Sections in This Document

1. Purpose

This standard operating procedure describes the analytical procedure, data processing, and quality control requirements for analysis of strontium-90 (^{90}Sr) in food through Cerenkov liquid scintillation counting of yttrium-90 (^{90}Y).

2. Scope

This method is suitable for analyzing foods containing ^{90}Sr known to be in a radioactive equilibrium with its progeny ^{90}Y. Presence of ^{91}Y in the early stage of a nuclear explosion or nuclear power plant accident can interfere with ^{90}Y analysis. However, the apparent complication can be avoided a few years after the incident when ^{91}Y ($T_{1/2}$ = 58.5 days) has decayed to negligible levels in comparison with ^{90}Y. The method has the ability to analyze a wide range of foods up to 0.25 kg each. The minimum detectable concentration (MDC) and limit of quantification (LOQ) for the method are estimated to be 0.08 Bq/kg and 0.32 Bq/kg, respectively, based on 0.25 kg sample, 80% chemical recovery, 53% counting efficiency, 1.08 cpm blank count rate, and 100-minutes count time. The accuracy and precision for the method are found to be better than ±7.2% and 7.2% at 95% confidence level provided that the sample ^{90}Sr concentration is equal to or greater than the LOQ.

3. Responsibility

A. Supervisors

1. Ensure analysts who use this method receive appropriate training.
2. Ensure this procedure is properly implemented.
3. Ensures the analyst is capable of providing acceptable analytical results through proficiency evaluation.

B. Analysts

1. Adhere to this procedure when performing sample analysis.
2. Exercise proper safety precautions throughout the sample analysis.
3. Ensure acceptable quality control data supports the analytical results.
4. Document and report any safety and methodological problems encountered to the supervisor.

5. Properly disposes of chemical and radioactive wastes resulting from sample analysis.

4. Background

In this method, the ^{90}Sr activity is determined via its progeny ^{90}Y. Therefore, ^{90}Y in the sample must be in radioactive equilibrium with ^{90}Sr at the time of sample analysis. A 250-g portion of homogenized food sample is ashed, spiked with a known amount of stable Y carrier, digested in concentrated HNO_3, and filtered. The $^{90}Y(Y)$ in the sample filtrate of ~12M HNO_3 is selectively extracted using 1.5 g of DGA resin. The resin impregnated with $^{90}Y(Y)$ is filtered through a column and then washed using 0.05M HNO_3. The $^{90}Y(Y)$ is stripped from the resin using 0.5M HCl and the eluent is collected and evaporated to dryness. After treating and drying with concentrated HNO_3, the resulting sample is dissolved in 2 mL of 1M HNO_3 and then passed through a column filled with 1-mL TRU resin into a 20-mL polyethylene liquid scintillation vial. The column is washed with additional 8 mL of 1M HNO_3 and the sample solution in the vial is fully mixed for Cerenkov counting of ^{90}Y using a liquid scintillation counter (LSC). The sample ^{90}Y recovery is determined by quantifying Y carrier remaining in the LSC sample using an X-ray fluorescence (XRF) analyzer. The described method procedure is illustrated in Attachment A.

5. References

A. Section 2.1 - Food Edible Portion, Elemental Analysis Manual for Food and Related Products
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B. Section 2.2 - Food Homogenization, Elemental Analysis Manual for Food and Related Products
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F. Multi-Agency Radiological Laboratory Analytical Protocols Manual, NUREG-1576, EPA 402-B-04-001C, NTIS PB2004-105421, July 2004 http://www.epa.gov/radiation/marlap/manual.html

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H. National Nuclear Data Center, http://www.nndc.bnl.gov/nudat2/

I. Reference Manual, QuantaSmart for the TriCarb Liquid Scintillation Analyzer (Models 4810TR, 4910TR, 5110TR, and Quantulus GCT 6220), Version 5.2, Rev. A, PerkinElmer, 2017

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L. WinTrace User’s Guide, Ver. 4.0, Thermo Electron Corporation, 2005

M. Instrument Manual, Wallac 1220 Quantulus, Ver. 1.D, PerkinElmer, 2002

N. WinQ User’s Manual, WinQ user interface 1224-307, PerkinElmer, 2002

O. Easy View User’s Manual, Easy View spectrum analysis program 1224-534, Ver. 1.0, PerkinElmer, 2002

6. Procedure

6.1. Equipment

Note: Ensure that the following equipment is ordered from an approved vendor.

