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DETERMINATION OF CERIUM-144  
IN VEGETATION AND SOIL

By  
Doris C. Sutton

February 1964

Health and Safety Laboratory  
New York Operations Office  
New York, New York



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U. S. Atomic Energy Commission  
Health and Safety Laboratory  
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## ABSTRACT

An extraction method has been used to determine cerium-144 in vegetation and soil. The principle advantages of the method are, (1) equilibration of cerium carrier and activity in the initial step of the procedure, (2) provision for the determination of strontium-90 and cesium-137 in vegetation and strontium-90 and normal cerium in soil.

The results of measurements on fourteen wheat samples collected from sites in the United States during 1962 are reported. Data for replicate analyses of two soil types and duplicate and single analyses for a small varied group of soils collected in 1960 are listed.



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## INTRODUCTION

Cerium-144 and its daughter, praseodymium-144 are the major contributors to fission product atmospheric radioactivity from 1-3 years after nuclear detonations.<sup>1</sup> Studies show that fallout cerium-144 is deposited as both soluble and insoluble material.<sup>2,3</sup> It also appears from measurement of cerium-144 at various depths in soil (0-12 inches) that the soluble portion is not fixed, after deposition, but is exchangeable.<sup>4</sup> It is reasonable to assume that a soluble and exchangeable element will eventually be distributed in environmental samples.

Plant uptake studies with Sr<sup>90</sup>, Cs<sup>137</sup>, Ru<sup>106</sup>, Ce<sup>144</sup> and Y<sup>91</sup> by Neel et al.<sup>5</sup> conducted on various artificially contaminated soils showed negligible concentrations of cerium-144 in all observed plants (barley, lettuce, carrot, bean and radish). A similar plant study by Romney et al.<sup>6</sup> indicated that Sr<sup>90</sup>, Cs<sup>137</sup> and Ce<sup>144</sup> increased in uptake as the hydrogen-ion concentration increased in soils on which the plants grew. The observed Cs<sup>137</sup> and Ce<sup>144</sup> uptake was small in comparison to that of Sr<sup>90</sup>. Gulyakin and Yuditseva<sup>7</sup> observed, from a study of the uptake of fission products by peas and summer wheat grown as sand crops and water crops, that 89, 74 and 15 per cent of the experimental total

activity concentrations of  $\text{Sr}^{90}$ ,  $\text{Cs}^{137}$  and  $\text{Ce}^{144}$ , respectively, were found in the above ground part of the mature plant for peas grown as a sand crop. Wheat samples grown as sand crops contained 90, 60 and 3 per cent, respectively,  $\text{Sr}^{90}$ ,  $\text{Cs}^{137}$  and  $\text{Ce}^{144}$ . Young pea plants (~40 days old) grown as water crops accumulated 90, 50 and 5.6 per cent, respectively,  $\text{Sr}^{90}$ ,  $\text{Cs}^{137}$  and  $\text{Ce}^{144}$  in above ground parts. Summer wheat samples grown as a water crop for the same period showed an accumulation of 81, 59 and 0.79 per cent respectively of  $\text{Sr}^{90}$ ,  $\text{Cs}^{137}$  and  $\text{Ce}^{144}$ . Of this above ground percentage, the mature water grown plants concentrated 1.58, 22.74, 1.12 and 5.10, 15.20 and 1.96 per cent of the  $\text{Sr}^{90}$ ,  $\text{Cs}^{137}$  and  $\text{Ce}^{144}$ , respectively in the pea seed and wheat grain.

Measurements of cerium-144 have been reported in various food samples, animal bone and soil.<sup>8-10</sup> Studies by Nezu et al.<sup>8</sup> show a notable occurrence of this nuclide in animal bone and clams. This is not in agreement with observations of Van Dilla<sup>11</sup> which indicate that animals grazing on vegetation contaminated with large amounts of cerium-144 and other radionuclides showed very little evidence of the retention of cerium-144.

The work reported here is primarily concerned with the radiochemical measurement of the concentration of cerium-144 in vegetation samples. Wheat, being a world wide principal food crop,

was selected for this study. The results of measurement of cerium-144 on fourteen wheat samples collected from sites in the United States during 1962 are reported. Radiochemical methods are presented for the determination of cerium-144, cesium-137 and strontium-90 in vegetation ash. Methods are also presented for the determination of normal cerium, cerium-144 and strontium-90 in soil. Measurements are reported for ten soil sites.

#### DETERMINATION of Ce<sup>144</sup>, Sr<sup>90</sup> and Cs<sup>137</sup>

##### Cerium

Radiochemical methods for the determination of cerium-144 in environmental samples have been devised in order to measure the concentration of this relatively short lived nuclide (285 days) due to fallout from nuclear weapons testing. Samples to be analyzed may be conveniently classified as follows, (1) soluble or insoluble, (2) single or several nuclides, and (3) matrix.

The initial sample preparation of soluble samples for cerium analysis is quite varied. Samples of natural waters,<sup>9</sup> rain water,<sup>3,12,13</sup> tap water,<sup>3,13</sup> sea water<sup>9,14</sup> and fission product solutions<sup>15-19</sup> are reduced in volume by evaporation in the presence of dilute acid, and particulate material removed by filtration,<sup>12,13</sup> the solution is evaporated in the presence of dilute acid and sodium bromate<sup>3</sup> and cerium coprecipitated and collected

as hydroxide.<sup>3,9,12-14</sup> Cerium is collected from fission product mixtures which have high acid concentrations, by precipitation as fluoride<sup>15</sup> or oxalate<sup>16</sup> and by extraction with organic solvents.<sup>17-19</sup> Purification is accomplished by zirconium iodate scavenging,<sup>3,9,12,13,16</sup> fluoride collection<sup>3,9,12-14</sup> and ion exchange.<sup>14</sup> Final precipitation is with oxalic acid. Yield is determined by converting the oxalate to oxide and weighing.

