Add ~ 2 ml of 6% H_2SO_3 and 5 drops of 55% HI. Heat on hot-water bath for ~ 5 minutes. Cool and add carbonate-free NH_4OH until solution is just basic (pH ~ 8). Centrifuge and discard the supernate. Wash the precipitate twice with ~ 15 ml of water, discarding the washes.
2. To the precipitate, add ~ 10 ml of 3 M carbonate-free KOH and sufficient Br_2 dropwise to make the supernate yellow. Heat for 5 minutes in a boiling water bath. Centrifuge and pour the supernate into a new 40-ml centrifuge cone. Repeat the hot leach with 10 ml 3 M KOH and a few drops of Br_2 .
3. To the combined yellow OH^- supernates add ~ 1 ml of saturated $BaCl_2$ solution. Centrifuge and discard the supernate. Wash the precipitate with ~ 10 ml of water. Centrifuge and discard the wash.
4. Dissolve the barium vanadate precipitate in ~ 25 ml of 4 M HCl. Add ~ 1 ml of 6 M H_2SO_4 . Heat for ~ 3 minutes on hot-water bath. Centrifuge and discard the $BaSO_4$ precipitate, pouring supernatant into a clean cone.
5. To the HCl-vanadium solution add ~ 1 ml of saturated $K_4Fe(CN)_6$ solution. Let stand ~ 5 minutes at room temperature. Centrifuge and discard the supernate. Wash the precipitate with 15 ml of 4 M HCl. Centrifuge and discard the wash.
6. Transfer the precipitate to a 40-ml glass centrifuge cone with 20 ml 3 N NaOH. Add Br_2 dropwise until a yellow color persists. Heat for 20 minutes in a hot-water bath to oxidize the $Fe(CN)_6^{-4}$ complex. Cool and centrifuge the $Fe(OH)_3$ precipitate. Pour supernatant into clean cone and save.
7. Dissolve the precipitate in a few drops of conc. HNO_3 (heat if necessary) and dilute to 15 ml with water. Add NaOH solution until alkaline. Centrifuge precipitate and combine supernatant with that from Step 6.
8. Repeat Step 3.
9. Dissolve the barium vanadate in ~ 5 ml of 6 M HCl. Heat on hot-water bath for ~ 5 minutes. Cool. Add ~ 3 ml of conc. HCl. Centrifuge the $BaCl_2$ and pour supernate through a Dowex A-1 anion column (6-mm i. d. $\times$ 7-cm resin bed), collecting the effluent in a clean cone. Wash the $BaCl_2$ with ~ 10 ml of 8 M HCl and pour the wash through the anion column. (Vanadium does not adhere to the Dowex A-1 resin in 8 M HCl.) Wash the column with another 10 ml of 8 M HCl.

10. To the combined effluents, add ~5 drops of saturated NaClO_3 solution. Equilibrate for 3 minutes with ~25 ml of 100% tributylphosphate ("TBP") in a 60-ml cylindrical separatory funnel. Wash the TBP layer twice for three minutes each time, with 15 ml of 6 M HCl containing 2 drops saturated NaClO_3 solution.
11. Equilibrate the TBP for 3 minutes with ~15 ml of a solution about 6 M in HCl and 1 M in HI (2 ml 55% HI in 13 ml 6 M HCl). Repeat the back-extraction. Combine aqueous layers.
12. Add ~2 mg of Sc , ~1 mg Zr , and ~2 mg Y to the V solution and make basic with conc. NH_4OH . Centrifuge and discard the supernate. Wash the precipitate with ~10 ml of water using a clean platinum wire stirrer.
13. Dissolve the precipitate in ~30 ml of 0.1 M HNO_3 and transfer the solution to a 50-ml HF -resistant centrifuge tube. Add ~3 mg of Mo , ~1 mg Te^{+4} , 2 drops of saturated BaCl_2 solution. Mix well. Add ~1 ml of conc. HF and pass H_2S gas through the solution for 5 minutes. Heat on a hot-water bath for ~3 minutes. Cool to room temperature. Centrifuge and discard the combined precipitates of YF_3 , ScF_3 , BaZrF_6 , MoS_3 and Te^0 .
14. Pour supernate into a clean tube and add ~2 mg Y carrier dropwise. Stir vigorously, centrifuge, and discard the YF_3 precipitate. Pour the supernate into another 50-ml HF -resistant centrifuge tube.
15. Repeat Step 5, adding 3 drops of saturated NaNO_2 solution, 2 ml saturated $\text{K}_4\text{Fe}(\text{CN})_6$. Allow the solution to stand at room temperature for 10 minutes before centrifugation.
16. Repeat Steps 6 and 7.
17. Repeat Steps 3 and 9.
18. Add sufficient (~10 ml) water to the combined 8 M HCl eluates to make the final solution ~6 M in HCl . Then repeat Step 5. Use a clean Pt wire stirrer.
19. Repeat Steps 6 and 3.
20. Dissolve precipitate in ~15 ml of 0.1 M HClO_4 . Add 1 ml of fresh 6% H_2SO_3 solution. Heat on the hot-water bath for ~5 minutes. Cool, centrifuge and discard the BaSO_4 precipitate.
21. Add 1 drop of 6% H_2SO_3 to the supernate, then pour it through a Dowex 50W-X8 (100-200 mesh), 6-mm i. d., 6-7 cm resin column which has been washed with 0.1 M HClO_4 . Wash the resin column with ~10 ml of 0.1 M HClO_4 , using 3- or 4-ml portions at a time, and discard the washes.
22. Elute the vanadium with ~10 ml of 0.01 M HClO_4 containing ~1% H_2O_2 , collecting the eluate in a clean 40-ml glass cone. Add 15 ml water to the eluate.
23. Add ~1 ml of ~1 M lead acetate in 0.1 M HAc solution. Adjust pH to 4-5 with 2 M NH_4Ac if necessary. Heat on a hot-water bath for 15-20 minutes. Centrifuge and discard the supernate (lead vanadate does not filter well). Wash precipitate once with ~10 ml of water with a clean Pt wire stirrer.

24. Dissolve precipitate in ~20 ml of 0.1 M HClO_4 . Centrifuge and discard any residual precipitate.
25. To the supernate add ~1 ml of ~1M lead acetate in 0.1 M HAc solution and sufficient (1-2 ml) 2 M NH_4Ac solution to make the pH 4-5. Heat on a hot-water bath for ~20 minutes. Centrifuge and discard the supernate. Wash precipitate twice with ~10 ml of water, twice with ~10 ml of methanol, and once with acetone.
26. Dry precipitate in the glass cone in an oven at ~110°C for ~20 minutes. Transfer to a tared aluminum planchet and distribute precipitate evenly in it. Weigh, add a few drops of 4% Duco cement in ethyl acetate, dry, mount, and count.

If greater purification is needed, the following steps can be added between Steps 11 and 12:

NOTE 1: To the V solution add ~10 mg of Y. Nearly neutralize with NH_4OH , cool solution, then make the solution basic with conc. NH_4OH . Cool, centrifuge and discard the supernate. Wash precipitate with ~10 ml of water using a clean Pt wire stirrer.

NOTE 2(a): Dissolve precipitate in 3 or 4 drops of conc. HCl. Add 15-20 ml of saturated NH_4SCN solution. The pH should be ~0.4. Transfer the solution to a 60-ml cylindrical separatory funnel which contains ~30 ml of hexone. Stir layers vigorously for 3 minutes with a motor-driven glass stirring paddle. Drain and discard the aqueous layer.

(b): Wash the organic layer for 2 minutes twice using 15-20 ml of saturated NH_4SCN solution each time. Discard the wash solutions.

(c): Back-extract the V into 10 ml of 6 M HCl for ~2 minutes. Repeat the back extraction and combine the 6 M HCl solutions.

(d): Repeat Step 12, then continue with the regular procedure.

IRON

W. H. Hutchin

Purification: 10^7 atoms of Fe^{59} from a 9-day-old solution of 4×10^{13} fissions showed no evidence of contamination by foreign radionuclides through a detectable limit of three half-lives.

Yield: About 70%.

Separation Time: ~4-5 hours.

Reagents: Anion exchange resin (Dowex 1X8, 50-100 mesh). Diisopropyl ether. A solution of dilute ammonia saturated with NH_4NO_3 .

Remarks: The washes of the FeS precipitate are made with $\text{NH}_4\text{OH-NH}_4\text{NO}_3$ solution to prevent peptization.

1. To the active solution add 10 mg Fe carrier and 1-2 ml saturated tartaric acid solution. Make basic with NH_4OH . Saturate with H_2S , centrifuge, and discard supernatant. Wash precipitate twice with a solution of dilute NH_4OH saturated with NH_4NO_3 .
2. Dissolve FeS in about 1/2 ml conc. HCl . Boil to drive off H_2S . Add one drop HNO_3 . Boil to get rid of NO_3^- , adding more HCl if necessary. Reduce volume to ~0.5 ml. Bring volume to 4 ml with conc. HCl .
3. Pass solution through an anion column (Dowex 1X8, 50-100 mesh, 3-in. $\times$ 1/4-in. i. d.) that has been washed with conc. HCl . Wash column with 4 ml of 12 M HCl , then with 4 ml 6 M HCl . Discard effluents.
4. To column add 4 ml of 0.5 M HCl . Collect eluate in clean 40-ml cone.
5. To the eluate add 5 mg Cu^{++} carrier, and one drop each of Sb^{+3} , As^{+3} , and Te^{+4} carriers. Add 0.5 M HCl to bring volume to 20 ml. Saturate with H_2S . Centrifuge and discard precipitate.
6. Add 1-2 ml saturated tartaric acid solution, make basic with NH_4OH , and saturate with H_2S . Centrifuge and discard supernatant. Wash precipitate twice with $\text{NH}_4\text{OH-NH}_4\text{NO}_3$ solution.
7. Dissolve precipitate in conc. HCl . Add a drop of conc. HNO_3 to complete solution. Boil to destroy HNO_3 and reduce volume to ~1 ml. Add 6 N HCl to bring volume to ~15 ml and add Hg metal (0.5 ml). Stir for ~5 minutes to reduce Fe^{+3} to Fe^{++} . Extract twice with equal volumes of diisopropyl ether. Discard ether layers.
8. Pipet aqueous layer away from Hg and centrifuge off the remaining Hg. Add 1 ml of saturated tartaric acid solution and make basic with NH_4OH . Saturate with H_2S , centrifuge, and discard supernatant. Wash precipitate twice with $\text{NH}_4\text{OH-NH}_4\text{NO}_3$ solution. Discard washes.
9. Dissolve in about 1 ml conc. HCl . Add 2 or 3 drops of conc. HNO_3 and boil to small volume. Add more conc. HCl and boil to small volume again (0.5 ml). Bring volume to 15 ml with 8 M HCl and extract twice with equal volumes of

diisopropyl ether. Combine ether layers and wash once with 8 M HCl. Discard wash.

10. Equilibrate the combined ether layer with two 10-ml portions of water. Separate Phases and discard ether layer.
11. Make the aqueous layer ammoniacal, centrifuge, and discard supernatant. Wash precipitate twice with acetone, stir moist precipitate while in hot-water bath until dry and finely divided. Ignite for 10 minutes in a porcelain crucible over a Fisher burner. Cool and weigh as Fe_2O_3 .

COBALT

G. M. Iddings

Purification: When a cobalt fraction was isolated from a solution of mixed activities (mainly fissions), no activities other than those of Co^{57} , Co^{58} , and Co^{60} could be detected either by radioactive decay or by gamma pulse-height analysis.

Yield: ~90%.

Separation Time: About 3 hours.

1. To an acid solution of mixed activities, add 20 mg of cobalt carrier and about 2 mg each of Fe^{+3} and Y^{+3} . Add conc. NH_4OH until solution is basic to phenolphthalein. Stir vigorously to ensure air oxidation of the cobalt to the trivalent state. (Note: Significant losses of Co can occur if oxidation is not complete, due to the precipitation of $\text{Co}(\text{OH})_2$.) Centrifuge and discard the precipitate. Repeat the hydroxide scavenge by adding the iron and yttrium carriers dropwise while stirring the ammoniacal solution vigorously with a motor-driven Pt wire stirrer. Centrifuge and discard the precipitate.
2. Adjust pH of supernate to 9, and saturate with H_2S . Add a few drops of aerosol, heat on a hot-water bath to coagulate the precipitate. Centrifuge and discard the supernate. Wash the precipitate once with water.
3. Dissolve the CoS in a small amount of boiling HCl . Add a few drops conc. HNO_3 and fume to near-dryness. Add a few drops of conc. HCl and boil to near-dryness. Repeat HCl addition and fume to near-dryness.
4. Dissolve the residue in ~5 ml of 10 M HCl and pour solution through a Dowex-1 anion exchange column (6-mm i. d. $\times$ 7 cm) preconditioned with 10 M HCl . Wash the column with ~5 ml of 10 M HCl .
5. Elute the Co with 10-15 ml of 5 M HCl . Collect eluate in clean cone.
6. Add ~1 ml of saturated $\text{K}_4\text{Fe}(\text{CN})_6$ solution to the Co- HCl solution and allow resultant solution to stand ~5 minutes at room temperature. Centrifuge and discard the supernate. Wash the precipitate with a mixture containing ~10-15 ml of 4 M HCl and 1/2 ml of saturated $\text{K}_4\text{Fe}(\text{CN})_6$ solution. Centrifuge and discard the wash solution.
7. Add ~20 ml of 3 M NaOH and a few drops of Br_2 . Heat on a hot-water bath for ~5 minutes. Cool and centrifuge. Discard the supernate. Wash the precipitate twice with ~10 ml of 0.1 M NaOH . Centrifuge and discard the wash solutions.
8. Dissolve the precipitate in a few drops of conc. HCl and one drop of conc. HNO_3 . Boil to dryness. Add ~10 ml of 0.1 M HCl and ~10 ml of saturated NH_4SCN solution. Add this solution to an equal volume of 4-heptanone (di-n-propyl ketone) in a 60-ml cylindrical separatory funnel. Stir layers rapidly for ~2 minutes. Drain and discard the aqueous layer. Wash the organic layer twice with equal volumes of 50% saturated NH_4SCN solution each time. (No acid is added to the wash solution.)

9. Back-extract the Co with three 5-ml portions of 6 M HCl for 2 minutes each time. Dilute combined aqueous layers with water to make ~4 M in HCl.
10. Repeat Steps 6 and 7.
11. To the precipitate add 1 ml each of conc. HCl, conc. HNO₃, conc. HClO₄ and conc. H₂SO₄. Heat and fume carefully to near-dryness. Cool and take up in a few ml of water. Make basic with an excess of conc. NH₄OH. Centrifuge and pour the supernate into an electrodeposition cell with a clean tared platinum disc as a cathode.
12. Electrodeposit the Co metal at ~100 mA for ~24 hours. Wash the disc with water and acetone, dry in an oven at 110°C for 10 minutes. Cool, weigh, mount, and count.

COPPER

W. H. Hutchin

Purification: 5×10^8 atoms Cu^{67} from a 6-day-old solution of 4×10^{13} fissions showed no evidence of contamination from foreign radionuclides over seven half-lives.

Yield: About 65%.

Separation Time: ~6-8 hours.

1. To a 4 N HCl solution of the activity, add 10 mg Cu carrier, and saturate with H_2S . Wash precipitate twice with 4 N HCl.
2. Dissolve precipitate in 2 ml conc. HNO_3 and bring to a boil. Dilute to 10 ml, add 5 mg Ag carrier and 2 drops conc. HCl. Centrifuge and discard precipitate.
3. Add 3 mg Fe^{+3} and a mg each of La, As, and Cr. Make ammoniacal. Centrifuge and discard precipitate.
4. Acidify supernatant with HCl and repeat Step 3.
5. To supernatant add several drops of Sr and Ba holdback carriers, and saturate solution with H_2S . Centrifuge and discard supernatant.
6. Dissolve CuS in several drops of conc. HNO_3 , boil to 0.5 ml or less, and bring volume to ~15 ml with water. Heat and swirl while adding 1 N NaOH dropwise until permanent black precipitate forms. If on the addition of a few drops more of 1 N NaOH no further precipitate forms, add 4 or 5 drops more in excess. Centrifuge and wash precipitate in 0.01 N NaOH. Discard wash.
7. Dissolve precipitate in minimum HCl and dilute to ~5 ml with 0.1 N HCl. Pass solution through Dowex 1X8, 50-100 mesh, anion exchange column that has been washed with 0.1 N HCl (anion column 2-1/2 in. long, 1/4 in. i.d.). Wash column with ~10 ml 0.1 N HCl. Combine all effluents in clean cone.
8. Saturate solution with H_2S . Centrifuge and wash precipitate with 0.1 N HCl. Dissolve precipitate in several drops of conc. HNO_3 . Boil to dryness, add HCl, and boil to dryness again.
9. Add 2 or 3 drops conc. HCl, dilute to ~10 ml, neutralize with NH_4OH , and add one drop HCl in excess. Saturate with SO_2 . Add 4-8 drops of a saturated solution of KCNS slowly while swirling. Centrifuge and wash precipitate twice with 0.1 N HCl.
10. Dissolve CuCNS in minimum conc. HNO_3 and repeat Steps 2 through 9, eliminating Steps 7 and 8.
11. Dissolve CuCNS in minimum conc. HNO_3 and add water to 15-ml volume. Heat and swirl, adding 1 N NaOH dropwise until permanent black precipitate forms. If on the addition of a few drops more of 1 N NaOH no further precipitate forms, add 4 or 5 drops more in excess. Centrifuge and wash precipitate in approximately 0.01 N NaOH.

12. Dissolve precipitate in 10 ml of 1.5 N H_2SO_4 , add two drops of freshly boiled HNO_3 , and electroplate with slow stirring on platinum disc at about 2.5 V and 50 mA for 2-1/2 hours. Weigh as Cu metal.

ZINC

W. E. Nervik and D. R. Nethaway

Decontamination: 10^{10} atoms of Zn^{72} isolated from a 1-day-old mixture containing 10^{15} fissions exhibited no contamination when decay was followed through three half-lives.

Yield: 60%.

Separation Time: 6 hours for eight samples.

- 1(a). Add several mg Cd^{++} carrier, 2 ml conc. HClO_4 , and boil to HClO_4 fumes. Cool and adjust to about 1 M HClO_4 (approximately 20 ml) (see NOTE 1).
- (b). Cool solution in an ice bath, add 5 ml mercuric thiocyanate reagent (8 g HgCl_2 , 9 g NH_4CNS , 100 ml water), and 2 drops conc. HClO_4 . Digest cold for 10 minutes, centrifuge and wash precipitate with 1 M HClO_4 .
- 2(a). Dissolve precipitate in 0.5 ml hot conc. HNO_3 and dilute to 20 ml. Add 3 drops each of Y^{+++} , Zr^{+4} and Cd^{++} holdbacks (each ~10 mg/ml) and make ~1 M in OH^- with NaOH pellets. Centrifuge and discard precipitate.
- (b). Pour supernatant liquid into a cone containing 2 drops each of Fe^{+++} and Cd^{++} carriers (~10 mg/ml). Centrifuge and discard precipitate.
- 3(a). Adjust supernatant liquid to 3 M H^+ with 12 N HCl . Pass solution through a 6 mm $\times$ 10 cm Dowex-21K column (pre-equilibrated with 3 M HCl).
- (b). Wash column with 10 ml 0.5 M HCl containing 3 drops of 5 M KI .
- (c). Elute with 40 ml boiling 0.1 M KI .
- 4(a). Pass eluate through a Dowex-50 column (6 mm $\times$ 5 cm) directly from Step 3(c).
- (b). Wash Dowex-50 column with 10 ml 1 M HClO_4 . Discard wash.
- (c). Elute zinc from Dowex-50 column with 20 ml 3 M HClO_4 .
5. Add 5 ml 6 M NaOH to eluate, 2 drops Cd holdback and repeat Steps 1(b), 2, 3, and 4.
- 6(a). Neutralize solution to methyl red endpoint with fresh NaOH pellets and 6 M NaOH . Add 3 ml 0.5 M $(\text{NH}_4)_2\text{HPO}_4$ (buffered to pH 6.8) and digest hot for 10 minutes.
- (b). Wash twice with dilute $(\text{NH}_4)_2\text{HPO}_4$ buffer, twice with acetone, dry at 110°C for 10 minutes, weigh as $\text{Zn}(\text{NH}_4)\text{PO}_4$.

NOTE 1: If excess iron is present in the original solution, make basic with NaOH after Step 1(a), and discard $\text{Fe}(\text{OH})_3$. Make supernatant 1 M in HClO_4 and proceed with Step 1(b).

GALLIUM

G. M. Iddings

Purification: $\sim 10^9$ atoms of Ga^{72} isolated from a 36-hour-old solution of 2.6×10^{14} fissions showed no detectable foreign radionuclides when followed through 11 half-lives on an end-window-type flow proportional counter ($\sim 45\%$ geometry).

Yield: About 70%.

Separation Time: $\sim 3\text{-}1/2$ hours.

1. To a 6 N HCl solution of mixed activities in a 125-ml Erlenmeyer flask add 10 mg Ga carrier and 1 mg Te^{+4} carrier. Boil solution for ~ 10 minutes. Final solution should be ~ 20 ml (NOTE 1).
2. Cool and transfer solution to a 60-ml cylindrical, open-top, separatory funnel (with stem detached immediately below the stopcock). Add 4 ml conc. HCl to the 20 ml of 6 N HCl to make solution 7 N in HCl. Add ~ 25 ml isopropyl ether, and stir rapidly with a motor-driven glass rod stirrer (paddle) for 2 minutes. Record time of this separation of Zn^{72} from Ga^{72} for later correction if necessary.
3. Drain and discard aqueous layer. Wash organic layer with ~ 15 ml of 7 N HCl for 2 minutes. Repeat 7 N HCl wash.
4. Stir the ether layer with ~ 15 ml of water for 2 minutes. Repeat the back-extraction with ~ 10 ml of water and combine water layers in a 40-ml centrifuge cone.
5. Add ~ 5 mg Fe^{+3} carrier and sufficient 10 N NaOH to bring pH to ~ 13 (0.1 N in OH^-). Heat on boiling water bath 3 minutes to coagulate $\text{Fe}(\text{OH})_3$ precipitate. Cool to room temperature, centrifuge, and discard the $\text{Fe}(\text{OH})_3$ precipitate (filter the $\text{Fe}(\text{OH})_3$ if it does not centrifuge well).
6. Transfer the supernate to a new 60-ml cylindrical, separatory funnel (identical with the one used previously). Add a measured amount (~ 4 or 5 drops) of conc. formic acid until the pH is ~ 7 as determined by Bromothymol Blue indicator. Then add twice this amount of conc. formic acid. (The solution is thus buffered at pH 3.3-3.6 by this treatment.) Add 30 ml of 0.40 M TTA* in benzene and equilibrate phases by rapid stirring for 15 minutes.
7. Drain and discard aqueous layer. Wash organic layer with ~ 15 ml of 1 N NaOH for ~ 10 seconds. Immediately drain aqueous layer and repeat 1 N NaOH wash. Wash organic layer with ~ 15 ml of water for 10 seconds. Wash organic layer with ~ 15 ml of 1 N HCl for 3 minutes. Immediately drain aqueous layer and repeat 1 N HCl wash twice. (Step 7 should be done quickly because Ga slowly back-extracts from the organic layer during these washes. See NOTE 2.)

