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PROCEDURES FOR THE ANALYSIS OF SOME
RADIONUCLIDES ADSORBED ON SOIL

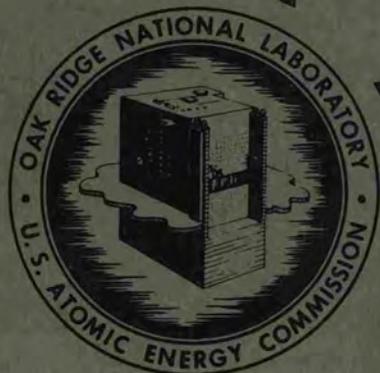
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HEALTH PHYSICS DIVISION

K.Z. Morgan, Director

PROCEDURES FOR THE ANALYSIS OF SOME
RADIONUCLIDES ADSORBED ON SOIL

Bernd Kahn*

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ABSTRACT

A procedure is presented for the determination of radionuclides of cesium, strontium, yttrium, the lanthanum rare earths, ruthenium, zirconium, niobium, and cobalt, which have been adsorbed on soil. The radionuclides are leached from the soil with nitric, sulfuric, or a combination of oxalic and hydrochloric acids; isotopic carriers are added to the leach solutions; and the carriers and radioisotopes are purified by a series of precipitations.

INTRODUCTION

The procedure for the analysis of radionuclides adsorbed on soil consists of leaching the soil with acids, to transfer the radionuclides to the liquid phase, followed by standard radiochemical methods for separating the radioelements. Experiments to determine the most effective leach solutions and procedures have been described,¹ and values of the per cent leached from soil with the two consecutive leaches indicated in the method are given in Table I. Leaching efficiencies of 97% to 99% may be obtained for the nuclides indicated in Table I by using three consecutive leachings instead of two.

The radiochemical separations of the fission products are essentially those devised for use with aqueous fission product solutions.^{2,3,4} In the ruthenium analysis, the leaching and distillation steps have been combined; to the cesium analysis an initial silicowolframate precipitation has been added; in the analyses of the rare earths, zirconium, niobium, and cobalt, the hydroxides of these elements are initially precipitated; and strontium is initially precipitated as the carbonate. Other modifications of the fission products analyses consist of the precipitation of zirconium as the phenylarsonate to separate it from the large amount of iron in solution, a fuming nitric acid precipitation of the calcium in solution to remove it from the rare earths fraction, and a fuming nitric acid precipitation, in the presence of some water, followed by an acetone wash, to eliminate calcium from the strontium fraction.

The method for determining cobalt is based on standard cobalt separations.⁵ The final precipitation with 1-nitroso-2-naphthol was made because the precipitate was sufficiently voluminous that little cobalt carrier need be used, and because the cobaltinitrite precipitate is difficult to retain on filter paper. Tracer studies with ^{60}Co and the major long-lived fission products (See Table II)

TABLE I

Per Cent Recovery of Radionuclides in Radiochemical Analysis

Element	% Recovered by Leaching Soil (Average of 4 samples) (two leachings)	% of Carrier Recovered (Average of 40 samples)
Cs	90	65
Sr	97	80
Y and Trivalent rare earths	93	80
Ce	97	80
Ru	99	60
Zr	97	60
Nb	95	65
Co	91	70

TABLE II

Behavior of Radioactive Tracers in Cobalt Analysis*

Radionuclide	Total Activity on Soil	Total Activity leached with $1M$ HNO_3	Total Activity in Co_3O_4 Precipitate (Corrected for yield)
Cs^{137}	5,460,000 c/m	1,170,000 c/m	0 c/m
Sr^{90}	423,000	403,000	0
Y^{90}	426,000	392,000	11
Ce^{144}	1,520,000	1,460,000	11
Ru^{106}	2,330,000	1,640,000	124
$Zr-Nb^{95}$	7,530,000	397,000	461
Co^{60}	3,840,000	3,490,000	3,410,000

* 10 gram soil samples

indicate that decontamination of the fission products from the latter is satisfactory and that essentially complete exchange takes place between cobalt carrier and tracer. The carrier used was $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, in a concentration of approximately 15 mg/ml in terms of the final precipitate of Co_3O_4 . The concentration of the carrier was determined by precipitating triplicate 2 ml aliquots with 1-nitroso-2-naphthol solution, made slightly acid with HCl, filtering, and igniting to the oxide.

