Sections in This Document

1. Purpose

This standard operating procedure (SOP) describes a radiochemical method (see Section 5.A) used to analyze for strontium-90 (Sr-90) in foods. This method was modified (9/23/03) to reduce analysis time, labor, and hazardous waste. An additional modification (5/8/2012) was made to further reduce labor and hazardous waste. This procedure also describes the differences in sample processing between regulatory samples and Total Diet Samples.

2. Scope

This method is applicable for the analysis of about 15g of ash from food or biological material. The minimum detectable activity concentrations provided in the Quality Control Section (6.7.K) are established with sample composite weights of 500g (Scheme 1) and 250g (Scheme 2) when strontium-90 (Sr-90) in the sample is in equilibrium with yttrium-90 (Y-90) and the sample is free of fresh fission products. The procedure provides a reliable and reproducible analysis for Sr-90 by detecting its Y 90 daughter product at activities above the detection limit with typical chemical and radiological interferences found in foods. When elevated levels of fresh fission products are present, the analysis of Sr-90 in the sample must be performed using the original method (see Sect 5.A).

This SOP presents two schemes for routine food analysis. Scheme #1 (9/23/03 modification) is typically used for samples with elevated ash weights. When scheme 1 is used for TDS samples, about 250g of sample are typically used. For regulatory samples, the analyst may opt to use more sample to achieve a certain data quality objective (DQO) or to use less sample to ensure that the ash amount will fall within the scope of the method. Because it involves a significantly higher volume of hazardous reagents and generates more waste, this scheme is reserved for samples that have higher amounts of ash. DQO's are typically defined by a combination of Compliance Policy Guidance and guidance from the work requester. The preferred method for routine sample analysis (e.g., Total Diet Survey samples, Toxic Element in Foods Radionuclide samples) is to use scheme #2 which is applicable for lower ash weights and a suggested sample composite weight of 250g. Sample composite weight suggestions are made for the purpose of ensuring that minimum detection objectives are met and may be varied when this is not an issue. However, for certain sample matrices (examples are grains, cereals, rice, cheeses, or samples that previously yielded low recoveries) it may be appropriate to employ scheme #1 for improved sample recoveries. When sample ash is greater than ~7g, scheme 1 may improve recoveries.

3. Responsibility

A. Supervisor

1. Ensures the analyst performing this procedure is properly trained and knowledgeable in radiochemical analysis.

2. Ensures the analyst is capable of providing acceptable analytical results through proficiency evaluation.
3. Ensures proper implementation of this procedure.

B. Analysts

1. Must adhere to this SOP when performing Sr-90 analysis.
2. Ensure all analytical results are fully supported by acceptable quality control data.
3. Complete the analytical worksheet package and report results for regulatory samples as instructed in ORA-LAB.5.10, Reporting Laboratory Data.
4. Complete the raw data analysis record (see H:\Analytical Branch\Radiochemistry\Analysis Database.accde) for Total Diet Samples as instructed in the database.
5. Report any problems encountered during the sample analysis to their supervisor and document the situation.
6. Handle and dispose of the chemicals and radioactive materials in accordance with the safety guidelines detailed in WEAC-LAB.14.0, Waste Management Program; WEAC-LAB-RS.002, WEAC Radiation Safety Manual; and WEAC-LAB-RS.004, Radioactive Waste Handling Procedure.

4. Background

Upon receipt, the food sample is weighed, ashed, and digested in nitric acid. The resultant solution is mixed with nitric acid equilibrated with tributylphosphate (TBP) where the Y-90 is separated from Sr-90 and the sample matrix. After iron and rare earth elements are removed by fluoride (F-) and hydroxide (OH-) precipitation, the purified Y-90 is deposited onto a glass fiber filter as yttrium oxalate and is counted using a low-background internal gas-flow proportional counter. The concentration of Sr-90 in the sample is equal to the concentration of Y-90 calculated from the observed Y 90 count rate, attenuation-corrected counting efficiency, chemical yield, decay correction factor, and sample weight.

Scheme #1 implements the following procedural modifications from the original method (see Sect 5.A):

1. Carriers are added prior to sample digestion;

2. The ice bath isn't needed because the fuming HNO3 precipitation step was eliminated;
3. The start of the first TBP extraction is designated as the separation time of Sr-90 and Y-90;
4. The volume of water and acid for back extraction is reduced;
5. The recovery is determined with yttrium oxalate instead of yttrium oxide; and
6. The calculated sample Sr-90 concentration is based on the original fresh sample weight rather than the sample ash weight.

Scheme #2 further modifies the original method as follows:

1. The total sample amount is decreased to 250 g;
2. Uses less 2:1 nitric acid for the digestion;
3. Requires less fuming nitric acid to achieve the appropriate final molarity;
4. Uses less equilibrated TBP in the first extraction so less 14N nitric acid rinse is required;
5. Uses less water in the back extraction so less 3N nitric acid rinse is required; and
6. Uses less ammonium hydroxide to adjust the pH of the first hydroxide precipitation.

5. References

A. Baratta, E.J. and Reavey, T.C., "Rapid Determination of Strontium-90 in Tissue, Food, Biota, and Other Environmental Media by Tributyl Phosphate" Journal of Agricultural and Food Chemistry, Vol. 17, No. 6, Nov.-Dec. 1969, pp. 1337-1339.

B. Douglas, G.S., Editor, "Radioassay Procedures for Environment-al Samples", U.S. Government Printing Office, Washington, 1967. Public Health Service Publication No. 999-RH-27.

C. Standard Methods for the Examination of Water and Waste water, 20th Edition, Method 7500-Sr, pp.7-45.

D. Currie, Lloyd A. "Limits for Qualitative Detection and Quantitative Determination – Application to Radiochemistry", Analytical Chemistry, Vol. 40, No. 3, March 1969, pp. 586-593.

E. Multi-Agency Radiological Laboratory Analytical Protocols Manual, NUREG-1576, EPA 402-B-04-001C, NTIS PB2004-105421, July 2004.

F. Northeast Radiological Health Laboratory, Analysis of Environmental Samples, Chemical and Radiochemical Procedures, NERHL-64-1, 1964. (Appendix III).

G. Rucker, T.L., “Methodologies for the Practical Determination and Use of Method Detection limits”, Journal of Radioanalytical and Nuclear Chemistry, Vol. 192, No. 2, pp. 345-350, 1995.

H. Validation of Excel Spreadsheets for the Calculations Set Forth by the Method SOP-WEAC.RN.Method.2.0, Feb. 2006, WEAC

6. Procedure

6.1. Equipment

- Fume hood
- Top loading balance: Mettler XS8001S or equivalent
- Analytical balance: Mettler XP205 or equivalent
- Scale: Harvard Trip balance or equivalent
- Large capacity drying oven maintained between 100 °C and 130 °C: Chromalox AR-2514 or equivalent
- Drying oven maintained at a constant temperature between 105 °C and 130 °C: Fisher IsoTemp oven or equivalent
- Programmable ashing oven/furnace: Nabertherm N450/DB, N450/G or N100G with automatic temperature ramping and holding control or equivalent
- Programmable muffle furnace: Thermolyne Furnace with Barber-Coleman control unit or equivalent
- Digital timer: Thomas Scientific Model 30490U0 or equivalent
- Large volume centrifuge: Thermo Sorvall LEGEND XT, Thermo IEC Centra-CL3R, or equivalent
- Small volume centrifuge: Thermo IEC Centra-CL2 or equivalent
- Low-background internal gas-flow proportional counter: Protean WPC-9550; Protean MPC-9604 or equivalent