8. Stir organic layer with ~15 ml of conc. HCl for 20 minutes. Repeat conc. HCl back-extraction for 20 minutes.
9. Combine conc. HCl solutions and dilute with water until the solution is ~7 N HCl (add ~21 ml of water to 30 ml of conc. HCl). Transfer 7 N HCl solution to a 125-ml cylindrical, open-top, separatory funnel (with stem detached immediately below the stopcock). Add ~30 ml of isopropyl ether and equilibrate layers by rapid stirring for 2 minutes.
10. Drain and discard aqueous layer. Wash organic layer with ~15 ml of 7 N HCl for 2 minutes. Repeat 7 N HCl wash.
11. Stir the ether layer with ~20 ml of water for 2 minutes. Repeat the back-extraction with ~10 ml of water.
12. Combine water solutions of Ga in a 40-ml centrifuge cone. Add ~0.5 ml of a saturated solution of NH_4Ac . Add NH_4OH dropwise until the solution is ~pH 6 (Bromocresol Purple indicator). Heat in boiling water bath for 10 minutes.
13. Filter $\text{Ga}(\text{OH})_3$ with Whatman No. 42 filter paper. Wash precipitate with 2% NH_4NO_3 . Ignite filter paper in a porcelain crucible for 20 minutes at 900° - 1000°C.
14. Powder the Ga_2O_3 , weigh, and mount for counting.

NOTE 1: Boiling in 6 N HCl reduces Te^{+6} to Te^{+4} and ensures complete exchange between Te carrier and radioactive Te.

NOTE 2: The 1 N NaOH washes the "free" TTA from the benzene layer, leaving the Ga-TTA chelate. In subsequent washes with 1 N HCl, the equilibrium is shifted so that the Ga tends to leave the organic layer. However, in low acid (1 N HCl) the rate is slow. Elements, such as Zr, Np^{+4} , rare earths, Pa, Th, etc., are rapidly back-extracted into the 1 N HCl when there is essentially no "free" TTA in the benzene.

* Thenoyltrifluoroacetone.

ARSENIC

N. A. Bonner

Purification: Arsenic separated from $\sim 10^{15}$ fissions and counted on a 2-in. thick NaI crystal (400-700 keV) decayed with 39-hour half-life to < 20 c/m of long-lived activity.

Yield: $\sim 75\%$.

Separation Time: ~ 5 hours for four samples.

1. Add active solution in 6 M HCl to 20 mg arsenic carrier in 40-ml glass centrifuge cone. Add an equal volume of conc. HCl, 2 ml conc. HF, and 1 drop 47% HI. Add ~ 10 mg of Te^{+4} . Pass in H_2S gas and heat on hot-water bath for 5 minutes. Add a few drops 1% aerosol solution. Centrifuge and discard supernate.
2. To the As_2S_3 (+Te) precipitate, add 1 ml conc. HCl and heat on hot-water bath ~ 1 minute. Add 1 ml conc. HNO_3 + 1 ml conc. HClO_4 and heat to fumes of HClO_4 . Cool.
3. Add 10 ml 3 M HCl and heat to boiling. Add 3 drops of 47% HI and 1 ml 6% H_2SO_3 , and boil. Add another 1 ml of H_2SO_3 and again boil. Add a few drops of 6% H_2SO_3 and centrifuge. Grease lip of cone and filter through 7-cm Whatman No. 42 filter paper into 60-ml, open-top, separatory funnel. Do not wash filter. To the solution in the separatory funnel, add 2 ml 6 M HCl, 30 ml benzene, and 7 ml of 47% HI. Stir for at least 2 minutes with motor-driven glass paddle. Drain and discard aqueous phase.
4. Stir benzene solution 1-2 minutes with a solution of 5 ml 3 M HCl + 2 ml 47% HI. Discard aqueous phase.
5. Repeat Step 4.
6. Stir benzene solution for 2 minutes with 10 ml water, and drain aqueous layer containing the arsenic into a 50-ml beaker. Add 2 mg Te carrier. Discard benzene layer and rinse separatory funnel with acetone and 3 M HCl.
7. Pour aqueous solution back into separatory funnel, add 3 ml of conc. HCl and 7 ml of 47% HI. Add 30 ml of benzene and stir for at least 1 minute. Drain and discard aqueous phase.
8. Repeat Step 4 twice.
9. Stir benzene layer with 10 ml of water and drain aqueous phase into a 40-ml centrifuge cone. (Particles of broken glass or other foreign debris must be eliminated at this point since there is no further separation from arsenic before the final weighing step.) Discard benzene phase.
10. Add 20 ml of conc. HCl and a few drops of 1% aerosol, and pass in H_2S at room temperature. Centrifuge and discard supernate.

11. Repeat Step 2.
12. Add 10 ml 6 M HCl and a few drops 1% aerosol. Heat to boiling and add 4-5 ml CrCl_2 solution (oxsorbent). Heat to boiling again and add another 4-5 ml CrCl_2 .
13. Heat on water bath for 3 minutes and cool. Filter through a tared, 1-in. diam Whatman No. 542 filter paper. Wash once with water and once with methanol (the portion of the filter extending beyond the chimney should also be washed). Draw air through the filter for ~2 minutes. Dry at 110°C for 10 minutes, and weigh as As^0 .

SELENIUM

G. M. Iddings

Purification: 3.5×10^9 atoms of Se^{75} (2800 γ c/m) isolated from a 3-month-old mixture containing 10^{15} fissions had no foreign radioactive nuclides as determined by gamma-pulse-height analysis and beta counting.

Yield: ~60%.

Separation Time: ~3 hours.

1. To the solution in ~25 ml of 6 M HCl in a 40-ml glass centrifuge cone, add 20 mg of selenium, ~2 mg of Rh^{+3} , and ~2 mg Te^{+4} . Add ~3 ml of 6% H_2SO_3 . Let stand at room temperature for ~3 minutes. Centrifuge and discard the supernatant. Dissolve the precipitate in ~4 drops of conc. HNO_3 and 1 ml of conc. HCl in a hot-water bath for ~5 minutes. Boil, if the Se has not dissolved. Cool to room temperature.
2. Add ~20 ml of conc. HCl and 2-3 ml of 6% H_2SO_3 . Let stand for 3 minutes. Add a few drops of 1% aerosol solution and centrifuge. Discard the supernatant. Wash the Se^0 with a mixture of ~20 ml of conc. HCl and 1 ml of 6% H_2SO_3 . Centrifuge and discard the supernatant.
3. Dissolve the Se^0 in ~4 drops of conc. HNO_3 and 1 ml of conc. HCl in a hot-water bath for ~5 minutes or boil over flame. Cool to room temperature.
4. Repeat Steps 2 and 3 five times.
5. Add ~15 ml of 2 M HCl and ~10 mg of Ba carrier. Add 1/2 ml conc. H_2SO_4 . Heat in a hot-water bath for ~3 minutes and cool to room temperature. Filter and discard the BaSO_4 .
6. To the filtrate add an equal volume of conc. HCl and ~3 ml of 6% H_2SO_3 . Let stand for ~3 minutes. Centrifuge and discard the supernatant. Add ~5 ml of water to the Se^0 and filter through a tared filter paper disc. Wash with water and methanol. Dry at 110°C for 10 minutes. Weigh, mount, and count.

RUBIDIUM AND CESIUM

J. B. Niday

Decontamination: 10^{10} atoms of Rb^{86} isolated from a 3-day-old solution of 10^{15} fissions showed no detectable foreign radioactivity over nine half-lives. 10^{11} atoms of Cs^{136} isolated from a 3-day-old mixture of 10^{15} fissions showed no detectable contamination over four half-lives.

Yield: About 60-70%.

Separation Time: Two days for four samples.

1. To the active solution add 20 mg each of rubidium and cesium carriers. If the solution contains negligible amounts of other elements, boil the solution to near-dryness and take up in 20 ml of water. Other solutions should be treated in such a way that the final solution is free of such elements and contains less than about 15 milliequivalents of acid in a volume of about 20 ml. An example might be the solvent extraction of large masses of uranium into ether, with subsequent boiling down of the aqueous residue to a volume of 20 ml at the specific acid concentration. Another example might be the precipitation of large amounts of ferric hydroxide. In the latter case the presence of large quantities of ammonium salts will probably be inevitable. In this event proceed from Step 3 directly to Step 4(b), omitting Step 4(a).
2. To the adjusted solution containing not more than 15 milliequivalents of acid in a volume of 20 ml, add 5 mg Fe^{+3} , one drop aerosol, and 2 drops of Bromothymol Blue indicator. Make solution slightly basic with NH_4OH . Centrifuge and discard precipitate.
3. Add another 2-4 mg Fe^{+3} and 1 mg La^{+3} . Centrifuge and discard precipitate.
- 4(a). Transfer supernatant to a 125-ml Erlenmeyer flask. Add NaOH pellets and boil until no further test (with moist pH paper) for ammonia fumes is obtained upon addition of more NaOH. Proceed to Step 5.
- (b). If large quantities of ammonium salts are present (i.e., more than can be removed by addition of 0.8 g NaOH), add aqua regia to the solution, and boil down just to dryness several times, first with an excess of HCl, then with an excess of HNO_3 . (This treatment serves to oxidize the NH_4^+ .) Proceed to Step 5.
5. Acidify with HNO_3 , add 2-5 ml conc. HClO_4 , and fume to 1-3 ml of HClO_4 . Cool and chill in an ice bath with occasional swirling. Transfer to a 40-ml centrifuge cone, centrifuge, and wash precipitate twice with 30 ml ethyl acetate. Wash down walls and macerate precipitate with a glass rod. Discard supernatant and washes.

6. Add 15 ml water to the precipitate and boil briefly to ensure solution. Add 5 mg Ba^{++} and 2 mg Fe^{+3} carriers. Add about 5 drops 6 M NaOH and 1.5 milliequivalents of Na_2CO_3 . Centrifuge and discard precipitate.
7. Add 3 mg Ba^{++} and 3 mg Fe^{+3} carrier to the supernatant. Centrifuge and discard precipitate.
8. Transfer supernatant to a 125-ml Erlenmeyer flask, boil, and check for completeness of NH_4^+ removal with pH paper.
9. Neutralize the solution with 6 N HCl and add 18-20 drops in excess. Boil out CO_2 and transfer to a 40-ml cone. Dilute to 30 ml (0.2 N in HCl), add 10 drops Sb carrier and saturate with H_2S . Centrifuge and pass supernatant through fast filter paper. Collect filtrate in a 125-ml Erlenmeyer flask.
10. Boil filtrate until H_2S is completely removed. Add 1 ml conc. HNO_3 and 2-3 ml conc. HClO_4 . Repeat Step 5.
11. Dissolve the precipitate in 20 ml water, boil out residual ethyl acetate, and pass solution through a $\sim 1 \text{ cm}^2$ (cross section) Duolite C-3 (BioRex 40) cation exchange resin column. (The resin should have previously been washed with HCl and several times with water.) Reject effluent from column.
12. Pass 0.3 N HCl through the column at a rate not to exceed 1 ml per minute per cm^2 . Collect the eluate in fractions of 5-10 ml and test each fraction by removing 2 or 3 drops and by adding a drop or two of 0.1 M phosphotungstic acid solution. Cs precipitates rapidly but low concentrations of Rb may not precipitate for several minutes. As soon as the second series of precipitates (Cs elution) begins, strip the column with 3 M HCl. (Normally the Rb fraction will be contained in 60-70 ml, beginning after about 55 ml of 0.3 N HCl have been eluted, and there will be a gap of 30-60 ml before Cs begins to elute.)
13. Rb fraction: Boil down in a 250-ml Erlenmeyer flask, add 2 ml each of conc. HNO_3 and conc. HClO_4 , and proceed as in Step 5. Wash precipitate at least twice with 30 ml ethyl acetate to remove all traces of HClO_4 . Slurry the precipitate with a few drops of ethyl acetate and transfer to a suitable weighed counting plate. Dry under an infrared lamp, then dry in an oven at 110°C . Cool and weigh as RbClO_4 .
14. Cs fraction: Cs may be handled in the same manner as Rb or alternatively, precipitated as Cs_2PtCl_6 . See Cesium (Alternate Procedure), this report, by H. M. Perdue.

STRONTIUM

R. Gunnink

Purification: 2×10^{13} atoms of Sr^{89} , Sr^{90} and daughter separated from 10^{15} fissions show no detectable foreign radionuclide contamination. Ba decontamination exceeds a factor of 10^5 .

Yield: 70-80%.

- 1(a). The starting solution must be free of such anions as sulfate and oxalate.
- (b). To the active acidic solution add 10 mg Sr^{+2} and 10 mg Ba^{+2} carriers.
- (c). Add carbonate-free NaOH, centrifuge, and discard any precipitate. Pour supernate into clean cone. Centrifuge, wash twice with water.
2. Add Na_2CO_3 solution to the supernate until further addition causes no further precipitation. Centrifuge and discard supernate. Wash precipitate twice with water.
- 3(a). If Ca is present add 5 ml 70% HNO_3 slowly to the precipitate. Stir, add 20-25 ml 90% HNO_3 , cool in an ice bath, and digest for 10 minutes. Centrifuge, and discard supernate, and wash with 20-25 ml 70% HNO_3 . Dissolve precipitate in 5 ml water and neutralize with NH_4OH . Add HAc if the solution becomes basic. Proceed with Step 4.
- (b). If Ca is absent, dissolve precipitate in 3 ml 9 M HCl and transfer to preconditioned Dowex 21K column. Wash cone with 1 ml 9 M HCl and pass through column. Wash column with an additional 2 ml 9 M HCl. To the eluate add NaOH and Na_2CO_3 solution until precipitation is complete. Wash once with water. Add sufficient HAc to dissolve the precipitate, heat to boiling, and dilute to ~5 ml.
4. Transfer the solution to a column 6 mm i. d. $\times$ 9 cm, containing 100-200 mesh Dowex 50-X8 resin. Wash cone with 1 ml of water and add to column.
5. Elute the column as follows:
 - (a) 8 ml 1 M α -HIBA (α -hydroxyisobutyric acid), pH 5.0. (This step removes residual amounts of Ca.)
 - (b) 8 ml of 1.5 M α -HIBA, pH = 5.0. (The strontium will be removed from the column during the elution.)
 - (c) If it is desirable to remove the barium from the column, this can be done with 5 ml 4 M HNO_3 .
6. To the Sr fraction add NaOH and Na_2CO_3 solutions until precipitation is complete. Wash precipitate and dissolve in minimum amount of HNO_3 . Boil to remove CO_2 .
7. Dilute to 25 ml, add 1 mg each of Fe^{+3} and La^{+3} scavenge, make basic with NH_3 gas. Centrifuge and pour into a cone containing Fe-La scavenge. Mix, centrifuge, and filter through No. 4 paper. (Note: This is the last step in which a Sr-Y separation is made, should the information be necessary for counting purposes.)

8. Add NaOH and Na_2CO_3 solution until SrCO_3 precipitation is complete. Digest briefly. Centrifuge, and wash twice with water, once with acetone. Dry at 110°C and weigh.

YTTRIUM

G. M. Iddings and W. H. Hutchin

Purification: From a 2-week-old solution containing 1.1×10^{15} fissions, a sample was obtained which had a gamma activity of about 27γ c/m between 1700 keV and 2000 keV through $\sim 1100 \text{ mg Cu cm}^{-2}$ absorber on a 2-in-thick $\times$ 1-3/4-in-diam NaI crystal. For other purification, see NOTES.

Yield: $\sim 65\%$.

Separation Time: ~ 5 hours.

1. To a 25-ml solution of mixed activities in 6 M HCl in a 40-ml glass centrifuge cone, add 20 mg of yttrium carrier and ~ 2 mg each of Sc, Zr, Te^{+4} , and Ce^{+3} . Add conc. NH_4OH in excess. Centrifuge the $\text{Y}(\text{OH})_3$ and discard the supernatant. Wash the precipitate twice with ~ 20 ml of water to remove salts (particularly Na salts). Centrifuge and discard the wash solutions.
2. Dissolve the precipitate in a few drops of 6 M HNO_3 . Transfer the solution to a 50-ml lusteroid tube with ~ 5 ml of water. Add an equal volume of Na-free 6 M NH_4F solution, making the final solution ~ 3 M in NH_4F and pH 4-5. Check the pH with narrow-range pH paper. Heat in a hot-water bath for ~ 3 minutes. Cool and centrifuge. Discard the supernatant (Sc). Add ~ 10 ml of 3 M NH_4F to the precipitate. Heat for 3 minutes. Cool and centrifuge, discarding the supernate (Sc). Wash the YF_3 with ~ 5 ml of water. Centrifuge and discard the supernate (Sc). (See NOTE 1.)
3. Dissolve the YF_3 in ~ 3 ml of saturated H_3BO_3 , ~ 10 ml of water and an equal volume of 12 M HNO_3 , making the final solution ~ 6 M in HNO_3 . Add ~ 3 ml of 5 M HNO_3 . Transfer the solution to a 40-ml glass centrifuge cone. Add ~ 1 ml of saturated NaBrO_3 solution. Stir well and heat in hot-water bath for ~ 5 minutes. Cool to room temperature and centrifuge. Discard the $\text{Ce}(\text{IO}_3)_4$ precipitate.
4. To the supernate add ~ 2 mg of Zr carrier while stirring the solution. Centrifuge well and discard the $\text{Zr}(\text{IO}_3)_4$ precipitate. Repeat the Zr iodate scavenge. Pour the supernatant into a 50-ml lusteroid tube (see NOTE 2).
5. Add 12-15 ml of 6 M NH_4F (solution is now ~ 3 M in H^+). Heat on hot-water bath for 5 minutes. Cool to room temperature in a cold-water bath for 5 minutes. Centrifuge and discard the supernatant (Np^{+6}). Wash the YF_3 with ~ 5 ml of water and discard the wash solution.
6. To the YF_3 add 2 ml of saturated H_3BO_3 . Mix thoroughly and add 3 ml of conc. HCl. Heat on hot-water bath for ~ 3 minutes. Dilute to ~ 30 ml volume with water and make solution basic to phenolphthalein conc. NH_4OH . Cool, centrifuge, and discard the supernatant. Wash the $\text{Y}(\text{OH})_3$ with ~ 20 ml of water. Centrifuge and discard wash.

7. Dissolve the precipitate in ~20 ml of ~pH 1.0 buffer solution, which is 0.5 M in Na_2SO_4 and 0.5 M NaHSO_4 , and transfer solution to a 40-ml glass cone.
8. Add 5 ml of toluene and ~10 ml of CrCl_2 solution (Oxsorbent). Add ~10 mg Ba (1 ml of solution as the chloride) dropwise while stirring slowly. Heat in hot-water bath (~95°C) for 5 minutes. Cool in a cold-water bath for 5 minutes. Add ~10 mg Ba dropwise while stirring slowly. Centrifuge well and pour supernatant into a cone containing ~10 mg of Ba. Heat for ~2 minutes in hot-water bath. Cool in cold-water bath for ~3 minutes. Add ~10 mg Ba dropwise while stirring slowly (see NOTE 3).
9. Pour supernatant into a 50-ml lusteroid tube containing ~2 ml of conc. HF. Stir, centrifuge, and discard supernatant. Wash the YF_3 with ~20 ml of water, and discard the wash solution.
10. Dissolve the YF_3 in 1 ml of 5% H_3BO_3 . Mix thoroughly. Heat in hot-water bath for ~3 minutes. Add ~2 ml conc. HNO_3 . Transfer the solution to a 40-ml glass cone rinsing lusteroid with 2 ml of 6 M HClO_4 .
11. Heat carefully to strong fumes of HClO_4 . Cool and dilute to ~20 ml with water. Add an excess of conc. NH_4OH . Centrifuge the $\text{Y}(\text{OH})_3$ and discard the supernatant. Wash the precipitate with 30 ml of water.
12. Dissolve the $\text{Y}(\text{OH})_3$ in ~15 ml of buffer solution of 0.1 M formic acid and 1 M Na formate (pH ~ 4.6). Transfer the solution to a 60-ml cylindrical separatory funnel and equilibrate the solution with ~30 ml of 0.40 M TTA* solution in benzene for 3 minutes stirring with a motor-driven glass paddle. Wash the organic layer 3 times with ~15 ml of buffer solution of 0.4 M formic acid and 1 M Na formate - (pH ~4.0) for 3 minutes. (See NOTE 4.)
13. Stir organic layer for 3 minutes with ~20 ml of 0.05 M HCl . Allow layers to settle for several minutes and then drain the aqueous layer into a 40-ml glass cone. Centrifuge and discard any precipitate, broken glass, etc.
14. Add ~5 ml of saturated oxalic acid and stir vigorously. Let stand for 5 minutes; then centrifuge and discard the supernatant. Wash the yttrium oxalate with a few ml of 0.2 M oxalic acid and filter through 7-cm No. 42 Whatman paper. Ignite at ~900°C for ~20 minutes in a Coors No. 00 porcelain crucible. Weigh, mount, and count.

NOTE 1: Steps 2, 8, 12, and 14 give an overall separation factor of $> 10^6$ of Sc from Y.

NOTE 2: Steps 3, 4, and 12 have an overall separation factor of $> 10^6$ of Ce from Y.

NOTE 3: Step 8 has a separation factor of $\sim 10^4$ of Eu tracer from Y. Nd, Pr and other cerium earths, if carrier-free, will also separate from Y at this point.

NOTE 4: Three washes in Step 12 will separate mass La from Y by a factor of ~100. More washes will give a greater separation factor, but there is about a 5% loss of Y at each wash step.

* Thenoyltrifluoroacetone.

ZIRCONIUM

G. M. Iddings and W. H. Hutchin

Purification: From a 4-day-old solution containing 10^{15} fissions and other activities, a sample was obtained which had no detectable foreign radionuclides when counted on a trochoidal-type analyzer for positrons associated with Zr^{89} . The procedure does not separate from hafnium.

Yield: 85-90%.

Separation Time: ~3-1/2 hours.

1. To a solution of mixed activities in 6 M HCl in a 125-ml Erlenmeyer flask, add 10 mg of zirconium carrier and ~2 mg each of Ce^{+3} and Te^{+4} . Heat to boiling and add 1 ml of 1 M H_3PO_4 . Cool to room temperature and centrifuge the precipitate in a 50-ml lusteroid tube. Discard the supernatant. Wash the precipitate with ~10 ml of 2 M HCl. Centrifuge and discard the supernatant.
2. To the precipitate add ~25 ml of 2 M HCl, ~1 ml of 6% H_2SO_3 , ~2 mg of Ce^{+3} , and ~1 ml of saturated KCl solution. While stirring vigorously, add ~1 ml of conc. HF. Centrifuge and discard the CeF_3 precipitate (separation from Np).
3. To the supernatant add ~4 drops of saturated $BaCl_2$ solution. Centrifuge the $BaZrF_6$ precipitate and discard the supernatant. Wash the precipitate with ~5 ml of water. Centrifuge and discard the wash solution.
4. To the precipitate add ~4 ml of saturated H_3BO_3 solution, ~5 ml of water, and ~3 ml of conc. HNO_3 . Heat the solution in a hot bath for ~3 minutes, then add ~0.5 ml of conc. H_2SO_4 . Dilute to ~25 ml volume with water. Cool, centrifuge, and discard the $BaSO_4$.
5. To the supernatant add conc. NH_4OH in excess. Centrifuge and discard the supernatant. Wash the precipitate with ~25 ml of 0.1 M NH_4OH - 1 M NH_4Cl solution. Centrifuge and discard the supernatant.
6. Dissolve the precipitate in 8 ml of conc. HCl and pass the resulting solution (~10 M in HCl) through a Dowex-1 anion exchange resin column (50-100 mesh), 6 mm i. d. $\times$ 7 cm. Pass 3 ml of 10 M HCl through the column. (Ti does not adhere in 10 M HCl.) Repeat the wash twice (carefully washing the walls of the column). Discard the effluents.
7. Pass 15-20 ml of 7 M HCl through the column and collect eluate in a clean cone.
8. To the 7 M HCl solution of Zr, add ~5 ml of saturated (~1 M) mandelic acid and heat in a hot-water bath for ~45 minutes. Cool to room temperature, centrifuge, and discard supernatant. Wash precipitate with water and filter the zirconium mandelate onto a tared 1-in. No. 542 Whatman paper, using a filter-chimney setup. Wash the precipitate with water and methanol. Dry at 110°C for 10-15 minutes. Cool to room temperature. Weigh, mount, and count.

