

RESL TECHNICAL PROCEDURE

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CHEM-TP-SR.7

CARBONATE METHOD FOR ⁹⁰Sr

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TITLE: CHEM-TP- SR.7, CARBONATE METHOD FOR ⁹⁰Sr

PURPOSE

The purpose of this procedure is to determine ⁹⁰Sr in environmental and biological samples.

APPLICABILITY

This procedure is applicable to low-activity environmental water samples such as United States Geological Survey (USGS) well water samples, surface, production, and monitor well waters and to large environmental water samples, such as the 4-L production well water samples from INTEC. It is applicable to no more than 5 g of ash from foodstuffs and vegetation; to the ash of 1 L of milk and of 500 g of wheat. This procedure is applicable to urine samples when ⁹⁰Sr-only is requested, and when actinide analyses such as uranium, plutonium, americium, plus ⁹⁰Sr in a urine sample are requested.

RESPONSIBILITIES

RESL staff responsible for implementing this procedure are:

Radiochemist(s)

DEFINITIONS

H₂O - Distilled or demineralized water.

PROCEDURE

1 ABSTRACT

The procedure is designed to accommodate a wide range of sample types while providing high and reliable decontamination factors from a host of other fission and activation products. An initial separation of strontium as the carbonate is carried out on the majority of the liquid samples. Stable elements, particularly calcium and magnesium, limit the size of aliquant which can be analyzed or make it necessary to employ additional steps in the procedure. Some types of samples contain relatively large quantities of calcium and phosphate and are wet or dry ashed. After treatment of the ashed residue, strontium is separated with calcium by one or more phosphate precipitations. The carbonate and phosphate precipitates are treated with nitric acid to convert strontium to the nitrate and separate it from calcium, phosphate and many fission and activation products. After a suitable ingrowth period, ⁹⁰Y is separated and counted on Y₂(C₂O₄)₃•9H₂O. The ⁹⁰Sr concentration is determined by beta counting its ⁹⁰Y daughter. The chemical yield for strontium is determined by gamma counting ⁸⁵Sr tracer and only the ingrown ⁹⁰Y is counted on Y₂(C₂O₄)₃•9H₂O.

2 ES&H PRECAUTIONS

2.1 Take proper precautionary measures. This procedure calls for the use of 49% HF, 70% HClO₄, 15 M H₃PO₄, 12 M HCl, 21 M HNO₃, 16 M HNO₃, 10 M NaOH, and

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15 M NH_4OH . Use a fume hood. Wear gloves, safety glasses/face shield, and protective clothing.

- 2.2 Refer to RE SL-TP-IH.9 when working with hydrofluoric acid.
- 2.3 Refer to RE SL-TP-IH.4 for the handling of corrosive chemicals.
- 2.4 Refer to RE SL-TP-IH.1 for eye protection.
- 2.5 Refer to RE SL-TP-IH.2 for general laboratory safety.
- 2.6 Refer to CHEM-AP.11 for proper management of chemicals.
- 2.7 Refer to RE SL-TP-IH.15 for acid and base neutralization.
- 2.8 Refer to RE SL-AP.10 for waste management.

3 APPARATUS

- 3.1 Perchloric acid fume hood
- 3.2 Hotplate, 3600 W, 46 x 61 cm
- 3.3 Fiberglass mat, 1.6-mm thick, to cover hotplate
- 3.4 Centrifuge with a 12-place rotor, trunnions, and cups for 100-mL centrifuge tubes; 6-place rotor and cups for 1-L polypropylene bottles with the necks cut off just below the shoulder.
- 3.5 Glass fiber GF/A filters, 2.4 cm
- 3.6 Filtering apparatus: custom-made, black Teflon filtering chimney, 2-cm inside diameter (i.d.); custom-made, white Teflon filter holder and frit, or a comparable Commercial filtering apparatus, for example: Fisher Scientific (Vacuum Filter Holder #09-753E)
- 3.7 Heat lamp, 250 W
- 3.8 PVC filter holder: custom-made, with locking ring for 2.4-cm GF/A glass fiber filters
- 3.9 DM-450 filter, 47 mm
- 3.10 4-L beakers
- 3.11 2-L beakers
- 3.12 600-mL beakers