- Glassware: 100-mL, 150-mL or 250-mL beakers and watch glasses, 250- and 1000-mL or 2000-mL separatory funnels and filtration flasks.
- Class A Volumetric Glassware: 25-mL, 100-mL, 500-mL, 1000-mL and 2000-mL graduated cylinders and 1000-mL and 2000-mL volumetric flasks.
- Plastic ware: 250-mL leak-proof polypropylene centrifuge bottles, 50-mL conical polypropylene centrifuge tube with cap, 120-mL polypropylene specimen cup with cap, and 2-mL disposable polyethylene transfer pipets
- Lab-Line Multi-Wrist Shaker Model 3589 (or equivalent) may be used to automate 250-mL separatory funnels shaking.
- Separatory funnel racks
- Centrifuge tube racks
- Automatic pipettes: 1-mL QC calibrated
- Bottle top dispensers: 10-mL, 25-mL and 50-mL
- Teflon stirring rods
- Ashing dishes: Corning Ware® and Coors® (or equivalent) dishes appropriate to accommodate the sample size
- pH papers with indicating ranges from 1.0 - 1.5 and 8 - 10
- Hot plate
- Desiccators
- Glass fiber filters: Whatman 24- and 55-mm circles or equivalent
- Buchner filter funnels with 63 mm inside diameter
- Filtration apparatus: Thomas WITT filter jar, Fisher filter dome, or equivalent
- Stainless steel vacuum filter assembly
- Mylar film in 0.01 mm thickness
- Tweezers
- Aluminum weighing trays
- Vacuum pump or equivalent
- Nylon source mounting platform and ring

- Gloves, safety glasses, dust masks and lab coats
- Permanent marker
- High temperature marker
- Labeled waste containers (Refer to Sect 6.8.L)
- Firm-bristle, flat (~1cm) nylon brush for ash transfer or equivalent
- Autopipette dispensers used for safety and convenience, but not for delivery of critical reagents.

6.2. Reagents and Standards

- Laboratory grade water
- Fuming nitric acid (HNO3), reagent grade or equivalent
- Concentrated nitric acid (HNO3), reagent grade or equivalent
- Concentrated hydrochloric acid (HCl), reagent grade or equivalent
- Concentrated hydrofluoric acid (HF), 48%, reagent grade or equivalent
- Boric acid (H3BO3), reagent grade or equivalent
- Oxalic acid ((COOH)2·2H2O), dihydrate, reagent grade or equivalent
- Concentrated ammonium hydroxide (NH4OH), reagent grade or equivalent
- Tributylphosphate (TBP), laboratory grade or better
- Absolute ethanol
- 10% methane and 90% argon (aka: P-10) reaction gas – information on the particular gas tank used for an instrument at a particular time is maintained in LIMS.
- Strontium nitrate (Sr(NO3)2), reagent grade or equivalent
- Yttrium oxide (Y2O3), reagent grade or equivalent
- NIST traceable Sr-90 spike standard.
  Note: Must be stored in an air tight container. Any aliquots taken from the stock bottle should be stored in an air tight container.
- Food ash serving as laboratory control sample (LCSs) matrix

Note: Class A volumetric glassware must be used for reagent preparation, except for TBP equilibration.

- Strontium carrier solution (90 mg/mL): dissolve 217 g of Sr(NO3)2 in 800 mL of laboratory grade water. Add 1 mL of concentrated HNO3, dilute to 1 L with laboratory grade water, and mix well (Douglas, G.S., 1967, Appendix B.2, pp. B-8)
- Strontium-90: NIST-traceable standard solution (0.5-2Bq/mL)
  Note: Must be stored in an air tight container. Any aliquots taken from the stock bottle should be stored in an air tight container.
- Standardized yttrium carrier solution (20 mg/mL) (Douglas, G.S., 1967, Appendix B.2, pp. B-8 – B-9)
  Note: Must be stored in an air tight container. Any aliquots taken from the stock bottle should be stored in an air tight container.
- Nitric acid (2:1): mix 2 volumes of concentrated HNO3 with 1 volume of laboratory grade water
- 14N HNO3: measure 880 mL of concentrated HNO3 and dilute to 1 L with laboratory grade water
- 3N HNO3: measure 189 mL of concentrated HNO3 and dilute to 1 L with laboratory grade water
- Saturated H3BO3: dissolve 50 g of H3BO3 in 400 mL warm laboratory grade water. Allow solution to cool to room temperature and dilute to 500 mL.
  Note: Some H3BO3 may recrystallize after the solution reaches room temperature.
- 2N (COOH)2: dissolve 126 g (COOH)2·2H2O in 900 mL warm laboratory grade water. Allow the solution to cool to room temperature and dilute to 1 L (Douglas, G.S., 1967, Appendix B.4, pp. B-20).
- Freshly prepared TBP: shake TBP with an equal volume of 14N HNO3 for five minutes. Wait for complete phase separation and discard the aqueous phase to a waste container labeled “14N Nitric acid saturated with TBP”. Record the waste disposal on WEAC-TMPL-198 Hazardous Waste Log Sheet as required by WEAC.Lab.14.0, Hazardous Waste Management Program. Repeat the process once. The TBP must be freshly prepared to ensure the proper separation of Y (Y-90) from Sr (Sr-90) and sample matrix.

6.3. Analytical Procedure

A. Upon receipt, properly store sample in a secure area, and initiate the analytical worksheet applicable to the analysis required (Sect. 8.A).

B. Determine which scheme to use for your sample analysis.

1. **Scheme #2** is typically used for routine analysis of samples food samples unless there are sample matrix or DQO considerations (e.g. elevated ash amounts).
2. **Scheme #1** (the 9/23/03 method modification,) is used for samples that require a larger amount of sample to meet the data quality objective (DQO) of the analysis or because of the amount of resultant ash.

C. When unsure if a sample is homogeneous, prepare a representative analytical portion following the guidance in WEAC-AB.8.0, Laboratory Sub Sampling.

D. Ensure that the balance used meets WEAC-LAB.6.0, Laboratory Balances. For each sample being analyzed, weigh a suitable sample portion to the nearest 0.1 gram in a clean ashing dish, label the dish using a high temperature marker, and record the dish ID and sample weight on the analytical worksheet. Typically, use about 250 g of sample composite.

E. **The Ashing Process**

Note: To ensure safety, use the specially designed Nabertherm N450/DB programmable furnace when ashing oily samples.

1. Ensure that the ashing oven/furnace you're using meets QC requirements.
2. Place the samples in an ashing oven programmed with the following temperature profile: 120 ºC for 48 hours, 250 ºC for 4 hours, 315 ºC for 4 hours, 480 ºC for 8 hours, and 575 ºC for 16 hours. Complete the sample ashing procedure following the operation instructions described in the applicable ashing oven's SOP. Review the temperature chart and fill in the Furnace FV/PM sheet as directed. If temperature charting capability is not available, refer to WEAC-MEMO-2014-06-18.01 for guidance.
3. Remove your dishes from the furnace and visually examine the ash quality. If sample ashing appears to be incomplete (shows obvious carbon soot residue), return the sample to the ashing oven/ furnace s at 575 ºC for additional 16 hours. Inform your supervisor if the problem persists. After cooling, weigh the ashing dish with ash and record the weight on the worksheet.

F. **LCS**: If a valid LCS analysis has been performed on the counting instrument within the previous seven-days of your sample analysis (Refer to Sect 6.7.C), continue onto the next step. If not, weigh a known amount of LCS ash, equivalent to 250 grams into a clean, labeled Coors dish or glass beaker, then add a known amount of Sr 90 spike standard. Record the dish ID and LCS ash weight on the analytical worksheet. If both scheme #1 and scheme #2 are being run in the same batch, either LCS may be used.

G. **Method Blank**: Use a clean, labeled glass beaker for your method blank preparation. Record the beaker ID on the analytical worksheet.

Note: Procedures below "Scheme 1" and "Scheme 2" describe the differences for higher ash/sample verses lower ash/sample. A batch may contain samples that are analyzed using both scheme 1 and scheme 2. QC samples for a batch must be analyzed using a scheme that was applied to at least one sample in that batch. If two schemes are used in one batch, it is not necessary to run QC by both schemes. However, the LCS and method blank must be run using the same scheme.

H. **Analysis – Scheme #1**

1. Sect 6.3.H describes digestion through the forward extraction and apply only to Scheme #1. This scheme is typically used to accommodate elevated ash weights. Using an automatic pipette QC calibrated per WEAC-AB.6.0 Gravimetric Calibration Check of Pipettes, add 1 mL of Sr carrier, 1 mL of standardized Y carrier, and 70 mL of 2:1 HNO3 to each sample, method blank, and LCS.