Sample preparation for insoluble samples requires drying and/or ashing, blending and aliquoting. Multi-isotope analyses of samples of air filters,<sup>13,20,21</sup> vegetation,<sup>8,9</sup> soil<sup>8,10</sup> and ion exchange resin<sup>22</sup> effect equilibration of cerium carrier and activity by acid extraction,<sup>10</sup> extraction with acid and peroxide,<sup>9,20</sup> extraction with acid and hydroxylamine hydrochloride<sup>21</sup> alkaline fusion and acid extraction<sup>13,22</sup> and acid extraction preceding alkaline fusion.<sup>8</sup> Cerium is collected as hydroxide<sup>8,13,20-22</sup> fluoride<sup>9</sup> or cerium potassium nitrate.<sup>10</sup> Purification is accomplished by ion exchange,<sup>8,22</sup> zirconium iodate<sup>8-10,13,22</sup> phosphate<sup>20</sup> and phenylarsonate<sup>21</sup> scavenge, fluoride precipitation<sup>13,20,22</sup> and organic solvent extraction.<sup>20,21</sup> Final precipitation is as oxalate<sup>8-10,13,20,21</sup> or iodate.<sup>22</sup> Yield is determined by weighing ceric oxide<sup>8,10,13,20</sup> or ceric iodate.<sup>22</sup>

In the method reported here for cerium determination, carrier is added to vegetation ash prior to extraction and finally weighed to determine yield. Soil samples are analyzed carrier free with  $Ce^{141}$  as tracer to measure normal cerium and to determine overall chemical yield. The sample is extracted with hydrochloric acid-hydrogen peroxide and the insoluble material is removed by filtration. The filtrate is evaporated and silica removed by filtration in samples where necessary. Alkaline earths and rare earths are collected as oxalates and ignited. The combined carbonates and oxides are dissolved in nitric acid-hydrogen peroxide. Manganese is removed by precipitating manganese dioxide. Zirconium and thorium are scavenged as iodates. Cerium and the rare earths are collected as fluorides. Cerium is isolated as ceric iodate.

Praseodymium-144 is allowed a three hour period to reach equilibrium with its parent cerium-144 after the final iodate precipitation and prior to counting. The cerium-144 disintegration rate is calculated from the counting rate of praseodymium-144 through a  $232 \text{ mg/cm}^2$  aluminum absorber. Corrections are made for background, chemical yield, decay, counting error, and counter efficiency.

Normal cerium in soil is determined by weighing the ceric iodate precipitate and calculating the weight of the ceric ion. This value, expressed as milligrams of cerium, is corrected for overall chemical yield, which is determined by gamma counting the ceric iodate precipitate for cerium-141.



## Strontium

Strontium carrier is equilibrated with strontium-90 in vegetation ash by sodium carbonate fusion of the insoluble fraction from the hydrochloric acid-hydrogen peroxide extraction and the alkaline earth fraction from the soluble fraction. The alkaline earth carbonates are separated by water leach and filtration. Strontium and barium are removed from the mixed carbonates by precipitation in the presence of 75% nitric acid. Barium is separated from strontium by precipitation as chromate. Strontium-90 is allowed two weeks to reach equilibrium with its daughter, yttrium-90. Yttrium is separated from strontium by ammonium hydroxide collection, dissolved and precipitated as oxalate.

Strontium is precipitated as carbonate and weighed to determine yield for vegetation samples. Strontium is precipitated as carbonate and strontium-85 gamma counted to determine chemical yield for soil samples. The strontium-90 disintegration rate is calculated from the counting rate of yttrium-90 with corrections made for background, chemical yield, decay, counter efficiency and counting error.

## Cesium

Added cesium carrier is equilibrated with cesium-137 in

vegetation ash by hydrochloric acid-hydrogen peroxide extraction. The insoluble material and silica are removed by filtration. Cesium is adsorbed from the filtrate by batch exchange onto ammonium phosphomolybdate. The ammonium phosphomolybdate is collected by filtration and dissolved with sodium hydroxide. EDTA is added to the solution to complex non-univalent cations. The solution is passed through a phenolsulfonic acid cation exchange column in the  $\text{Na}^+$  form. Ammonium, potassium and sodium ions (rubidium, if present) are removed from the resin by dilute hydrochloric acid. Cesium is selectively removed from the resin by increasing the hydrochloric acid concentration and precipitated as tetraphenylborate.

Cesium tetraphenylborate is weighed to determine yield. Cesium-137 is measured by beta counting the precipitate with a thin window beta counter. The instrument response is corrected for counter background and efficiency, self-absorption, and yield.

#### CHEMICAL PROCEDURES

##### Reagents and Special Apparatus

Cerium carrier - 10 mg/ml

Strontium carrier - 20 mg/ml

Cesium carrier - 30 mg/ml

Zirconium carrier - 5 mg/ml  
Yttrium carrier - 20 mg/ml  
Barium carrier - 20 mg/ml  
Hydrogen peroxide - 30%  
Aerosol OT - 0.1%  
Octyl alcohol  
Oxalic acid - solid  
Oxalic acid - saturated solution  
Sodium chlorate - solid  
Iodic acid - 0.35M  
Iodic acid wash solution - 0.175M - 10% HNO<sub>3</sub>  
Boric acid - saturated solution  
Sodium dichromate - 0.3M  
Ammonium oxalate - 5%  
Ammonium acetate - 6M  
Acetic acid - 6M  
Na<sub>4</sub>EDTA - 50%  
Sodium tetraphenylboron - 3%  
Ammonium phosphomolybdate - solid  
Sodium hydroxide - saturated  
Sodium chloride - 5%  
Ce<sup>141</sup> tracer - about 2000 cpm/ml

Sr<sup>85</sup> tracer - about 10,000 cpm/ml

Bio Rex 40 Cation exchange resin

Mechanical stirrer

Well-type gamma scintillation counter

Thin window beta counter

232 mg/cm<sup>2</sup> aluminum absorber

## VEGETATION

### Cerium (Section A)

1. Transfer 10 grams of vegetation ash to a 600 ml beaker. Add 1 ml each of cerium, strontium, and cesium carrier solutions. Add 100 ml of water. With mechanical stirring, add 100 ml of HCl and 60 ml of 30% hydrogen peroxide. Stir for 1 hour. Filter through #42 Whatman paper. Alternately wash the insoluble material with hot 10% HCl and hot water twice. Transfer paper and insoluble material to a platinum dish. Reserve for strontium determination.
2. Collect filtrate in a 600 ml beaker and evaporate the solution to ~10 ml. Add 100 ml of water and allow silica to settle. Filter through #42 Whatman paper. Alternately wash the silica precipitate with hot 10% HCl and hot water twice. Collect filtrate and washings in a 600 ml beaker. Add 1

gram ammonium phosphomolybdate to the solution and stir for 30 minutes. Filter through 4 cm glass fiber paper. Wash precipitate and paper with 5% HCl and a few drops of 0.1% Aerosol OT. Transfer the paper and precipitate to a 40 ml centrifuge tube. Reserve for cesium determination.