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4 REAGENTS

- 4.1 Calcium Carrier, 25 mg/mL: Dissolve 73.65 g of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ in H_2O and dilute to 500 mL with H_2O . Filter the solution through a DM-450 filter.
- 4.2 Metacresol Purple Indicator, 0.1%: Dissolve 0.25 g of m-cresolsulfonephthalein in H_2O and add 1 mL of 10 M NaOH. Dilute to 250 mL with H_2O and filter through a DM-450 filter.
- 4.3 Oxalic Acid-dihydrate, 5%: Dissolve 50 g of $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ in 1 L of H_2O and filter through a DM-450 filter.
- 4.4 Phenolphthalein Indicator, 1%: Dissolve 2.5 g of phenolphthalein in 125 mL of 95% ethyl alcohol and dilute to 250 mL with H_2O .
- 4.5 Sodium Carbonate, Saturated: Add 200 to 235 g of Na_2CO_3 to 1 L of H_2O in a plastic bottle. Seal the bottle and shake vigorously. Let the solution come to equilibrium and filter it through a DM-450 filter.
- 4.6 Sodium Hydroxide, 10 Molar: Slowly add 400 g of NaOH pellets to 1 L of H_2O in a 2-L beaker while stirring the solution vigorously. This reaction is very exothermic. Therefore, use all necessary safety precautions and prepare this reagent on a heat-resistant surface. After the NaOH is dissolved in the water and the solution has cooled, filter the solution through a DM-450 filter. Store in a plastic bottle.
- 4.7 Strontium Carrier, 50 mg/mL: Add 42.5 g of reagent grade SrCO_3 to 450 mL of 1.5 M HNO_3 . Transfer the solution to a 500-mL volumetric flask and dilute to 500 mL with H_2O . Filter the solution through a DM-450 filter.
- 4.8 Yttrium Carrier, 10 mg/mL: Dissolve 6.35 g 99.9% of Y_2O_3 in 40 mL of 8 M HNO_3 by heating in a bath of boiling water. Allow the solution to cool. Transfer the solution to a 500-mL volumetric flask and dilute to volume with 0.5 M HNO_3 . Filter the solution through a DM-450 filter. Determine the $\text{Y}_2(\text{C}_2\text{O}_4)_3 \cdot 9\text{H}_2\text{O}$ gravimetric factor for the current yttrium carrier solution as described in CHEM-TP-CA.9.

5 SAMPLE PREPARATION

5.1 Environmental Water Samples

- 5.1.1 Perform analysis of aliquants corrected to compensate for acidification. For example, take 400 mL to obtain 392 mL of an environmental water sample which has been acidified to 2% v/v with 12 M HCl. Add the 400-mL sample to a 600-mL beaker containing 1 mL of strontium carrier, 25 mg of calcium carrier and approximately 30,000 cpm of ^{85}Sr tracer.
- 5.1.2 Prepare a reagent blank in the same way using H_2O in place of the sample.