ZIRCONIUM (ALTERNATE PROCEDURE)

H. G. Hicks

Purification: 5×10^{13} atoms of Zr^{97} isolated from a 1-day-old solution containing 10^{15} fissions exhibited no trace of foreign radioactivity when followed through six half-lives. The procedure does not separate from hafnium.

Yield: 60%.

Separation Time: 8 hours for four samples.

Remarks: If no Fe present, begin at Step 3.

1. Transfer sample to "Auto Clear" plastic tube with water, diluting to ~2 M acid. Add ~20 mg standardized Zr carrier, 5 ml conc. HF and stir. If precipitate forms at this point, centrifuge and discard it, pouring supernate into clean tube. Add 2 ml saturated BaCl_2 solution and digest 5 minutes at room temperature. Centrifuge and wash precipitate with dilute HF, discarding wash.
2. To precipitate add 5 ml saturated H_3BO_3 solution, and about 0.5 ml conc. HNO_3 . Stir until dissolved, heating in hot-water bath if necessary.
3. When dissolution is complete add conc. NaOH dropwise until basic to phenolphthalein. Centrifuge and discard supernatant, washing precipitate once with dilute NaOH solution.
4. Dissolve precipitate in conc. HNO_3 , adjusting solution to 4 M HNO_3 with water. Transfer solution to 125-ml separatory funnel with 4 M HNO_3 .
5. Add 20 ml 0.4 M TTA* (in toluene) and extract 30 minutes. Discard aqueous layer.
6. Wash TTA twice for 10 minutes each time with 15 ml 4 M HNO_3 + 1 ml 1 M KNO_2 . Discard aqueous layers.
7. Wash TTA once with 15 ml 4 M HCl for 10 minutes. Discard aqueous layer.
8. Add 15 ml of a solution 9 M in H^+ (HCl) and 2 M in HSO_4^- (H_2SO_4). Fill funnel to within one inch of the top with toluene and stir 30 minutes. Remove aqueous through water-wet filter paper. Add 5-10 ml water to funnel as a rinse. Filter and combine aqueous layers.
9. Dilute aqueous to 30-35 ml and add 4 ml conc. H_3PO_4 . Stir, digest hot, centrifuge, and discard supernate.
10. Wash precipitate twice with 2 M HCl and twice with acetone.
11. Dry precipitate in hot-water bath with constant stirring to produce a fine powder. Transfer powder to porcelain crucible.
12. Dry further in 120°C oven for 10 minutes. Ignite (carefully at first) at bright red heat for 20 minutes. Cool and weigh as ZrP_2O_7 .

*Thenoyltrifluoroacetone.

NIOBIUM

G. M. Iddings and E. T. Chulick

Purification: Separation factor of $> 6 \times 10^4$ of Nb from Ta. From a 6-day-old mixture containing $\sim 10^{14}$ fissions a sample of Nb⁹⁵ was isolated which exhibited no contaminating activity when gamma-pulse-height-analyzed.

Yield: $\sim 50\%$.

Separation Time: About 6 hours, including the column step.

1. To ~ 50 ml of mixed activities in ~ 6 M HCl in a 250-ml Erlenmeyer flask, add 20 mg Nb, ~ 10 mg Zr, and ~ 1 mg Te⁺⁴ carriers. Heat to boiling and add 1.0 ml of 1 M H₃PO₄. Boil to ~ 15 ml volume. Add an equal volume of water. Cool to room temperature and centrifuge the precipitate in a 50-ml lusteroid tube. Discard the supernate. Wash the precipitate with ~ 10 ml of 2 M HNO₃. Centrifuge and discard wash.
2. To the precipitate in the 50-ml lusteroid tube, add ~ 25 ml of 1 M HCl, 1 ml of 6% H₂SO₃, and ~ 2 mg Ce⁺³. Make the solution ~ 1 M in HF by adding 1 ml of conc. HF. Stir well, then add ~ 4 drops of saturated BaCl₂ (~ 35 mg of Ba) solution. Centrifuge and discard precipitate. (Zr-Nb separation as well as rare earth-Nb separation.)
3. Make the supernate basic (pH 8-9) with an excess of concentrated NH₄OH. Cool to room temperature. Centrifuge and discard the supernate. Wash the precipitate twice with ~ 15 ml of 0.1 M NH₄OH - 1 M NH₄Cl solution. Centrifuge and discard wash solutions.
4. To the precipitate add $\sim 1/2$ ml of 30% H₂O₂ and ~ 30 ml of 1 M HCl. Stir thoroughly to dissolve. Transfer solution to a 40-ml glass cone and add ~ 2 mg of Zr, ~ 1 mg Fe⁺³, and ~ 1 mg of Te⁺⁴ carriers. Make the solution basic with ~ 3 ml of concentrated NH₄OH. Cool to room temperature, centrifuge, grease lip of cone, and filter through 9-cm No. 42 Whatman filter paper into a 40-ml glass cone.
5. Heat filtrate on a hot-water bath for 5 minutes. Add sufficient 55% HI (~ 1 ml) dropwise to make the solution acidic. Heat on a hot-water bath 5 minutes more with occasional stirring to destroy the peroxide. Cool to room temperature. Make the solution basic (pH 8-9) with ~ 1 ml of concentrated NH₄OH. Centrifuge the precipitate and discard the supernate.
6. Dissolve the precipitate in ~ 5 ml of 12 M HCl. Using 32 cm $\times$ 7 mm i. d. polypropylene tubing, prepare a 16-cm long bed of Dowex 1X8 ion exchange resin (200-400 mesh) supported by a Dacron wool plug. Pretreat the column with 5 ml of 12 N HCl and add the Nb solution to the pretreated resin column. Continuously elute the column with 50 ml of 9 M HCl - 0.20 M HF solution using a nonglass

reservoir at 55-60 cm above the resin bed and connected to it with a tygon tubing. Discard the first 20 ml of the 9 M HCl - 0.20 M HF eluate. Collect the next 20-30 ml in two 40-ml lusteroids in equal portions. (See NOTE 1.)

7. To each of the solutions in the lusteroid tubes add concentrated NH_4OH to pH ~8-9 until precipitates form. This will require ~15 ml. Centrifuge and discard supernates. Combine precipitates after slurring in ~15 ml of 0.1 M NH_4OH - 1 M NH_4Cl solution. Centrifuge and discard the supernate. Repeat the wash with another 15 ml 0.1M NH_4OH - 1 M NH_4Cl . Centrifuge and discard supernate.
8. Add ~20 ml of 4 M HNO_3 to the precipitate. Stir thoroughly and transfer to 40-ml glass cone. Heat in a boiling water bath for 5 minutes. Cool to room temperature and centrifuge. Discard the supernate, wash precipitate with 4 M HNO_3 , and filter through 7-cm No. 42 Whatman filter paper.
9. Ignite to Nb_2O_5 in a furnace at 1000°C for 20 minutes in a No. 00 Coors porcelain crucible. Transfer the oxide to a tared counting receptacle and weigh. Add a few drops of Duco (diluted 20-fold with ethyl acetate).

NOTE 1: At this point, Ta remains on the column; it can be eluted from the resin with 1 M H_2SO_4 if desired. The Ta-Nb separation factor is $> 2 \times 10^4$ in this step.

MOLYBDENUM

H. G. Hicks

Purification: 2×10^{13} atoms of Mo^{99} isolated from 1-day-old solution of 10^{15} fissions showed no detectable foreign radionuclide when followed through six half-lives.

Yield: 50%.

Separation Time: 6 hours for eight sample.

Remarks: This procedure was developed with emphasis on separation from gold and tungsten radionuclides (NOTE 1).

1. The active solution should be adjusted to 1-2 M HCl.
2. Add 2 drops Au carrier, transfer to separatory funnel with water, and extract 5 minutes with an equal volume ethyl acetate.
3. Filter aqueous layer through a water-wet filter paper into 125-ml Erlenmeyer flask.
4. Make basic with NH_4OH (see NOTE 2) and add 1 mg W carrier and 1 ml saturated tartaric acid.
5. Saturate with H_2S gas until orange color is formed.
6. Add conc. H_2SO_4 dropwise until acid (pH 2-3) and heat to boiling on hot plate to digest black MoS_3 precipitate.
7. Transfer contents to a 40-ml centrifuge cone. Centrifuge and wash precipitate once with a mixture of water, H_2SO_4 , and tartaric acid in approximately the same proportions as those in Step 6. Preheat the wash to boiling in the sample flask. Digest in hot-water bath and centrifuge. Discard supernate.
8. Dissolve precipitate in 2 ml conc. HCl and 2 drops conc. HNO_3 . Heat, if necessary. Then add 5 ml 6 M HCl.
9. Transfer to separatory funnel using water wash. Agitate with 25 ml amyl acetate saturated with α -benzoin oxime for 5 minutes (below 1 M H^+ , ethyl acetate can be used). (Mo gives milk-white precipitate in organic layer.) Add ethyl acetate to maintain volume of organic phase at about 25 ml. Separate phases, drain and discard aqueous layer.
10. Wash organic layer twice with 20 ml 1 M HNO_3 and twice with 20 ml water.
11. Add 10 ml ethyl acetate and 5 ml 3 M NH_4OH to the organic phase. Stir until both layers are clear. Drain and filter aqueous layer through a water-wet filter (be sure aqueous is basic). Wash organic briefly with 5-10 ml water and combine with ammoniacal aqueous fraction
12. Pass ammoniacal solution through an anion column (Dowex 21K, 50-100 mesh, 9 cm $\times$ 0.6 cm) previously washed with 3 N NH_4OH .
13. Wash column with 5 ml water and 15 ml 0.1 M HNO_3 . Discard washing.
14. Elute with 15 ml 4 M HNO_3 , into a 40-ml cone.

15. Add 2 drops special scavenge solution* and 3 drops KNO_2^{**} . Make basic with NH_4OH , centrifuge and discard precipitate.
16. Pour supernate into cone containing 1 mg each of Fe^{+3} and La^{+3} . Centrifuge and discard precipitate. Pour supernatant through No. 4 filter paper into rinsed cone.
17. Adjust supernate to pH 5 with glacial acetic acid, add 2 ml 0.1 M $\text{Pb}(\text{NO}_3)_2$ and digest PbMoO_4 in hot-water bath. Wash precipitate twice with water containing aerosol, and once with acetone. Dry and weigh as PbMoO_4 .

NOTE 1: If Au separation is not required, begin at Step 4 with W separation. If neither W nor Au separations are required, begin at Step 8 with 6 M HCl solution.

NOTE 2: If sample contains hydroxide-insolubles, centrifuge, discard precipitate, and continue with supernatant.

*Special scavenge solution contains 10 mg/ml each of Fe (III), La (III), and Zr (IV) and 2.5 mg/ml of Te (VI) in 6 M HCl .

**If Np is not present, KNO_2 may be eliminated.

MOLYBDENUM (ALTERNATE PROCEDURE)

H. G. Hicks

Purification: 2×10^{13} atoms of Mo^{99} isolated from 1-day-old solution of 10^{15} fissions showed no detectable foreign radionuclide when followed through six half-lives.

Yield: 75%.

Separation Time: 4 hours for eight samples.

1. Adjust to 6 M in HCl (if NO_3^- present, boil with HCOOH for removal), and pass solution through on Dowex 21 K, 50-100 mesh resin column (if 21 K is unavailable, Dowex 1-X8 may be used in its place).
2. Wash with 10 ml 0.1 M HCl, 1 ml water, 5 ml 3 M NH_4OH , and 1 ml water. Discard washes.
3. Elute Mo with 20 ml 4 M HNO_3 , collecting eluate in clean cone.
4. Add 3 drops of scavenge solution* to eluate and 3 drops of 1 M KNO_2 if Np is present. Make pH 10 with conc. NH_4OH . Centrifuge and discard precipitate.
5. Pour supernatant into cone containing 1 mg each of Fe^{+3} and La^{+3} . Stir, centrifuge, and pour supernate through No. 4 filter paper.
6. Add glacial HAc to adjust filtrate to pH 5, and add 2 ml 0.1 M $\text{Pb}(\text{NO}_3)_2$. Digest resultant precipitate in hot-water bath for 10 minutes. Wash twice with water containing 1 drop aerosol and once with acetone. Dry in air oven at 110°C and weigh as PbMoO_4 .

*Scavenge solution contains 10 mg each of Fe^{+3} , La^{+3} , and Zr^{+4} and 2.5 mg Te^{+6} per ml in 6 M HCl.

RHODIUM

N. A. Bonner and G. M. Iddings

Purification: A sample containing less than 5 c/m of impurity (β counter, ~70% counting efficiency on Pt.) was obtained from a 7-week-old mixture of 10^{15} fissions.

Yield: ~70%.

Separation Time: ~5 hours exclusive of electrodeposition.

1. Add the active solution in 6 M HCl to 20 mg of Rh carrier, ~2 mg of Te carrier, and 20 mg of Tl carrier in a 125-ml Erlenmeyer flask. Add ~8 ml of conc. HClO_4 and 1 ml conc. HNO_3 . Evaporate to fumes of HClO_4 . Fume vigorously until volume of HClO_4 is about 4 ml. Add 10 ml of conc. HCl and evaporate until total volume is 10 ml or less.
2. Transfer to a 40-ml centrifuge tube with water and dilute to 20 ml. Add 5 drops of 57% HI (containing no H_3PO_2) and 2 ml of 6% H_2SO_3 solution. Stir vigorously. Centrifuge and discard TH; pour supernate into a 125-ml Erlenmeyer flask.
3. To solution in flask, add 10 ml of conc. HCl and 2 ml of 57% HI, and boil vigorously for at least 20 minutes to precipitate RhI_3 . Add 6 M HCl, if necessary, to keep volume between 15 and 30 ml. Centrifuge in a 40-ml centrifuge cone. Wash RhI_3 precipitate with 10-15 ml of 6 M HCl.
4. To RhI_3 , add 3 ml conc. HCl + 1 ml 30% H_2O_2 . Mix thoroughly. Warm slightly. After solution has become clear (but very dark), boil to about 1 ml volume. Dilute to 20 ml with water. If a precipitate of RhI_3 remains, centrifuge, and dissolve it in $\text{HNO}_3 + \text{HClO}_4$. Evaporate this to ~1/2 ml and combine with main body of supernate. Add 5 ml of saturated NaNO_2 (~9 M). After most of the effervescence has stopped, heat on hot-water bath for at least 10 minutes. Add 1 mg of Fe^{+++} carrier, stir vigorously, and heat on water bath for one minute. Centrifuge and pour supernate into a clean 40-ml cone and discard precipitate.
5. Add 1 mg of La carrier + a few drops of phenolphthalein and stir. Add 1 M NaOH dropwise until solution turns pink (1 or 2 drops is usually enough). Stir and heat on water bath about one minute. Centrifuge, pour supernate into a clean centrifuge cone, and discard precipitate.
6. Add to the supernate 2 drops of 6 M HCl and 2 ml of saturated KNO_3 (~3 M). Stir vigorously and heat in water bath for at least 2 minutes. Cool to room temperature, and centrifuge. Wash white precipitate of $\text{K}_3\text{Rh}(\text{NO}_2)_6$ with 15 ml of 0.1 M HCl (NOTE 1).
7. To precipitate, add 3 ml of 6 M HCl and boil to dissolve. Evaporate just to dryness. If the solution is not deep ruby red and clear before dryness is approached, take nearly to dryness, add more 6 M HCl and carefully evaporate again to dryness (NOTES 2 and 3).

8. Dissolve the residue in ~5 ml of 0.1 M HCl and transfer to a Dowex-1 anion column (Cl^- form, water washed, 50-100 mesh, X-8 in a column 6 mm in diam and 5-6 cm long). Rinse contents of tube onto column with another few ml of 0.1 M HCl. Put small glass wool pad on top of ion bed when the percolation of solution is complete. Wash column with 15 ml of 0.1 M HCl. Discard washes.
9. Pass 20 ml of conc. HCl through column. Collect eluate in a 125-ml Erlenmeyer flask. Add 1 mg of Te carrier and boil for 10 minutes. If volume becomes too small, add 6 M HCl.
10. Cool to room temperature. Add 5 ml of 55% HI and immediately transfer solution to a 60-ml open-top, separatory funnel containing 30 ml of hexone. Use 6 M HCl for transfer. Aqueous volume should be 20-30 ml. Stir vigorously for 2 minutes and allow to settle for a short time after phases have separated. Extreme care should be taken to prevent any of the organic phase from passing through with the aqueous phase when it is removed (this step removes the last traces of Te and Ag fission product activities).
11. Withdraw aqueous phase into a 125-ml Erlenmeyer flask and boil vigorously for at least 20 minutes to precipitate RhI_3 . Add 6 M HCl, if necessary, to keep volume between 15 and 30 ml. Centrifuge, wash once with 15 ml of 6 M HCl (NOTE 4).
12. Dissolve precipitate in 3 ml of conc. H_2SO_4 by heating to fumes of SO_3 until I_2 vapor is no longer visible. Cool to room temperature, add 3 ml water, and 1 ml of conc. HCl. Boil for brief time, then add 1 ml each of conc. HNO_3 and conc. HClO_4 . Continue heating until all HClO_4 has evaporated and SO_3 fumes are evolved strongly.
13. Cool to room temperature, dilute with ~5 ml of water. Transfer to plating cell with water. Plate onto tared Pt plate for at least 8 hours at 100 mA. Rinse, dry and weigh. Count as appropriate.

NOTE 1: Do not allow the $\text{K}_3\text{Rh}(\text{NO}_2)_6$ to stand longer than necessary in the 0.1 M HCl wash solution. Do not heat the solution. The salt dissolves slowly in 0.1 M HCl.

NOTE 2: If more than one sample is being worked up, it saves time to place in the hot-water bath those samples which have not yet been evaporated while the first samples are being evaporated.

NOTE 3: From the time the rhodium chloride is dissolved in 0.1 M HCl (Step 8) until it has been eluted from the column (Step 9), the procedure should be carried on without stopping. Any appreciable delays will cause loss of Rh.

NOTE 4: If large amounts of iridium radioactivity were thought to be present in the original solution, omit Step 12 and proceed as follows: Dissolve the RhI_3 in 1 ml conc. HNO_3 and 2 ml of conc. HClO_4 . Heat to strong fumes of HClO_4 . Cool, add 1 ml conc. HBr and 1 ml conc. HCl . Heat just to fumes of HClO_4 . Cool, add 1 ml conc. HNO_3 , and heat to strong fumes of HClO_4 . Continue heating until the solution is clear and the volume is ~ 1 ml. Cool, add ~ 5 ml of 6 M HClO_4 . Transfer the 6-8 M HClO_4 solution to a Dowex 50 (100-200 mesh) cation exchange column (6 mm i. d. and 7 cm in length) which has been preoxidized with 8 M HNO_3 and washed with 6M HClO_4 . Wash the Rh through the resin column with two or three 5-ml portions of 6 M HClO_4 into a 125-ml Erlenmeyer flask (Ir^{+4} adheres to the resin).
Add 2 ml of conc. HNO_3 and 3 ml of conc. H_2SO_4 to the Rh solution in the flask. Heat until all HClO_4 has evaporated, and SO_3 fumes are evolved strongly. Continue to Step 13 (which also separates from Ir).

RHODIUM (ALTERNATE PROCEDURE)

W. H. Hutchin

Purification: A sample containing 7 c/m Rh¹⁰² (β counter, 30% geometry) was obtained from an 8-week-old fission product mixture containing 10^{15} fissions. There was no detectable contamination (see NOTE 1).

Yield: 60%.

Separation Time: About 4 hours exclusive of electrodeposition.

1. To a solution of the activity add ~4 ml HClO₄, 2 mg Te⁺⁴, 2 mg La, ~10 mg Zr, 20 mg Rh, ~10 mg Tl, and 5 mg Ru carrier. Take to fumes of HClO₄ and fume strongly for 20 or 30 seconds.
2. Cool, dilute to ~25 ml with water, and add ~3 drops H₃PO₄. Stir, centrifuge, and discard precipitate.
3. To the supernatant add ~2 ml 6% H₂SO₃ and 3 or 4 drops 55% HI. Stir and centrifuge, discarding precipitate. Pour supernatant into a 125-ml Erlenmeyer. Add 5 mg Ru.
4. To the supernatant add 20 ml conc. HCl, ~5 ml 55% HI, 1 mg Te⁺⁴. Boil for 20 minutes. Add 6 M HCl if volume becomes too small. Transfer to a 40-ml cone with 6 M HCl. Centrifuge and discard supernatant. Wash precipitate once with 6 M HCl.
5. Add 2 mg Te⁺⁴, 1 ml conc. H₂SO₄, 1 ml HNO₃, and ~0.5 ml conc. HClO₄. Take to fumes of SO₃. Cool to room temperature. Cautiously dilute to 15 ml volume with 6 M HCl. Stir and add ~1.5 ml H₃PO₂. Bring to a boil for 10 or 15 seconds to coagulate Te metal. Filter through a 9-cm No. 42 filter paper and wash filter with 6 M HCl. Collect filtrate in a 125-ml Erlenmeyer.
6. Add ~5 ml H₂O₂ (30%) and warm slightly. If reaction becomes too violent, quench with 6 M HCl (see NOTE 2). After reaction subsides, boil for at least 5 minutes to destroy H₂O₂.
7. Repeat Steps 4, 5, and 6 twice (use 10 ml conc. HCl instead of 20 ml).
8. After cooling solution from Step 6, transfer to a 40-ml cone. Make basic with 6 M NaOH. Add 3 ml of a saturated solution of Na₂S and heat in hot-water bath for ~15 minutes. Centrifuge and discard supernatant. Wash once with water. Dissolve in 3 ml conc. H₂SO₄, 1 ml conc. HNO₃, and 0.5 ml conc. HClO₄. Take to fumes of SO₃. Dilute with water and electroplate overnight at 80 mA (see NOTE 3).

NOTE 1: The separation from Ir is at least a factor of 10^3 . However, if large amounts of Ir activity are thought to be present, added separation may be accomplished as follows (Rh - Ir separation by G. M. Iddings): After fuming the activities in HClO₄, pass the solution through a Dowex 50X12 (100-200 mesh) column

that has been washed with 8 M HNO_3 and then with 6 M HClO_4 (column dimensions are ~5 mm i. d. $\times$ ~10 cm). Ir will remain on resin.