3. Transfer the solution to an 800 ml beaker. Add 5 grams of oxalic acid and dissolve by stirring. Adjust solution to pH 4.5 - 5.0 (Hydrion paper) with  $\text{NH}_4\text{OH}$ . Allow precipitate to settle and filter by gravity through #42 Whatman paper. Wash the precipitate with 5% ammonium oxalate (pH 4.5 - 5.0). Transfer the paper and precipitate to a platinum dish. Dry at  $100^\circ\text{C}$  and ignite at  $600^\circ\text{C}$ .
4. Transfer the ash to a 250 ml beaker. Add 5 ml water and 25 ml  $\text{HNO}_3$ . Heat solution to boiling. Cautiously add 5 ml 30% hydrogen peroxide dropwise while solution is boiling. Evaporate to ~20 ml. Cautiously add 1 gram sodium chlorate to the boiling solution and allow solution to boil until reaction ceases.
5. Transfer the solution to a 40 ml centrifuge tube with water. Centrifuge and decant the supernate into a 90 ml centrifuge tube. Wash the precipitate with 10 ml 10%  $\text{HNO}_3$ . Centrifuge



- and combine supernates. Discard the precipitate. Adjust the solution to pH 8 with  $\text{NH}_4\text{OH}$ . Centrifuge and decant supernate into a 250 ml beaker. Reserve for strontium determination.
6. Dissolve the precipitate with 10 ml  $\text{HNO}_3$ . Add 3 drops of 30% hydrogen peroxide and heat solution until peroxide decomposes. Cool solution to room temperature. Add 20 ml of 0.35M iodic acid and 1 ml zirconium carrier solution. Centrifuge and decant supernate into a 40 ml Lusteroid tube. Discard precipitate. Add 5 ml HF to supernate and allow to stand for 1 hour. Centrifuge, decant, and discard supernate.
  7. Slurry the precipitate with 2 ml of saturated boric acid solution and 5 ml  $\text{HNO}_3$ . Transfer the slurry to a 40 ml glass centrifuge tube. Digest in water bath at  $95^\circ\text{C}$  to dissolve precipitate. Add 10 ml water and adjust solution to pH 8 with  $\text{NH}_4\text{OH}$ . Add 3 drops of 30% hydrogen peroxide and heat solution until peroxide decomposes. Cool. Add 20 ml of 0.35M iodic acid and immerse tube in ice bath. Add 3 ml of 1M sodium bromate. Allow precipitate to coagulate and settle.
  8. Filter through weighed 2.4 cm glass fiber paper. Wash the

precipitate with 0.175M iodic acid - 10% nitric acid wash solution and finally with water. Dry at 110°C for 2 hours. Weigh, mount, and beta count praseodymium-144 through a 232 mg/cm<sup>2</sup> aluminum absorber.

### Strontium (Section B)

1. To the strontium fraction from Section A, Step 5, add 25 ml saturated sodium carbonate. Digest at 95°C for 10 minutes. Filter through #42 Whatman paper. Wash precipitate with 5% sodium carbonate. Transfer paper and carbonate precipitate to the platinum dish containing strontium fraction from Section A, Step 1. Dry at 110°C and ignite at 600°C.
2. Add 20 grams sodium carbonate to the platinum dish and fuse at 900°C. Cool. Transfer the melt from the dish to a mortar and grind. Transfer the ground material to a 400 ml beaker. Add 250 ml water and digest just below boiling for 2 hours.
3. Filter through #42 Whatman paper. Wash the precipitate with 5% sodium carbonate. Discard filtrate and washings. Dissolve the carbonate precipitate with hot 1:1 HCl. Alternately wash the paper with hot water and hot 1:1 HCl and collect the solution in a 250 ml beaker. Evaporate the solution to dryness.

4. Add 23 ml water to the residue. With mechanical stirring, add 77 ml 90% HNO<sub>3</sub>. Stir for 45 minutes. Filter with suction through 4 cm glass fiber paper. Discard filtrate. Dissolve the precipitate with water and collect the solution in a 100 ml beaker. Evaporate the solution to dryness.
5. Dissolve the residue with 12 ml water. With mechanical stirring, slowly add 40 ml 90% HNO<sub>3</sub>. Stir for 45 minutes. Filter with suction through 4 cm glass fiber paper. Discard filtrate. Dissolve the precipitate with water. Collect the solution in a 40 ml centrifuge tube.
6. Add 1 ml yttrium carrier solution. Adjust pH to 8 with NH<sub>4</sub>OH. Centrifuge and decant supernate into a 40 ml centrifuge tube. Dissolve the precipitate with HCl, dilute to 10 ml with water and re-precipitate the yttrium with NH<sub>4</sub>OH. Centrifuge and combine supernates. Discard precipitate.
7. Add 1 ml of barium carrier solution to the centrifuge tube. Add to the solution 2 ml of 6M ammonium acetate and 1 ml of 6M acetic acid. Adjust the pH to 5.5 with NH<sub>4</sub>OH or HCl. Add 1 ml 0.3M sodium dichromate. Digest at 95°C for 30 minutes. Cool, centrifuge, add 1-2 drops of 0.1% Aerosol OT to tube, and decant supernate into a 1 oz polyethylene

bottle containing 1 ml yttrium carrier and 5 drops of HCl. Discard precipitate. Store polyethylene container for two weeks.