All procedures were checked with blanks--leach solutions containing no carriers--to detect contamination of the final precipitate with elements leached from the soil. No contamination was found. Precipitates of the carriers from the leach solutions obtained from soils containing no added activity were counted to detect naturally occurring radionuclides, but no significant amount of these was found to remain with the carriers. The procedures have been utilized in the radiochemical analysis of 20 samples of mud from White Oak Lake, the Clinch River, and the Tennessee River.⁶

Approximately 20 mg of the carriers, prepared and their concentration determined as described elsewhere²⁻⁴ were used in all procedures. The same references are suggested for a discussion of radiochemical procedures and lists of reagents and laboratory equipment. The percent recovery of carrier is given in Table I. The sample mounting technique and method of calculating counts/minute/gram of soil are described in the cesium procedure.

ACKNOWLEDGEMENT

Acknowledgement is made to D. K. Smith and L. M. Lawless for testing the radiochemical procedures.

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CESIUM

1. Weigh duplicate 10.0 gm samples of soil in 50 ml glass centrifuge tubes.
2. Add 25 ml 9M sulfuric acid to the soil, stir to suspend the soil, heat to boiling, and stir while heating 3-4 minutes. Centrifuge immediately, before the solution cools, and decant into 150 ml beaker. Stir moist residue while heating gently, and again centrifuge and decant into 150 ml beaker.
3. Add 25 ml water to the supernate to prevent precipitation of salts upon cooling.
4. Repeat Step # 2. Combine supernates.
5. Add approximately 30 mg cesium carrier (weighed as CsClO_4) to the supernate and stir. Transfer to two 50 ml lusteroid centrifuge tubes and centrifuge to remove any soil remaining in supernate. Decant into glass centrifuge tubes.
6. Add to supernate 2 ml (1 ml into each centrifuge tube) 1/8M silicowolframic acid to precipitate cesium silicowolframate. Stir and cool several minutes in ice bath. Centrifuge and discard supernate.
7. Wash precipitates twice with 10 ml portions of cold 6M hydrochloric acid, using the acid to combine the precipitates. Centrifuge and discard supernates.
8. Dissolve precipitate in 3 ml 6M sodium hydroxide, heating if necessary. Dilute to 10 ml and add approximately 5 mg. Fe holdback carrier (ferric nitrate) to precipitate ferric hydroxide, carrying any remaining zirconium, niobium, or rare earth activities. Heat, centrifuge, and decant supernate into glass centrifuge tube.
9. To the precipitate from the last step, add 1 ml 6M sodium hydroxide to dissolve any remaining Cs precipitate, dilute to 5 ml, heat, stir, centrifuge, and combine supernate with that from step # 8.

10. Add 20 ml 70% perchloric acid to supernate and heat until dense white and yellow precipitates (silicon dioxide and wolfram trioxide) form. While hot, centrifuge and decant supernate into 125 ml Erlenmayer flask.
11. Heat on hot plate until dense white perchloric acid fumes form and volume is approximately 20 ml. Decant into 50 ml glass centrifuge tube, let cool in ice bath, and add 20 ml absolute (200 proof) ethanol. Stir and cool ten minutes to precipitate cesium perchlorate and sodium perchlorate. Centrifuge and discard supernate.
12. Wash precipitate twice with 20 ml cold absolute ethanol to dissolve sodium perchlorate. (If bulky precipitate remains, wash a third time). Centrifuge and discard supernate.
13. Take up the fine white precipitate in 5 ml cold absolute ethanol and filter on tared 2 cm. dia. fine filter paper in a Buchner funnel. Wash with 10 ml cold absolute ethanol and let dry. (Use filter paper which has been weighed on 2.5. cm. diameter watchglass.)
14. Place filter paper containing CsClO_4 on watch glass, and weigh. Calculate weight of precipitate.
15. Mount watch glass and paper on cardboard and cover with polystyrene.
16. Count on second shelf of end-window Geiger- Mueller counter.
17. (a) Calculate counts/minute. (b) Calculate fraction of Cs carrier recovered. (c) Divide counts/minute by the fraction Cs recovered to obtain counts/minute in original solution. (d) Divide this value by the fraction of Cs leached from soil to obtain counts/minute in 10 gm. sample of soil. (e) Divide by 10 to obtain counts/minute per gm. of dried soil.