- NOTE 2: The reaction between H_3PO_2 and H_2O_2 will not become too violent if, while warming, the flask is removed from the heat as soon as bubbles start to form. At this point the reaction will become self-sustaining for several minutes. Thereafter, there is little danger of excessive effervescence.
- NOTE 3: Electroplating cells and electrodes must be clean and free from all organics (including soap) in order that a satisfactory "plate" be obtained.

PALLADIUM

H. G. Hicks

Purification: 10^8 atoms of Pd^{109} isolated from a 1-day-old solution containing 10^{12} fissions exhibited no trace of foreign radioactivity when followed through nine half-lives.

Yield: 40-50%.

Separation Time: 6 hours for four samples.

Remarks: If Au not present, start at Step 3.

1. Add 2 drops Au carrier, wash into separatory funnel with sufficient water to adjust sample to 1-2 M HCl. Extract 5 minutes with an equal volume ethyl acetate.
2. Filter lower aqueous layer through water-wet filter paper into 125-ml Erlenmeyer flask.
3. Evaporate by boiling on hot plate to 2.5 ml volume. Transfer with water to 40-ml centrifuge cone and dilute to 30 ml (solution should now be less than 0.5 M HCl; if sample is small and contains no Au, dilute directly with water).
4. Heat, add 1 drop Ag carrier, digest hot for 5 minutes, centrifuge, and discard precipitate.
5. Add 2 drops Zr carrier, 1 ml conc. H_3PO_4 , and digest in hot-water bath. Centrifuge and filter through No. 4 paper.
6. Add 1 drop Cd holdback carrier and sufficient HCl to adjust to 1 M HCl. Add 2 ml 4% KI and digest at room temperature for 10 minutes. Centrifuge and wash PdI_2 with 0.1 M HCl.
7. Dissolve PdI_2 in 2 ml conc. HCl and 1 drop conc. HNO_3 . Boil and destroy NO_3^- cautiously with HCOOH . Color of solution must be brownish, not red. Cool.
8. Pass solution through a Dowex 21K anion exchange column conditioned with at least 0.5 M HCl. Wash with 10 ml 0.1 M HCl, 1 ml water, 15 ml 4 M HNO_3 , and 1 ml water. Discard washes.
9. Elute Pd with 5 ml 3 M NH_4OH and collect in clean centrifuge cone.
10. Acidify with HCl, add 3 drops of scavenge solution* and make basic with NH_4OH . Centrifuge and discard precipitate.
11. To the supernate add 2 mg each of Fe^{+++} and La^{+++} carriers. Digest, centrifuge, and discard precipitate.
12. Acidify supernate with HAc, add 3 drops Zr and 1 ml H_3PO_4 . Digest hot and filter through No. 4 filter paper. Collect filtrate in clean cone.
13. To filtrate, add 5 ml saturated dimethylglyoxime solution (ethanol), wash Pd DMG twice with 5 ml alcohol in 25 ml water and twice with alcohol. Dry and weigh.

* Scavenge solution contains 10 mg each of Fe^{+3} , La^{+3} , Zr^{+4} and 2.5 mg Te^{+6} per ml in 6 M HCl.

PALLADIUM (ALTERNATE PROCEDURE)

G. M. Iddings

Purification: 2.5×10^7 atoms of Pd^{103} (270 γ c/m under the geometry employed) isolated from a 3-month-old mixture containing 10^{15} fissions had $< 1 \beta$ c/m through 7 mg Al cm^{-2} (~45% geometry).

Yield: ~70%.

Separation Time: ~3 hours.

1. To the solution in ~25 ml of 6 M HCl, add 20 mg Pd and ~1 mg Te carriers. Boil the solution for ~10 minutes to reduce Te to +4 state.
2. Transfer to a 40-ml centrifuge cone and dilute with water so that the solution is ~1 M in HCl. Add ~5 drops of 55% HI and ~2 ml of 6% H_2SO_3 . Centrifuge the PdI_2 and wash with a mixture containing ~5 ml 2 M HCl, a few drops of HI, and 1 ml of 6% H_2SO_3 .
3. Dissolve precipitate in 3 ml of conc. HCl and 1 ml 30% H_2O_2 . Boil to destroy excess H_2O_2 . Add ~15 ml of 6 M HCl. Cool and transfer the solution to a 60-ml cylindrical, open-top, separatory funnel containing ~25 ml of methyl isobutyl ketone. Stir vigorously for ~2 minutes with motor-driven glass stirrer.
4. Drain off the aqueous layer into a 40-ml centrifuge cone. Discard the organic layer. Rinse the separatory funnel with acetone.
5. Repeat the methyl isobutyl ketone extraction twice, adding ~5 mg of Te^{+4} before each extraction and then discarding the organic layer each time.
6. Add sufficient conc. HI (without preservative) to make the aqueous solution ~1 M in HI (4 ml 55% HI for 30 ml volume). Extract Pd iodide complex into methyl isobutyl ketone. Drain and discard the aqueous layer.
7. Wash the organic layer three times with ~20 ml of 1 M H_2SO_4 . Drain and discard the aqueous wash solution each time.
8. Stir the organic layer with a mixture containing ~10 ml of 6 M HCl and ~3 ml of saturated KBrO_3 . Repeat the back-extraction without the bromate and combine aqueous layers in a 125-ml Erlenmeyer flask. Add 1/2 mg of Te^{+4} and 3 mg of Fe^{+3} carriers.
9. Boil solution to a volume of ~10 ml and transfer to a 40-ml centrifuge cone. Add conc. NH_4OH until pH is ~8. Cool and centrifuge. Discard the precipitate. Make the supernatant 0.1 - 0.5 M in HCl (~25 ml total volume) by adding ~2 ml of conc. HCl. Let stand in cold-water bath for ~10 minutes. Centrifuge the $\text{Pd}(\text{NH}_3)_2\text{Cl}_2$ precipitate and wash with a mixture containing 10 ml of 50% saturated NH_4Cl and a few drops of conc. HCl. Add a few drops of methanol to lower surface tension.
10. Dissolve $\text{Pd}(\text{NH}_3)_2\text{Cl}_2$ in 1 ml conc. HNO_3 and 2 ml conc. HClO_4 . Heat to strong fumes of HClO_4 . Fume until the solution is clear.

11. Cool and add ~20 ml of 2 M HClO_4 . Add ~5 mg Ag carrier and 5 or 6 drops conc. HCl . Centrifuge and discard AgCl precipitate.
12. To the supernatant, add another 5 mg of Ag. Stir well with a Pt wire. Centrifuge and discard precipitate. Repeat the AgCl scavenge, filtering the final AgCl .
13. To the supernatant in a 40-ml centrifuge cone, add ~10 ml of formaldehyde, and let stand in a boiling water bath for ~15 minutes to reduce the Pd.
14. Centrifuge the Pd metal and wash with water. Filter onto a 1-in. diam tared filter paper and wash with methanol. Dry at 110°C for 10 minutes. Weigh and add a few drops of diluted zapon. Mount and count.

SILVER

H. B. Levy

Purification: 3×10^{12} atoms Ag^{111} isolated from a 4-day-old solution containing 10^{15} fissions showed no detectable foreign radionuclide when followed through 3 half-lives.

Yield: 75%.

Separation Time: About 6 hours for eight samples.

1. Add active solution to Ag carrier. Add 1 drop of Pd carrier. Should AgCl precipitate, add conc. NH_4OH until solution is alkaline. Then reacidify with conc. HNO_3 and a few drops conc. HCl . Centrifuge the AgCl and wash twice with 4 M HNO_3 .
2. Dissolve the AgCl in ~3 ml warm conc. NH_4OH and pass resultant solution through Dowex 21K (50-100 mesh) resin column which has been preconditioned with 3 M NH_4OH . Rinse centrifuge tube with ~1 ml 3 M NH_4OH and add rinse to top of column. Wash column with 2 ml 3 M NH_4OH (no more). Collect and combine effluents in clean cone.
3. Heat effluents in hot-water bath for at least 5 minutes and no more than 10 minutes. Cool and add 10 ml conc. HCl . If necessary, heat in hot-water bath to dissolve the AgCl.
4. Transfer to Dowex 21K (50-100 mesh) resin column preconditioned with conc. HCl . Wash cone with 1 ml conc. HCl and add wash to top of column. Wash column with 4 ml conc. HCl .
5. Collect eluates and heat in boiling hot-water bath for 30 minutes or longer.
6. Cool in an ice bath. Add 3 drops of a special scavenge.* Make basic with excess NH_4OH . Centrifuge and discard precipitate.
7. Pour supernate into cone containing 1 mg each of Fe^{+3} and La^{+3} . Stir, centrifuge and discard precipitate, pouring supernate thru No. 4 filter paper.
8. To filtrate add excess fresh ascorbic acid solution until precipitation of metallic silver is complete. Centrifuge and wash twice with water, once with acetone.
9. Dry for 10 minutes at 110°C , and weigh as Ag metal.

*The special scavenge contains 10 mg each of Fe (III), La (III), and Zr (IV), and 2.5 mg Te (VI) per ml in 6 M HCl .

SILVER (ALTERNATE PROCEDURE)

G. M. Iddings

Purification: A sample of Ag^{111} isolated from an 11-day-old mixture containing 5×10^{15} fissions exhibited no contaminating activity when gamma-pulse analyzed, or when decay was followed for 15 half-lives. The only gamma activity present of higher energy than 1100-keV was that associated with $\text{Ag}^{110\text{m}}$.

Yield: 80%.

Separation Time: ~2 hours (excluding the electrodeposition).

1. To a 25-ml solution of mixed activities in 4 M HNO_3 in a 40-ml glass centrifuge cone, add 20 mg of silver carrier and ~1 ml of 1 M HCl . Stir and centrifuge the AgCl precipitate. Discard the supernate.
2. Dissolve the AgCl with ~2 ml of conc. NH_4OH and add ~25 ml of water. While stirring vigorously, add ~2 mg of Fe^{+3} dropwise. Centrifuge and discard the $\text{Fe}(\text{OH})_3$ precipitate. Repeat the $\text{Fe}(\text{OH})_3$ scavenge.
3. To the supernate add ~2 ml conc. HNO_3 , ~1 ml of 1 M HCl , and a few drops of dilute aerosol. Centrifuge and discard the supernate. Proceed to Step 4 immediately.
4. Dissolve the AgCl in ~4 ml of conc. HCl by stirring vigorously, and pass the solution through a Dowex-1 anion-exchange column (100-200 mesh, 6 mm i. d. $\times$ 7 cm), collecting effluent in a clean cone. Wash the column with ~5 ml of 10 M HCl , collecting effluent in same cone as original effluent.
5. To the combined solutions add an equal volume of water and 3-5 drops (and no more) of 55% HI . Centrifuge the AgI and discard the supernate.
6. Dissolve the AgI in 2 ml of fresh 55% HI . Add ~15 ml of 6 M H_2SO_4 and transfer the solution to a 60-ml cylindrical separatory funnel. Add ~30 ml of hexone and equilibrate phases for ~2 minutes, stirring with a motor-driven glass paddle. Discard aqueous layer and wash the organic layer once with 15-20 ml of 1 M HI - 6 M H_2SO_4 (same as above solution) and three times with 15-20 ml of 6 M HCl .
7. Stir the organic phase for ~3 minutes with 20 ml of 8 M HCl and sufficient (~3 ml) saturated KClO_3 solution to decolorize the organic phase. Drain aqueous layer into clean cone.
8. To the 8 M HCl - KClO_3 solution, add ~3 drops of 55% HI and 5-10 ml of acetone. Stir thoroughly and centrifuge. Carefully decant and discard the supernate and wash the precipitate with water-methanol (1:1) mixture.
9. Dissolve the AgI in ~5 ml of 3 M NaOH and ~5 ml of 1 M NaCN . Transfer the solution to a plating cell with water and electrodeposit the Ag onto a tared platinum plate with a current of ~40 mA for 4 (or more) hours.
10. Rinse the plate with water and acetone. Ignite the plate only enough to melt the Ag . Weigh, mount, and count.

CADMIUM

H. G. Hicks

Purification: 10^{11} atoms of Cd^{115} and $\text{Cd}^{115\text{m}}$ isolated from 1-day-old solution of 10^{15} fissions showed no detectable foreign radionuclide when the decay is followed for 3 weeks.

Yield: 70%.

Separation Time: 6 hours for eight samples.

Remarks: If Au not present, start with Step 3.

1. To the mixed activities in HCl solution, add a known weight of cadmium carrier and 2 drops Au carrier. Wash solution into separatory funnel with sufficient water to adjust sample to 1-2 M HCl. Extract 5 minutes with an equal volume of ethyl acetate.
2. Drain lower (aqueous) layer through a water-wet filter paper and collect filtrate in 40-ml cone.
3. To filtrate add 1 drop each of Mo and Sb holdback carriers. Add NaOH solution* until alkaline. Centrifuge precipitate and wash once with 3 M NaOH solution.
4. To precipitate add 10 ml of 2 M HCl. Add 1 drop each of Sb and Pd carriers and saturate the solution with H_2S gas. Centrifuge and discard precipitate.
5. Decant supernate through No. 4 filter on to a Dowex 21K column (preconditioned with 2 M HCl). Wash column twice with 5 ml 2 M HCl and once with 10 ml of 0.1 M HCl.
6. Elute Cd with 20 ml of 1.5 M H_2SO_4 .
7. Add several drops of special scavenge** and an excess of NH_4OH . Centrifuge and discard precipitate.
8. Pour supernatant into cone containing 1 mg Fe^{+3} . Stir, centrifuge, and filter through No. 4 paper.
9. Make filtrate just acid with HAc and saturate the solution with H_2S gas. Centrifuge precipitate and wash twice with a solution of 1 drop HAc in 15 ml water. Wash twice with acetone and dry at 110°C . Weigh as CdS.

*The success of this step will depend upon the absence of ammonium salts in the original solution in Step 1.

**Scavenge solution contains 10 mg each of Fe^{+3} , La^{+3} , and Zr^{+4} and 2.5 mg Te^{+6} per ml in 6 M HCl.

INDIUM

N. A. Bonner and G. M. Iddings

Purification: When isolated from 10^{15} fissions an indium fraction showed only indium activities by radioactive decay and by gamma-ray pulse-height analysis.

Chemical Yield: 60-70%.

Separation Time: About 8 hours.

1. To the HCl solution of mixed radioactivities in a 40-ml glass centrifuge cone, add 20 mg of indium, ~10 mg of yttrium, ~10 mg of scandium, and ~2 mg of tellurium (IV). Add CO_2 -free NH_4OH until solution is just basic (~pH 8). Heat on hot-water bath for 10 minutes. Centrifuge and discard the supernate. Wash the precipitate twice with ~15 ml of water each time, and discard the washes.
2. Dissolve the precipitate in ~1-2 ml of 6 M HNO_3 . Add ~5 ml of water, and an equal volume of (Na-free) 6 M NH_4F , making the final solution ~3 M in NH_4F . Mix thoroughly. Check to be sure pH is 4-5. Heat in hot-water bath for ~5 minutes. Cool, centrifuge, and pour supernate into a new 50-ml lusteroid tube. Wash the precipitate with 10 ml of 3 M NH_4F , heating for 5 minutes in a hot-water bath. Cool, centrifuge, and combine wash with original supernate. Wash precipitate once with ~5 ml of water and combine with previous supernates. Discard YF_3 precipitate.
3. To the combined supernates, add 1 ml of saturated KBrO_3 solution and heat in a hot-water bath for ~5 minutes. Add 10 ml conc. HNO_3 (the pH should be ~0.3 or less) and heat for an additional 5 minutes. Cool, centrifuge, and pour supernate into clean cone. Wash the precipitate with ~5 ml of water, centrifuge, and combine wash with original supernate. Discard the ScF_3 precipitate.
4. Divide the solution into two roughly equal parts, adding 3 ml of 10 M NaOH and 4 ml of conc. NH_4OH to each part. Finally, adjust pH to 5-6 with HNO_3 or NH_4OH as required. Cool to room temperature and centrifuge Na_3InF_6 precipitates. Wash the two precipitates each with 5-10 ml of 1% NaF solution, using the wash solutions to slurry the two precipitates into a single tube. Centrifuge and discard supernate.
5. To the precipitate add 4 ml saturated H_3BO_3 + 4 ml H_2O . Stir vigorously and heat. Dilute to 25 ml with 0.5 M HCl . Precipitate should be completely dissolved at this point.
6. Add 10 mg Sb (III) carrier, 1 mg Te (IV) carrier and 2 drops of 47% HI . Saturate solution with H_2S . Add a few drops of dilute aerosol, heat on hot-water bath for ~3 minutes, and cool. Centrifuge (or filter through 11-cm diam Whatman No. 40 paper) and discard precipitate.
7. To supernate, add 2-3 ml of saturated NH_4Ac (the pH should be 3-5, preferably closer to 3). Saturate again with H_2S . Centrifuge and wash precipitate with dilute HAc-Ac buffer solution (pH 3-5).

8. Dissolve In_2S_3 in ~2 ml 48% HBr. Heat on hot-water bath for 5 minutes to expel H_2S . Add sufficient 48% HBr to make total volume 18 ml. Transfer with 7 ml water to 60-ml cylindrical separatory funnel containing 30 ml of isopropyl ether. Stir for 2 minutes, and allow layers to separate. Discard aqueous phase.
9. Stir the ether phase twice with 25-ml portions of 6 M HBr for 2 minutes each time, and twice with 25-ml portions of 1 M HI for 2 minutes. Discard all washes.
10. Stir ether phase for 2 minutes with 10 ml of 6 M HCl, and drain aqueous phase into clean tube. Dilute the aqueous phase with an equal volume of water, and filter the solution through 11-cm diam No. 40 Whatman paper to remove extraneous solid material.
11. Add 5 ml conc. NH_4OH and adjust to pH 8. Heat on boiling water bath for 10 minutes. Centrifuge and discard the supernate. Dissolve the precipitate in a few drops of conc. HNO_3 . Dilute to ~15 ml with water and adjust the pH to ~8 with NH_4OH .
12. Filter through 9-cm Whatman No. 42 paper (or equivalent). Wash precipitate and paper thoroughly with ~1% NH_4NO_3 (do not use NH_4Cl). Ignite at 800°C (avoid higher temperatures) for 1/2 hour in an oxidizing atmosphere in a muffle furnace. Transfer to tared Al counting receptacle, weigh, mount, and count.

TIN

W. E. Nervik

Purification: 2×10^{12} atoms Sn^{121} and Sn^{125} isolated from a 4-day-old solution containing 10^{15} fissions showed no detectable foreign radionuclide when followed through 3 half-lives.

Yield: 50%.

Separation Time: About six samples in 5 hours.

Remarks: Allow all Sn^{127} to decay before chemistry is started.

1. To the active solution add Sn carrier, several mg each of Sb, Mo, Te (VI), and several mg Te metal. Adjust to 6 M HCl. Add some solid NaClO_3 and digest hot for 10 minutes.
2. Extract 3 times with 10 ml methylisobutylketone ("hexone") each time. Wash combined hexone layers twice with 10 ml 6 M HCl.
3. Back-extract hexone layer 3 times with 10 ml F^- solution (30 ml 1 M HCl + 2 ml 6 M $\text{NH}_4\text{F} \cdot 3\text{H}_2\text{O}$). Wash F^- layer once with 20 ml hexone, and filter through water-wet filter paper.
4. Saturate cold filtrate with H_2S and centrifuge. Add several mg As carrier, saturate with H_2S and filter. Add 5 ml saturated H_3BO_3 solution to filtrate. Digest in hot-water bath and saturate with H_2S . Centrifuge, wash SnS_2 once with 1 M HCl saturated with H_2S .
5. Dissolve SnS_2 in 2 ml 6 M HCl with 2 ml 1 M NaBrO_3 . Add 1 mg Mo carrier, reduce excess BrO_3^- with addition of solid $\text{NH}_2\text{OH} \cdot \text{HCl}$ and dilute to ~30 ml with 0.5 M HCl. Add 5 ml of a saturated alcohol solution of alpha benzoinoxime. Digest, centrifuge and filter. Saturate filtrate with H_2S in hot-water bath. Centrifuge and discard supernate.
6. To SnS_2 add 2 ml saturated Na_2S solution and 15 ml water. Stir and pass solution through a Dowex 21K anion column prewashed with water. Wash column with 15 ml water. Elute Sn with 15 ml saturated Na_2S solution. Collect eluate in 125-ml Erlenmeyer flask. Acidify eluate carefully with 6 M HCl. Centrifuge and wash SnS_2 once with water.
7. Dissolve SnS_2 in $\text{BrO}_3^- + 6 \text{ M HCl}$ as in Step 5. Reduce excess BrO_3^- with $\text{NH}_2\text{OH} \cdot \text{HCl}$. Dilute to 25 ml and filter if necessary. Adjust solution to ~pH 6 with about 10 ml HAc - Ac^- buffer. Digest in hot-water bath. Centrifuge, wash once with dilute pH 6 buffer solution and once with acetone. Dry, ignite, and weigh as SnO_2 .

ANTIMONY

W. E. Nervik

Purification: 10^{12} atoms of Sb^{127} isolated from a 4-day-old fission mixture containing 10^{15} fissions showed no evidence of foreign radioactivity over three half-lives.

Yield: 50%.

Separation Time: ~5 hours for four samples.

1. To the active solution add 30 mg Sb carrier plus 1 mg Mo, Te, Sn carriers (solution at this point should be free of oxidizing agents; if it is not, Sb should be precipitated with pH 6-7 acetate buffer, washed once with dilute acetate buffer, and dissolved in 8 ml 6 M HCl).
2. Adjust to ~2 M HCl (25-30 ml volume), cool in an ice bath, and add 10 ml CrCl_2 solution ("oxsorbent"). Digest cold for 5 minutes and saturate with O_2 gas for 1 minute. Centrifuge the Sb^0 , precipitate, and wash twice with water (NOTE 1).
3. Dissolve Sb^0 in 4 ml conc. HCl + about 1/2 ml liquid Br_2 . Add 1 mg each of Sn, Mo carriers, and sufficient $\text{NH}_2\text{OH} \cdot \text{HCl}$ to reduce excess BrO_3^- . Dilute to 30 ml, add 1 ml 5 M NaI, and digest for 15 minutes in a hot-water bath.
4. Add 2 ml 6 M $\text{NH}_4\text{F} \cdot \text{HF}$, cool in an ice bath, and saturate with H_2S . Digest in a hot-water bath for 15 minutes (no longer), saturating with H_2S every 5 minutes. Centrifuge and wash precipitate once with a solution of dilute $\text{NH}_4\text{F} \cdot \text{HF}$ in 1 M HCl saturated with H_2S .
5. Dissolve Sb_2S_3 in 15 ml hot 6 M HCl. Add 1 mg Te carrier and saturate with H_2S . Centrifuge and pour supernate into clean cone.
6. To supernate add 1 mg As^{+3} and saturate with H_2S . Digest in hot-water bath and filter As_2S_3 , collecting filtrate in clean cone.
7. Pass filtrate through a Dowex 21K resin column (50-100 mesh, 6 mm $\times$ 10 cm) or through a resin column of Dowex 1X8 (50-100 mesh), pre-equilibrated with 6 M HCl. Wash column with 5 ml 6 M HCl, 15 ml 1 M HCl, and finally with 10 ml 9 M H_2SO_4 . Discard all washes.
8. Elute Sb with 2 ml water followed by 15 ml boiling 2 M NaOH, collecting eluates in clean cone.
9. Adjust combined eluates to 1 M in HCl and filter any insoluble matter through a Whatman No. 4 paper. Cool filtrate in an ice bath and add 10 ml CrCl_2 solution. Digest cold for 10 minutes and saturate with O_2 for 1 minute. Centrifuge and discard supernate. Wash precipitate twice with 0.5 M HCl, once with acetone, dry at 110°C , and weigh as Sb^0 .