8. After the two week equilibration period, transfer the solution to a 40 ml centrifuge tube. Heat in water bath at 90°C. Adjust the pH to 8 with NH<sub>4</sub>OH. Add 6 drops of 30% hydrogen peroxide and continue heating until peroxide is decomposed. Centrifuge and decant supernate into a 150 ml beaker. Record date and hour. Dissolve the precipitate with HCl, dilute to 10 ml with water, and re-precipitate the yttrium with NH<sub>4</sub>OH. Centrifuge and combine supernates. Reserve for strontium yield determination.
9. Dissolve hydroxide precipitate by adding HCl dropwise. Dilute solution to 20 ml with water. Add 1 ml saturated oxalic acid and digest at 90°C for 10 minutes. Cool. Filter through 2.4 cm glass fiber paper. Dry at 110°C for 30 minutes. Mount and beta count yttrium-90.
10. Add 10 ml saturated sodium carbonate to the combined supernates from Step 8. Digest at 90°C for 10 minutes. Filter through a tared fine fritted crucible. Wash with 5% sodium carbonate and finally with 3-5 ml water. Dry at 110°C for

one hour. Weigh and calculate strontium yield.

Cesium (Section C)

1. Add 5 ml water to 40 ml centrifuge tube from Section A, Step 2. Macerate paper with a stirring rod. Add saturated sodium hydroxide dropwise and with stirring until ammonium phosphomolybdate is dissolved. With suction, filter through 4 cm glass fiber paper. Wash with water. Collect filtrate in 40 ml centrifuge tube.
2. Add 10 ml of 50%  $\text{Na}_4$  EDTA solution. Mix well. Pass solution through Bio Rex 40 cation exchange resin column (converted to  $\text{Na}^+$  form with 200 ml of 5% sodium chloride) at a flow rate not exceeding 1 ml per minute. Wash the column with 100 ml water at a flow rate not exceeding 3 ml per minute. Wash the column with 1 liter 0.12N HCl at a flow rate not exceeding 5 ml per minute. Discard all waste and wash solutions.
3. Elute cesium from the resin column with 200 ml of 2N HCl. Collect the effluent in a 400 ml beaker. Evaporate solution to dryness. Pass two column volumes of water through the ion exchange resin bed. Reserve column until ready for next use.



4. Dissolve residue with 5 drops of HCl and transfer solution with water to 40 ml centrifuge tube. Dilute to 20 ml. Adjust pH to 12.0 - 12.5 (Hydrion paper) with saturated sodium hydroxide. Add 10 ml of 3% sodium tetraphenylboron solution. Mix well. Add HCl dropwise with stirring until precipitate coagulates. Allow solution and precipitate to stand for 30 minutes. Adjust pH to 12.0 - 12.5 with sodium hydroxide (Hydrion paper). Allow precipitate to settle. Filter with suction through weighed 2.4 cm glass fiber paper. Dry at 110°C for 1 hour. Weigh, mount and beta count cesium-137.

#### Resin Preparation

Transfer 500 grams of Bio-Rex 40 (50-100 mesh) to a 3 liter beaker. Add 2 liters of water and stir for 30 minutes. Decant and discard wash solution. Repeat the washing procedure with 2 liters of each of the following solutions: (1) 1N sodium hydroxide, (2) water (twice), (3) 1N hydrochloric acid and (4) water (twice). Transfer the resin to a bottle with water and store.

The ion exchange resin is prepared for the analytical separation using the column illustrated in Figure 1 (pg. 37). Fill the column with water and place a wad of glass wool at the bottom of the column. Transfer 15 ml of the wet settled prepared resin

to the column. Place a second wad of glass wool at the top of the resin. Open the stopcock and allow the water level to reach the top of the upper glass wool. Place in position a 1 liter separatory funnel which has been fitted to the top of the column with a rubber stopper. Transfer 200 ml of 5% sodium chloride to the separatory funnel and allow the solution to flow at a rate not exceeding 5 ml per minute. The column is now ready for the sample solution.

#### SOIL

#### Cerium<sup>23</sup> (Section D)

1. Transfer a 500 gram soil aliquot to a 3 liter beaker. Add 1 ml each of cerium-141 and strontium-85 tracer solutions and 5 ml of strontium carrier. Add 500 ml distilled water. With mechanical stirring add 500 ml HCl and 300 ml 30% H<sub>2</sub>O<sub>2</sub>. Stir for 1 hour (Octyl alcohol added dropwise during the extraction will prevent frothing of the sample). Filter by suction through 24 cm #42 Whatman paper. Alternately wash the soil twice with hot 1:1 HCl and hot water. Transfer the filtrate and wash solutions to a 3 liter beaker. Discard insoluble material.
2. Evaporate the filtrate to 300-400 ml. Cautiously add 500 ml HNO<sub>3</sub>. Cover and digest until the organic material is

decomposed. Evaporate solution to 50-75 ml then add 300-400 ml water. Filter through #42 Whatman paper. Alternately wash the precipitate with hot 1:1 HCl and hot water. Transfer the combined filtrate and washings to a 3 liter beaker. Discard insoluble material.

3. Dissolve 150 grams of oxalic acid in the solution. Adjust to pH 4.5 - 5.0 (Hydrion paper) with  $\text{NH}_4\text{OH}$ . Allow precipitate to settle and remove liquid by siphoning. Filter precipitate through #42 Whatman paper and wash with 5% ammonium oxalate (pH 4.5 - 5.0). Transfer precipitate to a 600 ml pyrex beaker. Dry at  $110^\circ\text{C}$  and ignite at  $600^\circ\text{C}$  until ash is free of carbon. Cool.
4. Add 15 ml water to the beaker. Dissolve the ash by adding 50 ml  $\text{HNO}_3$ . Heat the solution to boiling. Add 30%  $\text{H}_2\text{O}_2$  dropwise to dissolve insoluble material. Boil the solution for ~5 minutes to destroy  $\text{H}_2\text{O}_2$ . Add sodium chlorate (~3 - 4 grams) in 1 gram increments to the boiling solution until manganese dioxide precipitation is complete. Cool. Transfer the solution and precipitate with water to a 90 ml centrifuge tube. Centrifuge and decant supernate into 800 ml beaker. Wash precipitate with 10 ml 10%  $\text{HNO}_3$ .

Centrifuge and combine supernates. Discard manganese dioxide precipitate.