- Fume hoods with utilities: vacuum, water, and power outlets
- Analytical balances with 0.1, 0.001, and 0.00001 g readabilities
- Top loading balances with 0.1 g readabilities
- Freezer
- Refrigerator
- Furnaces programmable up to 1000 °C
- Milli-Q water purification system
- Welch™ DryFast™ and DryFast Ultra™ diaphragm vacuum pump
- Welch™ Inlet/Exhaust Traps
- Digital hotplates or equivalent
- Multi-position stirrer
- Digital waving rotator
- Ultra-low background liquid scintillation counters
- Energy dispersive X-ray fluorescence analyzer (ARL QUANT'X, Thermo Scientific)
- Automated powder dispenser (QS30, Mettler-Toledo, LLC)
- MicroVac™ portable cleanroom vacuum cleaner, handheld
- Handheld density meter (Mettler-Toledo, LLC)
- Sarstedt Inc 30-mL flat bottom polycarbonate tube, Sarstedt Inc 30-mL polypropylene tube or equivalent
- XRF sample cups (Cat. # 2131, Chemplex)
- XRF prolene® thin film (Cat. # 426, Chemplex)
- Test paper for leak test of XRF sample cups (Cat. # 6150, Chemplex)
- Plastic trays for XRF samples
- LSC vial holder block
- 500 mL Coors@ dishes
- 20-mL Teflon-coated, anti-static polyethylene LSC vials

  Note: The LSC vial should be stored at room temperature and away from light exposure.

- 20-, 50-, and 100-mL vial racks
- 25-well HotBlock digestion system or equivalent
- Racks for 100-mL digestion cup
- 100-mL FlipMate digestion cups with screw caps
- 100-mL DigiTube
- Filter assembly with 1.5 µm glass fiber filter
- 12-port FlipMate vacuum manifold

- NPT barbed to port fitting
- Digestion cup, FlipMate 100 systems
- Plastic watch glasses
- 10-cm Plastic columns
- 145-mL plastic funnels
- 2-mL plastic columns
- Column racks
- 12-hole or 24-hole vacuum box/rack
- Column/funnel assembly plate holder
- White inner support tubes
- Yellow outer tips
- 1-way polycarbonate stopcocks
- 250-mL glass beakers and matching watch glasses
- Class A glass graduated cylinders used for reagent preparation
- 50-mL bottle top dispensers
- 250-mL Teflon spray bottles
- Top cutout 1000-mL high density polypropylene square bottles
- 120-mL polypropylene specimen cups with caps
- 2-mL plastic transfer pipettes
- 1-mL fine-tip plastic transfer pipettes
- 5-mL long-stem plastic transfer pipettes
- 1-mL calibrated automatic pipette and tips
- 5- and 10-mL adjustable pipettes and tips
- Paper towels
- Gloves, safety glasses, mask and laboratory coat, and full-face shield
- Fine-tip and ultra-fine tip permanent markers
- Digital clock and timer
- Teflon-coated stir bars
- Stir bar retrievers
- Firm-bristle, flat nylon brush for ash transfer or equivalent
- Polypropylene vented caps (Performance Systematix Inc)
- Wax-coated glass waste bottles
- Chemical waste labels
- Radioactive labels
- Inspection mirrors
- Acid spill kit
- Aluminum weighing trays

6.2. Reagents and Standards

- 10” round pre-cut parchment paper liners
- Copper (Cu) XRF energy adjustment disk (Cat. # OFHC, Thermo Scientific)
- DGA resin (50–100 μm, N, N, N’, N’-tetra-n-octyldiglycolamide, Normal, Part #DN-B50-S or #DN-B01-S, Eichrom):

  Note: DGA resin must be fully wetted before use. To aliquoted and wet DGA resin, weigh 1.5 g of DGA resin into a clean plastic vial (Automated powder dispenser may be used for batch preparation according to Attachment H) and then add 10 mL of 3M HNO_3 into the vial. Wet the resin using a waving rocker overnight or at least one day ahead of sample analysis. The wetted resin can be stored up to one year. Each vial of DGA resin should be labeled with resin quantity, lot #, and date of dispensing. A DGA resin sign-up sheet is provided to track resin use.