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- 5.1.3 Add the same quantity of ^{85}Sr tracer to a 90-mL polystyrene counting bottle; then add 10 mL of 8 M HNO_3 and 10 mL of H_2O and save for use as a standard in Step 5.5.4.
- 5.1.4 Add 2-3 boiling chips and evaporate the solution to a volume of 30 to 50 mL on the hotplate. Transfer the hot sample to a 100-mL centrifuge tube. Use 5 to 10 mL of H_2O from a wash bottle to complete the transfer of the solution and to rinse the beaker. Combine the rinse with the sample solution.
- 5.1.5 Add 2 drops of phenolphthalein indicator and adjust the pH to the indicator endpoint (pink) with 10 M NaOH while vigorously swirling the solution. Continue swirling and add 5 mL of saturated Na_2CO_3 . Digest the sample solution for 5 min in a boiling water bath and then cool to room temperature in a bath of cold, running tap water. Balance the tubes and centrifuge the sample for 5 min at 2400 rpm. Decant and discard the supernate.
- 5.1.6 Dissolve the carbonate precipitate with 5 mL of 8 M HNO_3 .
- 5.1.7 Continue at Step 5.4 with the nitrate precipitation.
- 5.2 INTEC Water Samples Or Large Volume Environmental Water Samples
- 5.2.1 Use a 2-L graduated cylinder to measure, in 2 portions, 4 L of the water sample. Add the first 2 L of water to a 4-L beaker containing 1 mL of strontium carrier, approximately 30,000 cpm of ^{85}Sr tracer and 4 boiling chips. Use 30 mL of 12 M HCl to rinse the sample bottle. Combine this acid rinse with the sample in the beaker. Rinse the bottle with approximately 20 mL of H_2O and again combine it with the sample in the beaker. Evaporate the first 2-L portion of the sample and rinse to about 500 mL before adding the second 2-L portion of the sample to the beaker. Evaporate the entire sample to 200 to 300 mL.
- 5.2.2 Prepare a reagent blank in the same way, using H_2O in place of the sample and add 100 mg of Ca^{2+} .
- 5.2.3 Add the same quantity of ^{85}Sr tracer to a 90-mL polystyrene counting bottle; then add 10 mL of 8 M HNO_3 and 10 mL of H_2O and save for use as a standard in Step 5.5.4.
- 5.2.4 Transfer the sample (from Step 5.2.1) to a 1-L polypropylene centrifuge bottle. Use 10 to 20 mL of H_2O from a wash bottle to complete the transfer of the sample into the centrifuge bottle. Combine the rinse with the sample.
- 5.2.5 Add 4 drops of phenolphthalein indicator to the sample and adjust the pH to the indicator endpoint (pink) with 15 M NH_4OH while stirring the solution with a stirring rod.
- 5.2.6 Add 15 to 20 g of solid $(\text{NH}_4)_2\text{CO}_3$ and cover the 1-L centrifuge bottle with a watch glass. Digest the sample for 15 min in a boiling water bath. Cool the sample overnight (if possible) or to room temperature in a bath of cold, running tap water.

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- 5.2.7 Balance the 1-L centrifuge bottles and centrifuge the sample for 10 min at 2000 rpm. Decant and discard the supernate.
- 5.2.8 Dissolve the carbonate precipitate with 5 mL of 6 M HCl. Transfer the solution to a 100-mL centrifuge tube. Use 5 to 10 mL of H₂O from a wash bottle to complete the transfer of the sample into the centrifuge tube. Total volume of the sample and rinse should not exceed 40 to 50 mL.
- 5.2.9 Add 2 drops of phenolphthalein indicator and adjust the pH to the indicator endpoint (pink) with 15 M NH₄OH while swirling the solution.
- 5.2.10 Add 3 to 5 g of solid (NH₄)₂CO₃ and cover the centrifuge tube with a watch glass. Digest the sample for 15 min in a warm (30-40°C) water bath so that the CO₂ which will form does not cause the sample to foam out of the centrifuge tube.
- 5.2.11 Cool the sample to room temperature in a bath of cold, running tap water. Balance the tubes and centrifuge the samples for 5 min at 2400 rpm. Decant and discard the supernate.
- 5.2.12 Dissolve the carbonate precipitate with 5 mL of 8 M HNO₃.
- 5.2.13 Continue at Step 5.4 with the nitrate precipitation.
- 5.3 Samples Which Require Phosphate Precipitation
- 5.3.1 Except for milk and wheat, ashed foodstuff and vegetation samples require total dissolution of the ash followed by an initial phosphate separation of strontium and calcium. Add calcium carrier to some ashed samples to help precipitate strontium; for example, lettuce ash contains little calcium and 100 mg of Ca²⁺ must be added.
- 5.3.1.1 Add the ash to a 400-mL Teflon beaker containing calcium carrier (if needed), 1 mL of strontium carrier, and approximately 30,000 cpm of ⁸⁵Sr tracer. Add 40 mL of 16 M HNO₃ and 40 mL of 12 M HC 10₄.
- 5.3.1.2 Prepare a reagent blank in the same way.
- 5.3.1.3 Add the same quantity of ⁸⁵Sr tracer to a 90-mL polystyrene counting bottle; then add 10 mL of 8 M HNO₃ and 10 mL of H₂O and save for use as a standard in Step 5.5.4.
- 5.3.1.4 Cover the beaker with a watch glass and heat until perchloric acid fumes evolve. Wash the solids and organic distillates from the cover and beaker sides, as needed, with 5 to 10 mL of 16 M HNO₃.