5. Evaporate the solution to ~20 - 25 ml. Cool. With mechanical stirring, add 250 ml of 90%  $\text{HNO}_3$ . Stir for 45 minutes. With suction, filter through glass fiber paper. Transfer filtrate to a 1 liter beaker. Dissolve nitrate precipitate with water and transfer solution to a 600 ml beaker.
6. Evaporate the solution in the 600 ml beaker to ~20 - 25 ml and repeat Step 5. Combine  $\text{HNO}_3$  filtrates. Reserve solution from dissolved nitrate precipitate for strontium determination.
7. Evaporate the combined  $\text{HNO}_3$  filtrates to ~10 ml. Transfer the solution to a 90 ml centrifuge tube with water. Adjust the solution to pH 8 with  $\text{NH}_4\text{OH}$ . Centrifuge, decant and discard supernate. Dissolve precipitate with 5 ml  $\text{HNO}_3$ . Transfer solution with water to a 40 ml Lusteroid centrifuge tube. Add 10 ml HF and allow solution to stand 30 minutes. Centrifuge, decant and discard supernate.
8. Slurry the precipitate with 2 ml saturated boric acid. Add 10 ml  $\text{HNO}_3$  to dissolve precipitate. Transfer solution to a

- 90 ml centrifuge tube with water. Adjust the solution to pH 8 with  $\text{NH}_4\text{OH}$ . Centrifuge, decant and discard supernate.
9. Dissolve precipitate with 10 ml  $\text{HNO}_3$ . Add 3 drops of 30% hydrogen peroxide and heat solution until peroxide decomposes. Cool solution to room temperature. Add 20 ml 0.35M iodic acid. Add 1 ml zirconium carrier solution. Centrifuge and decant supernate into a 40 ml Lusteroid tube. Add 5 ml HF. Allow solution to stand 30 minutes. Centrifuge, decant, and discard supernate.
  10. Slurry the precipitate with 2 ml saturated boric acid. Add 10 ml  $\text{HNO}_3$  to dissolve precipitate. Transfer solution to a 40 ml centrifuge tube with water. Adjust the solution to pH 8 with  $\text{NH}_4\text{OH}$ . Centrifuge, decant, and discard supernate.
  11. Dissolve precipitate with 10 ml  $\text{HNO}_3$ . Add 3 drops of 30% hydrogen peroxide and heat solution until peroxide decomposes. Cool. Add 20 ml 0.35M iodic acid and immerse tube in ice bath. Add 3 ml 1M sodium bromate. Allow precipitate to coagulate and settle.
  12. Centrifuge, decant, and discard supernate. Gamma count cerium-141 by inserting the 40 ml centrifuge tube into the well of



a gamma scintillation counter. Standardize by counting 1 ml of cerium-141 tracer precipitated as iodate, as in Step 11. Calculate relative per cent recovery.

13. Add 10 ml  $\text{HNO}_3$  and 3 drops of  $\text{HCl}$  to the centrifuge tube. Dissolve the precipitate by adding 30% hydrogen peroxide dropwise while heating at  $95^\circ\text{C}$  and constantly stirring. Continue adding hydrogen peroxide dropwise until iodine is no longer expelled and the solution is colorless. Boil to remove excess peroxide.
14. Cool the solution in an ice bath. Add 20 ml of 0.35M iodic acid and 3 ml of 1M sodium bromate. Allow the precipitate to coagulate and settle. Filter over weighed 2.4 cm glass fiber paper. Wash the precipitate with 0.175M iodic acid - 10%  $\text{HNO}_3$  wash solution and finally with water. Dry the precipitate at  $110^\circ\text{C}$  for 2 hours. Weigh, mount and beta count praseodymium-144 through a  $232 \text{ mg/cm}^2$  aluminum absorber.

#### Strontium (Section E)

1. Evaporate solution from the dissolved nitrate precipitate, Step 6, Section D to dryness. Dissolve the salts in 60 ml water. With mechanical stirring slowly add 220 ml 90%  $\text{HNO}_3$ .

Stir for 45 minutes. Filter through glass fiber paper.

Discard filtrate.

2. Dissolve precipitate with water and collect solution in a 250 ml beaker. Evaporate the solution to dryness. Dissolve the residue with 23 ml water. With mechanical stirring, slowly add 77 ml 90%  $\text{HNO}_3$ . Stir for 45 minutes. Filter through glass fiber paper. Discard filtrate. Dissolve precipitate with water and collect solution in a 40 ml centrifuge tube.
3. Add 1 ml of yttrium carrier solution. Adjust pH to 8 with  $\text{NH}_4\text{OH}$ . Centrifuge and decant the supernate into a 40 ml centrifuge tube. Dissolve the precipitate by adding  $\text{HCl}$  dropwise. Dilute the solution with 10 ml water and reprecipitate yttrium with  $\text{NH}_4\text{OH}$ . Centrifuge and combine supernates. Discard precipitate.
4. Add 1 ml of barium carrier solution to the centrifuge tube. Add 2 ml of 6M ammonium acetate and 1 ml of 6M acetic acid. Adjust solution to pH 5.5 (Hydrion paper). Add 1 ml of 0.3M sodium dichromate solution. Digest for 30 minutes at  $90^\circ\text{C}$ . Cool, centrifuge, add 1-2 drops of 0.1% Aerosol OT to tube, and decant supernate into a 2 oz polyethylene bottle containing

1 ml yttrium carrier solution and 5 drops of HCl. Store the polyethylene bottle and solution for fourteen days.

Discard precipitate.

5. Transfer the solution from the polyethylene container after fourteen days to a 40 ml centrifuge tube. Adjust the pH to 8 with  $\text{NH}_4\text{OH}$ . Centrifuge and decant the supernate into a 150 ml beaker. Record date and hour. Dissolve the precipitate by adding HCl dropwise. Dilute the solution with 10 ml water. Reprecipitate yttrium with  $\text{NH}_4\text{OH}$ . Centrifuge and combine supernates. Save for strontium yield determination.
6. Dissolve the precipitate by adding HCl dropwise. Dilute the solution with water to ~20 ml. Add 1 ml of saturated oxalic acid. Digest for 10 minutes at  $90^\circ\text{C}$ . Cool in a cold water bath. Filter through 2.4 cm glass fiber paper. Dry at  $110^\circ\text{C}$  for 30 minutes. Mount and beta count yttrium-90.
7. Evaporate the combined supernates to ~15 ml. Transfer the solution to a 40 ml centrifuge tube and adjust pH to 8 with  $\text{NH}_4\text{OH}$ . Add 15 ml saturated sodium carbonate and digest at  $90^\circ\text{C}$  for 30 minutes. Centrifuge, decant and discard supernate.

