

Analytical Method	<b>DETERMINATION OF SELECTED ACTINIDES AND STRONTIUM-90 IN WATER</b>	Identifier: ACMM-3815
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## 1. ABSTRACT

A liquid phase extraction technique is described for the selective separation of americium, curium, strontium, plutonium and uranium from water samples. Strontium and actinides are separated from water samples using the TEVA and TRU columns in a serial configuration. Strontium is separated by classical sulfate and carbonate precipitations and then the yttrium-90 is allowed to grow before being quantified by beta proportional counting. The actinides are coprecipitated with neodymium fluoride, mounted and quantified by alpha spectrometry.

## 2. APPLICABILITY

This method is designed to selectively separate strontium and actinides from water samples. Isotopic tracers are added to determine yield.

A Job Safety Analysis (JSA) was developed for this procedure in accordance with a determination made using MCP-3562 and MCP-3480.

## 3. DISCUSSION

The actinides are concentrated using an  $\text{Fe}(\text{OH})_3$  co-precipitation. The  $\text{Fe}(\text{OH})_3$  is dissolved in nitric acid. The Pu is adjusted to the +4 state using ascorbic acid and sodium nitrite. The actinides are purified using TEVA and TRU columns. The TEVA columns collect the thorium and plutonium, while the TRU columns collect the americium, curium, and uranium. The actinides are eluted separately from the columns except for americium and curium which are eluted together. The actinides are mounted for alpha counting using a neodymium fluoride co-precipitation.

Sr90 is collected and purified using  $\text{Fe}(\text{OH})_3$ ,  $\text{SrCO}_3$ , and  $\text{SrSO}_4$  precipitations. The Y90 daughter of the Sr90 is allowed to ingrow and then is purified and counted by gas proportional counting.

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#### 4. SAFETY PRECAUTIONS

- 4.1 Hot surfaces may be present. Use appropriate gloves and exercise caution to avoid contact. [JSA]
- 4.2 Acids and bases may cause chemical burns. Use care and appropriate PPE when handling. [JSA]
- 4.3 RCT coverage may be required when handling radiological samples as specified on the applicable Radiological Work Permit (RWP) [JSA]
- 4.4 The solutions being analyzed may be highly radioactive and care must be taken to limit personal exposure. [JSA]
- 4.5 Samples, not previously identified as radioactive, that are determined to be radioactive shall be handled under the appropriate RWP. [JSA]
- 4.6 When tracers must be added to the samples, this activity shall be in an RBA under a current RWP. [JSA]
- 4.7 All chemicals must be handled according to MCP-3635, Chemical Hygiene Plan. [JSA]
- 4.8 After samples have been concentrated, dried, muffled, desiccated, filtered and dried or other method or procedure that may have changed the concentration of radioactivity, an RCT must survey prior to moving sample to avoid the spread of contamination. [JSA]

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## 5. APPARATUS AND REAGENTS

### 5.1 Apparatus

- 5.1.1 0.100 µm polypropylene filters, 25 mm
- 5.1.2 Alpha spectroscopy system with multichannel analyzer
- 5.1.3 Avery stickers, ¾ inch, or equivalent
- 5.1.4 Balance, capable of reading 0.001 to 200 grams
- 5.1.5 Beakers, Pyrex, assorted sizes
- 5.1.6 Centrifuge
- 5.1.7 Centrifuge Tubes, 50 ml conical polypropylene
- 5.1.8 Glass fiber filters, 25-mm
- 5.1.9 Hot Plate, stirring
- 5.1.10 Infrared heat lamp
- 5.1.11 Gas flow proportional counter
- 5.1.12 Pipets, Eppendorf or equivalent, assorted sizes, with tips
- 5.1.13 Stir bars
- 5.1.14 pH paper
- 5.1.15 Tongs
- 5.1.16 Vacuum manifold and filtering apparatus for 25-mm filters.
- 5.1.17 Vortex mixer
- 5.1.18 Water bath
- 5.1.19 TEVA extraction columns available from EIChroM Industries, Inc. (Evanston, IL).
- 5.1.20 TRU extraction columns available from EIChroM Industries, Inc. (Evanston, IL).