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5.3.1.5 Add 40 mL of 12 M HCl, 15 mL of 49% HF, and approximately 10 mL of 16 M HNO₃ when the organic material has been destroyed. Evaporate the solution in the uncovered beaker to approximately 10 mL in volume.

CAUTION: TAKE PROPER SAFETY PRECAUTIONS WHEN USING THESE STRONG ACIDS AND EVAPORATING THEM TO A SMALL VOLUME.

5.3.1.6 Transfer the solution to a 2-L beaker using H₂O from a wash bottle to complete the transfer of the sample and to rinse the Teflon beaker. Combine the rinse with the sample solution.

5.3.1.7 Continue at Step 5.3.5 with phosphate precipitation.

5.3.2 Milk and Wheat Ash

5.3.2.1 Add the milk or wheat ash to a 600-mL beaker containing 1 mL of strontium carrier and approximately 30,000 cpm of ⁸⁵Sr tracer.

5.3.2.2 Prepare a reagent blank in the same way, except 100 mg of Ca²⁺ must also be added.

5.3.2.3 Add the same quantity of ⁸⁵Sr tracer to a 90-mL polystyrene counting bottle; then add 10 mL of 8 M HNO₃ and 10 mL of H₂O and save for use as a standard in Step 5.5.4.

5.3.2.4 Add 250 mL of 2 M HCl to the ash slowly. Cover the beaker with a watch glass and boil the solution for at least 15 min on the hotplate.

5.3.2.5 Use a large bell jar to filter the warm solution with suction through a 47-mm DM-450 filter into a 2-L beaker. Rinse the 600-mL beaker with 50 mL of boiling H₂O, and wash the insoluble material by filtering this rinse into the 2-L beaker.

5.3.2.6 Continue at Step 5.3.5 with phosphate precipitation.

5.3.3 Urine Samples Requiring Only ⁹⁰Sr Analysis

5.3.3.1 Add up to 1800 mL of raw urine (in two or more portions with evaporation between additions) to a 2-L beaker containing 1 mL of strontium carrier and approximately 30,000 cpm of ⁸⁵Sr tracer.

5.3.3.2 Prepare a reagent blank in the same way, except 100 mg of Ca²⁺ must also be added.

5.3.3.3 Add the same quantity of ⁸⁵Sr tracer to a 90-mL polystyrene counting bottle; then add 10 mL of 8 M HNO₃ and 10 mL of H₂O and save for use as a standard in Step 5.5.4.

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- 5.3.3.4 Use 50 mL of 16 M HNO₃ to rinse the sample bottle. Combine the rinse with the sample. Add 250 mL of 16 M HNO₃ to the urine and cover the beaker with a watch glass.
- 5.3.3.5 Evaporate the sample to dryness, and carefully heat it to a char.

CAUTION: THE SAMPLE CAN IGNITE DURING CHARRING. REMOVE THE BEAKER FROM THE HOTPLATE IF RAPID CHARRING BORDERING ON IGNITION TAKES PLACE, AND ALLOW THE SAMPLE TO COOL.

- 5.3.3.6 Add 50 mL of 16 M HNO₃ and heat the sample again to a char in the beaker covered with a watch glass. Repeat until the entire sample has charred, leaving a light brown to white or pale yellow residue.
- 5.3.3.7 Add 250 mL of 2 M HCl and 5 mL of calcium carrier to the residue in the beaker.
- 5.3.3.8 Continue at Step 5.3.5 with phosphate precipitation.