   Note: This step may be performed in the Coors dish if the ash can't be easily transferred to a beaker prior to adding the carriers. Allow the sample to come to a low boil on a hot plate, and then transfer the sample solution and residue to a clean, labeled glass beaker. Rinse the dish multiple times using a total of 10 mL of 2:1 HNO3, adding the rinses to the beaker.

   Note: This reaction is *exothermic* and will add additional heat to your solution.

   Cover the beaker with a watch glass and digest the sample by refluxing on a hot plate (at just below boiling) for 10 minutes.

2. Assemble a filtration apparatus. Record the cup ID (Digest ID) on the analytical worksheet. Place a clean 55-mm glass fiber filter to fully cover the perforated holes. Attach the vacuum pump to the

chamber and vent the output hose to the hood. Pour the warm sample solution through the filter. Rinse the beaker with 2:1 HNO3 and pour the rinse through the filter.

3. Retrieve the cup from the filtration apparatus and adjust the sample volume to 100 mL with 2:1 HNO3. At this point, the sample may be stored until a later date.
4. Transfer the sample solution to a clean, labeled 250-mL separatory funnel. Add 50 mL of fuming HNO3 to the funnel.

   Note: Verify that the fuming nitric acid portion added to the sample is 50 ± 1mL.

   While holding the loosely capped funnel vertical, gently swirl the funnel to liberate the built-up gases. Record the funnel ID as the sample Analysis ID on the analytical worksheet. Steps 6.3.H.4 and 6.3.H.5 should have a minimal time interval (preferably <30 minutes).
5. Add 50mL of freshly prepared TBP to the funnel. Take the same precautions as described in the previous step to liberate additional built up gases. Ensure that the clock you're using meets WEAC-AB-RN.8.0 QC requirements. With both the funnel cap and drain stopper securely closed, shake the funnel for 3 minutes. **Record the Sr-90/Y-90 separation time** at the start of funnel shaking.
6. Wait for complete phase separation. Transfer the aqueous (bottom) phase to another clean, labeled 250-mL separatory funnel and save the TBP (top) portion.
7. Add another 50mL of freshly prepared TBP to the funnel containing the aqueous phase, securely close the funnel cap and drain stopper, then shake for 3 minutes. Wait for complete phase separation. Discard the aqueous (bottom) phase from non-radioactive samples to a waste bottle labeled "14N HNO3 saturated with TBP". Discard the aqueous (bottom) phase from radioactive samples to a waste bottle labeled "14N Nitric Acid saturated with Tributylphosphate+Sr90/Y90". Record the radioactive waste disposal on form WEAC TMPL.191, Radiological Waste Record as required by WEAC-LAB-RS.004, Radioactive Waste Handling Procedure. *Note that there should be an entry per each disposal of 1mL of original radioactive solution.*
8. Combine the TBP portions from Sect 6.3.H steps 6 and 7 in one separatory funnel.

9. Add 40 mL of 14N HNO3 to the separatory funnel, secure the funnel cap and drain stopper, and shake for 2 minutes. Wait for complete phase separation. Discard the aqueous (bottom) phase from method blank and samples to a waste bottle labeled "14N Nitric Acid saturated with Tributylphosphate". Repeat the TBP wash two more times. Discard the aqueous (bottom) phase from each rinse of the LCS to a waste bottle labeled "14N Nitric Acid saturated with Tribuylphosphate and containing Sr-90/Y-90". Record the radioactive waste disposal on form WEAC TMPL.191, Radiological Waste Record as required by WEAC-LAB-RS.004, Radioactive Waste Handling Procedure. *Note that there should be an entry per each disposal of 1mL of original radioactive solution.*

   Note: From this point on, the wastes from the radioactive samples can be disposed of along with non-radioactive wastes.

10. Add 50 mL of laboratory grade water to the separatory funnel and carefully liberate the built up gases. After securing the funnel cap and drain stopper, shake the funnel for 2 minutes. Wait for complete phase separation, then transfer the aqueous (bottom) phase to a clean, labeled 250 mL centrifuge bottle.

11. Add 25 mL of 3N HNO3 to the separatory funnel, secure the funnel cap and drain stopper, and shake for 2 minutes. Wait for complete phase separation and then dispense the aqueous (bottom) phase into the centrifuge bottle saved in step 10 above. Repeat two more times. Discard the TBP (top) portion to a waste bottle labeled "Tributylphosphate saturated with 3N Nitric Acid".

12. Ensure that the centrifuges you're using meets WEAC-AB-RN.10.0 QC requirements and fill in the FV/PM charts. Add ~50mL of concentrated NH4OH to the centrifuge bottle, adjust the solution pH to between 8-10 using concentrated NH4OH, and centrifuge the solution at approximately 3000 rpm for 15 minutes. To prevent precipitate loss, carefully decant the supernatant to a waste bottle labeled "72% 3N Nitric Acid+28% concentrated ammonium hydroxide". Record the waste disposal on the Hazardous Waste Log Sheet. Proceed to 6.3.I. step 12.

I. **Analysis - Scheme #2**

1. Section 6.3.I., which describes the digestion through the forward extraction apply only to Scheme #2. If possible, transfer all the ash to a clean labeled glass beaker using an ash brush. If you can't remove all the ash from the dish, add the carriers directly to the

Coors dish. Using an automatic pipette QC calibrated per WEAC-AB.6.0, add 1 mL of Sr carrier, 1 mL of standardized Y carrier, and 20 mL of 2:1 HNO_3 to each sample, method blank, and LCS.

Note: If the carriers are added to the ash in the Coors dish, warm the 2:1 HNO_3 and divide the 20mL of 2:1 HNO_3 into at least three separate portions totaling 20mL. Agitate each addition to the dish with a Teflon stirrer if needed, and then decant to a glass beaker.

Cover the beaker with a watch glass and digest each sample by refluxing on a hot plate (at just below boiling) for 10 minutes. If the ash is totally submerged in the 2:1 nitric acid and has broken down into smaller particles (use a Teflon stirrer, if needed), proceed to step 2 below. Otherwise, add enough 2:1 nitric to submerge all ash and digest another 10 minutes while mixing the ash with a Teflon stirrer.

Caution: Strontium nitrate will begin to precipitate from solution at a molarity of 12N. Avoid an evaporation loss of >10% of the reflux volume.

Note: This reaction is *exothermic* and will add additional heat to your solution. Allow any bubbling following the addition of 2:1 nitric to subside prior to refluxing.

2. Assemble a filtration apparatus. Record the tube ID (Digest ID) on the analytical worksheet. Place a clean 55-mm glass fiber filter and connect to a vacuum pump and vent the output to the hood. Pour the warm sample solution through the filter. Rinse the beaker with a total of 10 mL of 2:1 HNO_3 (divided into 2-3 rinses) and pour each rinse through the filter. If necessary, adjust the sample volume appropriately with 2:1 HNO_3.

   Note a) Unless additional 2:1 nitric was used, samples and method blanks will measure 32 mL and the LCS 33 mL.

   Note b) If additional 2:1 nitric acid was used, evaporate the filtered digest to near dryness (~2-3)mL and reconstitute with 30 mL of 2:1 nitric acid on a hot plate.

3. Transfer the sample solution to a clean, labeled 250-mL separatory funnel. Add 20 mL of fuming HNO_3 to the funnel. Note: Verify that the fuming nitric acid portion added to the sample is 20 mL ± 1 mL. While holding the loosely capped funnel vertical, gently swirl the funnel to liberate the built-up gases. Record the funnel ID as the sample Analysis ID on the analytical worksheet. Steps 6.3.I.3 and

6.3.I.4 should have a minimal time interval (preferably <30 minutes). In order to maintain DQO's for minimum detectable concentrations (MDC's), the remainder of the analysis (scheme 2) is typically completed within a day.