STRONTIUM

1. Weigh duplicate 10.0 gm. samples in 50 ml glass centrifuge tubes.
2. Add 25 ml 1M nitric acid to the soil, stir to suspend the soil, heat to boiling, and stir while heating 3-4 minutes. Centrifuge and decant into 50 ml lusteriod centrifuge tube. Stir moist residue while heating gently, and again centrifuge and decant into lusteriod tube.
3. Repeat step # 2. Combine supernates.
4. Add approximately 20 mg. strontium carrier (weighed as $\text{SrC}_2\text{O}_4 \cdot 4\text{H}_2\text{O}$) and stir. Centrifuge to remove any soil remaining in supernate. Decant into 150 ml beaker.
5. Heat to boiling, add 19M sodium hydroxide to make solution basic, and add 5 ml 2M sodium carbonate to precipitate strontium carbonate (as well as insoluble hydroxides of iron, etc.) Stir, cool in ice bath for 5 minutes, centrifuge and discard supernate.
6. Wash precipitate with 30 ml water. Centrifuge and discard supernate.
7. Dissolve precipitate with 5 ml 6M nitric acid, heating if necessary. Slowly, with stirring, add 25 ml fuming nitric acid (90% HNO_3) to precipitate strontium nitrate. Cool, centrifuge and discard supernate.
8. Wash precipitate with 20 ml acetone. Centrifuge and discard supernate.
9. Dissolve precipitate in 5 ml water. Add 20 fuming nitric acid, stir, cool, centrifuge and discard supernate.
10. Repeat step # 8, to remove all calcium leached from soil.
11. Dissolve precipitate in 10 ml water, add approximately 10 mg. iron carrier, and add 2 ml concentrated ammonium hydroxide to precipitate ferric hydroxide. Centrifuge and decant supernate into 50 ml glass centrifuge tube. Discard precipitate.

12. Heat supernate to boiling and slowly add 5 ml saturated ammonium oxalate to precipitate strontium oxalate.
13. Take up precipitate in 10 ml hot water, stir to dissolve all ammonium oxalate, and filter on tared 2 cm. diameter filter paper (Whatman #40) in Buchner Funnel. Wash with 20 ml hot water, and dry with consecutive portions of 20 ml ethanol and 20 ml diethyl ether.
14. Proceed as in the Cesium method, steps # 14-17, weighing strontium as $\text{SrC}_2\text{O}_4 \cdot 4\text{H}_2\text{O}$.

CERIUM, THE TRIVALENT RARE EARTHS, AND YTTRIUM

1. Weigh duplicate 10.0 gm. samples in 50 ml glass centrifuge tubes.
2. Add 25 ml 1M nitric acid to the soil, stir to suspend the soil, heat to boiling, and stir while heating 3-4 minutes. Centrifuge and decant into 50 ml lusteroid centrifuge tubes. Stir moist residue while heating gently, and again centrifuge and decant into lusteroid tube.
3. Repeat Step # 2. Combine supernates.
4. Add approximately 20 mg. cerium carrier and 20 mg. lanthanum carrier (weighed as $Ce_2(C_2O_4)_3 \cdot 10H_2O$ and $La_2(C_2O_4)_3 \cdot 8H_2O$, respectively) and stir. Centrifuge to remove any soil remaining in supernate. Decant into 150 ml beaker.
(Lanthanum is added to carry yttrium and all rare earth radionuclides except cerium).
5. Add 19M sodium hydroxide to make solution strongly basic (pH ~10) to precipitate insoluble hydroxides (cerium, lanthanum, iron, and some calcium). Centrifuge in lusteroid tubes and discard supernate (containing aluminum).
6. Wash precipitate with 30 ml water. Centrifuge and discard supernate.
7. Dissolve precipitate with 3 ml concentrated nitric acid and dilute with 15ml water. Add 1 ml hydrogen peroxide, approximately 10 mg. zirconium "holdback" carrier, and 3 ml hydrofluoric acid (49% HF) to precipitate insoluble fluorides (cerium, lanthanum and calcium). Stir, let stand 5 minutes, centrifuge and discard supernate.
8. Add 30 ml water and transfer water and precipitate to glass centrifuge tube. Stir, centrifuge, and discard supernate.
9. Dissolve the precipitate in 0.5 ml saturated boric acid and 2 ml fuming nitric acid (90% HNO_3). Add 25 ml fuming nitric acid slowly, with stirring, and cool in ice bath to precipitate calcium nitrate. Centrifuge and decant into

lusteroid tube containing 20 ml water. Discard precipitate.