5.3.4 Urine Samples after Actinide Separation

- 5.3.4.1 Add 1 mL of the strontium carrier and approximately 30,000 cpm of ⁸⁵Sr tracer to the sample prior to wet ashing and actinide separations in CHEM-TP-A. 16.
- 5.3.4.2 Prepare a reagent blank in the same way, except 100 mg of Ca²⁺ must also be added.
- 5.3.4.3 Add the same quantity of ⁸⁵Sr tracer to a 90-mL polystyrene counting bottle; then add 10 mL of 8 M HNO₃ and 10 mL of H₂O and save for use as a standard in Step 5.5.4.
- 5.3.4.4 Add 5 mL of calcium carrier and 5 mL of 15 M H₃PO₄ to the acetate-buffered solution in a 1-L polypropylene centrifuge bottle after the actinide separations. Heat in a boiling water bath for 15 min and cool in a bath of cold, running tap water to room temperature.
- 5.3.4.5 Add 4 drops of phenolphthalein indicator, then slowly add 15 M NH₄OH, while stirring vigorously, until a copious precipitate forms. The solution may become very dark brown. Cover with a watch glass and allow the sample to sit overnight.
- 5.3.4.6 Balance the bottles and centrifuge the sample for 10 min at 2000 rpm. Decant and discard the supernate.
- 5.3.4.7 Continue at Step 5.3.5 of the Phosphate Precipitation.

5.3.5 Phosphate Precipitation

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- 5.3.5.1 Adjust the sample volume to 1 L with H₂O and add 5 mL of 15 M H₃PO₄. Cover the 2-L beaker with a watch glass and heat the solution to boiling on the hot plate. Remove the sample from the hot plate and cool to room temperature.
- 5.3.5.2 Add 4 drops of phenolphthalein indicator and adjust the pH to the indicator endpoint (pink) by slowly adding 200 to 250 mL of 15 M NH₄OH while stirring the solution vigorously. Cover the 2-L beaker with a watch glass and allow the sample to sit overnight.
- 5.3.5.3 Add 1 drop of calcium carrier to check the sample for excess phosphate. If the drop of calcium carrier forms a white phosphate precipitate, go to the next step. If a precipitate does not form, add 1 mL of 15 M H₃PO₄ and adjust the pH to the indicator endpoint (pink) by slowly adding 15 M NH₄OH while stirring. Allow the sample to sit for an additional 1 to 2 hrs.
- 5.3.5.4 Decant off as much of the supernate as possible without disturbing the precipitate. Swirl the remaining solution and transfer it to a 1-L polypropylene centrifuge bottle. Rinse the beaker with 10 to 15 mL of H₂O from a wash bottle and combine the rinse with the sample. Do not fill the centrifuge bottle more than 1 in. from the top. Balance the bottles and centrifuge the sample for 10 min at 2000 rpm. Decant and discard the supernate. If the sample is too large, centrifuge approximately 600 mL of the solution, decant and discard the supernate, add the remaining sample to the precipitate in the centrifuge bottle, and centrifuge again.
- 5.3.5.5 Add approximately 200 mL of boiling H₂O to the phosphate precipitate in the 1-L centrifuge bottle. Slurry the precipitate with a stirring rod. Balance the bottles and centrifuge the sample for 10 min at 2000 rpm. Decant and discard the supernate.
- 5.3.5.6 Dissolve the phosphate precipitate with 20 mL of 16 M HNO₃ and transfer the solution into a 250-mL beaker. Use up to 5 mL of 16 M HNO₃ from a wash bottle to rinse the centrifuge bottle and complete the transfer of the sample to the beaker. Combine the rinse with the sample in the beaker. Evaporate the solution to 20 to 25 mL on the hotplate.
- 5.3.5.7 Transfer the hot solution to a 100-mL centrifuge tube. Use 16 M HNO₃ from a wash bottle to complete the transfer of the sample and combine the rinse with the sample solution. The total volume in the tube should not exceed 25 to 30 mL.

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5.3.5.8 Continue at Step 5.4 with the nitrate precipitation.

5.4 Nitrate Precipitation

5.4.1 Add 20 to 30 mL of 21 M HNO₃ while swirling the solution so that a nitric acid concentration of at least 16 M is achieved. Heat the solution in a boiling water bath for 5 to 10 min and cool the sample to room temperature in a bath of cold, running tap water. Balance the tubes and centrifuge the sample for 5 min at 2400 rpm. Carefully decant and discard the supernate. Dissolve the nitrate precipitate with 5 to 10 mL of H₂O.