4. Add 15 mL of freshly prepared TBP to the funnel. Take the same precautions as described in the previous step to liberate additional built up gases. Ensure that the clock you're using meets WEAC-AB-RN.8.0 QC requirements. With both the funnel cap and drain stopper securely closed, shake the funnel for 3 minutes. Record the Sr-90/Y-90 separation time at the start of funnel shaking.
5. Wait for complete phase separation. Transfer the aqueous (bottom) phase to another clean, labeled 250-mL separatory funnel and save the TBP (top) portion.
6. Add another 15 mL of freshly prepared TBP to the funnel containing the aqueous phase, securely close the funnel cap and drain stopper, then shake for 3 minutes. Wait for complete phase separation. Discard the aqueous (bottom) phase from non-radioactive samples to a waste bottle labeled "14N HNO3 saturated with TBP. Discard the aqueous (bottom) phase from radioactive samples to a waste bottle labeled "14N Nitric Acid saturated with Tributylphosphate+Sr90/Y90". Record the radioactive waste disposal on form WEAC TMPL.191, Radiological Waste Record as required by WEAC-LAB-RS.004, Radioactive Waste Handling Procedure. *Note that there should be an entry per each disposal of 1mL of original radioactive solution.*
7. Combine the TBP portions from Sect 6.3.I steps 5 and 6 in one separatory funnel.
8. Add 10 mL of 14N HNO3 to the separatory funnel, secure the funnel cap and drain stopper, and shake for 2 minutes. Wait for complete phase separation. Discard the aqueous (bottom) phase from method blank and samples to a waste bottle labeled "14N Nitric Acid saturated with Tributylphosphate". Repeat the TBP wash two more times. At the end of this step, discard the aqueous (bottom) phase from each rinse of the LCS to a waste bottle labeled "14N Nitric Acid saturated with Tribuylphosphate and containing Sr-90/Y-90 Record the radioactive waste disposal on form WEAC TMPL.191, Radiological Waste Record as required by WEAC-LAB-RS.004, Radioactive Waste Handling Procedure. *Note that there should be an entry per each disposal of 1mL of original radioactive solution.*

Note: From this point on, the wastes from the radioactive samples can be disposed of along with non-radioactive wastes.

9. Add 25 mL of laboratory grade water to the separatory funnel and carefully liberate the built-up gases. After securing the funnel cap and drain stopper, shake the funnel for 2 minutes. Wait for complete phase separation, then transfer the aqueous (bottom) phase to a clean, labeled 250 mL centrifuge bottle.
10. Add 8 mL of 3N HNO3 to the separatory funnel, secure the funnel cap and drain stopper, and shake for 2 minutes. Wait for complete phase separation and then dispense the aqueous (bottom) phase into the centrifuge bottle saved in step 9 above. Repeat two more times. Discard the TBP (top) portion to a waste bottle labeled "Tributylphosphate saturated with 3N Nitric Acid".
11. Ensure that the centrifuges you're using meets WEAC-AB-RN.10.0 QC requirements. Add ~14-mL of concentrated NH4OH to the centrifuge bottle, adjust the solution pH to between 8-10 using concentrated NH4OH, and centrifuge the solution at approximately 3000 rpm for 15 minutes. Observe the precipitate and ensure that it is gelatinous and not opaque. To prevent precipitate loss, ***carefully*** decant the supernatant to a waste bottle labeled "72% 3N Nitric Acid+28% concentrated ammonium hydroxide". Record the waste disposal on the Hazardous Waste Log Sheet.
12. Add 2 mL of concentrated HCl to dissolve the precipitate in the centrifuge bottle. Transfer the dissolved precipitate into a 50-mL poly-propylene centrifuge tube and rinse the 250-mL centrifuge bottle multiple times using a total of 30 mL of laboratory grade water. Combine the rinses with the solution in the 50-mL centrifuge tube. Add 2 mL of concentrated HF. Stir thoroughly using a Teflon stirring rod, then centrifuge the solution at approximately 3000 rpm for 15 minutes. ***Carefully*** decant the supernatant to a plastic waste bottle labeled "6% HCl+6% HF+88% H2O".
13. Dissolve the precipitate in 2 mL of saturated H3BO3 and 2 mL of concentrated HCl. Add 20 mL of laboratory grade water and about 2 mL of concentrated NH4OH then stir with a Teflon rod. Adjust the solution pH with concentrated NH4OH to between 8–10, and then centrifuge the solution at about 3000 rpm for 15 minutes. ***Carefully*** decant the supernatant to a plastic waste bottle labeled "8% HCl+8% boric acid+8% ammonium hydroxide+76% H2O".

14. Dissolve the precipitate in 2 mL of concentrated HCl. Add 20 mL of laboratory grade water and 10 mL of 2N (COOH)2 to the centrifuge tube, then stir thoroughly. Adjust the solution pH to between 1.0-1.5 with concentrated NH4OH, then place the tube in a hot-water bath (~80 °C) for 20 minutes. Remove the tube from the water bath.
15. Ensure that the drying oven and thermometer you're using meet WEAC-AB.3.0 and WEAC-AB.7.0 QC requirements. Place a blank 25-mm Whatman glass fiber filter in a clean, labeled aluminum weighing tray for each sample being analyzed. Dry the filters in a drying oven at a temperature between 120 °C and 130 °C for about 20 minutes. Cool the filters and trays to room temperature in a desiccator. Ensure that the analytical balance you use meets WEAC-LAB.6.0 QC requirements. Weigh the filter and tray to the nearest 0.00001gram and record the filter ID and weight under "unloaded filter weight" on the analytical worksheet.
16. Assemble a stainless-steel filter assembly and connect the assembly to a vacuum pump (or equivalent).
17. Load a pre-weighed blank filter and turn on the pump.
18. Select the centrifuge tube (saved in step 14) corresponding to the filter on the filtration assembly and pour the slurry through the filter to achieve uniform deposition.
19. Rinse the precipitate on the filter with ~20 mL of warm laboratory grade water followed by three portions of ~10-mL absolute ethanol.
20. After turning off the pump, carefully retrieve the loaded filter from the filtration assembly and place in its aluminum weighing tray. Dry the filter and tray in a drying oven at a temperature between 120 ºC and 130 ºC for about 20 minutes. Transfer them into a desiccator and allow to cool to room temperature. Discard the solution in the filtration flask into a waste bottle labeled "66% 0.3N oxalic acid and 33% ethanol".
21. Weigh the loaded filter and tray to the nearest 0.00001 gram and record the weight under "Yttrium oxalate loaded filter weight" on the analytical worksheet.
22. Place the loaded filter on a clean, labeled nylon source mount disk and cover it with a piece of Mylar film. Secure the filter and film with a mounting ring. Trim the film along the lower edge of the mounting ring.

23. Ensure the low-background internal gas-flow proportional counter is calibrated and QC checked per WEAC-AB-RN.019, Gas-Flow Low-Level Alpha/Beta Proportional Counter. When editing a sample batch counting file, pair each sample source with its associated counting cartridge and then load each sample source on its counting cartridge for analysis. Count the sample for 100 minutes using the appropriate counting protocol. Record the counting cartridge ID assigned to each sample on the analytical worksheet.
24. Retrieve all samples from the counter, dismount the sources, and discard the filters, including the radioactive ones, in a waste container labeled “Yttrium oxalate+glass fiber filters”. All counted sources should initially be treated as radioactive waste in the category of “Storage-for-Decay”. After Y-90 has decayed away, they are then reclassified as hazardous chemical waste by the radiation safety officer.
25. Retrieve the counting result sheet and calculate result.
26. Enter the counting data and associated analytical data into Analytical Database (see Sect 8.A). The results are noted as not detected (ND) if the calculated activity concentration is less than the reported minimum detectable activity concentration (MDC). If the reported result is greater than MDC but less than the limit of quantification (LOQ), the result will be noted as trace amount (TR). Attach the spreadsheet to the worksheet.
27. Calculate the LCS and method blank results using the Analytical Database (see Sect 8.A).
28. Verify that all data entries on the analytical worksheet are accurate. Enter the analytical findings, i.e., the Sr-90 activity concentrations and the associated measurement uncertainties for samples and LCS, into the FACTS database following the guidance provided in ORA-LAB.5.10, Reporting Laboratory Data.

6.4. Calculations

The equations listed in this section are used to calculation the analytical results.