8. Gamma count strontium-85 by inserting the 40 ml centrifuge tube into the well of a gamma scintillation counter. Standardize by counting 1 ml of Sr<sup>85</sup> tracer precipitated with strontium carrier solution as carbonate as in Step 7. Calculate relative per cent recovery.

## EXPERIMENTAL

### Vegetation

After the initial sample preparation for vegetation samples, cerium-144 is most likely to be present as ceric oxide. Solubilizing ceric oxide and equilibration with carrier are effected by extracting with hydrochloric acid and hydrogen peroxide. This mixture produces a temperature increase which remains at 85 - 90°C during the extraction. The extraction process also effects equilibration of cesium-137 with carrier in vegetation samples. The non-uniformity of the amount of sulfate present in various vegetation samples necessitates further steps for equilibration of strontium-90 and added strontium carrier. This latter step is accomplished by combining the insoluble residue after extraction and the alkaline earth fraction in the soluble portion and performing a sodium carbonate fusion and water leach.

Experimental samples consisting of one spinach and five

wheats were analyzed for cerium-144, strontium-90 and cesium-137 by (1) sodium carbonate fusion and (2) hydrochloric acid-hydrogen peroxide extraction. These data are listed in Tables 1 and 2. The values of cerium-144 are consistently lower in each type sample after sodium carbonate fusion as compared to samples extracted. Cesium-137 values are comparable in each type sample when fused or extracted. Strontium-90 values are considerably lower for extracted wheat samples but are comparable for the spinach sample. The agreement between the strontium-90 values in spinach when fused or extracted may be attributed to the lower sulfate content compared to wheat.

Table 1. Results of Ce<sup>144</sup>, Sr<sup>90</sup>, and Cs<sup>137</sup> by Sodium Carbonate Fusion

Sample	HASL No.	pc/gm of ash			
		Ce <sup>144</sup>	Sr <sup>90</sup>	Cs <sup>137</sup>	
Spinach	581	1	15.98 ± .41	1.97 ± .09	1.16 ± .08
		2	14.04 ± .45	2.03 ± .09	1.14 ± .09
		3	16.38 ± .54	1.93 ± .12	1.07 ± .10
		4	15.12 ± .50	2.09 ± .09	1.23 ± .09
		5	13.73 ± .05	1.98 ± .05	1.24 ± .09
	Average		15.05 ± .37	2.00 ± .06	1.17 ± .07
Wheat	171 172 177 185 189		lost	6.31 ± .13	21.42 ± .41
			4.25 ± .75	5.62 ± .21	15.12 ± .23
			4.01 ± .26	4.29 ± .12	15.57 ± .32
			8.51 ± .32	6.12 ± .13	10.94 ± .23
			7.53 ± .44	5.70 ± .10	13.64 ± .27

Table 2. Results of Ce<sup>144</sup>, Sr<sup>90</sup>, and Cs<sup>137</sup> by Hydrochloric Acid-Hydrogen Peroxide Extraction.

Sample	HASL No.		pc/gm of ash		
			Ce <sup>144</sup>	Sr <sup>90</sup>	Cs <sup>137</sup>
Spinach	581	1	19.08 ± .36	1.97 ± .10	1.29 ± .09
		2	18.63 ± .45	2.03 ± .07	1.16 ± .07
		3	18.59 ± .50	1.86 ± .09	1.14 ± .08
		4	19.22 ± .45	2.01 ± .09	1.18 ± .09
		5	19.08 ± .45	2.04 ± .09	1.20 ± .09
	Average		18.92 ± .29	1.98 ± .07	1.19 ± .06
Wheat			Ce <sup>144</sup>	Sr <sup>90</sup>	Cs <sup>137</sup>
		171	3.00 ± .41	3.46 ± .27	21.74 ± .36
		172	4.86 ± .35	4.38 ± .18	14.76 ± .27
		177	5.18 ± .27	5.09 ± .149	15.80 ± .23
		185	10.58 ± .72	1.56 ± .22	9.99 ± .18
189	9.06 ± .44	3.87 ± .18	13.10 ± .23		

Table 3 compares the results obtained for strontium-90 by (1) hydrochloric acid-hydrogen peroxide extraction, (2) sodium carbonate fusion and (3) hydrochloric acid-hydrogen peroxide extraction followed by sodium carbonate fusion. Good agreement is shown between the latter two methods.

Table 3. Results of Sr<sup>90</sup> by Hydrochloric Acid-Hydrogen Peroxide Extraction and Sodium Carbonate Fusion.

Sample	HASL No.	pc/gm of ash		
		Extraction	Fusion <sup>24</sup>	Extraction & Fusion
Wheat	171	3.46 ± .27	6.31 ± .13	6.08 ± .11
	172	4.38 ± .18	5.62 ± .21	5.29 ± .11
	177	.509 ± .149	4.29 ± .12	4.32 ± .10
	185	1.56 ± .22	6.12 ± .13	5.67 ± .11
	189	3.87 ± .18	5.70 ± .10	5.80 ± .11

The range of recovery for added cerium carrier in vegetation samples is 75 - 90 per cent. Cesium and strontium recoveries range from 84 - 94 and 81 - 89 per cent, respectively. The purity of each cerium sample has been established by beta absorption measurement and by following the decay of the samples over several months. The purity of the cesium fraction was ascertained by the elution characteristics of the ammonium and alkali metal ions from the cation exchange column and the selective removal of cesium. Yttrium-90 samples were counted immediately after separation and recounted after two days. The error term associated with each result in Tables 1, 2, and 3 is expressed as a single Poisson counting error. All error terms associated with average results are expressed as a Gaussian error of the mean.

## Soil

Cerium-141 has been used as a tracer in the determination of cerium-144 in soil to determine overall chemical yield because of the existence of normal cerium in this type sample. The chemical form in which normal cerium exists is not known for specific soil types; however, it is assumed that it does exist as some accessory mineral constituent. Considering a large number of mineral forms in which cerium is present, either in large or small amounts, or in various proportions with other elements, many are soluble in their natural form in hydrochloric acid. The exceptions are those minerals containing niobates, tantalates, and titanates.