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## 5.2 Reagents

Use only Analytical Reagent Grade chemicals and ASTM Type II or better water for preparation of all reagents. Instructions may be provided for forming the various reagents.

5.2.1 Below is a list of radionuclide tracer solutions and the approximate activities that are needed. The QC lab at INTEC can provide these solutions.

Am243:	0.1 Bq/ml
Pu242	0.1 Bq/ml
U232	0.1 Bq/ml
Sr90	1.5 Bq/ml
Am241:	0.1 Bq/ml
Pu240	0.1 Bq/ml
U238	0.1 Bq/ml
Sr85	30.0 Bq/ml

5.2.2 5% NaNO<sub>2</sub>: Dissolve 1 g sodium nitrite in 20 mL of water.

5.2.3 Acetic Acid: glacial.

5.2.4 Aluminum nitrate solution (50% by weight): 500 g of Al(NO<sub>3</sub>)<sub>3</sub> • 9H<sub>2</sub>O, per 1L of water

5.2.5 Ammonium bioxalate, (NH<sub>4</sub>)HC<sub>2</sub>O<sub>4</sub>, 0.1 M: Dissolve 14 g of ammonium oxalate, (NH<sub>4</sub>)<sub>2</sub>C<sub>2</sub>O<sub>4</sub> • H<sub>2</sub>O, and 7 g of oxalic acid, HOOC-COOH • 2H<sub>2</sub>O in 2L of water.

5.2.6 Ammonium hydroxide, NH<sub>4</sub>OH, concentrated.

5.2.7 Ammonium hydroxide, NH<sub>4</sub>OH, 3M: Dilute 20 mL of concentrated ammonium hydroxide to 100 mL.

5.2.8 Ascorbic acid solution, 10%: Dissolve 2 g in 10 mL of water. Prepare fresh before each use.

5.2.9 Bromocresol green (BCG), 0.04%: Dissolve 40 mg of the sodium salt of bromocresol green in 100 mL of water.

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5.2.10 Hydrochloric acid, HCl

- 12M: concentrated (38%, 12M)
- 9M: 750 mL concentrated HCl diluted to 1 L with water
- 6M: 500 mL concentrated HCl diluted to 1 L with water
- 4M: 330 mL concentrated HCl diluted to 1 L with water
- 1M: 83 mL concentrated HCl diluted to 1 L with water
- 0.5M: 42 mL concentrated HCl diluted to 1 L with water.

5.2.11 Hydrofluoric acid, HF, concentrated (49%).

5.2.12 Iron chloride, FeCl<sub>3</sub>, 10% solution: Dissolve 10 grams of FeCl<sub>3</sub> in 20 mL of 9M HCl and dilute to 100 mL with water.

5.2.13 Lithium metaborate, LiBO<sub>2</sub>

5.2.14 Lithium sulfate, LiSO<sub>4</sub>

5.2.15 Lithium sulfate, 5%: Dilute 250mL of 10% lithium sulfate to 500mL with water in a 500mL wash bottle.

5.2.16 Lithium sulfate, 10%: Dissolve 100 g of lithium sulfate in 1 L of water.

5.2.17 Neodymium carrier: 0.5mg/ml: Dissolve 0.583 g of neodymium oxide with 20 ml of 4M HCl and dilute to 1 liter with water.

5.2.18 Nitric acid, HNO<sub>3</sub>,

- 16M concentrated (69%, 16 M)
- 4M: 250 mL of concentrated HNO<sub>3</sub> diluted to 1 L with water
- 2.5M: 156 mL of concentrated HNO<sub>3</sub> diluted to 1 L with water
- 2.0M: 125 mL of concentrated HNO<sub>3</sub> diluted to 1 L with water.