NOTE 1: If difficulty is experienced in centrifugation of the Sb^0 , it should be filtered, washed on the filter paper and dissolved as in Step 3.

TELLURIUM

H. G. Hicks

Purification: 5×10^{13} atoms Te^{132} isolated from a 4-day-old solution containing 10^{15} fissions showed no detectable foreign radionuclide when followed through three half-lives.

Yield: 70%.

Separation Time: 8 hours for eight samples.

1. Make solution, containing a known weight (~ 10 mg) of tellurium carrier, basic with NaOH. Add 2 drops 5% NaOCl solution and digest in a hot-water bath for 10 minutes. Then make solution at least 2 M in HCl. If NO_3^- is present, add HCOOH and heat on hot-water bath until effervescence ceases to remove NO_3^- . Add 5 ml 10% SnCl_2 and digest for 5 minutes in a hot-water bath. Centrifuge and wash precipitate twice with 2 M HCl.
2. Dissolve precipitate in 2 ml conc. HCl + 2-3 drops HNO_3 , heat in a hot-water bath until reaction is complete. Add 1 ml conc. HCOOH and heat to destroy NO_3^- .
3. Pass solution through a Dowex 21K resin column (50-100 mesh) conditioned with 6 M HCl; wash cone with 2 ml 6 M HCl and add wash to column. Wash column alone with 2 ml of 6 M HCl, then 20 ml of 1 M H_3PO_4 in ethanol (1 ml H_3PO_4 + 44 ml 95% ethanol). Discard all washes.
4. Elute with 20 ml 1 N H_3PO_4 (in water), collecting eluate in clean cone.
5. Add 2 mg each of Fe^{+3} and La^{+3} to eluate. Stir, add 5 ml 6 M NaOH, and digest in hot-water bath for 5 minutes. Centrifuge and discard precipitate, pouring supernate into clean cone containing 1 mg each of Fe^{+3} and La^{+3} . Stir, centrifuge, and filter through No. 4 filter paper.
6. Adjust filtrate to 2 M in HCl, add 5 ml 6% H_2SO_3 solution and heat in hot-water bath until precipitate has completely coagulated. Wash twice with 2 M HCl, once w/acetone, dry, and weigh as Te^0 .

IODINE

K. V. Marsh

- Purification: 10^{10} atoms of I^{131} from a 16-day-old solution of 10^{12} fissions showed no evidence of contamination by longer-lived nuclides after 10 half-lives.
- Yield: 80-90%.
- Separation Time: The first eight steps can be performed on duplicate samples in about an hour.
- Special Reagents: 1 M $NaClO_3$ water solution, 1 M $FeCl_3$ in 1 M H_2SO_4 solution, 0.05 M $PdCl_2$ in 1 M HCl , toluene, n-butyl acetate ("BuAc").
- Remarks: Evidence for complete exchange between the active species and inactive carrier is that equal weights of I carrier added to equal aliquots of the same fission product solution produce equal specific activities of PdI_2 regardless of the chemical form of the carrier, iodide, iodate, or periodate.
1. Starting with a sample containing only inorganic materials and no strong reducing agents, add 10-20 mg of I carrier as iodide, iodate, or periodate, add 1 ml of 1 M $NaClO_3$ in water solution, and adjust the HCl concentration to 6-10 M (see NOTE 1).
 2. When the reaction is complete in about 2-3 minutes (NOTE 1), extract the ICl with a suitable volume of butyl acetate and discard the aqueous phase (see NOTE 2). Wash the BuAc with half its volume of 6 M H_2SO_4 . Discard the wash.
 3. Add 1 ml of 6% H_2SO_3 solution to the BuAc. Dilute the aqueous phase with water to approximately one-half the volume of the BuAc. Extract until the I^0 color completely disappears. Discard the organic phase.
 4. To the aqueous add 2 ml of 1 M $FeCl_3$ in 1 M H_2SO_4 solution. Add a volume of toluene about equal to the final aqueous volume, and sufficient conc. H_2SO_4 to bring H_2SO_4 concentration to about 4 M. Mix aqueous phase thoroughly and wait 2-3 minutes for the complete oxidation of I^- to I^0 . Extract I^0 into the toluene. Discard the aqueous phase.
 5. Wash the toluene with about one-half its volume of 4 M H_2SO_4 . Discard the aqueous.
 6. Add 1 ml of 6% H_2SO_3 to the toluene, dilute the aqueous phase with water to make its volume about one-half the volume of the toluene, and stir until all I^0 is reduced to I^- and extracted into the aqueous phase. Discard the toluene.
 7. Make the aqueous about 1 M in HCl and add 5 ml of 0.05 M $PdCl_2$ solution. Digest the precipitate in hot-water bath to coagulate the PdI_2 . Centrifuge and wash precipitate once with water and once with alcohol. Dry at $110^\circ C$ and weigh as PdI_2 . Mount in any convenient manner for counting.

- NOTE 1: With iodide carrier the solution will become darker as I^0 is produced, then lighter as the I^0 is oxidized to ICl . The final color is a yellowish green. With iodate or periodate carriers the solution will change from colorless to the green color of ICl directly.
- NOTE 2: The distribution coefficient of ICl between BuAc and 6 M HCl is about 600.
- NOTE 3: If gold carrier is present in the starting solution, modify the procedure as follows:
- (a). Instead of extracting the ICl as in Step 2, heat the solution in a hot-water bath to expel excess Cl_2 . Add three times the quantity of 6% H_2SO_3 required to reduce the Au^{+3} to Au^0 and the I^+ to I^- . For 20 mg of Au and 20 mg of I, the volume of 6% H_2SO_3 required is about 2 ml.
 - (b). Heat the solution in the water bath to coagulate the gold and expel the excess SO_2 . Centrifuge and filter the supernate into a clean vessel.
 - (c). Add 1 ml of 1 M $NaClO_3$ and adjust the HCl concentration to 6 M with concentrated HCl. Allow 2-3 minutes for oxidation of I^- to I^+ , then extract the solution with butyl acetate as described in Step 2. Proceed to Step 3.

CESIUM

J. B. Niday

Purification: 10^{10} atoms of Cs^{136} isolated from mixed fission products showed no detectable contamination from other radioelements by decay or by gamma-ray spectroscopy.

Yield: about 70%.

Separation Time: 2-1/2 days for 24 samples.

1. Adjust solution containing Cs carrier to 6 M in HCl (NOTE 1). Add 2 ml of 1/8 M silicotungstic ("HSiW") acid, and allow to stand for several hours (NOTE 2). Centrifuge the cesium silicotungstate ("CsSiW") precipitate and wash thoroughly with about 30 ml 6 N HCl at least twice. Discard supernates and place cone in hot-water bath.
2. Add 2-4 ml of 2 M LiOH to the cone while in hot-water bath. Stir quickly to dissolve, and rapidly add ~2 ml water. Continue to heat for a few minutes, adding a little water if necessary. Dilute to 10-15 ml.
3. Filter through No. 1 paper onto a 6-cm $\times$ 6-mm column of 100-200 mesh BioRex 40 (Li^+ form, NOTE 3) ion exchange resin. Wash filter paper with a small amount of 0.2 M LiOH. Wash column with 35-40 ml of 2 M LiOH. The eluate contains K^+ , which may be saved if desired. Test the eluate at intervals to ensure removal of K^+ without premature loss of Cs^+ (NOTE 4). Wash resin with 2-3 ml of water.
4. Elute Cs with 25-30 ml 6 N HCl, passing the eluate through a preconditioned Dowex 21K ion exchange resin column into a 125-ml Erlenmeyer flask.
5. Evaporate eluate to dryness and dissolve residue in hot water. Transfer solution to a 40-ml centrifuge cone and dilute to 25-30 ml. Add 2 drops Fe^{+++} , 1 drop La^{+++} , and ~5 drops Ba^{++} carriers (all in Cl^- form). Add one drop aerosol and 2 drops of Bromothymol Blue indicator. Make solution slightly basic with NH_4OH , digest, and centrifuge. Pour supernate into cone containing 2-3 drops of Fe^{+++} - La^{+++} . Digest, centrifuge, and discard precipitate, pouring supernate into an Erlenmeyer flask.
6. Add about 2 drops 10 M NaOH to the solution (NOTE 5) and boil until all traces of NH_4^+ are eliminated. Add more NaOH dropwise if necessary, testing the vapors with moist pH paper. Acidify solution slightly with HCl, add 2 drops Fe^{+++} and 1 drop La^{+++} (volume ~30 ml). Add a slight excess of NaOH and 3-4 drops Na_2CO_3 . Centrifuge after letting stand a few minutes. Pour supernate into cone containing 2-3 drops of Fe^{+++} - La^{+++} . Centrifuge.
7. Pour supernate through No. 1 filter paper onto 6-cm $\times$ 6-mm BioRex 40 column (NOTES 3 and 6). Wash column with ~10 ml 0.2 M LiOH and ~2-3 ml water.
8. Repeat Step 4.

9. Before commencing Step 9, refer to Comment C. Boil eluate to dryness, add 3-5 ml each of conc. HNO_3 and HClO_4 , and fume to a volume of about 1 ml. Cool in ice bath, add about 30 ml anhydrous ethyl acetate, and cool for 15 minutes with occasional stirring to break up any massive precipitate. Transfer to a clean, dry cone, centrifuge and wash at least twice with 30 ml anhydrous ethyl acetate to remove all traces of HClO_4 (wash down the walls and macerate precipitate with a glass rod).
10. Slurry the precipitate with a few drops of ethyl acetate and transfer to a suitable weighed counting plate. Dry at 110°C , cool, and weigh as CsClO_4 .

NOTE 1: The procedure outlined here is adapted to the handling of large numbers of samples which contain K and dissolved solids, especially where the use of an organic extractant may not be desired (see Cesium (Alternate Procedure), this report, by H. M. Perdue). For other types of samples there are more convenient alternatives:

- (a). For samples containing no K or HNO_3 but appreciable solids and/or undesirable levels of radioactivity, precipitate, wash, and dissolve CsSiW as in Steps 1 and 2. Separate the Cs in the manner of Step 3 except that the cation column (NOTE 3) need be only about 2.5 cm long and the only washing is with 10-15 ml 0.2 N LiOH and then with 2-3 ml water. Continue the regular chemistry, beginning with Step 4.
- (b). For samples containing very little K and other solids (Sr carrier is permissible), boil to dryness, dissolve in hot water (a few drops of 6 N HCl may be added if necessary), and carry out Steps 5 and 6 substituting LiOH reagents for NaOH . Add conc. HCl until the solution is 6 M in HCl . Precipitate, wash, and dissolve CsSiW as in Steps 1 and 2. Separate the Cs in the manner of Step 3 except that the cation column (NOTE 3) need be only about 2.5 cm long and the only washing is with 10-15 ml 0.2 N LiOH and then with 2-3 ml water. Continue with Steps 4, 9, and 10.
- (c). For samples containing some K but little else, follow alternative (b) except that the Cs is recovered on a 6-cm column as in Step 3. Continue Steps 3, 4, 9, and 10. In this alternative, the complete removal of NH_4^+ by boiling is less critical.
- (d). For samples initially dissolved in other acids such as H_2SO_4 or HClO_4 , adjust to ~6 N and carry out the CsSiW precipitation of Step 1. If HNO_3 is present and alternatives (b) or (c) are not practical, the Cs can be recovered by precipitation as the phosphotungstate from dilute acid and recovery of Cs on a 6-cm column after dissolution in NaOH .

- NOTE 2: The CsSiW precipitation is inhibited by HNO_3 and by large amounts of Na^+ ion. K, Rb, NH_4^+ are carried to varying extents. Precipitation is best from $>5 \text{ N}$ HCl and at room temperature.
- NOTE 3: All Bio-Rex-40 (Duolite C-3) columns may be made up to proper length in advance but should be converted to the Li^+ form only shortly before use. Wash with 2 or 3 times the resin volume of 2 M LiOH and then with 0.2 M LiOH solution. Stir and resettle the resin bed for reasonable flow rates. These solutions exceed the recommended pH range of the resin and result in deterioration with time.
- NOTE 4: Test for K and/or Cs in basic solutions by treating 2 drops of the eluate with one or two drops of basic tetraphenylboron reagent. This test is sensitive to $< 5 \mu\text{g/ml}$ K. To discriminate between Cs and K, acidify 2 drops of eluate with HCl ($< 6 \text{ M}$) and add a drop or two of 0.1 M phosphotungstic acid reagent (PW). In dilute HCl solutions (preferably $\sim 1\text{-}3 \text{ M}$), the PW test is sensitive to $\sim 2\text{-}5 \mu\text{g/ml}$ of Cs but reacts to K only in high concentrations.
- NOTE 5: Use NaOH and Na_2CO_3 reagents selected for low K content.
- NOTE 6: A column length of $\sim 6 \text{ cm}$ is required here because of the presence of Na^+ ion. One of the prime purposes of this step is removal of silica introduced in the scavenges. Care is necessary to avoid introduction of silica or other foreign matter after this step. (The LiOH should be silica-free, and all reagents should be examined for traces of foreign matter.)

COMMENTS

- A. This procedure has been found to give very pure samples with yields $>50\%$. No detectable impurities are found in samples of $\text{Cs}^{136, 137}$ separated from the products of thermal neutron fission of U and Pu. In case radically different distribution of activities are present, such as from Cf^{252} fission, it may be desirable to add a sulfide scavenging step (e. g., Ag_2S precipitation from 0.2 N HCl solution).
- B. Carrier quantities of K and/or Rb may be separated from Cs (prior to the use of any SiW step, of course) by chromatographic elution from somewhat wider columns with weaker eluting agents. It is possible to separate K from Rb quantitatively in a few hours on a column containing 10 ml of basic, Li-form, Bio-Rex-40 resin by starting with 0.4 N LiOH as eluant.
- C. The final precipitation and mounting of Cs as CsClO_4 (Steps 9 and 10) gives clean and highly reproducible counting samples but is somewhat tedious for large numbers of samples. The alternative chloroplatinate precipitation described below has been tested and gives apparently pure samples.

Collect the eluate from Step 8 in a 40-ml cone. Heat, add 2-3 ml of 10% H_2PtCl_6 , digest 30 minutes while cooling, centrifuge, wash twice with EtOH and once with acetone. Dry at 110°C for 10 minutes, cool, and weigh.

CESIUM (ALTERNATE PROCEDURE)

H. M. Perdue

- Purification: $\sim 10^{12}$ atoms of cesium isolated from a 1-day-old solution of 10^{15} fissions showed no detectable radioactivities other than Cs^{136} or Cs^{137} when decay was followed for 6 months.
- Yield: $\sim 75\%$.
- Separation Time: 8 hours for 16 samples.
- Special Reagents: 1/8 M silicotungstic acid in ~ 5 M HCl, 6 M NaOH, 1 M NaOH, 6 M HCl, scavenge solution containing 10 mg/ml each of FeCl_3 , LaCl_3 , and BaCl_2 , saturated Na_2CO_3 solution, 0.5 M solution (125 g/l) of 4-sec. butyl-2 (α methylbenzyl) phenol (BAMBP) in cyclohexane. 10% H_2PtCl_6 in 4 M HCl, Dowex 21K anion resin, 50-100 mesh.
- Remarks: All solutions must be NH_4^+ -free. If NH_4^+ is present, boil with excess NaOH. For starting solutions which are basic, or which contain NO_3^- , make ~ 1 N in NaOH and treat as in Step 4 first. Then proceed to Step 1.
1. Pipet 20 mg standardized carrier into sample, adjust to 6 M HCl, and mix well. Add 4 ml silicotungstic acid reagent (1/8 M in 5 M HCl). Let stand for at least 1 hour. Centrifuge in 40-ml cone, and wash precipitate twice with 6 M HCl.
 2. Add 5 drops scavenge solution (Fe^{+3} , La^{+3} , and Ba^{+2}), several drops of 6 N HCl and stir. Add 7 ml 6 M NaOH and stir well to dissolve. Dilute to 15 ml with water.
 3. Add 5 ml Na_2CO_3 , stir well, digest in a hot-water bath and centrifuge. Pour supernatant into 40-ml cone containing 3 drops scavenge solution. Digest hot, centrifuge, and filter through No. 42 paper (pre-wet with 1 M NaOH solution). Discard precipitates.
 4. Add 1 ml saturated tartaric acid, stir, and chill in ice bath. Transfer chilled solution to a 125-ml separatory funnel and add 20 ml 0.5 M BAMBP in cyclohexane. Equilibrate layers for 2 minutes and discard aqueous. Wash organic 3 times with 20 ml 1 M NaOH for 1 minute each. For last wash, rinse walls of funnel with small amount of water into NaOH wash. Drain well to remove wash solution.
 5. Back-extract cesium by stirring 2 minutes with 10 ml 6 M HCl. Drain aqueous layer through a water-wet filter paper. Wash organic again with 10 ml 6 M HCl and add to first aqueous through filter paper.
 6. To combined aqueous layers add ~ 1 ml conc. HCl, and pass the solution through a Dowex 21K anion resin column (6 mm i. d. $\times$ 9 cm) preconditioned with 6 M HCl. Collect effluent in clean 40-ml cone.
 7. Add 2-1/2 ml 10% H_2PtCl_6 solution and stir. Digest at room temperature 30 minutes. Centrifuge and wash once with 20 ml 6 M HCl. Wash twice with ethyl alcohol and once with acetone.

8. Dry 10 minutes at 110°C, and weigh as Cs_2PtCl_6 .

NOTE 1: For volumes above 25 ml, reduce by boiling, and use 6 ml reagent.

BARIUM

H. M. Perdue and H. G. Hicks

Purification: $\sim 10^{13}$ atoms of Barium isolated from a day-old solution of 10^{15} fissions showed no detectable foreign radionuclide when followed through three half-lives.

Yield: 60%.

Separation Time: 6 hours for eight samples.

Remarks: If Au is absent, adjust to 4 M in HCl and begin with Step 4.

1. To sample containing ~ 20 mg standardized Ba carrier add ~ 1 ml H_2SO_4 , mix well, and digest until BaSO_4 settles out. Centrifuge and wash precipitate once with water. Discard supernate and wash.
2. Add ~ 2 ml 50% NaOH solution, 10 ml saturated solution K_2CO_3 and stir well. Digest hot for one hour with occasional stirring. Centrifuge and wash precipitate once with water. Discard supernate and wash.
3. Dissolve BaCO_3 precipitate in ~ 2 ml 6 M HCl (if there is a residual precipitate of BaSO_4 , re-digest this separately as in Step 2 and combine final HCl solutions).
4. Add 5 ml 4 M HCl, and transfer solution to Dowex 21K column (which has been preconditioned with 4 M HCl), collecting eluate in clean cone. Wash column with 5 ml 4 M HCl. Combine wash with eluate.
5. Cautiously neutralize combined eluates with 50% NaOH and add 2 ml saturated K_2CO_3 solution. Digest hot, centrifuge, and discard supernate. Wash precipitate once with water. Centrifuge and discard wash.
6. Dissolve precipitate in minimum 2 M HCl, add 30 ml of a mixture of 20% diethyl ether and 80% conc. HCl, and digest in ice bath for 10 minutes. Centrifuge and discard supernate.
7. Dissolve BaCl_2 in 2-3 ml water and repeat Step 6 twice.
8. Dissolve in 15-20 ml water and add 2-3 drops scavenge solution.* Make solution basic with NH_3 gas. Centrifuge and discard precipitate.
9. To supernate add 3 drops each of Fe^{+3} and La^{+3} carriers. Stir, filter, and discard precipitate.
10. Adjust filtrate to pH 5 with conc. HAc. Add 1-2 ml 1.5 M Na_2CrO_4 solution and digest hot for 10 minutes. Centrifuge and discard supernate.
11. Wash precipitate twice with water, once with acetone, and dry at 110°C for 10 minutes.

* Scavenge solution contains 10 mg each of Fe^{+3} , La^{+3} , Zr^{+4} and 2.5 mg Te^{+6} per ml in 6 M HCl solution.

CERIUM

H. G. Hicks and W. E. Nervik

Purification: 6×10^{11} atoms of Ce^{141} isolated from a 21-day-old solution containing 10^{13} fissions exhibited no trace of foreign radioactivity when followed through two half-lives.

Yield: 60%.

Separation Time: 8 hours for four samples

Remarks: Better yields are likely if the initial fluoride precipitate is allowed to digest 2-3 hours and is then set aside overnight.

1. To sample in "Auto-Clear" plastic tube containing Ce carrier, add 5 ml 1.5 M Na_2CrO_4 and 1 drop Zr holdback. Stir, add 5 ml conc. HF, and dilute to 35 ml with water. Digest hot for 30 minutes (see Remarks above), centrifuge, and discard supernatant. Wash precipitate once with solution of 0.5 M NH_4NO_3 in 0.1 M HNO_3 (+ drop HF). Centrifuge and discard wash.
2. To precipitate add 3 ml conc. HNO_3 , 3 ml saturated H_3BO_3 and stir (heating in hot-water bath may be required to dissolve precipitate). Add 6 ml water (solution is now 4 M in HNO_3), 4 drops 1 M KNO_2 , and digest in hot-water bath for 10 minutes (solution should now be colorless).
3. Transfer solution to separatory funnel with 10 ml 4 M HNO_3 . Add 20 ml 0.1 M HDEHP* in CCl_4 , and stir phases for 5 minutes. Discard organic phase (lower), and repeat extraction with 20 ml 0.1 M HDEHP in CCl_4 . Discard organic layer and add 20 ml CCl_4 . Stir 30 seconds and discard CCl_4 .
4. Add 20 ml 0.1 M HDEHP in heptane. Add 4 ml 1 M NaBrO_3 and 2 ml conc. HNO_3 . Stir 5 minutes and discard aqueous (lower) phase.
5. Wash organic phase 3 times with 10 ml 4 M HNO_3 for 1 minute each, and once with 10 ml 0.5 M HNO_3 , discarding all washes (lower phases).
6. Add 10 ml 0.5 M HNO_3 , 2 drops 30% H_2O_2 and stir 5 minutes. Both phases should be colorless. Withdraw aqueous layer, filter through water-wet No. 4 filter paper, and save. Wash organic layer with 10 ml 0.5 M HNO_3 for 30 seconds and filter into first aqueous extract.
7. Pass combined aqueous phases through a Dowex 50WX8 cation-exchange column (6 mm i. d. $\times$ 4 cm, 50-100 mesh). Wash column twice with 10 ml water. Discard all column effluents.
8. Elute Ce from column with 30 ml pH 7, 1 M ammonium lactate solution.
9. To eluate add 3 ml saturated oxalic acid solution and digest in hot-water bath for 10 minutes. Centrifuge and discard supernate. Wash precipitate once with water, once with acetone and dry at 110°C . Ignite and weigh as CeO_2 .

*Di-2-ethylhexyl phosphoric acid.

RARE EARTHS

W. E. Nervik and M. M. Zeligman

Yields: 60-70%.

Separation Time: 36 hours for 16 samples, including time for cation column separations.