A. Sample, Method Blank, and Laboratory Control Sample (LCS) Activity

The Sr-90 activity in samples, method blank, and LCS is calculated as follows:

$$A_i = \frac{R_i - R_B}{E_Y \times Y_Y \times D_Y \times D_{Sr} \times 60} \qquad (1)$$

where,

A_i = Activity of Sr-90 in sample, method blank, or LCS, Bq

i = S, M and C for sample, method blank, and LCS respectively

R_i = Gross count rate of sample, method blank, or LCS, cpm

R_B = Count rate of instrument background with cartridge assembly including stainless steel tray and nylon ring, cpm

E_Y = Counting efficiency of Y-90, cpm/dpm

Y_Y = Chemical yield of Y-90, fractional

D_Y = Decay correction factor for Y-90

D_{Sr} = Decay correction factor for Sr-90

60 = Conversion factor from minutes to seconds

Furthermore, E_Y, Y_Y, D_Y, and D_{Sr} are calculated as follows:

$$E_Y = I + S \times W_{YOX} \quad (2)$$

$$W_{YOX} = W_L - W_{UL} \quad (3)$$

$$Y_Y = \frac{W_{YOX} \times k_1}{C_{YOX} \times V_{YOX}} \quad (4)$$

$$D_Y = e^{(-\lambda_Y \times \Delta T_1)} \quad (5)$$

$$D_{Sr} = e^{(-\lambda_{Sr} \times \Delta T_2)} \quad (6)$$

where,

I = Intercept of counting efficiency attenuation curve, cpm/dpm

S = Slope of counting efficiency attenuation curve, cpm/dpm/mg

W_{YOX} = Net weight of yttrium oxalate on filter, mg

W_L = Weight of yttrium oxalate loaded filter, mg

W_{UL} = Weight of unloaded filter, mg

k_1 = Ratio of molecular weight of method precipitate to molecular weight of precipitate in carrier standardization

C_{YOX} = Concentration of yttrium carrier as yttrium oxalate, mg/mL

V_{YOX} = Volume of yttrium carrier solution added, mL

λ_Y = Decay constant of Y-90, min^{-1}

λ_{Sr} = Decay constant of Sr-90, min^{-1}

ΔT_1 = $T_M - T_S$, time interval between midpoint of counting period and Sr-90/Y-90 separation, min.

ΔT_2 = $T_S - T_R$, time interval between Sr-90/Y-90 separation and the reference date, min.

T_M = Midpoint of counting period $T_M = T_A + 1/2 T_C$

T_S = Time of Sr-90 and Y-90 separation

T_A = Acquisition start time

T_R = Reference date

T_C = Live time for sample, method blank, or LCS counting, min

B. Typical Sr-90 Activity in Method Blanks

The typical Sr-90 activity in method blanks for correcting the sample activity is calculated as follows:

$$A_{TMB} = \frac{\sum_{i=1}^{N} A_{M_i}}{N} \quad (7)$$

where,

A_{TMB} = Typical activity of Sr-90 in method blanks, Bq

A_{Mi} = Activity of Sr-90 in individual method blank calculated using Equation (1), Bq

N = Number of individual method blanks

C. Minimum Detectable Sr-90 Activity Concentration in Sample or LCS

The minimum detectable activity concentration (MDC) for the sample or LCS is estimated from the sample weight or LCS spike solution volume, counting efficiency, chemical yield, counting time, and typical method blank as follows:

$$MDC_S = \frac{t^2 + 2 \times t \times \sigma_{TMB}}{E_{YS} \times W_S \times Y_{YS} \times T_{CS} \times D_{YS} \times D_{SrS} \times 60}, \text{ Bq/kg} \quad (8)$$

$$MDC_C = \frac{t^2 + 2 \times t \times \sigma_{TMB}}{E_{YC} \times V_C \times Y_{YC} \times T_{CC} \times D_{YC} \times D_{SrC} \times 60}, \text{ Bq/mL} \quad (9)$$

where,

σ_{TMB} = Typical standard deviation of net method blank counts

t = t factor for df = (n -1) at α = 0.05

n = Number of method blanks used in the calculation of σ_{TMB}
W_S = Sample weight, kg
V_C = LCS spike solution volume, mL
E_Y = Counting efficiency of Y-90 for sample or LCS, cpm/dpm
Y_Y = Chemical yield of Y-90 for sample or LCS, fractional
T_C = Live time for sample or LCS counting, min.
D_Y = Decay correction factor for Y-90 for sample or LCS
D_{Sr} = Decay correction factor for Sr-90 for sample or LCS
60 = Conversion factor from minutes to seconds

D. Sr-90 Limit of Quantification in Sample or LCS

The limit of quantification (LOQ) for the sample or LCS is estimated from the sample weight or LCS spike standard volume, counting efficiency, chemical yield, counting time, and typical method blank as follows:

$$LOQ_S = \frac{50 \times \left(1 + \sqrt{1 + \left(\frac{\sigma_{TMB}^2}{12.5}\right)}\right)}{E_{YS} \times W_S \times Y_{YS} \times T_{CS} \times D_{YS} \times D_{SrS} \times 60}, \text{ Bg/kg} \quad (10)$$

$$LOQ_C = \frac{50 \times \left(1 + \sqrt{1 + \left(\frac{\sigma_{TMB}^2}{12.5}\right)}\right)}{E_{YC} \times V_C \times Y_{YC} \times T_{CC} \times D_{YC} \times D_{SrC} \times 60}, \text{ Bg/mL} \quad (11)$$

Where, the definitions for the variables in the equations are the same as those described in Sect 6.4.C.

E. Sr-90 Activity Concentration in Sample or LCS

The net activity concentration of Sr-90 in the sample or LCS is calculated as follows:

$$C_S = \frac{A_S - A_{TMB}}{W_S} \quad (12)$$

$$C_C = \frac{A_C - A_{TMB}}{V_C} \quad (13)$$

where,

C_S = Net Sr-90 activity concentration of sample, Bq/kg

C_C = Net Sr-90 activity concentration of LCS, Bq/mL

A_S = Activity of Sr-90 in sample, Bq

A_C = Activity of Sr-90 in LCS, Bq

A_{TMB} = Typical activity of Sr-90 in method blanks, Bq

W_S = Sample weight, kg

V_C = LCS spike standard volume, mL

F. Measurement Uncertainties

Description of Major Sources of Standard Uncertainty, Standard Uncertainty Components, and Their Estimates

The major sources of standard uncertainty for the method, xi, and their typical values, are operationally defined and listed in Table 1 for Scheme #1 and Table 2 for Scheme #2. The standard uncertainty of xi, σ(xi), is estimated either from statistical method (A) or other method (B), such as manufacturer's specification or scientific references. The typical value for each standard uncertainty component is provided.

Table 1. Estimates of the Sr-90 Uncertainty Activity Concentration for the Method (for Schemes #1 and 2)

Description of Source of Standard Uncertainty x_i	Evaluation Method for $\sigma(x_i)$, The standard uncertainty of x_i (A) Statistical method (B) Other method	Value	Uncertainty	Sensitivity Correction Coefficient	Uncertainty contribution Bq/kg
Scheme #1					
Gross Count Rate of Sample, R_i	$\sigma_{Rs,}$ estimated (A)	4.00	0.20	0.1645	0.0329
Background Count Rate, R_B	$\sigma_{RB,}$ estimated (A)	0.36	0.06	0.1643	0.0099
Intercept of efficiency attenuation curve, I	$\sigma_{I,}$ estimated (A)	0.556	0.011	1.155	0.0127
Slope of efficiency attenuation curve, S	$\sigma_{S,}$ estimated (A)	-0.00069	0.00016	64.38	0.0103
Weight of Yttrium Oxalate Loaded Filter, W_L	$\sigma_{WL,}$ estimated (B)	1.18440	0.00005	1.00E+01	0.0005
Weight of Unloaded Filter, W_{UL}	$\sigma_{WUL,}$ estimated (B)	1.12849	0.00005	1.43E+02	0.0072
Concentration of Yttrium Carrier Solution, C_{YOX}	$\sigma_{CYOX,}$ estimated (A)	66.79	0.80	4.51E-03	0.0036
Volume of Yttrium Carrier Solution, V_{YOX}	$\sigma_{VYOX,}$ estimated (A)	1	0.016	0.598	0.0096
Midpoint point of count, T_m	$\sigma_{Tm,}$ estimated (A)	1/1/2020 3:00	3 min	0.000	0.0003
Midpoint point of count, T_s	$\sigma_{Ts,}$ estimated (A)	1/1/2020 9:00	20 min	0.000	0.0002
Decay Constant, λ_{Y90}	$\sigma_{DY,}$ estimated (A)	0.01083	0.00004	0.000	0.0001
Decay Factor, D_{Sr}	$\sigma_{DSr,}$ estimated (A)	1.0000	0	NA	0.0000
Method Blank Activity, A_{TMB}	$\sigma_{ATMB,}$ estimated (A)	0.012	0.009	3.66	0.0329
Ratio of YOX molecular weight for method versus carrier standardization preciptate, k_1	$\sigma_{k1,}$ estimated (A)	1.00	0.02	0.60	0.0120
Sample Weight, W_s	$\sigma_{Ws,}$ estimated (B)	0.2500	0.0001	2.000	0.0002
Total Combined Standard Uncertainty of C_i, %					0.055
Coverage Factor, k (k=2)					0.110
Activity Concentration					0.550
Relative Total Combined Uncertainty of C_i, %					10.00%
Relative Expanded Uncertainty of C_i, %					20.0%