5.2.19 Oxalic acid, 2%: Dissolve 10g of oxalic acid in 500mL of water in a 500mL wash bottle.

5.2.20 Oxalic acid, 5%: Dissolve 25g of oxalic acid in 500 mL of water in a 500 mL wash bottle.

5.2.21 Potassium hydroxide, KOH.

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5.2.22 Potassium carbonate,  $K_2CO_3$ , 2M: Dissolve 276 grams  $K_2CO_3$  in 1,000 mL water.

5.2.23 Reagent Alcohol.

5.2.24 Sodium ethylenediaminetetraacetate, 0.25M (EDTA), dissolve 73g of ethylenediaminetetraacetic acid in 50mL of 50% NaOH and about 500mL of water. Dilute to 1L with water and adjust the pH to 11.1. Filter through a 0.45  $\mu$ M DM- 450 membrane filter (Gelman Sciences, Ann Arbor, MI).

5.2.25 Sodium hydroxide, 50%, dissolve 500g of solid sodium hydroxide pellets in 750mL of water in a 2-L beaker. Dilute to a final volume of 1L and transfer to a 1-L PP bottle.

5.2.26 Sodium hydroxide, 0.25M, dilute 10mL of 50% sodium hydroxide to 500mL in a 500ml, wash bottle.

5.2.27 Strontium chloride,  
0.5%: Dissolve 5g of strontium chloride ,  $SrCl_2 \cdot (6H_2O)$ , in 100 mL of water.

5.2.28 Strontium carrier 100mg/mL, dissolve 30g of strontium chloride in 100 mL of water

5.2.29 Thymol blue indicator (TB) 0.04%: Dissolve 0.04 grams TB in 100 mL water

5.2.30 Titanous chloride solution 20%, available commercially.

5.2.31 Yttrium Carrier, 10mg/mL: Dissolve 6.456g of 99.9%  $Y_2O_3$  in 20mL of hot concentrated  $HNO_3$  and dilute to 500mL with water in a volumetric flask. Transfer the solution to two 250mL glass bottles with polyethylene lined screw caps.

## 6. SAMPLE HANDLING

6.1 Check water samples for a pH of 2 or less. If pH is greater than 2, add 5mL of concentrated nitric acid and wait 24 hours. Repeat if pH is still above 2.

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## 7. PROCEDURES

**NOTE:** *Not all sections are required to be performed. All steps within a given section are to be performed in sequence unless other instructions are provided.*

### 7.1 Fe(OH)<sub>3</sub> Preconcentration

7.1.1 Laboratory Analyst: Add the following tracer amounts to the sample

Am243 (for AM241):	0.5 Bq/ml
Pu242 (for Pu239/240 and Pu238)	0.5 Bq/ml
U232 (for U238, U235, and U234)	0.5 Bq/ml
Sr85 (for Sr90)	30.0 Bq/ml

7.1.1.1 Record the exact amount(s) of tracer used on the preparation log.

7.1.2 If sample is 1,000 mL or less, add 0.5 mL of 10% FeCl<sub>3</sub> solution to sample while stirring.

7.1.3 If sample is greater than 1,000 mL, add 1.0 mL of 10% FeCl<sub>3</sub> solution to sample while stirring.

7.1.4 Slowly add 50% NaOH to sample to establish a pH of 9 to 10 while stirring. (A rust-colored precipitate should form.)