1. To suitable aliquot of sample add ~5 mg of each rare earth of interest, 3 drops Zr carrier (~10 mg Zr/ml), and 2 ml 1.5 M $\text{CrO}_4^{=}$ solution. Adjust volume to 30 ml with HNO_3 such that final solution is 1-3 M in H^+ . Add 5 drops H_3PO_4 with stirring and maintain at room temperature for 2-3 minutes. Centrifuge and pour supernate into glass cone.
2. To supernate add 5-10 ml conc. HF. Stir, centrifuge, and discard supernate. Wash precipitate once with a solution made by mixing 5-10 drops conc. HF with 30 ml water. (Under no circumstances is the HF solution to be heated or allowed to stand more than 2-3 minutes.)
3. To precipitate add 3 ml saturated H_3BO_3 and 3 ml conc. HNO_3 . Stir well and digest in a hot-water bath for 2-3 minutes. Add 10 drops Ba carrier (~10 mg Ba/ml), 5 drops conc. H_2SO_4 , and dilute to 15 ml with water. Add another drop of Ba carrier and stir well. Wash down side walls of cone with water and dilute to 20 ml. Centrifuge.
4. Pour supernate into 15 ml conc. NH_4OH containing one drop of phenolphthalein. Centrifuge and discard supernate. Wash precipitate once with 30 ml water. Centrifuge and discard wash.
5. Dissolve precipitate in 5 ml conc. HCl and pass solution through a Dowex 21K anion exchange column (6 mm i.d. $\times$ 10 cm), collecting eluate in a clean cone. Wash cone with 2 ml conc. HCl and add wash to column. When HCl has run through, wash column with 2 ml conc. HCl (rare earths remain in the eluate).
6. Dilute combined eluates to 20 ml, add 5 drops Ba carrier, and make basic with conc. NH_4OH . Centrifuge and discard supernate. Wash precipitate once with 30 ml water.
7. To precipitate add 5-10 drops conc. HNO_3 , and digest in a hot-water bath until precipitate is dissolved. Add 1 ml of "settled" Dowex-50X8 resin (100-200 mesh). Add ~3-5 ml water. Stir for 1 minute.
8. Prepare ion-exchange column as described in UCRL-7729. * Load rare earth, resin, and water mixture on the top of the equilibrated column and close the system.
9. Allow 0.1 M α -hydroxyisobutyric acid to drip through column at a rate of 1 drop/second until yttrium is eluated (~5 hours). Collect all eluates in 10-minute fractions in an automatic fraction collector.
10. Start 0.4 M α -hydroxyisobutyric acid dripping into the 0.1 M acid eluating agent at a rate equal to the rate going through the column (i. e., 1 drop/7 sec). The separation is completed when the Nd elutes in ~20 hours.

11. Check for completeness of elutions by adding saturated oxalic acid solution to each tube in the collection. The rare earths will precipitate in the normal order, Y, Tb, Gd, Eu, Sm, Nd.
12. Combine all tubes of the same element in a 40-ml cone. Centrifuge and discard supernates.
13. Wash the precipitates once with water and once with acetone. Allow to stand in air until precipitates appear dry. Heat at 100°C for 10 minutes and ignite over Meker burner for 20 minutes.
14. Weigh as the rare-earth oxide, M_2O_3 , and count as appropriate.

* M. M. Zeligman, "Ion Exchange Separation of Fission Product Rare Earths With α -hydroxyisobutyric Acid," Lawrence Radiation Laboratory (Livermore) Rept. UCRL-7749, April 1964.

EUROPIUM

G. M. Iddings

Purification: See NOTES.
Yield: 20-30%.
Separation Time: About 8 hours.

1. To ~20 ml of mixed activities in a 40-ml glass centrifuge cone, add 20 mg of Eu^{+3} carrier. Add excess NH_4OH , centrifuge, and discard the supernate. Wash the precipitate once with water. Dissolve in ~20 ml of pH ~1 buffer solution which is 0.5 M in Na_2SO_4 and 0.5 M in NaHSO_4 . There should be no mass quantities of the cerium earths present (La, Ce, Pr, Nd or Sm).
2. Add ~20 mg of Ba (2 ml of solution as the chloride) dropwise while stirring slowly. Centrifuge and pour supernate into a cone containing ~10 mg of Ba. Add ~10 mg Ba dropwise while stirring slowly, then centrifuge. Pour supernate into clean cone.
3. Repeat Step 2, then to the supernate add 1 mg of Y (in HCl).
4. Add 5 ml of toluene and then ~10 ml of CrCl_2 solution (oxsorbent). Heat in a hot-water bath (~97°C) for 10 minutes until precipitation of EuSO_4 is complete. Cool in cold-water bath for ~5 minutes and add a few drops of 5% aerosol solution. Centrifuge and discard supernate.
5. Add ~1 mg of Y, then redissolve the EuSO_4 in 1 ml of conc. HNO_3 and 1 ml of Conc. HClO_4 . Do not use conc. H_2SO_4 . Heat to HClO_4 fumes and to near-dryness. Cool and add ~20 ml of pH ~2 sulfate buffer solution.
6. Repeat twice Steps 4 and 5 without the addition of Y carrier. Use 25 ml of 6 M HNO_3 instead of 20 ml of sulfate buffer solution at the conclusion of the repeat of Step 5.
7. Add ~2 mg Ce^{+3} carrier, ~3 ml of 5 M HIO_3 , and ~1 ml of saturated NaBrO_3 solution. Stir and heat in a hot-water bath for ~5 minutes. Cool to room temperature, centrifuge, and discard the precipitate. Pour the supernate into a 50-ml lusteroid tube.
8. Add 12-15 ml of 6 M NH_4F . Heat on hot-water bath for 5 minutes. Cool to room temperature in a cold-water bath for 5 minutes. Centrifuge and discard the supernate. Wash the EuF_3 with ~5 ml of water and discard the wash solution.
9. Dissolve the EuF_3 with 2 ml of saturated H_3BO_3 . Mix thoroughly and add 3 ml of conc. HCl. Heat on hot-water bath for ~3 minutes. Dilute to ~30 ml volume with water and add an excess of conc. NH_4OH . Cool, centrifuge, and discard the supernate. Wash precipitate with ~20 ml of water. Centrifuge and discard the wash solution.
10. Dissolve the precipitate in ~20 ml of 0.1 M HCl. Add ~5 ml of saturated oxalic acid and stir vigorously. Let stand for 5 minutes, then centrifuge and discard the supernate. Wash the europium oxalate with a few ml of 0.2 M oxalic acid and

filter through 7-cm No. 42 Whatman paper. Ignite at $\sim 900^{\circ}\text{C}$ for ~ 20 minutes in a Coors No. 00 porcelain crucible. Weigh, as Eu_2O_3 , mount, and count as appropriate.

NOTE 1: Steps 2 and 3 separate La^{140} tracer from mass Eu by a factor of $\sim 70,000$ if there are no mass amounts of cerium earths present.

NOTE 2: Steps 4, 5, and 6 separate Dy from mass Eu by a factor of $> 10,000$ and separate Y from mass Eu by a factor of $\sim 2 \times 10^5$.

HAFNIUM

R. J. Nagle

Purification: A hafnium fraction isolated from 5×10^{15} fissions showed no detectable gamma-ray impurities upon pulse-height analysis on a NaI detector. The principal component was 10^{10} atoms of Hf^{181} and a lesser amount of Hf^{175} . The limit of contamination due to Zr^{95} was set at $\leq 5 \times 10^5$ atoms.

Yield: About 50%.

Separation Time: For four samples about 5 hours — excluding column separation; about 5-7 hours per column.

1. To 10-20 ml of sample (in 6 N HCl) contained in a polyethylene or lusteroid cone add 30 mg of standardized hafnium carrier (as the nitrate) and 10 drops La^{+3} (~10 mg La/ml). Add 1/2 ml 5% $\text{NH}_2\text{OH}\cdot\text{HCl}$ and heat in hot-water bath for several minutes. Add 1 ml conc. Hf^+ and digest in hot-water bath for several minutes. Centrifuge and discard precipitate. To supernate add another 10 drops of La^{+3} . Centrifuge and discard precipitate.
2. To supernate add ~75 mg of Ba^{+2} (as saturated BaCl_2 solution) and let stand for 10 minutes. Centrifuge and discard supernate. Wash precipitate once with 10 ml of water.
3. To BaHfF_6 precipitate add 2 ml 5%- H_3BO_3 , 2 ml conc. HCl, and 2 ml water. Heat in hot-water bath until precipitate is dissolved. Add 10 ml water and 10 drops conc. H_2SO_4 . Let stand for 15 minutes. Centrifuge and discard BaSO_4 . Pour supernate into clean polyethylene cone.
4. To supernate add conc. NH_4OH until solution is basic to phenolphthalein. Digest in a hot-water bath for 10 minutes, and centrifuge and discard supernate. Wash precipitate once with dilute NH_4OH .
5. Dissolve hafnium hydroxide in 4-6 ml of 2 N HNO_3 and transfer to 40-ml glass cone. Heat in boiling hot-water bath for 20 minutes. Add 1-2 ml of 200-400 mesh Dowex-50 resin which has been briefly washed with 2 N HNO_3 . Continue heating in HWB for additional 30 minutes, stirring occasionally with a Pt wire stirrer. Centrifuge and transfer supernate to clean glass cone. Check supernate for completeness of Hf uptake either by comparison of activity to that of resin, or by precipitation of residual Hf with NH_4OH (in case resin uptake was incomplete, Steps 4 and 5 must be repeated, using a fresh portion of resin). Wash resin once with 10-15 ml water, and slurry onto top of a column of Dowex-50 (200-400 mesh) cation resin, 6 mm i. d. $\times$ 10 cm.
6. Pass a total of 40 ml of 0.97 N H_2SO_4 through the column, collecting effluents in 10-15-ml portions. Check effluents for absence of Hf by addition of NH_4OH . No precipitate should form, although small amounts of zirconium present in the original hafnium carrier will precipitate at this point.

7. To column add a total of 20 ml 3 N H_2SO_4 (in two portions), collecting eluates in one clean 40-ml cone.
8. Make eluate basic with conc. NH_4OH and digest for 10 minutes in a hot-water bath. Centrifuge and discard supernate. Wash precipitate once with dilute NH_4OH .
9. Repeat Steps 5 through 8 twice.
10. Dissolve the hafnium hydroxide in 4 ml of 3 N HCl . Heat in hot-water bath for 10 minutes, cool, and add 4 ml of 0.4 M TTA* in benzene. Equilibrate layers with a Pt wire stirrer for 25 minutes. Centrifuge to separate layers and transfer the organic layer with transfer pipet to a clean glass cone. Repeat TTA extraction with another portion of TTA if necessary.
11. Wash organic layer for 5 minutes with 3 ml of 9 N HCl . Centrifuge and transfer organic layer to a polyethylene cone. Stir with 4 ml of 6 N HCl - 0.1 N HF for 5 minutes and transfer aqueous layer to clean 40-ml cone.
12. Make solution basic with conc. NH_4OH . Wash precipitate twice with dilute NH_4OH , and twice with anhydrous acetone. Allow to dry at room temperature. When dry, pulverize and transfer to weighed platinum hat; ignite carefully, cool, and weigh as HfO_2 .

*Thenoyltrifluoroacetone.

TANTALUM

M. Lindner and K. V. Marsh

Purification: 10^{10} atoms of Ta^{182} isolated from a 3-day-old solution containing 10^{15} fissions showed no evidence of contamination by foreign radionuclides when decay was followed through one half-life.

Yield: 30-60%.

Separation Time: 2 hours for duplicate samples.

1. To the sample solution, which should be at least 1 N in either HCl or HNO_3 , add 20 mg standardized Ta carrier and 5 ml conc. HClO_4 . Fume to near-dryness. Cool, add 20 ml conc. HNO_3 , and boil until about 10 ml remain. Cool, centrifuge precipitate and wash once with conc. HNO_3 , transferring slurry to a 12-ml polyethylene centrifuge cone. Centrifuge and discard wash.
2. Dissolve the precipitate in 1 ml of 12 N H_2SO_4 - 0.4 N HF . Ignore any small quantities of undissolved foreign material.
3. Add 3 ml of pre-equilibrated diisopropylketone and equilibrate layers for about one-half minute with a platinum stirring wire. Centrifuge and discard aqueous phase.
4. Wash the ketone fraction with 1 ml of 12 N H_2SO_4 - 0.4 N HF , transferring the organic layer to a clean polyethylene cone.
5. To the ketone layer add 8 ml water and equilibrate for about one minute with a Pt stirring wire. Centrifuge layers. It may be necessary to centrifuge for 5-10 minutes at this point because the organic layer often exhibits emulsification. Transfer the aqueous layer to a clean polyethylene cone.
6. To the water layer add one drop of phenolphthalein and sufficient conc. NH_4OH to make the solution just alkaline. Centrifuge the precipitate, and wash once with either 5 ml conc. HNO_3 or with a mixture made by adding a few drops of saturated NH_4NO_3 followed by 5 ml dilute NH_4OH (in that order). Discard supernate and wash.
7. Repeat Steps 2 through 6 twice.
8. Wash the final precipitate twice with 5 ml of acetone, the second time transferring the slurry to a 12-ml glass centrifuge cone. Discard the wash and drain acetone thoroughly for 5 minutes, preferably until the precipitate appears to crack. With vigorous stirring motion of a Pt wire in the precipitate, place the cone in a boiling water bath and continue stirring until the precipitate appears powder-dry and the last traces of acetone have evaporated from the cone.
9. Cautiously shake the powder out of the cone into a weighted platinum hat. Ignite over a Meker burner. Cool and weigh as Ta_2O_5 .

RHENIUM

M. Lindner

Purification: A rhenium fraction isolated from 10^{15} fissions by this procedure showed no detectable contamination upon following radioactive decay. The principal components were approximately 10^{10} atoms each of Re^{188} and Re^{186} .

Yield: About 30-50%.

Separation Time: About 6 hours.

1. The solution containing the mixed activities should be at least 1 N in HCl. Nitrates must be absent. Add about 20 mg of standardized Re carrier. Heat on hot plate and saturate with H_2S for 10 minutes – or more, if necessary, to produce a sulfide precipitate. (The character of the Re_2S_7 is variable; it may be black and settle rapidly, or brown, finally divided and settle slowly; the former is preferable but not always attainable.) If total volume is very large, allow precipitate to settle for at least one hour, preferably longer. If supernatant liquid is reasonably clear, decant and discard as much as possible, centrifuging the remainder. Wash precipitate with 1-2 N HCl.
2. To Re_2S_7 add ~1 ml conc. NH_4OH and several drops 30% H_2O_2 . Place in hot-water bath and digest until the resultant mixture is colorless. Add more H_2O_2 as necessary. Dilute to ~20 ml, centrifuge, and discard any foreign material.
3. To supernate add conc. HCl until solution is acidic. Add 5 drops Fe^{+3} (~10 mg Fe/ml), and sufficient conc. NH_4OH to render the solution basic. Digest in hot-water bath for 10 minutes, centrifuge, and discard precipitate. Pour supernate into 125-ml Erlenmeyer flask.
4. Acidify solution with conc. HCl and add several drops of tetraphenylarsonium chloride solution (30 mg/ml). Swirl for short time and add 2 ml more of tetraphenylarsonium chloride. Allow to stand for ~15 minutes, swirling occasionally (cooling in an ice-bath may be helpful in enhancing yield). Add another 5 drops reagent, pour whole into 40-ml cone, and centrifuge. Discard supernate and wash precipitate with water, discarding wash.
5. Add 15 ml conc. H_2SO_4 to precipitate (orange solution should result). Transfer solution to a conventional rhenium distillation apparatus equipped with a water-cooled condenser. Use a 40-ml cone containing ~10 ml water as a "catcher." Heat until about half the H_2SO_4 has been distilled (it may be advisable to empty contents of catcher once during the distillation).
6. Pour distillate into 125-ml Erlenmeyer flask, add 10 ml 6 N HCl, and warm on hot plate. Saturate solution with H_2S for 10 minutes. Transfer slurry to 40-ml cone. Centrifuge and discard supernate. Wash precipitate once with 15 ml 1 N HCl (if Re_2S_7 should form a colloid, it may be necessary to warm the wash and saturate with H_2S). Discard wash.

7. To precipitate add 1 ml conc. NH_4OH and several drops H_2O_2 , heating in water bath until precipitate has dissolved. Heat until H_2O_2 is decomposed, and make solution acidic. Add 3 drops Fe^{+3} carrier and conc. ammonia until basic. Digest in hot-water bath for 10 minutes. Centrifuge and discard precipitate. Pour supernate into 125-ml Erlenmeyer flask.
8. Make solution slightly acidic with HCl (this is optional, as tetraphenylarsonium perrhenate is also insoluble in dilute ammonia), and add several drops of tetraphenylarsonium chloride reagent. Swirl, add another 2 ml of reagent and allow to stand for 10-15 minutes (see remarks in Step 4 above).
9. Filter precipitate on weighed 1-in. filter paper, wash with water, and dry in air oven at 110°C for 10 minutes. Let filter paper stand in air for ~30 minutes before weighing gross. Weigh as $(\text{C}_6\text{H}_5)_4\text{AsReO}_4$.

IRIDIUM

K. V. Marsh

Purification: From 10^{15} fissions an iridium fraction was obtained which contained $< 5 \times 10^8$ atoms of Ir^{194} or of Ir^{190} .

Yield: 25-50%.

Separation Time: About 15 hours for six samples.

1. To the radioactive solution in a 125-ml Erlenmeyer flask, add a specified weight (usually 10-20 mg) of standardized iridium carrier solution, 5 ml conc. HClO_4 , and fume to near-dryness (see NOTE 1). Solution or residue must be a reddish violet color before proceeding.
2. Take up moist residue in ~10 ml water (more if necessary) and add 10 N NaOH either to pH 5-7 or until the color of the solution changes noticeably from purple (color in HClO_4) to blue (xs NaOH). Then add conc. HClO_4 dropwise until solution turns just purple again. Add 1 N NaOH until precipitate forms. If solution turns blue again, add 1 N HClO_4 dropwise. Continue adjustment of (H^+) until supernatant liquid is colorless (either by centrifugation or settling). Centrifuge and discard supernate. If solution contained a high concentration of salts, wash precipitate once with water (see NOTE 2).
3. To precipitate add 5 ml conc. HCl and 10 drops conc. HNO_3 . Heat for a few minutes in a hot-water bath until precipitate has dissolved, then either boil gently (to avoid bumping) for 10 minutes in the cone over a Bunsen flame or transfer to an Erlenmeyer flask and heat for 10 minutes on a hot plate. Then boil solution to a volume of 2-3 ml. Solution at this point must have a reddish-brown color (NOTE 3). Dilute to 25 ml with water.
4. Pass solution through a Dowex 1X8 resin column 6 mm diam $\times$ 7 cm (50-100 mesh), previously washed with 0.5 N HCl (see NOTE 4). Wash column with 30 ml 0.5 N HCl , 15 ml 6 N HCl saturated with Cl_2 , (see NOTE 5) and finally with 30 ml 6 N HCl .
5. To column add 30 ml 6 N HCl - 1% HI solution (see NOTE 6). Collect eluate in clean 125-ml Erlenmeyer flask (NOTE 7).
6. Add 1-2 ml 30% H_2O_2 (NOTE 8) a few drops of conc. HNO_3 , and boil solution to near-dryness. Cool, add 10 ml water and 5 drops each of Fe and La carriers (~10 mg/ml).
7. Cautiously add 6 ml saturated NaNO_2 solution, dropwise at first. Heat carefully until effervescence ceases, then boil for several minutes. Cool, dilute to ~20 ml, and centrifuge in 40-ml cone (NOTE 9). Discard precipitate and pour supernate into clean cone.
8. Add 5 drops each of Fe and La carriers, 1 drop phenolphthalein, and sufficient 10 N NaOH to make solution alkaline. Heat for 5 minutes in hot-water bath, centrifuge, and pour supernate into 125-ml Erlenmeyer flask.

9. Add 2-3 ml conc. HCl and boil solution to near-dryness. If level of Ir activity is thought to be very low, proceed to Step 10. Otherwise proceed to Step 12.
10. Add 10 ml 1 N HNO₃, 3 drops each of Mo and Pd carriers (~10 mg/ml), and 1 ml of 2% α -benzoinoxime in EtOH solution. Filter through a coarse filter and collect filtrate in clean 40-ml cone. Wash precipitate with 5 ml 1 N HNO₃, and add wash to main filtrate.
11. Extract filtrate twice with 5 ml chloroform each time (NOTE 10). Discard chloroform phases.
12. Add 3-4 ml conc. HClO₄ and fume to near-dryness.
13. Repeat Steps 2 through 5.
14. Add 1-2 ml 30% H₂O₂, 0.5 ml conc. HNO₃, and boil gently for ~10 minutes to a volume of about 10 ml.
15. Cool and add 10 N NaOH until solution is just basic (NOTE 11), then 6 N HCl until solution is just acidic.
16. Add 5 ml saturated NaHCO₃ solution (NOTE 12) and 2 ml saturated NaBrO₃ solution (NOTE 13). Heat mixture at low heat on hot plate (if mixture is in Erlenmeyer) for 10 minutes or in hot-water bath for 20 minutes (if mixture is in 40-ml centrifuge cones). Centrifuge thoroughly in 40-ml cone and discard supernate. Wash precipitate and centrifuge well three times (NOTE 14) with 15 ml 0.1 N HNO₃ (NOTE 15). Then wash twice with 10 ml 95% EtOH each time. Drain precipitate for several minutes.
17. Dry precipitate in the cone at 105-110°C for 10 minutes. Precipitate may then be easily pulverized with a Pt or stainless steel wire (or rod).
18. Transfer powdered product into a weighed appropriate counting disc. Weigh (NOTE 16), cover powder with 5 drops 10% zapon, or an equivalent agent.

NOTE 1: When the HClO₄ fuming stage is reached, the solution should turn purple, indicating destruction of the Cl⁻ complexes of Ir. This can be important in ensuring completeness of precipitation in Step 2.

NOTE 2: Colloid formation at this point may occasionally occur. It can usually be eliminated by heating for 10 minutes in a hot-water bath and by alternate addition of a few drops of 1 N NaOH and 1 N HClO₄.

NOTE 3: While the brown color is probably due to a chloride complex, its formation is greatly enhanced by HNO₃. The success of Step 4 depends upon the development of this coloration.

NOTE 4: The Ir-Cl complex forms a rather narrow dark brown band (0.5-1 in.) at the top of the column. Quite frequently a greyish-green species will become evident on the column and even pass into the effluent. This loss usually represents a very small fraction of the total and can be ignored. It is minimized by careful adherence to Step 3.

- NOTE 5: Upon addition of the 6 N HCl-Cl₂ wash, the liquid above the column becomes yellow-brown, indicating increased Ir solubility in 6 N HCl. As percolation proceeds, the Ir band widens somewhat, but not sufficiently to cause loss.
- NOTE 6: The reagent should be made from HI solution containing no preservative. It consequently contains some brown I₃⁻.
- NOTE 7: The iridium is reduced by the I⁻ to a colorless state and is then eluted by 6 N HCl. However, the dark brown band at the top of the column appears to remain. This phenomenon is due to complex formation between I₃⁻ and the resin, and it can safely be ignored.
- NOTE 8: At this point the H₂O₂ oxidizes the reduced Ir to the familiar brown form.
- NOTE 9: Although the solution is only weakly alkaline, it is sufficiently so to cause precipitation of the La and Fe hydroxides. In Step 8 a similar precipitation is made, but from a more alkaline medium.
- NOTE 10: The α-benzoinoxime extracts into chloroform, and is therefore removed before the HClO₄ fuming stage in Step 12.
- NOTE 11: The brown color will give way to a colorless or pale green when the solution is basic.
- NOTE 12: The solution should turn light green.
- NOTE 13: A black precipitate (reported to be an oxide of iridium) should form slowly. A fairly long digestion period is desirable to promote completeness of precipitation.
- NOTE 14: Thorough centrifugation and washing are necessary to eliminate the very high levels of inorganic salts present at this point, since the next step is the final weight determination.
- NOTE 15: If water is used as a wash instead of 0.1 N HNO₃, there is considerable likelihood of troublesome colloid formation.
- NOTE 16: Although the exact composition of the "oxide" has not been determined the stoichiometric factor has been found to be

$$\frac{\text{Ir oxide}}{\text{Ir}} = 1.55 \pm 0.03$$

GOLD

N. A. Bonner

Purification: A gold fraction isolated from 10^{16} fissions 2-weeks-old gave about 45 c/m on a β counter (~70% counting efficiency).