NOTE: MOST ENVIRONMENTAL AND ICPP WATER SAMPLES REQUIRE ONLY 20 mL OF 21 M HNO₃. SAMPLES THAT ARE UNUSUALLY HIGH IN CALCIUM (SUCH AS MILK, WHEAT, AND SOME VEGETATION ASH) MAY REQUIRE UP TO 30 mL OF 21 M HNO₃. SEE EXHIBIT I FOR FURTHER GUIDANCE.

5.4.2 Repeat Step 5.4.1 a second time and then continue at Step 5.4.3.

5.4.3 Add 2 drops of phenolphthalein indicator and, while constantly swirling, adjust the pH to the indicator endpoint (pink) with 10 M NaOH. Continue swirling the solution and add 5 mL of saturated Na₂CO₃.

5.4.4 Digest the sample for 5 min in a boiling water bath and then cool it to room temperature in a bath of cold, running tap water. Balance the tubes and centrifuge the sample for 5 min at 2400 rpm. Decant and discard the supernate. Dissolve the carbonate precipitate in 5 mL of 8 M HNO₃. Repeat Step 5.4.1 if a very copious or gelatinous precipitate developed when the saturated Na₂CO₃ was added, otherwise, continue at Section 5.5.

5.5 Removal of Yttrium from Strontium Solution

5.5.1 Remove ⁹⁰Y from the sample by first adding 5 mL of H₂O and then 1 mL of yttrium carrier and swirling. Digest the sample solution in a boiling water bath for 3 min, then cool the sample to room temperature in a bath of cold, running tap water. Add 1 to 2 drops of MCP indicator and adjust the pH just to the purple endpoint of the indicator by slow, dropwise addition of 15 M NH₄OH while swirling. Wash down the sides of the 100-mL centrifuge tube with a few milliliters of H₂O from a wash bottle. Cool the sample to room temperature in a bath of cold, running tap water. Balance the tubes and centrifuge the sample for 5 min at 2400 rpm. Decant the supernate into a clean 90-mL polystyrene counting bottle.

5.5.2 Record in the sample logbook the Y(OH)₃ precipitation time as the starting time for ⁹⁰Y ingrowth.

5.5.3 Dissolve the Y(OH)₃ precipitate with 5 mL of 8 M HNO₃ and add 5 mL of H₂O. Add 1 to 2 drops of MCP indicator and adjust the pH just to the purple endpoint of the indicator by slow dropwise addition of 15 M NH₄OH while swirling. Wash down the sides of the 100-mL centrifuge tube with a few milliliters of H₂O from a wash bottle. Cool the sample to

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room temperature in a bath of cold, running tap water. Balance the tubes and centrifuge the sample for 5 min at 2400 rpm. Decant and combine with the supernate in the counting bottle from Step 5.5.1. Discard the $\text{Y}(\text{OH})_3$ precipitate.

5.5.4 Add 1 mL of yttrium carrier to the combined supernates. Adjust the pH to ≤ 1 (pink) with 16 M HNO_3 . With H_2O , adjust the volumes of the sample, blank, and ^{85}Sr standard to the same height in the counting bottles. Gamma count each for the same period of time in a 3 x 3-in. sodium iodide well counter to at least 20,000 counts. Record the counts and the background of the well counter in the sample logbook. The data are used to calculate the Sr yield by comparison with the standard solution. Store the solution 7 to 14 days to allow ^{90}Y ingrowth.

5.5.5 After 7 days, approximately 85% of the yttrium daughter product has grown into the strontium solution; after 14 days, ingrowth is 97% complete.

5.6 Preparation of Yttrium Oxalate after Ingrowth

5.6.1 Transfer the solution from the counting bottle to a 100-mL centrifuge tube after the ingrowth period. Use approximately 5 mL of H_2O from a wash bottle to complete the transfer of the sample and rinse the counting bottle. Combine the rinse with the sample solution. Add 1 to 2 drops of MCP indicator and adjust the pH just to the purple endpoint of the indicator by slow dropwise addition of 15 M NH_4OH while swirling the sample solution. Continue swirling until the copious precipitate of $\text{Y}(\text{OH})_3$ forms.

5.6.2 Record in the sample logbook the precipitation time as the end of the ^{90}Y ingrowth period.

5.6.3 Wash down the walls of the 100-mL centrifuge tube with a few milliliters of H_2O from a wash bottle. Balance the tubes and centrifuge the sample for 5 min at 2400 rpm. Then decant the supernate into the original counting bottle. Adjust the pH to ≤ 1 (pink) with 16 M HNO_3 and store the solution. Storage of the strontium fraction permits remilking the solution for a second set of ^{90}Y counts if necessary.