7.1.5 Continue stirring sample for 10 minutes.

7.1.6 Remove the stir bar, and let the precipitate settle.

7.1.7 Decant and save the solution.

7.1.8 Transfer the precipitate to a 50-mL centrifuge tube with water.

7.1.9 Centrifuge and decant the solution.

7.1.10 Combine solutions from Steps 7.1.7 and 7.1.9 and save for use in Section 7.8.

7.1.11 Dissolve the precipitate with 1.5 mL of concentrated HNO<sub>3</sub> and transfer solution to a 50 ml-beaker.

7.1.12 Add 6 mL of 50% Al(NO<sub>3</sub>)<sub>3</sub> • 9H<sub>2</sub>O solution and dilute to 20 mL with water.

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## 7.2 Actinide Separation

- 7.2.1 Laboratory Analyst: Add 1 mL of 10% ascorbic acid and heat near boiling until sample turns yellow or for 10 minutes, then remove sample from heat.
- 7.2.2 Carefully add 1 mL of 5% NaNO<sub>2</sub> and heat at or near boiling for 10 minutes.
- 7.2.3 Adjust volume to 30 mL, with water and cool to room temperature before loading onto columns.
- 7.2.4 Stack a TEVA with a reservoir extension above a TRU column with a reservoir.
- 7.2.5 Condition the TEVA and TRU columns with 7 mL of 4M HNO<sub>3</sub>.
- 7.2.6 Load the samples onto the columns.
- 7.2.7 After the samples have passed through the columns, rinse columns with 5 mL of 4M HNO<sub>3</sub>.
- 7.2.8 Collect the load solution and the rinse for Sr analysis for use in Section 7.7.
- 7.2.9 Rinse the columns with an additional 7.5 mL of 4M HNO<sub>3</sub>.
- 7.2.10 Collect the rinse as waste.
- 7.2.11 Separate the columns.
- 7.2.12 GO TO the appropriate Section (7.3, 7.4, or 7.5) to proceed with elution.

## 7.3 TEVA Columns (Pu analysis)

- 7.3.1 Laboratory Analyst: Elute Thorium from TEVA columns with two 7.5 mL aliquots of 6M HCl.
- 7.3.1.1 Collect this "Thorium fraction" as waste.



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7.3.2 Elute Plutonium from the TEVA columns with 15 mL of 0.5M HCl and 0.20 mL of TiCl<sub>3</sub>. (Mix the HCl and the TiCl<sub>3</sub> just before pouring through columns).

7.3.2.1 Collect this "plutonium fraction" in centrifuge tubes and save for final precipitation and mounting for use in Section 7.6.

7.3.3 GO TO the appropriate Section (7.4, 7.5, or 7.6) to continue.

#### 7.4 TRU Columns (Am analysis)

7.4.1 Laboratory Analyst: Rinse the TRU columns twice with 7.5 mL of 4M HNO<sub>3</sub>.

7.4.1.1 Collect the rinse as waste.

7.4.2 Elute Americium from the TRU columns with 2 mL of 9M HCl followed by 15 mL of 4M HCl.

7.4.2.1 Collect this "americium fraction" in centrifuge tubes and save for the final precipitation and mounting for use in Section 7.6.

7.4.3 GO TO the appropriate section (7.5 or 7.6) to continue.

#### 7.5 TRU Columns (U analysis)

7.5.1 Laboratory Analyst: After the Am is eluted, rinse the TRU columns with two 10-mL aliquots of 0.03M oxalic acid in 1M HCl.

7.5.1.1 Collect the rinse as waste.

7.5.2 Elute Americium from the TRU columns with 20 mL of 0.1M ammonium bioxalate.

7.5.2.1 Collect this "Uranium fraction" in centrifuge tubes and save for the final precipitation and mounting.

#### 7.6 Final Precipitation and Mounting

7.6.1 Laboratory Analyst For U analysis only, add 0.5 mL of 20% TiCl<sub>3</sub> to each tube.

7.6.1.1 Mix and let stand at least 5 minutes.

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7.6.2 For Pu analysis only, If the Ti purple color does not persist from the elution process, add 0.2 mL of  $TiCl_3$ .

7.6.2.1 Mix and let stand at least 5 minutes.

7.6.3 To all fractions (U, Pu, and Am), add 0.2 mL of the 0.5 mg/mL Nd carrier to each centrifuge tube and mix.

7.6.4 Add at least 5 mL of cone HF and mix.

7.6.5 Wet a 0.1 micron Metricel filter (or equivalent) with reagent alcohol.

7.6.6 Set up the filtration apparatus with the filter.

7.6.7 Filter the sample:

7.6.7.1 Wash with a small amount of water.

7.6.7.2 Wash with a small amount of reagent alcohol.

7.6.7.3 For Am samples, Wash with two additional small aliquots of reagent alcohol.

7.6.8 Mount the filter (precipitate side up) to a round self-adhesive numbered label with the ID of the sample written on it.