* The calculations for uncertainty are described in the SOP

Total Combined Standard Uncertainty of Sr-90 Activity Concentration

The total combined standard uncertainty of Sr-90 activity concentration, σCi, for a single measurement is estimated as the root sum square (RSS) of the identified standard uncertainty components (for RS-RB, WS, EY, and YY) at the concentration level close to the limit of quantification of Scheme #1 for the purpose of comparison. However, since the largest uncertainty component of both schemes at the LOQ level is due to counting statistics,

the total and expanded uncertainties of Scheme #2 at its LOQ is essentially the same as that for Scheme #1. It is assumed that the variables are independent and the time for sample and method blank counting is equal. The equations used for propagating the uncertainty are listed as follows (for LCS, the value of WS is substituted by VC):

$$\sigma_{C_i} = C_i \times \sqrt{\frac{\sigma_{A_i}^2 + \sigma_{A_{TMB}}^2}{(A_i - A_{TMB})^2} + \left(\frac{\sigma_{W_S}}{W_S}\right)^2} \quad (14)$$

Furthermore,

$$\sigma_{A_i} = |A_i| \times \sqrt{\left(\frac{R_i}{R_i - R_B}\right)^2 \times \left(\frac{\sigma_{R_i}}{R_i}\right)^2 + \left(\frac{R_B}{R_i - R_B}\right)^2 \times \left(\frac{\sigma_{R_B}}{R_B}\right)^2 + \left(\frac{\sigma_{E_Y}}{E_Y}\right)^2 + \left(\frac{\sigma_{Y_Y}}{Y_Y}\right)^2} \quad (15)$$

$$\sigma_{E_Y} = \sqrt{\sigma_I^2 + W_{YOX}^2 \times \sigma_S^2 + S^2 \times \sigma_{W_{YOX}}^2} \quad (16)$$

$$\sigma_{W_{YOX}} = \sqrt{\sigma_{W_L}^2 + \sigma_{W_{UL}}^2} \quad (17)$$

$$\sigma_{Y_Y} = \left(\frac{(W_L - W_{UL}) \times k_1}{C_{YOX} \times V_{YOX}}\right) \times \sqrt{\frac{\sigma_{W_L}^2 + \sigma_{W_{UL}}^2}{(W_L - W_{UL})^2} + \left(\frac{\sigma_{k_1}}{k_1}\right)^2 + \left(\frac{\sigma_{C_{YOX}}}{C_{YOX}}\right)^2 + \left(\frac{\sigma_{V_{YOX}}}{V_{YOX}}\right)^2} \quad (18)$$

$$\sigma_{A_{TMB}} = \sqrt{\frac{\sum_{i=1}^{N} (A_{M_i} - A_{TMB})^2}{N-1}} \quad (19)$$

Expanded Uncertainty for Activity Concentration of Sr-90 Measured in the Sample and LCS

The activity concentration of Sr-90 measured in the sample or LCS will be reported along with the expanded measurement uncertainty. The expanded measurement uncertainty at 95% confidence level is obtained by multiplying the total combined standard uncertainty, σCi, by a coverage factor of k=2.

6.5. Detection Limits

A. The instrument detection limit for Sr-90 measurement, also called the minimum detectable activity (MDA), is estimated using the modified Currie's detection limit expression (Currie, L.A., 1969) as described by Rucker (Rucker, T.L., 1995) (Sect 5.G) as follows:

$$MDA = \frac{t^2 + 2 \times t \times \sigma_{TMB}}{T_{IB} \times 60}, \text{ Bq} \quad (20)$$

where,

σ_{TMB} = Typical standard deviation of net method blank counts
t = t factor for df = (n -1) at α = 0.05
n = Number of method blanks used in the calculation of σ_{TMB}
T_{IB} = Live time of instrument background counting, min.
60 = Conversion factor from minutes to seconds

B. Method Detection Limit

The method detection limit for Sr-90 measurement, also called the minimum detectable activity concentration (MDCM), is estimated from the typical values for sample weight, counting efficiency, chemical yield, and method blank as follows:

$$MDC_M = \frac{t^2 + 2 \times t \times \sigma_{TMB}}{E_{TY} \times W_{TS} \times Y_{TY} \times T_{CMB} \times 60}, \text{ Bq/kg} \quad (21)$$

where,

σ_{TMB} = Typical standard deviation of net method blank counts
t = t factor for df = (n -1) at α = 0.05
n = Number of method blanks used in the calculation of σ_{TMB}
T_{CMB} = Live time for method blank counting, min.
W_{TS} = Typical sample weight, kg
E_{TY} = Typical counting efficiency of Y-90, cpm/dpm
Y_{TY} = Typical chemical yield of Y-90, fractional
60 = Conversion factor from minutes to seconds

C. Limit of Quantification of the Method

The quantification capability of the method, expressed as the Limit of Quantification (LOQM), is estimated from the typical sample weight, counting efficiency, chemical yield, and method blank as follows:

$$LOQ_M = \frac{50 \times \left(1 + \sqrt{1 + \left(\frac{\sigma_{TMB}^2}{12.5}\right)}\right)}{E_{TY} \times W_{TS} \times Y_{TY} \times T_{CMB} \times 60}, \text{ Bq/kg} \quad (22)$$

Where, the definitions for the variables in the equation are the same as those described in Sect 6.5.B.

6.6. Counting Efficiency Calibration

The instrument used for sample counting must be calibrated as described in WEAC-AB-TM.002, Use of Sr Resin to Generate Yttrium-90 Efficiency Attenuation Curve (see Sect 9.I).

6.7. Quality Control

A. General Requirements

The quality control (QC) samples, i.e., method blank, laboratory control sample (LCS), and sample duplicate; must be processed and analyzed to determine if the sample analysis meets QC criteria requirements. The QC criteria and the warning and control limits are pre-established according to the corresponding historical data or standard reference value(s) for each QC element.

The values of the limits and their supporting data are documented in the method QA logbook (WEAC.AB.Log.2.0). Any changes of QC criteria should also be clearly documented and kept in the method QA logbook (WEAC.AB.Log.2.0). All method QC data, reagent preparation sheets, and information related to QC are maintained in the method QA logbook (WEAC.AB.Log.2.0).

Upon completing each sample batch, promptly process samples and LCS and record data in the Analysis Database. Open the Sr90QCChart file (Sect 8.F) in order to verify specifications are met, identify any trends, abnormal changes, and/or outliers.

All QC sample results must meet their requirements and acceptance criteria as described in Sects 6.7.B – 6.7.E. If not, follow the corrective actions described in Sect 6.7.I.

B. Method Blank

A method/reagent blank must be included in each analytical batch to identify if contaminations from the reagents, laboratory ware, and surrounding environment have significantly affected the analysis. It must be prepared, processed, and analyzed in the same manner as the samples being analyzed.