Yield: 70%.

Separation Time: ~4 hours for six samples (without the electrodeposition).

1. To the sample add 2.00 ml of standardized Au carrier (~10 mg/ml), one mg Te carrier, and 1 ml conc. HClO_4 . Evaporate to fumes of HClO_4 . Cool, add 2 drops of 6 M HCl, a few drops of aerosol, and dilute to ~20 ml with water.
2. Add 2 ml of 6% H_2SO_3 and heat on boiling water bath for ~5 minutes. Add another ml of H_2SO_3 and heat 3 minutes more. Centrifuge and wash Au precipitate with 10 ml hot 2 M HCl.
3. Dissolve Au in 1 ml conc. HCl plus 2 drops conc. HNO_3 (NOTE 1). Heat on boiling water bath for ~3 minutes (longer if Au has not dissolved completely).
4. Dilute to 20 ml with 3 M HCl. Centrifuge and discard any foreign matter remaining. Add 2 ml 1 M hydroquinone in ethyl alcohol, heat on water bath for 5 minutes. Add 1 ml more hydroquinone and heat another 3 minutes. Centrifuge and wash Au precipitate once with 6 M HCl.
5. Repeat Step 3, then dilute to 20 ml with 1 M HCl. Add a few drops of 1% aerosol. Add 5 mg Ag carrier, heat on water bath ~2 minutes and centrifuge. Transfer solution to a clean 40-ml glass centrifuge cone and discard AgCl precipitate. Repeat AgCl scavenge.
6. To the solution at room temperature add 2 ml 1 M hydroquinone (NOTE 2), and allow to stand 10 minutes at room temperature. Add another 1 ml hydroquinone, and allow to stand 5 minutes more. Centrifuge and discard supernate. Wash precipitate twice with water and boil with 1 ml conc. HNO_3 . Dilute to ~20 ml, centrifuge (if necessary) and discard solution. Wash precipitate with 2 ml conc. NH_4OH , and dilute before pouring off supernate.
7. Dissolve the Au in 2 ml 48% HBr plus 2 drops of 30% H_2O_2 . Stir frequently until all Au is dissolved; heat on hot-water bath if necessary. Transfer with ~20 ml 3 M HBr to a 60-ml open-top, cylindrical, separatory funnel containing 40 ml of di-isopropyl ether. Stir one minute. Discard aqueous phase and wash ether phase twice with 10-ml portions of 3 M HBr.
8. Stir ether phase with four (more, if necessary) 5-ml portions of water, combining aqueous fractions. Discard ether phase.
9. Add 2 ml 6% H_2SO_3 to combined aqueous fractions. Heat on water bath ~5 minutes. Add 1 ml more H_2SO_3 and heat another 3 minutes. Centrifuge and wash once with water.
10. Dissolve Au in 1/2 ml conc. HCl plus 1/2 ml conc. HNO_3 and evaporate to near-dryness. Take up residue in a few drops of conc. HCl. Dilute with ~3 ml water,

add 5 ml 1 M KCN, and 1 ml 10 M NaOH (enough to make solution quite basic). Transfer to plating cell with few ml water. Electrolyze on tared Pt plate as cathode (use at least 40 mA for ~4 hours). After plating, rinse Pt plate with conc. HNO_3 , water, and acetone. Carefully heat the plate in a burner flame until the Au just begins to melt. Cool, weigh as Au, and mount.

NOTE 1: It is important to use as little HNO_3 as possible. If the hydroquinone is added to an oxidizing medium in Step 4, the gold is not precipitated quantitatively and large amounts of insoluble organic matter are formed.

NOTE 2: Hydroquinone at room temperature will reduce Au to the metal in dilute acid. This separates Au from Se, Te, Pt, Pd, Hg, Cu, Sb, Bi.

THALLIUM

N. A. Bonner

Purification: Tl isolated from 10^{15} fissions 1-week-old had ≤ 10 c/m of impurity. Tl X-rays were counted (50-95 keV) on 1/8-in. NaI crystal with a geometry of about 35%.

Yield: 75%.

Separation Time: ~6 hours.

1. Add exactly 2.0 ml of standardized Tl carrier and 1 drop of Te carrier to the sample in 6 M HCl in a 125-ml Erlenmeyer flask. Boil for at least 10 minutes and evaporate to 10 ml or less.
2. Transfer to a 40-ml centrifuge cone and dilute to 30 ml with water. Add 3 ml of 47% HI, then 1 ml 6% H_2SO_3 solution. Warm in hot-water bath, cool, and allow to stand 10 minutes. Centrifuge, and wash precipitate with 15 ml of water containing 1 drop of HI and 2 drops of 6% H_2SO_3 solution.
3. Dissolve TlI in 5 ml of 2 M HClO_4 + 2 drops of saturated NaNO_2 solution. Boil to drive off I_2 . Dilute to 20 ml.
4. Add 5 mg La carrier and 2 drops of SO_2 solution. Add 3 M NaOH until $\text{La}(\text{OH})_3$ just begins to precipitate. If precipitate is white, add 10 ml excess 3 M NaOH; if brown, add more SO_2 ; then add 10 ml of 3 M NaOH in excess. Centrifuge, transfer supernate to clean 40-ml cone, and discard precipitate.
5. Add a few drops of Br_2 and heat on a water bath for 5 minutes. It is important that the supernate be yellow and basic. Centrifuge and wash precipitate with 15 ml of water containing 1 ml of 3 M NaOH and 1 drop of Br_2 .
6. Dissolve precipitate with 20 ml of 1 M HBr containing a few drops of Br_2 (or saturated NaBrO_3 solution). Transfer to a 60-ml open-top, separatory funnel containing 30 ml of isopropyl ether. Stir vigorously for 1 minute. If solutions are not yellow, add more Br_2 , and stir again. Draw off and discard aqueous phase.
7. Wash ether phase twice with 20-ml portions of 1 M HBr, and twice with 20-ml portions of 0.5 M HBr, stirring one minute each time. Discard all aqueous phases.
8. Remove Tl from the ether by washing successively with 5 ml of 1 M NaOH, 5 ml of water, and 10 ml of 8 M HNO_3 . Combine all three solutions in a 125-ml Erlenmeyer flask, and evaporate to fumes of HNO_3 . Continue fuming until NO_2 and Br_2 fumes are no longer visible. Add conc. HNO_3 if necessary. (See NOTE 1.)
9. Transfer to 40-ml centrifuge cone with 20 ml of water, and repeat Steps 4 and 5.
10. Dissolve TlOOH in 1 ml of conc. HNO_3 , dilute to 15 ml, and add NH_4OH until brown TlOOH begins to precipitate again. Add 1 or 2 drops of 30% H_2O_2 to reduce TlOOH to Tl^+ (colorless solution). Reacidify with a few drops of conc. HNO_3 ,

and add 5 mg La carrier. Make solution basic with NH_4OH , and add ~2 ml conc. NH_4OH in excess. Centrifuge, grease lip of cone, and filter into 40-ml glass centrifuge cone through a 9-cm Whatman No. 40 filter paper in a 50-mm long-stemmed funnel.

11. To filtrate add 10 ml of 10% Na_2CrO_4 and allow to stand in a hot-water bath for 3 minutes. Filter through tared No. 542 filter paper (~1-in. diam). Wash filter paper once with water, and once with methanol (the portion of the filter paper extending outside of the filter chimney should also be washed). Dry at 110°C for 10 minutes. Cool, allow to stand for at least 1/2 hour, and weigh as Tl_2CrO_4 . Mount as appropriate for counting.

NOTE 1: This step removes excess bromine. Any bromide remaining will cause precipitation of TlBr in the $\text{La}(\text{OH})_3$ scavenge step.

LEAD

M. Lindner

Purification: 10^{10} atoms of Pb^{203} isolated from a 4-day-old fission mixture showed no evidence of foreign activities when decay was followed with a NaI counter over seven half-lives.

Yield: About 50%.

Separation Time: About 4 hours.

1. To the active solution add standardized lead carrier solution containing at least 10 mg Pb. Add 2 ml conc. H_2SO_4 and boil to fumes of H_2SO_4 . Cool, very cautiously add water with vigorous agitation, centrifuge, and discard supernate. Wash precipitate once with 15 ml 1 $\underline{\text{N}}$ H_2SO_4 . (See NOTE 1.)
2. Dissolve the PbSO_4 in ~ 2 ml 6 $\underline{\text{N}}$ NH_4Ac . Dilute to 10 ml with water and saturate with H_2S . Centrifuge and wash the PbS once with ~ 0.5 $\underline{\text{N}}$ NH_4Ac saturated with H_2S .
3. Dissolve the PbS in about 1 ml 12 $\underline{\text{N}}$ HCl , and fume to dryness. Take up the residue in 0.3 $\underline{\text{N}}$ HCl , heating, if necessary, to dissolve the residue. Saturate the solution with H_2S . Centrifuge and wash the precipitate with 0.3 $\underline{\text{N}}$ HCl saturated with H_2S .
4. To the precipitate add a few drops 16 $\underline{\text{N}}$ HNO_3 and boil to near-dryness. Add 10 ml fuming HNO_3 . Cool in an ice bath for 10 minutes. Centrifuge and wash precipitate with 10 ml fuming HNO_3 , discarding supernate and wash.
5. To the precipitate, add about 2 ml conc. HCl and fume to dryness. To the residue add 1-2 ml 6 $\underline{\text{N}}$ NH_4Ac , and warm, if necessary, to bring about dissolution. Dilute to 10 ml and saturate with H_2S . Centrifuge and wash precipitate once with ~ 0.5 $\underline{\text{N}}$ NH_4Ac saturated with H_2S .
6. Repeat Steps 3 and 4.
7. To the final $\text{Pb}(\text{NO}_3)_2$ add about 2 ml water and ~ 1 ml conc. H_2SO_4 , and boil to fumes of H_2SO_4 . Cool, cautiously dilute with about 15 ml water, centrifuge, and wash final PbSO_4 , first with about 5 ml water and twice with about 5 ml portions of ethyl alcohol.
8. Slurry precipitate onto suitable weighed counting disc, dry under heat lamp and weigh as PbSO_4 .

NOTE 1: If this initial step is not applicable, an alternative procedure might involve the precipitation of lead sulfide from an ammoniacal tartrate solution.

BISMUTH

W. H. Hutchin

Purification: 3×10^8 atoms of Bi^{210} from a 1-day-old solution of 10^{15} fissions exhibit no foreign radionuclides through eight half-lives.

Yield: About 50%.

Separation Time: 6 hours.

Reagents: Dowex anion exchange resin (1 $\times$ 8, 50-100 mesh).
Acid sulfide holdbacks (Ca^{++} , Mn^{++} , Fe^{+3} , Zn^{++} , In^{+3} , and Cd^{+2}).
Basic sulfide holdbacks (Sb^{+3} , As^{+3} , Te^{+4} , Sn^{+2} , Ba^{+2} , and Sr^{+2}).
Ammonia holdbacks (Ni^{++} , Co^{++} , Cu^{++} , Ag^{+} , and Cd^{++}).

1. To a 2 N HCl solution of the activity, add 10 mg Bi carrier and saturate with H_2S . Centrifuge and discard supernatant.
2. Dissolve precipitate in 1 ml conc. HNO_3 and fume to small volume (0.5 ml or less). Bring volume to ~30 ml with 2 M HCl. Add 1 or 2 ml formic acid and place in hot-water bath. Add 1 ml La carrier (~10 mg La/ml) and five drops of 27 N HF. Centrifuge and discard precipitate.
3. Add more La carrier, centrifuging and discarding the precipitate.
4. To the supernate add acid sulfide holdbacks (see "Reagents") and saturate with H_2S . Centrifuge and wash precipitate with 2 M HCl.
5. Dissolve precipitate in 1 ml conc. HNO_3 and fume to near-dryness (0.5-ml volume). Add two drops conc. HCl, then add 1 N HNO_3 to bring volume to ~20 ml. Add 0.5 ml Mn^{++} carrier and three drops MNO_4^- carrier. Heat in hot water bath for ~5 minutes. Centrifuge and discard precipitate.
6. To the supernate add acid sulfide holdbacks and saturate with H_2S . Centrifuge and discard supernate. Wash Bi_2S_3 with 2 N HCl.
7. Repeat Steps 5 and 6 once.
8. Dissolve precipitate in several drops of conc. HNO_3 , cool and add ~1 ml of saturated tartaric acid solution. Add basic sulfide holdbacks. Dilute with water to ~20-ml volume. Add 1 drop phenolphthalein, make basic with NH_4OH , and saturate with H_2S . Centrifuge and wash precipitate with dilute NH_4OH .
9. Repeat Step 8 twice.
10. Dissolve precipitate in conc. HNO_3 . Fume to dryness and take up in 10 ml 6 N HCl. Pass solution through a 3-in. $\times$ 1/4-in. i. d. anion column (Dowex 1X8, 50-100 mesh) that has been washed with 6 M HCl. Wash column once with 10 ml 6 M HCl and once with 10 ml 1 M HCl. Discard washes.
11. Elute Bi with 15 ml 3 N H_2SO_4 , collecting eluate in clean cone.
12. To eluate add NH_4OH holdbacks, and make basic with NH_4OH . Centrifuge and discard supernate. Wash precipitate once with 0.1 N NH_4OH . Discard wash.

13. Dissolve precipitate in a few drops conc. HNO_3 , add several drops of NH_4OH holdback carriers, dilute with water to ~20 ml and make basic with NH_4OH . Centrifuge and wash precipitate with 0.1 N NH_4OH .
14. Dissolve precipitate in no more than three drops conc. HCl , and dilute to no more than 2.5 ml with water. If at this point a small precipitate is present, centrifuge and discard this precipitate. If there is no precipitate, dilute slowly with water to 25 ml. Centrifuge and wash precipitate twice with water, once with acetone and slurry onto weighing vehicle. Dry under heat lamp. Dry in oven at 110°C for 10 minutes. Weigh as BiOCl .

POLONIUM

M. S. Coops

Decontamination: 10^7 atoms of Po^{210} isolated from a 1-day-old mixture containing 10^{13} fissions showed no evidence of foreign alpha radioactivity on pulse analysis or on following decay through one half-life.

Yield: about 60%.

Separation Time: About 4 hours.

1. To a solution containing the activity in 6 N HCl in a 125-ml Erlenmeyer flask, reduce the volume to about 5 ml, or to the point at which "salting out" occurs if large amounts of solids are present. Add conc. HCl until the concentration of HCl is about 7-8 M.
2. Pass the solution through a Dowex 1X10 anion exchange column (about 2 mm $\times$ 4 cm) previously washed with water, conc. HCl, 0.1 M HCl, and finally conc. HCl. Wash column with 2-3 ml conc. HCl, 2 ml 6 N HCl, 2 ml 0.1 N HCl, 2 ml conc. HCl, and finally with 2 ml 0.1 N HCl.*
3. Pass about 3 ml conc. HNO_3 through the column over a period of at least 20-30 minutes. Collect eluate.
4. To the eluate add 3 ml conc. HClO_4 and fume until about 1 ml of conc. HClO_4 remains. Cool and dilute to 8-10 ml with 5% citrate-HCl solution at a pH $\sim$ 1.0.
5. With continuous stirring, plate the polonium on a silver disc (previously washed with acetone and gently flamed) for about 2 hours.
6. Remove disc, wash twice with water, and rinse with acetone. Dry under a heat lamp at low temperature for several minutes.**

* A trace of nitrate or perchlorate does not appear to affect the procedure.

** The samples at this point may contain considerable other beta activity, although this is not sufficient to interfere with alpha counting. However, if further separation from beta contamination is desired, the silver discs may be placed in a "volatilizer" cell. Essentially, the silver disc is separated by a copper gasket from a platinum disc that is cooled on the back side. Heating this silver disc for 30 seconds with a microburner is sufficient to volatilize the polonium almost quantitatively onto the platinum.

ACTINIUM

G. M. Iddings

Purification: This procedure yields actinium samples suitable for alpha counting in most cases. It does not provide a high degree of purification from lanthanum, and is therefore not necessarily satisfactory for beta- and gamma-counting samples of actinium.

Yield: About 50%.

Separation Time: 4 hours.

1. To the Ac tracer in mixed activities add ~5 mg of pure lutetium carrier. Add NaOH solution until pH $\geq$ 13 (see NOTE 1). Wash the precipitate once with water.
2. Dissolve the precipitate in ~15 ml of a pH 5.7 buffer solution 0.1 M in acetic acid and 1 M in sodium acetate. Transfer the solution to a 60-ml cylindrical, separatory funnel and equilibrate with ~30 ml of 0.40 M TTA* in benzene for 3 minutes, stirring with a motor-driven glass paddle. Wash the organic layer twice with ~15 ml of pH 5.7 buffer solution. Discard aqueous layer and washes.
3. Equilibrate TTA layer with ~10 ml of pH 4.5 buffer solution 0.1 M in formic acid and 1 M in sodium formate. Repeat the back-extraction and combine the aqueous solutions (see NOTE 2). Discard TTA phase (containing Lu).
4. Adjust the pH of the aqueous phase to ~5.7 with saturated sodium acetate solution and again equilibrate with 30 ml of 0.40 M TTA in benzene. Discard aqueous phase and wash the organic layer with 15 ml of pH 5.7 buffer solution. Discard buffer phase, and wash the TTA phase with 15 ml of water without stirring. Discard aqueous phase.
5. Equilibrate TTA phase with 2-3 ml of 0.05 M HCl. Repeat the back-extraction with another 2-ml portion of 0.05 M HCl.
6. Evaporate or boil the combined HCl solutions to a small volume and mount remaining volume on a Pt plate under a heat lamp. Ignite gently to dryness and count.

NOTE 1: Ac is carried more quantitatively on lutetium at pH 13 or 14 with NaOH than at pH 8 or 9 with NH_4OH .

NOTE 2: The pH 4.5 back-extraction is an effective separation of Ac from Y earths and the heavier Ce earths. There is only a partial separation of Ac from La.

* Thenoyltrifluoroacetone.

THORIUM

N. A. Bonner, W. H. Hutchin, G. M. Iddings, and H. A. Tewes

Purification: From a 42-hour-old solution containing $\sim 7 \times 10^{14}$ fissions (from Pu), a thorium sample was obtained which had $< 70 \gamma$ c/m between 8 keV and 22 keV (Th L-X-rays) on a 1/8-in. thick NaI crystal after a 17 mg ThO₂ blank ($\sim 25 \gamma$ c/m) was subtracted from it.

Yield: $\sim 75\%$.

Separation Time: ~ 8 hours for four samples.

1. To a 25-ml solution of mixed activities in 6 M HCl in a 40-ml glass centrifuge cone, add 20 mg of standardized thorium carrier and ~ 2 mg each of Sc, Zr, Te⁺⁴, and Ce⁺³ carriers. Add conc. NH₄OH in excess. Centrifuge the Th(OH)₄ and discard the supernate. Wash the precipitate twice with ~ 20 ml of water to remove salts (especially Na⁺). Centrifuge and discard the wash solutions.
2. Dissolve the precipitate in a few drops of 6 M HNO₃. Transfer the solution to a 50-ml lusteroid tube with ~ 5 ml of water. Add an equal volume of Na-free 6 M NH₄F solution, making the final solution ~ 3 M in NH₄F at pH 4-5. Heat in a hot-water bath for ~ 3 minutes. Cool and centrifuge. Discard the supernatant (Sc). Add ~ 10 ml of Na-free 3 M NH₄F to the precipitate. Heat for 3 minutes. Cool and centrifuge, discarding the supernatant (Sc). Wash the ThF₄ with ~ 5 ml of water. Centrifuge and discard the supernatant (Sc). Wash the ThF₄ with ~ 5 ml of water. Centrifuge and discard the supernatant (Sc).
3. To the ThF₄ precipitate add ~ 3 ml of saturated H₃BO₃, ~ 10 ml of water, and mix thoroughly. Add an equal volume of 12 M HNO₃, making the final solution ~ 6 M in HNO₃. Add ~ 3 ml of 5 M HIO₃. Centrifuge the Th(IO₃)₄ and discard the supernatant (Ce⁺³). Wash the precipitate with ~ 5 ml of 6 M HNO₃ and ~ 1 ml of 5 M HIO₃. Centrifuge and discard supernatant.
4. Dissolve the Th(IO₃)₄ in ~ 10 ml conc. HCl. Add ~ 1 mg of Ce⁺³ carrier. Pass the solution through a Dowex-1 anion exchange resin column (50-100 mesh, 6-mm i. d. $\times$ 7 cm), collecting effluent in a clean cone. Wash the column with ~ 5 ml of conc. HCl and combine with original effluent.
5. To the combined effluents add ~ 2 drops of saturated NaBrO₃. Make solution basic with 10 M NaOH. Do not cool the solution unless it is necessary (the liberated heat and the NaClO oxidize Np⁺⁴ contaminant to higher valence states). Centrifuge and discard the supernatant. Wash the Th(OH)₄ with ~ 25 ml of water.
6. Transfer the thorium to a 50-ml lusteroid tube with ~ 25 ml of 2 M HNO₃. Add 1 ml saturated NaBrO₃. Heat in a hot-water bath for ~ 5 minutes and add 1 ml of 27 N HF. Cool and centrifuge, discarding the supernatant. Wash the ThF₄ once with ~ 5 ml of water.