7.6.9 Dry the filters.

7.6.10 Place the filters in the alpha chamber for counting.

## 7.7 **Sr-90**

7.7.1 Laboratory Analyst: Split solution from 7.2.8 into 50 mL centrifuge tubes containing no more than 30 mL per tube.

7.7.2 Add and dissolve 3.5 grams of  $Li_2SO_4$  in each tube.

7.7.3 Add 0.1 mL of 100 mg/mL Sr carrier to each tube.

7.7.3.1 Mix each and wait at least 3 minutes (A strontium sulfate precipitate will form).

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7.7.4 Add five 3-mL aliquots of 0.5% SrCl<sub>2</sub> to each tube.

7.7.4.1 Mix and wait at least 3 minutes after each addition.

7.7.5 Centrifuge, decant, and discard solution.

## 7.8 SrCO<sub>3</sub> Preconcentration

7.8.1 Laboratory Analyst: Adjust the pH of the supernate from the hydroxide precipitation from 7.1.10 to a pH of 12 using 50% NaOH and pH paper.

7.8.2 Add 40 mL of 2M KCO<sub>3</sub> for every liter of sample and stir for 5 minutes.

7.8.3 Add 0.1 mL of 100 mg/mL Sr carrier.

7.8.3.1 Mix and wait at least 3 minutes (a strontium carbonate precipitate will form).

7.8.4 Add five 3-ml aliquots of 0.5% SrCl<sub>2</sub>, mixing and waiting at least 3 minutes after each addition.

7.8.5 Centrifuge, decant, and discard the solution.

## 7.9 Sr-90 via Y-90

7.9.1 Laboratory Analyst: To the precipitate from Section 7.7.5, add 10 mL of 0.25M EDTA, 5 drops of TB, vortex, and heat in a boiling water bath until the precipitate has dissolved completely.

7.9.2 Transfer the solution to the centrifuge tube containing CO<sub>3</sub> precipitate from Section 7.8.5.

7.9.3 If the solution fades from blue during the dissolution, add 50% NaOH dropwise to the blue endpoint of the TB.

7.9.4 When all the precipitate has been dissolved, add 1 to 2 drops of 10% Fe Cl<sub>3</sub> and 1 to 2 drops of 50% NaOH.

7.9.5 Heat the sample in a boiling water bath for 2 to 3 minutes. (iron hydroxide precipitates and will gather any interfering actinides and about 50% of the Ra.)

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7.9.6 Centrifuge the solution for 20 minutes and decant into another 50-mL centrifuge tube.

7.9.6.1 Save the iron hydroxide precipitate for possible reanalysis.

7.9.7 Swirl the solution, and add 10 mL of 10% lithium sulfate and 4 drops of bromocresol green (BCG).

**NOTE:** *If the acidity of the solution is increased much further, calcium sulfate will precipitate with the strontium sulfate and might not dissolve in the limited amount of EDTA in the subsequent dissolution. If the pH of the solution is much higher than 4.0, the strontium sulfate will be precipitated incompletely.*

7.9.8 Add HCl dropwise until the solution turns light blue-green.

7.9.8.1 Add three 1-mL portions of glacial acetic acid to the yellow endpoint of the bromocresol green to precipitate strontium sulfate (pH of 4).

7.9.8.2 Record this time as the start time for the <sup>90</sup>Y ingrowth.

7.9.9 Heat the precipitated solution in a boiling water bath for 5 minutes.

7.9.10 Centrifuge the precipitate for 12 minutes.

7.9.11 Decant the supernate. If there is any question as to the completeness of precipitation, count the supernate for Sr-85 before discarding.