Two soil types which are available in large quantities in this laboratory were used in the experimental work with soils. These soils are commonly referred to as "red" and "black" and were taken from sample sites located at Raleigh, North Carolina, and McHenry County, Illinois, in the fall of 1958. Radiochemical analyses were performed during the latter part of 1962. The cerium-144 and strontium-90 results listed in Table 4 have been extrapolated to the sampling date and are reported as picocuries per kilogram. Normal cerium is reported in milligrams per kilogram.



Table 4. Replicate Analyses of Normal Cerium, Ce<sup>144</sup>,  
and Sr<sup>90</sup> in Two Soil Types.

Location	Sampling Date	Per Cent Recovered		mg Ce / kg	pc/kg		
		Ce <sup>141</sup>	Sr <sup>85</sup>		Ce <sup>144</sup>	Sr <sup>90</sup>	
Raleigh, N.C.	11-5-58	1	83	69	7.96	548 ± 61	64.1 ± 2.8
		2	90	74	7.80	554 ± 63	64.3 ± 2.2
		3	84	70	7.94	589 ± 68	65.8 ± 2.1
		4	88	71	9.22	576 ± 63	63.7 ± 2.6
		5	83	72	7.66	599 ± 67	64.0 ± 2.1
		6	92	70	7.72	546 ± 62	67.4 ± 2.2
		7	81	68	9.28	582 ± 61	66.0 ± 2.4
		8	87	73	9.04	572 ± 64	67.9 ± 3.3
		9	86	71	7.80	596 ± 67	64.2 ± 3.1
		10	89	72	8.64	548 ± 63	65.1 ± 2.5
	Average	86±4	71±2	8.31±0.66	571 ± 20	65.2 ± 1.4	
McHenry Co., Ill.	10-30-58	1	69	86	38.4	732 ± 91	86.3 ± 2.2
		2	67	84	37.7	769 ± 44	89.4 ± 2.8
		3	71	82	39.0	719 ± 67	78.5 ± 2.5
		4	65	87	38.8	724 ± 74	85.1 ± 2.7
		5	73	83	43.4	696 ± 77	86.7 ± 2.8
		6	69	87	39.6	741 ± 74	81.0 ± 2.6
		7	72	89	39.8	752 ± 48	83.2 ± 2.8
		8	74	81	36.7	708 ± 74	86.8 ± 2.8
		9	70	85	43.6	769 ± 54	81.5 ± 2.3
		10	68	80	41.7	702 ± 78	87.8 ± 2.7
	Average	73±3	84±3	39.9±2.3	731 ± 26	84.6 ± 3.5	

## MEASUREMENTS

In this report the measurement of cerium-144 is emphasized. The documentation of cerium-144 in various environmental samples is probably more significant when measured and compared with other nuclides of major importance, namely, strontium-90 and cesium-137.

Eight soil samples collected in 1960 were analyzed for normal cerium, cerium-144, and strontium-90 in the latter part of 1962. Cerium-144 and strontium-90 values are reported as picocuries per kilogram as of the sampling date. Normal cerium is reported in milligrams per kilogram. The results are reported in Table 5.

Radioecology studies agree that negligible concentrations of the rare earth nuclides are accumulated through the roots in plants. Also, measured amounts of these nuclides found with plants are the result of physical adherence. Wheat samples collected during the summer of 1962 were analyzed for cerium-144 in late 1963. The results are listed in Table 6. Also listed are strontium-90 and cesium-137 results obtained from the same samples. The reported values for each nuclide have been extrapolated to the middle of the sampling month. Of the fourteen

sites measured for cerium-144, 79 and 50 per cent of the values are higher than strontium-90 and cesium-137, respectively.

Table 7 reports the results of cerium-144, strontium-90, and cesium-137 for a sample of Kansas wheat separated into milling fractions. The material balance for this sample is poor for each of the nuclides. However, it is indicated that the patent flour fraction, which is used for human consumption, contains at least 10 per cent of the measured cerium-144. Bran, being the fraction exposed to the atmosphere contains the highest percentage. The reason for the presence of this percentage of cerium-144 in the patent flour fraction is not clearly known. Possibly, it may be attributed to cross mixing in the separation process for the different milling fractions.

#### SUMMARY

Using hydrochloric acid-hydrogen peroxide as extracting reagent, sequential radiochemical procedures are presented for the determination of  $Ce^{144}$ ,  $Sr^{90}$  and  $Cs^{137}$  in vegetation samples; and  $Ce^{144}$ , normal cerium and  $Sr^{90}$  in soils. Recoveries range from 75 - 90, 81 - 89 and 84 - 94 per cent respectively for cerium, strontium, and cesium in vegetation samples. Recoveries range from 68 - 92 for cerium and 68 - 89 per cent for strontium

in soils. The normal cerium content ranges from 8 - 137 milligrams per kilogram for the ten soils measured.

Table 5. Results of Normal Cerium, Ce<sup>144</sup> and Sr<sup>90</sup> in Soil.

Location	Sampling Date	BSL* No.	HASL No.	mg Ce / kg	pc/kg			
					Ce <sup>144</sup>		Sr <sup>90</sup>	
Manhattan, Kansas	11-2-60	6143	SO 123	39.3	414	± 24	133	± 13
St. Paul, Minnesota	8-4-60	60699	SO 008	23.9	256	± 11	82.8	± 7.1
		6155	SO 133	25.6	263	± 12	87.0	± 6.7
Singapore, Malaysia	12-12-60	611	SO 156	9.3	37.3	± 0.9	14.4	± 2.5
		6165	SO 185	10.0	43.5	± 5.0	12.1	± 2.2
Belem, Brazil	1-3-61	6113	SO 161	8.1	59.6	± 8.1	24.1	± 2.1
		6150	SO 183	7.5	63.2	± 9.6	23.0	± 2.0
Salisbury, So. Rhodesia	12-23-60	614	SO 159	48.9	11.8	± 11.8	9.5	± 2.2
		6174	SO 187	46.1	23.7	± 10.6	12.2	± 2.3
Durban, Union of S. Africa	12-21-60	6146	SO 126	27.9	35.2	± 5.9	17.9	± 1.8
Perth, Australia	12-6-60	6123	SO 166	130.4	69.7	± 9.1	16.0	± 1.7
		6179	SO 189	137.0	70.7	± 7.9	13.9	± 2.0
Alice Springs, Australia	12-6-60	6120	SO 163	33.3	119	± 15	17.7	± 2.1
		6178	SO 188	33.9	106	± 14	17.0	± 1.8

\*Beltsville Soil Laboratory, U.S. Dept. Agric., Beltsville, Md.