10. To supernate, add 3 ml hydrofluoric acid, stir, and let stand for 5 minutes to precipitate cerium fluoride and lanthanum fluoride. Centrifuge and discard supernate.
11. Wash precipitate with 30 ml water. Centrifuge and discard supernate.
12. Dissolve precipitate by swirling with 1 ml saturated boric acid and adding 8 ml concentrated nitric acid. Add 2 ml 1M sodium bromate to oxidize cerous to ceric ion, and slowly and with stirring add 20 ml 0.35M hydriodic acid to precipitate ceric iodate. Cool in ice bath for at least 5 minutes, centrifuge, and decant supernate containing lanthanum, (carrying yttrium and all rare earths except cerium) into glass centrifuge tube. Save supernate for step # 15.
13. Dissolve cerium precipitate with 3-4 drops concentrated hydrochloric acid, 1 drop hydrogen peroxide, and 8 ml concentrated nitric acid. Oxidize cerous ion with 2 ml 1M sodium bromate and slowly and with stirring add 20 ml 0.35M hydriodic acid to precipitate ceric iodate again. Cool in ice bath for at least 5 minutes, centrifuge and discard supernate.
14. Dissolve precipitate in 3-4 drops concentrated hydrochloric acid and add 1 ml hydrogen peroxide to reduce ceric ion. Add 8 ml concentrated nitric acid and approximately 10 mg. zirconium carrier. Slowly and with stirring add 20 ml 0.35M hydriodic acid to precipitate zirconium iodate "scavenger". Centrifuge and decant supernate into glass tube. Discard precipitate.
15. Follow identical procedure for separate supernates from step # 12 and # 14. Add 19M sodium hydroxide to make solutions basic and precipitate cerium hydroxide and lanthanum hydroxide. Centrifuge and discard supernates.

16. Wash each precipitate with 30 ml water. Centrifuge and discard supernates.
17. Dissolve precipitates in 1 ml concentrated hydrochloric acid (heating with a drop hydrogen peroxide, if necessary), dilute with 10 ml water, and reduce iodate in solutions by adding a few drops of 6% sulfurous acid until solutions are colorless. Add concentrated ammonium hydroxide to make solutions basic and to precipitate cerium hydroxide and lanthanum hydroxide. Centrifuge and discard supernates.
18. Wash each precipitate with 30 ml water. Centrifuge and discard supernates.
19. Dissolve precipitates in 1 ml 6M hydrochloric acid and dilute with 15 ml water. Heat to boiling and slowly add 15 ml saturated oxalic acid. Stir and let cool in ice bath for 10 minutes to precipitate cerium oxalate and lanthanum oxalate, (the latter carrying yttrium and all trivalent rare earths except cerium).
20. Add 10 ml hot water to the precipitates, stir to dissolve oxalic acid, and filter on tared 2 cm. diameter filter papers (Whatman # 40) in Buchner funnels. Wash with 20 ml hot water, and dry with consecutive portions of 20 ml ethanol and 20 ml diethyl ether.
21. Proceed as in the cesium method, steps # 14-17, weighing cerium and lanthanum as $Ce_2(C_2O_4)_3 \cdot 10H_2O$ and $La_2(C_2O_4)_3 \cdot 8H_2O$, respectively. The lanthanum carrier contains yttrium and all rare earth radionuclides except those of cerium.

RUTHENIUM

1. Weigh duplicate 10.0 gm. samples in 125 ml distillation flasks.
2. Add 25 ml 9M sulfuric acid and approximately 20 mg. ruthenium carrier (weighed as Ru metal) and swirl to suspend soil. Heat to boiling and boil, while shaking, for 1-2 minutes. Cool in ice bath.
3. Add 1 gm. potassium permanganate and immediately connect condenser having air inlet and vapor outlet. Admit air in a slow, steady stream, and submerge vapor outlet in 50 ml glass centrifuge tube, standing in ice bath, and containing 20 ml 6M sodium hydroxide. Heat flask until solution boils vigorously and brown ruthenium tetroxide vapor passes into sodium hydroxide solution. Let cool, add another gram potassium permanganate to solution, and again boil, to distill remaining ruthenium tetroxide. Discard contents of flask.
4. To the sodium hydroxide solution, add 3 ml ethanol, stir, and heat in boiling water bath to precipitate ruthenium dioxide. Centrifuge and discard supernate.
5. Wash precipitate with 20 ml boiling water containing 1 drop sodium hydroxide. Centrifuge, and discard supernate.
6. Dissolve precipitate by heating in 2 ml concentrated hydrochloric acid and dilute to 20 ml. Add powdered magnesium metal in small portions until black ruthenium metal precipitates. Boil to coagulate ruthenium metal, then add concentrated hydrochloric acid dropwise until all magnesium metal is dissolved. Boil, cool, centrifuge and discard supernate.
7. Take up ruthenium metal in 5 ml water and filter on tared 2 cm. diameter filter paper (Whatman # 40) in Buchner funnel. Wash with 20 ml water, and dry with consecutive portions of 20 ml ethanol and 20 ml diethyl ether.
8. Proceed as in cesium method, steps # 14-17, weighing ruthenium as Ru metal.