7.9.12 Add 10 mL of 0.25M EDTA, and 3 drops of thymol blue to the precipitate, vortex to suspend the precipitate, then add 50% NaOH dropwise to the blue endpoint of the indicator and vortex.

7.9.13 Place the centrifuge tube in a bath of boiling water for 5 minutes to dissolve the precipitate completely.

7.9.14 Allow to cool and then count for 300 seconds for Sr-85 to determine the strontium yield.

7.9.15 Cap the tube to prevent evaporation and set aside for at least 7 days to permit <sup>90</sup>Y to ingrow to at least 90% of equilibrium with the <sup>90</sup>Sr.

7.9.16 After <sup>90</sup>Y ingrowth, add 1.0 mL of yttrium carrier (10mg/mL) and mix.

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7.9.17 Add 5.0g of KOH.

7.9.18 Heat in a boiling water bath for 15 minutes to ensure complete precipitation of yttrium hydroxide.

7.9.18.1 Record this time as the end of the  $^{90}\text{Y}$  ingrowth.

7.9.19 Centrifuge the solution for 5 minutes while still hot.

7.9.20 Decant and save the supernate for possible reanalysis.

7.9.21 Wash the precipitate with 10 mL of 0.25M NaOH.

7.9.22 Centrifuge, decant and discard the wash.

7.9.23 Dissolve the hydroxide precipitate in 5 mL of 4M nitric acid then vortex.

**NOTE:** *The yttrium purification steps will purify the yttrium from radium isotopes, but not from the Ac228 daughter of Ra228.*

7.9.24 Swirl the solution and add 3 drops of thymol blue and 5mL of 5% oxalic acid.

7.9.25 Add 3M  $\text{NH}_4\text{OH}$  dropwise while swirling to the last shade of pink, but not the yellow endpoint of the indicator (pH).

7.9.26 Heat in a boiling water bath for 5 minutes

7.9.27 Allow to cool and then centrifuge for 5 minutes.

7.9.28 Decant and discard the supernate.

7.9.29 Add 5mL of 4M  $\text{HNO}_3$  to the centrifuge tube to dissolve the yttrium, oxalate and vortex.

7.9.30 Add 5 mL of 5% oxalic acid and 3 drops of thymol blue to the solution.

7.9.31 Swirl the solution and add about 1.5 mL of concentrated  $\text{NH}_4\text{OH}$

7.9.32 Let solution sit for 15 minutes, then continue to add concentrated  $\text{NH}_4\text{OH}$  dropwise to the pink endpoint of the indicator to reprecipitate yttrium oxalate.

7.9.33 Heat the precipitated solution in a boiling water bath for 5 minutes.

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7.9.34 Filter the precipitate on a well washed, tared, 25mm glass fiber filter paper in an all glass filtering chimney.

7.9.35 Wash the precipitate with 5 mL of 2% oxalic acid followed by 5mL of reagent alcohol.

7.9.36 Dry the precipitate at a distance of about 8 inches from a 250-watt infrared lamp for 20 to 25 minutes.

7.9.37 Weigh the dried filter paper to determine the yttrium yield.

7.9.37.1 Record weight to 0.1 mg on preparation log..

7.9.38 Mount the filter paper in a sample holder and count in a gas flow proportional counter for a time long enough to obtain the statistical precision desired.

#### 7.10 **Determination of Counting Efficiency for Yttrium-90 on Yttrium Oxalate**

7.10.1 Laboratory Analyst: Add the following to a 50-mL conical polypropylene centrifuge tube:

- 5 mL of 4M HNO<sub>3</sub>
- 1 mL of 10 mg/mL yttrium carrier
- 1.0 mL of 5 mg/mL strontium carrier
- 1 mL of a standard solution of strontium-90 at an activity of about 30,000 dpm/mL
- 5 mL of 5% oxalic acid
- 5 drops of 0.04% TB. and dilute to 20 mL with water.

7.10.2 Swirl the centrifuge tube to mix the solution thoroughly and add concentrated ammonium hydroxide dropwise until the red color of the indicator just fades to pink (pH 2).