5.6.4 Dissolve the $\text{Y}(\text{OH})_3$ precipitate with 5 mL of 8 M HNO_3 . Add 5 drops of strontium carrier and dilute the sample to approximately 30 mL with H_2O . Add 1 to 2 drops of MCP indicator and adjust the pH just to the purple endpoint of the indicator by slow dropwise addition of 15 M NH_4OH while constantly swirling. Wash down the walls of the 100-mL centrifuge tube with a few milliliters of H_2O from a wash bottle. Balance the tubes and centrifuge the sample for 5 min at 2400 rpm. Decant and discard the supernate.

5.6.5 Dissolve the $\text{Y}(\text{OH})_3$ precipitate with 5 mL of 8 M HNO_3 . Add 5 mL of oxalic acid and 2 to 3 drops of MCP indicator. Dilute the sample to approximately 25 mL with H_2O . Slowly add 15 M NH_4OH dropwise, with constant swirling, until the deep red of the indicator begins to become more pink in appearance. Complete the adjustment of pH to the salmon pink

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color of the indicator, ca. pH 1.5, by dropwise addition of dilute NH_4OH with constant swirling.

- 5.6.6 Digest the sample for 5 min in a boiling water bath and then cool to room temperature in a bath of cold, running tap water. Allow the $\text{Y}_2(\text{C}_2\text{O}_4)_3 \cdot 9\text{H}_2\text{O}$ precipitate to settle.
- 5.6.7 Weigh a 24-mm glass fiber GF/A filter. Record the filter weight in the sample logbook. Decant and filter the $\text{Y}_2(\text{C}_2\text{O}_4)_3 \cdot 9\text{H}_2\text{O}$ supernate with gentle suction, through the glass fiber filter mounted in the 2-cm i.d. filtering apparatus. Avoid letting the filter go totally dry until the acetone wash to reduce the possible collection of radon daughters from the air.
- 5.6.8 Wash the $\text{Y}_2(\text{C}_2\text{O}_4)_3 \cdot 9\text{H}_2\text{O}$ in the 100-mL centrifuge tube by swirling it with approximately 20 mL of H_2O from a wash bottle and filter the precipitate. Rinse the centrifuge tube again with approximately 1 mL of acetone and filter it just as the last of the water passes through the $\text{Y}_2(\text{C}_2\text{O}_4)_3 \cdot 9\text{H}_2\text{O}$ on the filter. Remove the chimney and suction just as the filter goes completely dry.
- 5.6.9 Place the filter on a numbered watch glass and dry under a heat lamp at a distance of about 18 cm for 10 min. Allow the filter to cool for 10 min and weigh. Record the weight in the sample logbook. (This data will be used to determine the yttrium yield by comparison with the yttrium gravimetric factor.)
- 5.6.10 Place the filter in a PVC filter holder, then place the holder in a numbered Tennelec carrier for beta counting. Operate the Tennelec beta counter according to procedure CHEM-TP-GB.1 to Count ^{90}Y .
- 5.6.11 Conduct a gamma frisk after completion of this procedure to check for contamination. Frisk gloves and surface areas where samples were handled.

6 CALCULATIONS

- 6.3 The calculations used to determine the ^{90}Sr concentration are described in CHEM-TP-SR.6.

REFERENCES

Sr-1, " $^{89,90}\text{Sr}$ in Environmental, Process Wastes, Foodstuffs, and Biological Samples." Refer to RESL-TP-IH.9 when working with hydrofluoric acid.
Refer to RESL-TP-IH.4 for the handling of corrosive chemicals.
Refer to RESL-TP-IH.1 for eye protection.
Refer to RESL-TP-IH.2 for general laboratory safety.
Refer to CHEM-AP.11 for proper management of chemicals.
Refer to RESL-TP-IH.15 for acid and base neutralization.