ZIRCONIUM AND NIOBIUM

1. Weigh duplicate 10.0 gm. samples in 50 ml glass centrifuge tubes.
2. Add 12 ml saturated oxalic acid, 4 ml 6M hydrochloric acid, and 9 ml water to the soil, stir to suspend the soil, heat to boiling, and stir while heating 3-4 minutes. Centrifuge and decant into 50 ml lusteroid centrifuge tube. Stir moist residue while heating gently, and again centrifuge and decant into lusteroid tube.
3. Repeat step # 2. Combine supernates.
4. Add approximately 20 mg zirconium carrier and 20 mg niobium carrier (weighed as ZrO_2 and Nb_2O_5 , respectively) and stir. Centrifuge to remove any soil remaining in supernate. Decant into 150 ml beaker.
5. Add 19M sodium hydroxide to make solution strongly basic (pH \sim 10) to precipitate insoluble hydroxides of zirconium, niobium, iron, and some calcium. Centrifuge in glass centrifuge tubes and discard supernate.
6. Wash precipitate with 30 ml water. Centrifuge and discard supernate.
7. Take up precipitate in 15 ml concentrated nitric acid. Add 3 gm. potassium chlorate and wash down sides of tube with 5 ml water. Heat carefully until thick white niobium pentoxide precipitate forms, then heat in boiling water bath for 15 minutes. Centrifuge and decant into glass centrifuge tube. Save supernate, containing zirconium, for step #15.
8. Wash precipitate with 10 ml "acid wash" solution (3 ml 6M nitric acid, 2 ml 6M ammonium hydroxide, 5 ml water). Heat to boiling, transfer solution and precipitate to lusteroid tube, centrifuge, and add supernate, containing some additional zirconium, to supernate from step # 7.
9. Dissolve niobium pentoxide precipitate in 1 ml 49% hydrofluoric acid and 2 ml 6M nitric acid. Add approximately 10 mg. zirconium carrier and 1 ml barium

- nitrate (50 gm./l Ba) solution to precipitate barium fluozirconate "scavenger."
Centrifuge and decant supernate into lusteroid tube. Discard precipitate.
10. Slowly add concentrated ammonium hydroxide until solution is basic, to precipitate niobium pentoxide. Stir, centrifuge, and discard supernate.
 11. Wash precipitate with 20 ml "basic wash" solution (6 ml 6M ammonium hydroxide, 2 ml 6M nitric acid, 12 ml water). Transfer to glass centrifuge tube, heat to boiling, centrifuge, and discard supernate.
 12. Again wash precipitate with 20 ml boiling "basic wash" solution. Centrifuge and discard supernate.
 13. Immediately dissolve precipitate by heating with 0.5 ml 6M nitric acid and 2 ml saturated oxalic acid. Cool, add 3 ml water, 10 ml concentrated nitric acid and 0.5 gm. potassium chlorate, and wash down sides of tube with a few ml of water. Heat carefully until white niobium pentoxide precipitates, then heat in boiling water bath for 15 minutes. Centrifuge and discard supernate.
 14. Take up precipitate in 30 ml "acid wash" solution, heat to boiling, stir, and filter hydrated niobium pentoxide on tared fine filter paper (11 cm. diameter). Save for step # 23.
 15. To supernate from step # 7 and # 8, containing zirconium, add 5 ml phenylarsonic acid (25 gm/l) solution and heat to precipitate zirconium phenylarsonate. Cool, centrifuge, and discard supernate.
 16. Wash precipitate with solution of 2 ml phenylarsonic acid and 20 ml 6M nitric acid. Centrifuge and discard supernate.
 17. Take up precipitate in 20 ml 6M nitric acid and transfer to lusteroid tube. Add approximately 10 mg. lanthanum carrier and 2 ml 49% hydrofluoric acid, to dissolve zirconium precipitate and to precipitate lanthanum fluoride "scavenger".