The batch-specific blank result must be evaluated against the warning and control limits that have been established from at least 20 method blank/instrument background results. The method blank result for a sample batch is acceptable only if it falls within the warning limits or

between the warning and the control limits not more than three consecutive times. If the blank result is not acceptable, follow the corrective actions described in Sect 6.7.I.

C. Laboratory Control Sample (LCS)

Prepare the LCS using food-based matrix. The bulk material must be ashed following Sect 6.3.E of this SOP as appropriate. The reagent preparation document should state the amount of ash which is equivalent to either 250 g or 500 g of the original food product. The reagent record must be documented in the method QA logbook (WEAC.AB.Log.2.0). Homogenize the ash.

An LCS must be processed to demonstrate the measurement accuracy of the analysis. At least one LCS should be analyzed in a seven-day period. If multiple sample batches will be processed within the seven-day time period, the LCS should be analyzed along with the first sample batch, and the acceptable LCS result is applicable to the rest of the sample batches analyzed by a team of qualified analysts using the procedures described in this document. If the LCS result is not acceptable, refer to Sect 6.7.I for appropriate actions.

Note: Each LCS is prepared from food ash equivalent to 250 g of original food sample and spiked with a known Sr-90 activity at a level near or above the LOQ.

The z-score of the LCS result, defined as the difference between the measured and expected values divided by the root sum square of the respective standard uncertainties (Sect 5.E), must be evaluated against warning and control limits that are set as ±2 and ±3, respectively. The LCS result is acceptable only if its z-score is within ±2, or between ±2 and ±3 not more than three consecutive times. If the result is not acceptable, follow the corrective actions described in Sect 6.7.I.

Promptly view reported LCS data in order to verify specifications are met, as described in 6.7.A.

D. Duplicate/Replicate Sample

The relative standard deviation (RSD) of Y recovery for each sample batch must be evaluated against the warning and control limits, i.e., 2 and 3 times the RSDs calculated from historical Y recovery data, respectively. The variation of Y recoveries for a sample batch, shown as RSD, is acceptable only if it's within the warning limits or between the warning and control limits not more than three consecutive times. If the

result isn't acceptable, a note of explanation must be added to the QC summary page.

E. Acceptable Y Recovery

The Y recovery for each sample must be greater than 40% and less than 105% to be acceptable. If the Y recovery for a sample falls outside the acceptable range, the analysis should be repeated using Scheme #1 unless there is a directly observable reason that is unrelated to matrix, in which case Scheme #2 may be used. The data from the analysis that resulted in the recovery failure should be entered into the Analysis Database and properly annotated. If a sample is reanalyzed due to failed recovery, the recovery point for that sample does not need to be used in the %RSD calculation for associated batch.

Promptly view Y recovery reported in order to verify specifications are met, as described in 6.7.A.

F. Proficiency Evaluation Sample

External proficiency evaluation samples must be analyzed following the QA schedule in compliance with the WEAC-QMS. 5.9, WEAC, Proficiency Testing. Unacceptable results must be investigated, documented, the appropriate corrective action implemented, and affected samples reanalyzed.

G. Preparation of Reagents and Standards

Document the preparation of each batch of reagent and/or standard using the Analysis Database. These reagent preparation sheets are maintained in the H:\Analytical Branch\ Radiochemistry\Reagent Prep Worksheets directory (see Sect 8.F).

H. Analytical Data Review

The analyst who performs the analysis is responsible for the initial review of their analytical results. The data review should verify the implementation of this method SOP and its quality assurance procedures. Follow the elements listed in Sect 6.15.D.1 of WEAC-QMS.2.0, WEAC Quality Assurance/Quality Control Program to review analytical data.

I. Corrective Action

Take corrective action according to WEAC-QMS.4.11 WEAC Corrective Action Procedures when nonconformity to any QC requirement is identified. The corrective action may include but is not limited to the following:

1. Check the results for any transcription/calculation error(s);
2. Confirm the measurement;
3. Reevaluate working standard(s) or reagent(s);
4. Review facility maintenance records for abnormal changes in the environment;
5. Check whether the instruments are set properly;
6. Recalibrate the instrument; and
7. Decontaminate the laboratory ware.

J. Method Precision and Bias

WEAC-RN-Method.2.0 has been tested using certified reference material and various proficiency test samples.

Table 2 summarizes the sample matrices, number of samples per matrix tested, and the associated activity range for matrices used to demonstrate method validation when the method was originally modified on 9/23/03.

The typical method precision estimated from the acceptable results was found to vary with the sample activity as shown in Table 3. In addition, the typical method bias, as shown by the ratio of the measured value and the known value, had a grand mean of 0.96.

Table 2. Typical Method Precision and Bias

Sample Matrix	Number of Sample	Activity Range (Bq/sample)	Typical Precision (1σ, %)	Typical Bias (Measured/Known)
Bone	24	0.06 – 0.4	9	1.02
Water	29	0.2 – 5	5	0.95
Milk	24	0.6 – 2	3	0.91

Table 3 provides an intercomparison of the accuracy and precision of laboratory control sample results from 2004 thru 2011. **Scheme #1** was used for measurement in the years between 2004 and 2010. Scheme #2 was used in year 2011. The LCS ash matrix used each year are as follows: water - 2004, pasta - 2005, green bean - 2006, spinach - 2007, water - 2008, dry milk – 2009, and berry juice - 2010. LCS samples in 2011 used either berry juice or dry milk ash matrices. Method accuracy results for **Scheme #1** varies between 99% and 105% while method accuracy results for **Scheme #2** was 102.4%.

Table 3. Interannual comparsion of precision, LCS z-scores, yield, and sample size (n)

MB Year	2004	2005	2006	2007	2008	2009	2010	2011
Sample Size (n)	51	45	44	28	18	32	41	32
precision (RSD)	5.10%	7.07%	4.92%	8.09%	4.87%	3.39%	4.11%	4.92%
z-score mean	1.20	0.10	-0.21	-0.03	0.87	-0.25	0.54	0.53
z-score SD	1.39	1.72	0.97	1.94	0.96	0.90	1.00	1.30
Sr-90 mean	104.52%	100.63%	99.00%	100.27%	104.84%	99.21%	102.17%	102.38%
Sr-90 SD	5.33%	7.12%	4.87%	8.11%	5.10%	3.37%	4.20%	5.04%

K. MDA, MDC, and LOQ

The typical instrument minimum detectable activity (MDA), minimum detectable activity concentration (MDCM), and limit of quantification (LOQM) for this method are estimated to be 0.012 Bq, 0.063 Bq/kg, and 0.3 Bq/kg, respectively for Scheme #1 and 0.023 Bq, 0.122 Bq/kg, and 0.6 Bq/kg, respectively for Scheme #2.

6.8. Safety and Hazardous Waste Management

The waste generated by this method must be disposed of in accordance with WEAC-LAB.12.0, Chemical Hygiene Plan; WEAC-LAB.14.0, Hazardous Waste Management Program; WEAC-LAB-RS.004, Radioactive Waste Handling Procedure; and WEAC-LAB-RS.002, WEAC Radiation Safety Manual. Consult the RSO, IH, or supervisor concerning the safety and waste disposal procedures. Analysts must comply with the following precautions:

A. Use well-ventilated fume hoods and wear protective laboratory gears including gloves, safety glasses, and a laboratory coat when performing radiochemical analysis.

B. Adequately dry samples before putting them into the ashing oven to prevent spattering and boiling over. Ensure sample ashing is performed by gradually ramping up the temperature to prevent samples from catching on fire. These precautions also reduce the amount of smoke escaping from the oven into the ashing room and surrounding areas.

C. Wear dust masks to avoid inhaling contaminants when transferring the ash between different containers.

D. Examine all glassware used for sample analysis for crack and damage prior to use.

E. Check the cap and drain stopper on each separatory funnel to ensure that they can function properly prior to use. This ensures that the internal pressure build up from the exothermic reaction can be vented without difficulty and prevents leakage during the extraction.

F. When using the automatic shaker, ensure that reagents stored within and below the hood (especially those in glass containers) are placed far enough away so vibrations from the shaker don't cause damage.

G. Make sure the shaker arms are properly tightened and that caps and o-rings are properly secured. Also keep watch that these parts haven't loosened up during shaking.