7. Dissolve the ThF_4 with ~3 ml of saturated H_3BO_3 and ~10 ml of water. Mix thoroughly and add ~13 ml of 12 M HNO_3 . Add 1 drop of 30% H_2O_2 (to reduce Ce^{+4}). Add ~3 ml of 5 M HIO_3 . Centrifuge and discard supernate. Wash the precipitate with a solution containing ~5 ml of 6 M HNO_3 , one drop of 30% H_2O_2 , and 1 ml of 5 M HIO_3 . Centrifuge and discard supernatant.
8. Dissolve the precipitate with ~2 ml of conc. HCl . Add ~1 mg of Ce^{+3} . Heat in hot-water bath for ~3 minutes. Dilute with ~20 ml of water and add ~4 ml of 6% H_2SO_3 solution. Add ~15 ml of 3 M NaOH . Centrifuge and discard the supernatant. Wash the precipitate once with ~30 ml of water.
9. To the $\text{Th}(\text{OH})_4$ add ~15 ml of 0.05 M HCl . Stir with Pt wire stirrer until solution is complete (the pH should be ~1.5). Transfer the solution to a 60-ml cylindrical, separatory funnel. Add ~30 ml of 0.40 M TTA* in benzene and stir vigorously for ~3 minutes with a motor-driven stirring paddle. Drain aqueous layer and wash organic layer four times with ~20 ml of 0.05 M HCl for ~3 minutes each time. Discard aqueous layer and washes.
10. Stir organic layer for 2 minutes with ~20 ml of 2 M HCl . Allow layers to settle a few minutes. Drain and save aqueous layer.
11. To the aqueous layer add 10 M NaOH until alkaline, and dilute to ~40-ml volume with water. Centrifuge and discard supernate. Wash the precipitate with ~30 ml of water and discard wash.
12. Dissolve the $\text{Th}(\text{OH})_4$ with ~10 ml of conc. HCl . Pass the solution through a Dowex-1 anion exchange resin column (50-100 mesh, 6 mm i. d. $\times$ 7 cm), collecting effluent in a clean 40-ml cone. Wash the column with ~5 ml of conc. HCl , and combine effluents.
13. To the combined effluents add an equal volume of water. Centrifuge and reject any foreign solid matter. Add ~5 ml of saturated (~1 M) oxalic acid. (Volume of solution should now be ~35 ml.) Heat for ~5 minutes in a hot-water bath. Cool and stir vigorously. Centrifuge the thorium and discard the supernate. Transfer the precipitate to a filter paper (7-cm diam No. 42 Whatman) with a few ml of ~0.2 M oxalic acid. Filter and ignite at ~900°C for ~30 minutes in a Coors No. 00 porcelain crucible. Weigh as ThO_2 and mount as appropriate for counting.

*Thenoyltrifluoroacetone.

PROTACTINIUM

R. J. Nagle

Purification: 2×10^6 atoms of Pa^{233} isolated from a 3-day-old mixture of 10^{15} fissions showed no evidence of contamination by foreign radionuclides when the decay was followed through two half-lives.

Yield: 30-40%.

Separation Time: ~8 hours per 4 samples, excluding mounting.

1. To the sample contained in a 125-ml Erlenmeyer add standardized Pa^{231} tracer (NOTE 1), 10-20 mg of Ta carrier, 5 ml conc. HClO_4 (NOTE 2), and 3 ml conc. HNO_3 . Take to fumes of HClO_4 for ~1/2 minute and cool. Add 10 ml conc. HNO_3 and boil ~1 minute. Dilute with 5 ml water, stir and transfer to 40-ml centrifuge cone. Centrifuge, and discard supernate.
2. Dissolve precipitate in 4 ml of 6 N HCl 0.4 N HF and transfer to 12-ml polyethylene cone. Equilibrate twice with 4-ml portions of diisopropylketone ("DIPK", pre-equilibrated with the 6 N HCl 0.4 N HF) with vigorous stirring. Separate phases by centrifugation and discard DIPK layers.
3. To aqueous phase add 4 ml of conc. HCl and 0.4 g of solid $\text{AlCl}_3 \cdot 6 \text{H}_2\text{O}$ (to complex F^-). Extract the Pa into DIPK by stirring with 4-, 3-, and 2-ml portions of DIPK for 1-3 minutes each. Combine DIPK layers and discard aqueous. Wash combined organic phases once with 2 ml of 6 N HCl . Discard wash.
4. Equilibrate DIPK once with 2 ml of 6 N HCl 0.1 N HF for 2-4 minutes. Transfer aqueous layer to 20-ml lusteroid cone. Discard DIPK layer.
5. To aqueous add 1.5 ml conc. HNO_3 , 8-10 mg Zr carrier, 1 ml conc. HF , and dilute to 10 ml with water. Add 10 mg La^{+3} and stir. Centrifuge and wash precipitate with 3 ml water, combining wash and supernate in clean 20-ml lusteroid cone. Discard precipitate.
6. Add ~45 mg Ba^{+2} (as saturated BaCl_2 solution) and allow to stand for ~5 minutes. Centrifuge and discard supernate. Wash precipitate once with ~4 ml water. Discard wash.
7. To precipitate add 1.5 ml (5%) H_3BO_3 , 0.5 ml conc. HNO_3 , and 1 ml water. Heat on hot-water bath until fluoro-zirconate is completely dissolved. Add 3.5 ml water and 3.5 ml of 2 M HIO_3 (NOTE 3), and allow to stand 5-10 minutes. Centrifuge and discard supernate. Wash precipitate once with ~5 ml water and discard wash.
8. Add 2.5 ml conc. HCl to precipitate and heat on hot-water bath for ~2 minutes. Add 1 ml water and continue heating until solution is complete. Transfer with 3 ml 6 N HCl to 12-ml glass cone.
9. With aqueous volume <7-ml equilibrate with two 4-ml portions of DIPK for 1-3 minutes each (NOTE 4). Wash combined DIPK phases in 12-ml glass cone once with 2 ml 6 N HCl for ~1 minute. Transfer DIPK to clean 12-ml glass cone.

10. Equilibrate DIPK layer with 2.5 ml of 2 N HClO_4 for 2-3 minutes. Transfer HClO_4 to 12-ml glass cone.
11. Equilibrate 2 N HClO_4 layer once with 4-5 ml of 0.4 M - TTA* in benzene for at least 20 minutes. Wash TTA phase twice with 3-ml portions of 9 N HCl , stirring for 3-5 minutes each. Transfer TTA phase to polyethylene cone after last wash.
12. Equilibrate TTA with 2-3 drops of 6 N HCl 0.1 N HF by stirring for 4-6 minutes.
13. Either evaporate solution directly onto a Pt disc or onto a tungsten filament first, for vacuum volatilization onto a Pt disc.

NOTE 1: The relative abundance of conversion electrons from the alpha tracer (Pa^{231}) is approximately 0.05 conv e^- per α . There is, therefore, a lower detection limit for Pa^{233} dependent upon amount of Pa^{231} tracer used.

NOTE 2: HClO_4 is necessary for the carrying of Pa on hydrous Ta oxide; HNO_3 alone gives less satisfactory results.

NOTE 3: HIO_3 gives a very voluminous precipitate.

NOTE 4: A slight precipitate may be noticed here after extraction with DIPK; it apparently does not interfere.

*Thenoyltrifluoroacetone.

URANIUM

Hugh Wilson

Purification: 3×10^8 atoms of U^{240} and 8×10^9 atoms of U^{237} isolated from a 1-day-old mixture containing $\sim 2 \times 10^{12}$ fissions showed no detectable foreign radioactivity when decay was followed through 10 half-lives of U^{240} and through 6 half-lives of U^{237} .

Yield: 30-40% through final volatilization step.

Separation Time: 8 hours

1. To the mixed activities in HCl add $\sim 2 \times 10^3$ α cpm U^{232} tracer. Double the volume with conc. HCl. Pass solution through a 6-mm $\times$ 4-cm Dowex 1X10 column (prewashed with conc. HCl). Wash column once with 4 ml 8 N HCl. Wash once with 3 ml 6 N HCl and once with 3 ml 4 N HCl. Discard eluate and washes.
2. Pass two 2-ml portions of 0.1 N HCl through column, collecting eluates in a 20-ml lusteroid tube. Dilute to 10-12 ml with water so that solution will be ~ 0.2 N HCl.
3. Pass solution through a 6-mm $\times$ 4-cm Dowex 1X10 column (prewashed with 0.1 N HCl). Wash column once with 2 ml 0.1 N HCl, collecting both eluate and wash in 20-ml lusteroid. Discard column.
4. Add 4 drops La^{+3} carrier (10 mg/ml La^{+3}), 1 drop uranyl carrier (100 μ g/ml UO_2^{++}), and 1 drop MoO_6^- carrier (10 mg/ml Mo^{+6}). To solution add ~ 3 ml conc. NH_4OH . Digest at about 60°C (to protect lusteroid), centrifuge, and discard supernate. Wash precipitate with ~ 5 ml water containing 2 drops NH_4OH . Centrifuge and discard wash.
5. Dissolve precipitate in minimum amount (~ 0.5 ml) of 1 M H_2SO_4 (warming in hot bath is usually necessary). Dilute to ~ 8 ml with 0.06 M H_2SO_4 .
6. Pass solution through a 6-mm $\times$ 4-cm Dowex 1 $\times$ 10 column (prewashed with 0.06 M H_2SO_4), and discard eluate. Wash column twice with 3-ml portions of 0.06 M H_2SO_4 , once with ~ 3 ml 6 N HCl, and once with 2 ml 4 N HCl.
7. To column add two 2-ml portions of 0.1 N HCl, collecting eluates in a 12-ml pyrex cone.
8. Add 4 drops La^{+3} and sufficient excess conc. NH_4OH to make solution basic. Digest, centrifuge, and wash as in Step 4 but at a temperature of $\sim 80-90^\circ C$.
9. Dissolve precipitate in minimum amount (0.5-1 ml) 1 N HNO_3 (warming in hot bath usually necessary). Add ~ 10 drops 5 M $NH_2OH \cdot HCl$. Make basic to pH ≥ 9 with NH_4OH and add 1 drop 1 M NaOH. Heat about 30 seconds in $\sim 90^\circ$ water bath, cool in ice bath for ~ 2 minutes, and centrifuge. Transfer supernate to clean 12-ml cone.

10. Dissolve precipitate as in Step 9 and repeat Step 9, combining supernate from Step 10 with that of Step 9. Discard precipitate.
11. Add 4 drops Zr^{+4} (10 mg/ml) and ~5-10 drops of 5 M $\text{NH}_2\text{OH} \cdot \text{HCl}$. Make basic to pH 9 with NH_4OH (~0.5 to 1 ml). Digest in hot-water bath, cool, and centrifuge as in Step 9. Transfer supernate to 50-ml Erlenmeyer flask and discard precipitate.
12. Place flask on hot plate and boil gently or evaporate to 1-2 ml. Remove from hot plate, add ~2 ml water and cool.
13. Double the solution volume with conc. HCl and pass through a 6-mm $\times$ 4-cm Dowex 1 $\times$ 10 column (prewashed with conc. HCl , then stripped with 0.1 N HCl , and washed again with conc. HCl). Wash column as in Step 1. Discard eluate and washes.
14. Pass three 1-ml portions of 0.1 N HCl through column and collect eluates in glass cone. Evaporate in hot-water bath to near-dryness using a gentle air stream. Transfer remaining liquid to a tungsten filament with a depression creased in center. Pass current through filament until solution is evaporated. Evacuate the system and "flash" residue onto a platinum disc for α , β and/or γ counting.

NEPTUNIUM

G. H. Coleman and K. V. Marsh

Purification: When 5×10^9 atoms of Np^{238} were added to 10^{15} fissions (1-week-old) and a neptunium fraction isolated by this procedure, no activities other than Np^{239} and Np^{238} could be detected either through decay of the gamma radiation for 3 weeks or through gamma-ray pulse-height analysis.

Yield: 30-60%.

Separation Time: About 20 hours for six samples. The procedure was designed for obtaining samples of ultra-high gamma purity. It may be shortened if less stringent radiochemical purity can be tolerated.

1. To sample add 10 mg of La^{+3} carrier, about $1-2 \times 10^3$ α cpm Np^{237} standardized tracer, 10-15 ml conc. HClO_4 and fume to near-dryness in a 125-ml Erlenmeyer flask.
2. Cool, add 5 ml conc. HCl and 2 ml conc. formic acid. Boil briefly, taking care not to boil again to fumes of HClO_4 . Transfer to 5-ml IEC "carbonate" centrifuge cone and dilute to about 30 ml with water. Add 2 drops Zr and Nb carriers.
3. Add 3 ml 27 N HF and place in hot-water bath for 10 minutes. Stir occasionally, then centrifuge, and pour supernate into clean cone (retain precipitate).
4. To supernatant add 5 drops La^{+3} carrier, digest 5 minutes in hot water bath, centrifuge, and discard supernatant.
5. Wash the first precipitate with 30 ml of a solution of 1 N HCl -0.5 N HF . Centrifuge and pour supernatant onto the second LaF_3 precipitate.
6. Stir second LaF_3 , centrifuge and discard wash.
7. To both precipitates add about 0.5 ml conc. HCl and 0.5 ml saturated H_3BO_3 solution. Place in hot-water bath for 5-10 minutes and stir occasionally, (or until precipitates have dissolved).
8. Add contents of "tube 2" to those of "tube 1," and dilute to 25 ml. Add 1 drop phenolphthalein solution, and neutralize with 10 N NaOH solution. Then add 10 drops excess NaOH solution. Heat in hot-water bath for 10 minutes (with occasional stirring), centrifuge, and discard supernatant. Wash precipitate twice with 20 ml water each time. (It is necessary to first add several drops 10 N NaOH solution to the precipitate before adding water so as to avoid possible peptization of the $\text{La}(\text{OH})_3$.) Transfer second 20-ml slurry to 40-ml glass centrifuge cone. Discard washes.
9. Add 1 ml conc. HCl and 1 ml conc. formic acid to precipitate and boil briefly and gently (caution: watch for "bumping"). Transfer solution to clean polyethylene (or carbonate) cone and dilute to 30 ml with water.
10. Repeat Steps 3 through 8, being careful to add two drops of Nb and Zr holdback carrier before adding HF .

11. To the $\text{La}(\text{OH})_3$ precipitate add 1 ml conc. HCl and 10 drops of conc. HI solution (no hypophosphite preservative). Transfer to 125-ml Erlenmeyer flask, boil gently to ~0.5 ml, cool, and add 15 ml conc. HCl .
12. Pass solution through a 2-in.-long column of Dowex A-1 ion exchange resin (50-100 mesh). Wash column with 15 ml conc. HCl + 5 drops conc. HI , then with 15 ml conc. HCl . Discard effluents.
13. Add 5 ml of 5 N HCl to column and collect eluate in clean 125-ml Erlenmeyer flask. Wait 10 minutes and add 10 more ml of 5 N HCl , collecting in same flask. Add 10 drops conc. HI solution to flask and boil to near-dryness. Cool, dilute to ~10 ml with 1 N HCl and transfer to 40-ml glass cone.
14. Add 5-10 ml 0.4 M TTA* (benzene solution) to the solution and equilibrate layers for 10-15 minutes. Separate layers, transfer organic layer to clean 40-ml cone, and equilibrate aqueous layer with another 5-10 ml of 0.4 M TTA. Combine organic layers.
15. Wash combined TTA solutions with 5 ml 1 N HCl , discarding the aqueous phase.
16. Equilibrate TTA with 3 ml 9 N HCl and separate layers. Repeat back-extraction with 3 ml 9 N HCl and combine aqueous layers in clean 40-ml cone. Discard TTA.
17. Dilute 9 N HCl to 20 ml with water and repeat Steps 14-16. (One extraction usually extracts all Np at this point; likewise one back-extraction usually suffices.) Transfer 9 N HCl to a 125-ml Erlenmeyer flask.
18. Boil contents of flask to near-dryness and cool. Add 1 drop methyl red indicator 1 ml saturated NH_4Cl solution and add conc. NH_4OH dropwise until color turns yellow. Add 3 N HCl dropwise until color turns red. Continue further adjustment with dropwise additions of 1 N NH_4OH and 1 N HCl until solution turns red with the addition of one drop of 1 N HCl . Solution volume should be 2-3 ml.
19. Transfer solution to an electroplating cell fitted with a 30-mil Pt wire anode and a 1-in. circular Pt counting disc as a cathode. Pass ~1-1.5 A at 4-6 V through cell for 15-20 minutes.
20. Add 1 ml conc. NH_4OH to unit while electrolysis is in progress. After 15-30 seconds remove solution from cell and transfer to a 40-ml cone. Test solution in "well" counter for completeness of electrolysis. If solution contains appreciable activity, transfer to 125-ml Erlenmeyer and repeat Step 18 (omit addition of saturated NH_4Cl solution).
21. Dismantle electrolysis cell, carefully rinse disc first with water, then with acetone. Allow disc to dry in air. Ignite disc in Bunsen- or Meker-burner flame. Count as appropriate.

*Thenoyltrifluoroacetone.

PLUTONIUM

R. J. Morrow

Purification: This procedure is designed primarily for separation of plutonium samples to be alpha-counted; it does not yield samples highly purified from beta- and gamma-emitters. Samples containing 10^{13} - 10^{14} atoms of Pu have been isolated from 4-day-old solutions of 5×10^{13} fissions. No foreign alpha activities could be detected, and the beta content was 10^5 dpm or less.

Yield: 60% if an electroplating technique is used for the final step, and 35% if vacuum volatilization is used.

Separation Time: 6 hours for six samples

1. To an acid solution of mixed activities in a plastic centrifuge cone add ~1 mg La^{+3} carrier and an appropriate amount of standardized Pu alpha-emitting tracer, the isotope added and its level of activity depending upon the isotope sought and its estimated level of activity. Add 2-3 drops saturated NaNO_2 solution and heat in a hot-water bath for five minutes.
2. Add sufficient conc. NH_4OH until the solution is basic. Stir, and digest at 60°C in a hot-water bath for five minutes. Centrifuge and discard supernate.
3. To precipitate add 5 ml 25% NaOH solution. Stir and digest for 5 minutes at 60°C in a hot-water bath. Centrifuge and discard supernate.
4. To precipitate add 2 mg Zr^{+4} carrier and sufficient 3 $\underline{\text{N}}$ HCl to dissolve the precipitate. Dilute to 5 ml or more with 3 $\underline{\text{N}}$ HCl .
5. Add 1 ml 27 $\underline{\text{N}}$ HF , stir and digest at 60°C in a hot-water bath until the LaF_3 is well coagulated. Centrifuge and discard supernate. Wash precipitate once with 5 ml 2 $\underline{\text{N}}$ HCl - 0.5 $\underline{\text{N}}$ HF . Centrifuge and discard wash.
6. To precipitate add sufficient 8 $\underline{\text{N}}$ HNO_3 saturated with H_3BO_3 (usually ~5-10 drops) to dissolve. Heat in hot-water bath if necessary.
7. Pipet the solution onto a 3-mm i. d. $\times$ 6-cm Dowex A-1 (1 $\times$ 8) anion exchange resin column (previously washed thoroughly with 8 $\underline{\text{N}}$ HNO_3) and discard effluent. Wash column twice with 2 ml 8 $\underline{\text{N}}$ HNO_3 each time, and twice with 1-ml portions of 10 $\underline{\text{N}}$ HCl . Discard all washes.
8. To column add 2 ml of a solution 10 $\underline{\text{N}}$ in HCl and 0.5 $\underline{\text{N}}$ in HI , collecting eluate in a 50-ml Erlenmeyer flask or a 40-ml centrifuge tube.
9. Either of two alternatives may now be followed:
 - (a). Boil the solution to a very small volume and pipet the remainder onto the central depression of a tungsten filament of vacuum bell jar apparatus. Carefully boil the remaining solution to dryness in the filament in air by application of current through filament. Evacuate system and flash the contents of the filament onto a Pt disc located about 1/4-1/2 in. from filament.

- (b). Evaporate the solution to dryness and redissolve residue with three drops of conc. HCl. Transfer to an electrolysis cell suitable for 1-in. diam plates. Rinse tube successively with three more drops of conc. HCl and three drops of water, adding the rinses to the solution in the cell. Carefully adjust acidity to the methyl red end point with conc. NH_4OH , then make barely acidic with two drops of 2 M HCl. With a 1-in. Pt disc as cathode and a volume of roughly 1-2 ml, plate at 5-6 V with about 2.5 A for roughly 15 minutes. Before stopping the current add 1 ml conc. NH_4OH . Shut off current, remove solution, and wash the disc first with water, then with acetone. Mild flaming of the plate is desirable.

AMERICIUM-CURIUM

R. W. Hoff, E. K. Hulet, and G. H. Coleman

Purification: This procedure does not separate americium from curium (NOTE 1). For curium alpha-emitting isotopes, a 1.5-day-old mixture of 10^{14} fissions yielded a final counting sample containing less than 10^5 β dpm. The determination of $\text{Am}^{242\text{m}}$ required additional decontamination from β - γ impurities. Samples containing 10^8 atoms of $\text{Am}^{242\text{m}}$ isolated from a 1.5-day-old fission mixture containing 10^{14} fissions showed only the 16-hour beta decay over 2-3 half-lives.

Yield: 75%, exclusive of the final volatilization step. Volatilization step is usually ~50%.

1. To the unknown solution (usually 25 ml or less) add an appropriate level of Am and Cm tracers, the choice and level of added tracers depending upon the isotope sought. If the unknown activity and the tracer are both alpha emitters, the more abundant activity should have the lower energy if possible, as an aid in resolution.
2. If the unknown solution (usually 25 ml or less) is ~ 6 M HCl, add an equal volume 12 M HCl plus a few drops 16 M HNO_3 . Let stand for 10 minutes. Pour the solution on a 6-cm $\times$ 6-mm diam Dowex 1 $\times$ 10 ion exchange column which has been washed with 12 M HCl containing a few drops of 16 M HNO_3 . Collect the eluate in a 40-ml cone and wash column with 12 M HCl containing a few drops 16 M HNO_3 , adding the wash to the main eluate.
3. To the eluate and wash, add 2 drops $\text{La}(\text{NO}_3)_3$ carrier solution (10 mgs La^{+3}/ml) plus 0.5 ml Zr^{+4} carrier solution (10 mg Zr/ml). Cool in an ice bath and add conc. NH_4OH until solution is alkaline. Digest, centrifuge, and wash precipitate once with dilute NH_4OH and once with water.
4. Dissolve the precipitate with 2-3 ml 6 M HCl, transfer to a 20-ml lusteroid cone, and dilute to 10-ml volume with water. Add 2 ml conc. HF and digest in a hot-water bath with stirring. Centrifuge and wash precipitate once with 2 M HCl-2 M HF.
5. Dissolve precipitate in 3 ml saturated H_3BO_3 -6 M HCl mixture. Transfer to a 12-ml glass centrifuge cone. Add conc. NH_4OH until solution is alkaline. Digest, centrifuge, and wash precipitate twice with water.
6. Dissolve the precipitate with HCl gas and saturate the resultant solution with HCl gas. Load the solution (~3 drops) on a 5-cm $\times$ 3-mm diam Dowex-50 $\times$ 12 colloidal-form ion exchange column which has been washed with 13 M HCl solution. Allow the loading solution to pass into the resin. Add to the column about 1.5 ml of 13 M HCl and force through the column at a rate of 1.5 min/drop (air pressure is generally required to reach this flow rate). Collect 3 drops of eluate in each of a series of culture tubes. Combine contents of tubes 4, 5, and 6 into a 12-ml cone and evaporate to dryness.

7. Repeat the column elution by adding 3 drops 13 M HCl to the dry material in the cone and loading this solution onto another Dowex 50 column as in Step 6.
8. Upon evaporating the combined tubes 4, 5, and 6 to a small volume after the second column elution, transfer and evaporate the solution on a tungsten filament. The counting sample is produced by volatilizing the material in a vacuum onto a 1-in. diam Pt plate (NOTES 2, 3).

NOTE 1: Although americium-curium separations found using this procedure are slight, appreciable separations can occasionally occur in the 13 M HCl Dowex-50 column step. Thus, tracers for both elements must be added if one is to determine chemical yields of each element accurately.

NOTE 2: The volatilization is necessary to obtain good resolution in the alpha particle pulse analysis and is also an aid in obtaining reproducible results in the beta counting.

NOTE 3: Some interference in the beta counting of $\text{Am}^{242\text{m}}$ might be encountered from the growth of the Np^{239} daughter of the alpha decay of Am^{243} (if present). One can readily correct for this growth of Np^{239} activity, subsequent to its removal during the chemical procedure.

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