7.10.3 Record the time as the beginning of the yttrium-90 decay.

7.10.4 If the indicator endpoint is overshot and yellow solution is obtained, quickly add up to 5 drops of 2M nitric acid to the first permanent pink color.

7.10.5 Heat the solution in a boiling water bath for 5 minutes.

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- 7.10.6 Cool solution and centrifuge solution for 5 minutes.
- 7.10.7 Decant and discard the supernate.
- 7.10.8 Wash the precipitate with a forceful jet of 5 mL of 2% solution of oxalic acid directed from a wash bottle and mix.
- 7.10.9 Centrifuge, decant, and discard the supernate.
- 7.10.10 Add 5 mL of 4M nitric acid and heat in a boiling water bath until the precipitate has dissolved.
- 7.10.11 Cool the solution to room temperature and add 5 mL of 5% oxalic acid, 3 drops of TB, and dilute to 20 mL with water.
- 7.10.12 Precipitate yttrium oxalate by adding concentrated ammonium hydroxide dropwise until the indicator fades to a light pink.
- 7.10.13 If the indicator endpoint is overshoot and yellow solution is obtained, quickly add up to 3 drops of 2M nitric acid to the first permanent pink color.
- 7.10.14 Mount the strontium-free yttrium oxalate nonahydrate precipitate on a tared 25-mm glass fiber filter paper in a all glass filtering chimney.
- 7.10.15 Wash the precipitate with 5 mL of 2% oxalic acid and then with 5 mL of reagent alcohol.
- 7.10.16 Dry the precipitate at a distance of about 8 inches from a 250-watt infrared lamp for 20 to 25 minutes.
- 7.10.17 Weigh the precipitate to determine the yttrium yield.
- 7.10.18 Count the mounted precipitate in the low background beta counter to determine the counting efficiency of yttrium-90 as 34 mg of yttrium oxalate nonahydrate.
- 7.10.19 Record the time at which half of the counting time has elapsed as the end of the yttrium-90 decay.

## 8. QUALITY CONTROL REQUIREMENTS

- 8.1 Analyze a blank with each batch. Blank values are used during calculations for accurate results.

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- 8.2 Analyze a control sample with each batch. Repeat any control that is beyond 20% of known values or beyond the acceptance criteria specified by the customer.
- 8.3 Analyze any additional QC samples as required by project requirements.
- 8.4 If the strontium yield is below 35% recovery, contact Technical Supervisor for possible reanalysis of saved fractions.

## 9. CALCULATIONS

The SUN/Analytical computer is programmed to calculate the activity of each isotope. The method requires that a daily control be passed before the analyst can enter results. If hand calculations are necessary, they can be performed according to the following equations:

NOTE: *If the following equations are used, the appropriate dilution factors must be used as applicable.*

### 9.1

Where:

cnts @ 5.499 MEV = total counts in Pu-238 peak.  
cnts @ 5.74 MEV = total counts in Pu-236 spike peak.  
SA = spike activity added in d/s.  
V = volume of sample in mL.

### 9.2

Where:

cnts @ 5.15 MEV = total counts in Pu-239 peak.  
cnts @ 5.74 MEV = total counts in Pu-236 spike peak.  
SA = spike activity added in d/s.  
V = volume of sample in mL.



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9.3

Where:

cnts @ 5.46 MEV = total counts in Am-241 peak.  
cnts @ 5.25MEV = total counts in Am-243 spike peak.  
SA = spike acitvity added in d/s.  
V = volume of sample in mL.

9.4

Where:

cnts @ 5.8 MEV = net counts in Cm-244 peak.  
cnts @ 5.25 MEV = net counts in Am-243 peak.  
SA = spike activity added in d/s (Am-243).  
V = volume of sample in mL

9.5

Where:

U-2XX = uranium isotope of interest (i.e., U-234,235,238).  
cnts @ 5.3 MEV = net counts in U-232 spike peak.  
SA = spike activity added in d/s.  
V = volume of sample in mL  
BR = branching ratio of the uranium isotope.