Table 6.  $Ce^{144}$ ,  $Sr^{90}$  and  $Cs^{137}$  in 1962 U.S. Wheat Crops.

<u>Location</u>	<u>Sampling Month</u>	<u>HASL No.</u>	<u>pc/kg</u>		
			<u>Ce<sup>144</sup></u>	<u>Sr<sup>90</sup><sup>25</sup></u>	<u>Cs<sup>137</sup><sup>26</sup></u>
California	June	174	97	14	41
Colorado	July	185	261	113	29
Idaho	August	186	69	21	70
Iowa	July	171	98	142	550
Kansas	June	179	126	92	280
Missouri	July	172	168	131	340
Montana	August	184	183	66	67
Nebraska	July	191	147	192	224
New York	August	178	88	103	269
North Dakota	August	177	163	103	157
Oklahoma	June	175	149	104	107
Texas	June	176	121	52	191
Utah	July	173	292	24	269
Washington	August	188	81	14	15

Table 7.  $Ce^{144}$ ,  $Sr^{90}$  and  $Cs^{137}$  in Kansas Wheat and Milling Products.

Sample Fraction	HASL No.	Relative Weights	pc/kg			pc in fractions		
			$Ce^{144}$	$Sr^{90^{25}}$	$Cs^{137^{26}}$	$Ce^{144}$	$Sr^{90}$	$Cs^{137}$
Wheat	179	1.00	126	92	280	126	92	280
Patent flour	181	0.58	39	17	58	23	10	34
Clear flour	183	0.14	256	58	76	36	8	11
Shorts	180	0.17	385	379	497	65	64	84
Bran	182	0.11	882	498	671	97	55	74
Total						221	137	203

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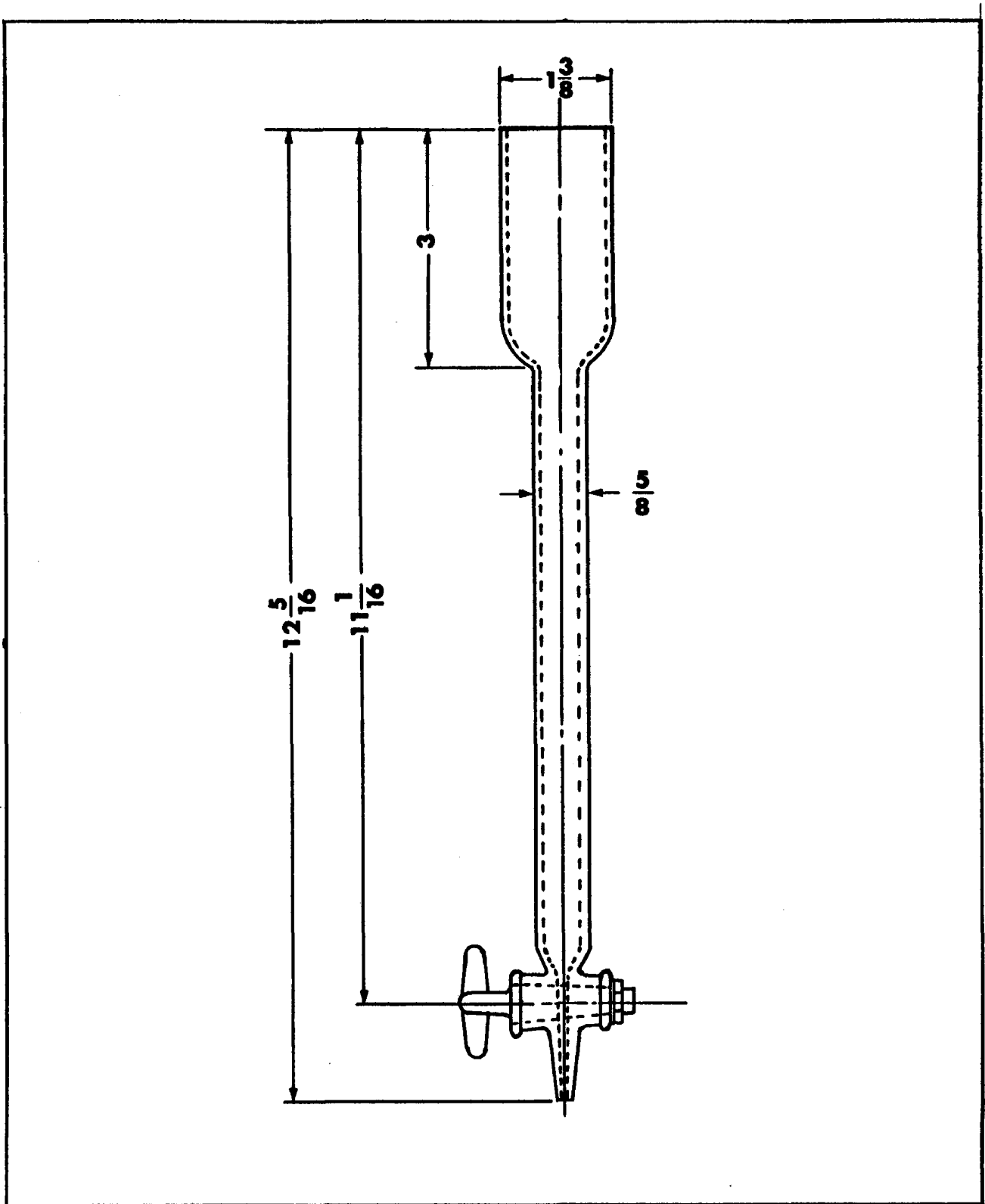


Figure 1. Ion Exchange Column