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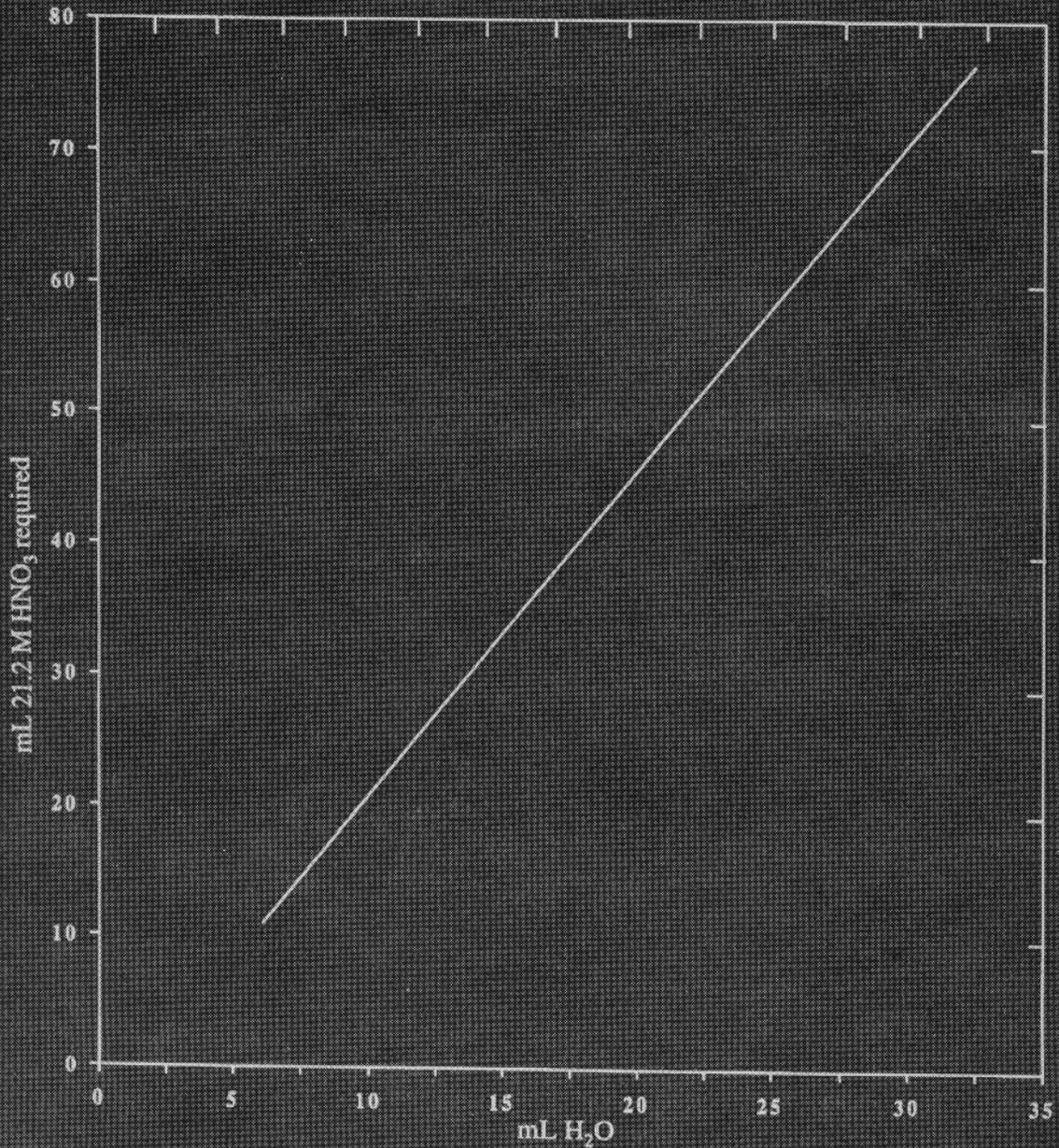
Refer to RESL-AP.10 for waste management.

QUALITY RECORDS

Data that are entered into the RESL database.

Laboratory sample logbook

Printout or electronic media generated by the Tennelec beta counter.



Volume of 21 M HNO₃ required versus volume of water
to obtain 15 HNO₃. 90% HNO₃, Sp. Gr. = 1.48, 21.2 M.

INEL 2 2656