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9.6

Use approved calculation program or the following equations:

$$\text{Sr-90, } \mu\text{Ci/g} = (Y - YB) / (CTY * CEY * g * SY * YY * YG * YD * 2.22 \text{ E6})$$

$$S^2 \text{Sr-90} = \text{SQR}(A^2 + B^2 + C^2 + D^2 + E^2 + F^2 + G^2) * \text{Sr-90}^2$$

Where:  $A = \text{SQR}(Y + YB) / (Y - YB)$

$$B = \text{SCEY} / \text{CEY}$$

$$C = \text{SG} / g$$

$$D = \text{SSY} / \text{SY}$$

$$E = \text{SYY} / \text{YY}$$

$$F = \text{SYG} / \text{YG}$$

$$G = \text{SYD} / \text{YD}$$

and:  $Y =$  Gross counts of Y-90

$YB =$  Background of Y-90

$CTY =$  Count time in minutes

$g =$  Sample size

$SG =$  Standard deviation of the sample size.

$SY =$  Strontium yield

$SSY =$  St. Dev. of SY

$YY =$  Yttrium yield

$SYY =$  St. Dev. of YY

$YG =$  Yttrium growth =  $1 - \exp(-\text{Ln } 2 * T1 / 64)$

$SYG =$  St. Dev.  $YG = (\text{Ln } 2 * T1 / 64) * \text{SQR}((\text{ST1} / T1)^2 + (.1 / 64)^2)$

$YD =$  Yttrium decay =  $\exp(-\text{Ln } 2 * T2 / 64)$

$SYD =$  St. Dev. of  $YD = (\text{Ln } 2 * T2 / 64) * \text{SQR}((\text{ST2} / T2)^2 + (.1 / 64)^2)$

$T1 =$  Time of Y-90 ingrowth in hours

$ST1 =$  St. Dev. of T1

$T2 =$  Time of Y-90 decay in hours

$ST2 =$  St. Dev. of T2

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CEY = Counting efficiency of Y-90 on yttrium oxalate.

SCEY = St. Dev. of CEY

SQY ( ) = Square root of the quantity in the ( )

exp- ( ) = The antilog of minus the quantity in the ( )

## 10. RECORDS

The records listed below are generated by the Analytical Laboratories Department and are managed in MCP-2007, Analytical Records Management.

- Data printouts
- Data reports
- Preparation logs
- Raw data files

## 11. REFERENCES

- 11.1 JSA ACMM-3815
- 11.2 MCP-2001, Control of Analytical Methods and Procedures
- 11.3 MCP-2007, Analytical Records Management
- 11.4 MCP-3562, Hazard Identification, Analysis & Control of Operational Activities
- 11.5 MCP-3635, Chemical Hygiene Plan
- 11.6 MCP-3480, Environmental Instructions for Facilities, Processes, Materials and Equipment

## 12. SUPPLEMENTAL INFORMATION

### 12.1 History of ACMM-3815:

Revision	Author	Date
0	Robert Hague	MAY 2000

### 12.2 REVISION SUMMARY

Existing RML method instructions have been converted into ACMM-3815 per MCP-2001. Revision number established as 0.

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**13. APPROVAL SIGNATURE BLOCK**

Position Title	Signature	Date
Method Author	<i>Robert H. Hays</i>	5/24/2000
Responsible Technical Leader	<i>Robert H. Hays</i>	5/24/2000
Responsible Supervisor	<i>Joseph P. Henshel</i>	5/24/00
ALD QA Officer	<i>Shelby A. ...</i>	5/24/00
ALD Manager	<i>RD ...</i>	5/24/00
ALD Facility Manager	<i>Joseph P. Henshel</i>	5/24/00

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**Appendix A**

**Method Basis**

Step/Section	Basis/Summary	Source
4.1 through 4.8,	Controls are implemented to adequately mitigate hazards identified by JSA.	JSA ACMM-3815