H. Always wear protective gloves and safety glasses when handling hydrofluoric acid (HF), work in a well-ventilated fume hood, and have an HF spill kit nearby.

WARNING: **NEVER** store or use HF in glassware.

I. Inspect all centrifuge tubes for leakage or damage and properly balance prior to centrifuging.

J. Rinse all laboratory ware that have been in contact with acids or bases with water before handing over for dish washing.

K. Clear out the fume hood after each use. Don't use the fume hood to store clean glassware and apparatus.

L. Store regular chemical wastes and radioactive wastes separately. Return labeled waste containers to their proper (labeled) storage area after use. The following nine waste containers must be designated for waste collection and safe disposal:

1. 14N HNO3 saturated with TBP
2. 14N HNO3 saturated with TBP+Sr90/Y90 (Rad Waste)
3. TBP saturated with 3N HNO3
4. 72% 3N HNO3+28% concentrated NH4OH
5. 6% concentrated HCl+6% concentrated HF+88% H2O
6. 8% concentrated HCl+8% saturated H3BO3+8% concentrated NH4OH+76% H2O
7. 6% concentrated HCl+29% 2N (COOH)2+6% concentrated NH4OH+59% H2O
8. 60% H2O+40% absolute ethanol

9. YOX+glass fiber filters (Rad Waste)

7. Glossary/Definitions

A. **DQO**: Data quality objective

B. **FV/PM**: Function Verification / Preventive Maintenance

C. **IH**: Industrial Hygienist

D. **Laboratory Grade Water**: Water that has been treated so it's free from traces of dissolved metal, bactericidal, and inhibitory compounds.

E. **LCS**: Laboratory Control Sample

F. **LOQ**: Limit of quantification

G. **MDA**: Minimum detectable activity

H. **MDC**: Minimum detectable concentration

I. **MDC_M**: Minimum detectable activity concentration (also called method detection limit)

J. **Method blank**: For the purposes of this procedure, a method blank is synonymous with a reagent blank and does not imply the addition of food ash.

K. **RSD**: Relative standard deviation

L. **RSO**: Radiation Safety Officer

M. **Sr-90**: Strontium-90

N. **TBP**: Tributylphosphate

O. **Y-90**: Yttrium-90

8. Records

A. Analysis Database

(File location: H:\Analytical Branch\ Radiochemistry\Analysis Database.accde)

B. Analytical worksheet generated from Analytical Database

C. Calculation Attachments (Sample and LCS) from Analytical Database

D. Counting data report for samples, method blank, and LCS

E. Instrument background report

F. Reagent Prep (Files location: H:\Analytical Branch\Radiochemistry\ Reagent Prep Worksheets)

G. Strontium-90 QC Charts (File location: H:\Analytical Branch\Radiochemistry\WEAC.AB.Log.2.0-Sr-90 Method\QC Charts)

H. WEAC.AB.Log.2.0 Strontium-90 in Foods by Internal Gas-Flow Proportional Counting logbook (File location: H:\Analytical Branch\ Radiochemistry\WEAC.AB.Log.2.0-Sr-90 Method)

I. WEAC-TMPL.003, Temperature Record Form

J. WEAC-TMPL.112, Analytical Branch Laboratory Balances Function Verification/Preventive Maintenance Sheet

9. Supporting Documents

A. WEAC-AB.3.0 Monitoring of Freezers, Incubator, Ovens, Water Baths & Refrigerators

B. WEAC-AB.6.0 Gravimetric Calibration Check of Pipettes

C. WEAC-AB-7.0 Verification of Thermometers and Thermocouples

D. WEAC-AB.8.0 Laboratory Sub Sampling

E. WEAC-AB-RN.3.0 Nabertherm Furnaces

F. WEAC-AB-RN.8.0 Calibration, Use and Maintenance of Clocks used for Measuring Time for the Purpose of Decay Correction

G. WEAC-AB-RN.10.0 Bench Top Centrifuges

H. WEAC-AB-RN.019 Gas-Flow Low-Level Alpha/Beta Proportional Counter

I. WEAC-AB-TM.002 Generation of Yttrium-90 Efficiency Attenuation Curve Using Sr Resin Extraction Chromatography

J. WEAC-LAB.6.0 Laboratory Balances

K. WEAC-LAB.12.0 Chemical Hygiene Plan

L. WEAC-LAB.14.0 Hazardous Waste Management Program

M. WEAC-LAB-RS.002 WEAC Radiation Safety Manual

N. WEAC-LAB-RS.004 Radioactive Waste Handling Procedure

O. WEAC-QMS.2.0 WEAC Quality Assurance-Quality Control Program

P. WEAC-QMS.4.11 WEAC Corrective Action Procedure

Q. WEAC-QMS.5.9 WEAC Proficiency Testing

10. Document History

Revision #	Status* (D, I, R)	Date	Author Name and Title	Approving Official Name and Title
1.0	I	12/3/04	KELLY GARNICK, CHEMIST ZHONGYU WU, CHEMIST	PAMELA MACKILL, ANALYTICAL BRANCH DIRECTOR
2.0	R	5/24/05	KELLY GARNICK, CHEMIST ZHONGYU WU, CHEMIST	PAMELA MACKILL, ANALYTICAL BRANCH DIRECTOR
3.0	R	7/29/05	KELLY GARNICK, CHEMIST ZHONGYU WU, CHEMIST	PAMELA MACKILL, ANALYTICAL BRANCH DIRECTOR
3.1	R	11/9/05	KELLY GARNICK, CHEMIST ZHONGYU WU, CHEMIST	PAMELA MACKILL, ANALYTICAL BRANCH DIRECTOR
3.2	R	2/11/06	KELLY GARNICK, CHEMIST ZHONGYU WU, CHEMIST	PAMELA MACKILL, ANALYTICAL BRANCH DIRECTOR
3.3	R	3/27/06	KELLY GARNICK, CHEMIST ZHONGYU WU, CHEMIST	PAMELA MACKILL, ANALYTICAL BRANCH DIRECTOR
3.4	R	5/11/06	ZHONGYU WU, CHEMIST	PAMELA MACKILL, ANALYTICAL BRANCH DIRECTOR
4.0	R	1/9/07	ZHONGYU WU, CHEMIST	PAMELA MACKILL, ANALYTICAL BRANCH DIRECTOR
4.1	R	6/5/07	ZHONGYU WU, CHEMIST	PAMELA MACKILL, ANALYTICAL BRANCH DIRECTOR
5.0	R	7/10/09	ZHONGYU WU, CHEMIST	PAMELA MACKILL, ANALYTICAL BRANCH DIRECTOR
5.1	R	9/30/09	ZHONGYU WU, CHEMIST	PAMELA MACKILL, ANALYTICAL BRANCH DIRECTOR
6.0	R	5/7/10	ZHONGYU WU, CHEMIST	PAMELA MACKILL, ANALYTICAL BRANCH DIRECTOR
7.0	R	5/8/12	KELLY GARNICK, CHEMIST	PAMELA MACKILL, ANALYTICAL BRANCH DIRECTOR
7.1	R	5/10/12	LESLEY KERR, QSM	PAMELA MACKILL, ANALYTICAL BRANCH DIRECTOR

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8.0	R	9/2/14	KELLY GARNICK, CHEMIST	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR
8.1	R	12/3/14	KELLY GARNICK, CHEMIST	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR
8.2	R	2/8/2016	ANTHONY WETHERBY, CHEMIST KELLY GARNICK, CHEMIST	CONG WEI, ACTING ANALYTICAL BRANCH DIRECTOR
8.3	R	12/23/2016	CHRISTINE KARBIWNYK, CHEMIST	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR
8.4	R	6/22/2017	KELLY GARNICK, CHEMIST	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR
09	R	1/9/2020	KELLY GARNICK, CHEMIST	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR
10	R	4/3/2020	JENNIFER SZYMANSKI, CHEMIST	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR
11	R	SEE INFOCARD	BRIAN SWEENEY, CHEMIST	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR

* - D: Draft, I: Initial, R: Revision

11. Change History

Revision #	Change
11	Updated Section 6.3.E.2 to add a reference to WEAC-MEMO-2014-06-18.01.

12. Attachments

None