Stir and let stand for 5 minutes, then centrifuge and decant supernate into lusteroid tube. Discard precipitate.

18. Add 2 ml barium nitrate solution to precipitate barium fluozirconate, stir well, let stand 5 minutes, centrifuge, and discard supernate.
19. Dissolve precipitate with 3 ml concentrated hydrochloric acid, 1 ml saturated boric acid, and 15 ml water. Add 2 drops concentrated sulfuric acid to precipitate barium sulfate. Stir, centrifuge, and decant supernate into glass centrifuge tube. Discard barium sulfate precipitate.
20. Cool supernate in ice bath. Add 2 ml cold 6% cupferron solution to precipitate zirconium cupferrate, stir, centrifuge, and discard supernate.
21. Wash precipitate with 20 ml 1M hydrochloric acid containing a drop 6% cupferron solution. Centrifuge and discard supernate.
22. Take up precipitate in 10 ml cold 1M hydrochloric acid containing a drop cupferron solution and filter (11 cm. diameter Whatman # 40).
23. Follow same procedure separately for precipitates from step # 14 and # 22. Place filter papers in tared porcelain crucibles and ignite in muffle furnace at approximately 800°C. for 45 minutes. Cool to room temperature.
24. Weigh crucibles and ignited oxides to obtain weight of ZrO_2 and of Nb_2O_5 .
25. Transfer precipitates to 2.5 cm. diameter watch glasses, mount on cardboard, and cover with polystyrene.
26. Proceed as in cesium method, steps # 16 and # 17.

COBALT

1. Weigh duplicate 10.0 gm. samples in 50 ml glass centrifuge tubes.
2. Add 25 ml 1M nitric acid* to the soil, stir to suspend the soil, heat to boiling, and stir while heating 3-4 minutes. Centrifuge and decant into 50 ml lusteroid test tube. Stir moist residue while heating gently, and again centrifuge and decant into lusteroid tube.
3. Repeat step #2. Combine supernates.
4. Add approximately 15 mg. cobalt carrier (weighed as Co_3O_4) and centrifuge to remove any soil remaining in supernate. Decant into 2 glass centrifuge tubes.
5. Add potassium hydroxide solution (1 mg/ml) to supernate to pH 10 or higher (approximately 10 ml) to precipitate cobalt hydroxide and other hydroxides. Centrifuge and discard supernate.
6. Wash precipitate twice with 30 ml water. Centrifuge and discard supernate.
7. Dissolve precipitate by heating with 5 ml acetic acid. Dilute with 20 ml water and cool. Add slowly 10 ml potassium nitrite solution (1 mg/ml) to precipitate potassium cobaltinitrite. Let stand at least 15 minutes, stirring frequently.
8. Prepare fresh 1-nitroso-2-naphthol solution by dissolving 1 gm reagent in 10 ml acetic acid and 10 ml water with boiling. Filter through dry funnel (Whatman #40 paper) into dry bottle.
9. Centrifuge solution from step #7 and discard supernate. Wash the precipitate twice with 20 ml cold 10% acetic acid. Centrifuge and discard supernate.
10. Add 2-3 drops concentrated hydrochloric acid and 15 ml water to precipitate, and dissolve precipitate by boiling the solution. Cool in ice bath, add approximately 20 mg. cerium carrier and 15 ml saturated ammonium chloride,

and stir. Add 1 ml concentrated ammonium hydroxide to make solution basic and to precipitate cerium hydroxide "scavenger". Stir, centrifuge immediately, and decant supernate into glass centrifuge tube. Discard precipitate.

11. Immediately acidify supernate with 3 ml concentrated hydrochloric acid. Add 10 ml warm 1-nitroso-2-naphthol solution, stir, and filter through Whatman #40 paper, using cold water to wash out centrifuge tube.
12. Wash precipitate with 20 ml cold water.
13. Place filter paper in tared porcelain crucible and ignite in muffle furnace at approximately 800°C for 45 minutes. Cool to room temperature.
14. Weigh crucible and ignited oxide to obtain weight of Co_3O_4 .
15. Transfer precipitate to 2.5 cm. diameter watch glass, mount on cardboard, and cover with polystyrene.
16. Proceed as in cesium method, steps #16 and # 17.

* By using 1M hydrochloric acid, 98% of the cobalt is leached (instead of 91% with 1M nitric acid). The subsequent procedure is not affected by the substitution of hydrochloric for nitric acid, hence the use of hydrochloric acid may be preferred.