  Note: Although DGA resin come with one-year manufacturer warranty, the manufacturer has instructed that the warranty begins with the time of purchase, not the time of manufacture. If the resin is kept dry in original bottle, the expiration day can be up to 2 years.

- TRU resin (100-150 μm, Part # TR-B50-A, Eichrom):

  Note: TRU resin must be pre-conditioned before use. To aliquoted and pre-condition TRU resin, weigh 0.37 g of TRU resin into a clean plastic vial (Automated powder dispenser may be used for batch preparation according to Attachment H) and then add 5 mL of 1M HNO_3 into the vial. Wet the resin using a waving rocker for about 10 minutes. The wetted resin can be stored up to one year. Each vial of TRU resin should be labeled with resin quantity, lot #, and date of dispensing.

  Caution: Pure resin must be used for effective separation. To prevent mixing DGA with TRU resin, each resin has a loading head for the powder dispenser reserved specifically for the resin type. The chance for contamination may be reduced by dispensing the entire contents of a resin bottle in one weighing session.

  Note: Although TRU resin come with one-year manufacturer warranty, the manufacturer has instructed that the warranty begins with the time of

purchase, not the time of manufacture. If the resin is kept dry in original bottle, the expiration day can be up to 2 years.

- Laboratory grade water
- Concentrated HNO_3 (15.8M), ACS reagent grade or equivalent
- 3M HNO_3: Dilute 190 mL of 15.8M HNO_3 to 1 L with laboratory grade water
- 1M HNO_3: Dilute 63 mL of 15.8M HNO_3 to 1 L with laboratory grade water

  Note: After preparation, the solution density and standard uncertainty must be determined and recorded on reagent preparation sheet.

- 0.05M HNO_3: Dilute 3.2 mL of 15.8M HNO_3 to 1 L with laboratory grade water
- Concentrated HCl (12M), ACS reagent grade or equivalent
- 0.5M HCl: Dilute 41.7 mL of 12M HCl to 1 L with laboratory grade water
- NIST traceable Y carrier standard, 1 mg/mL in 1M HNO_3, with known solution density, g/mL
- NIST traceable Y batch standard, 100 µg/mL in 1M HNO_3
- XRF standards for Y calibration curve (See Attachment G)
- NIST traceable ^{90}Sr LCS spike, 1-2 Bq/mL

  Note: Must be stored in an air-tight container. When ^{90}Sr LCS spike standard contains Y carrier, the bias in Y recovery caused by the ^{90}Sr spike must be <0.1%. Otherwise, the bias must be corrected. If the Y concentration in the standard is ≥1 µg/mL and not accurately quantified by the supplier, the Y concentration and associated uncertainty must be determined in-house by XRF analysis unless the standard certificate states that it is Y carrier free.

- Purified ^{90}Y standards for determining Cerenkov counting efficiency (See Attachment F)
- Food ash matrix blank for preparation of LCS
- Concentrated hydrogen peroxide, ACS reagent grade or equivalent

6.3. Sample Preparation

A. Handling Laboratory Sample

1. Upon sample receipt, verify sample against sample collection report for any non-conformance.

2. Preserve sample at proper temperature in a secure area until time of analysis.
3. Record sample information per sample chain of custody requirements.

B. **Compositing Analytical Sample**

When sample homogeneity is in doubt, a representative analytical portion must be prepared as follows:

1. Perform random subsampling to yield a composite sample from the received laboratory sample.
2. Obtain food edible portion as described in Reference A.
3. Homogenize edible sample portion and acquire representative analytical sample as described in Reference B.

C. **Ashing Analytical Sample**

1. Ensure that the balance and furnace to be used meet QC requirements.
2. Tare a clean Coors@ dish on balance.

   Note: If sample is known to bond to ashing dish at the end of ashing cycle, lay a piece of 10” round parchment paper in the ashing dish before weighing the sample into the dish.

3. Weigh ~250 g of sample into the dish to nearest 0.1 g.
4. Spread out the sample in the dish evenly.
5. Record sample weight and dish ID on analytical worksheet.
6. Ash sample per the ashing method described below.

Ashing Method	Temperature Profile	Food Type	Dish Load
A	120 °C (48 hrs) 250 °C (4 hrs) 315 °C (4 hrs) 480 °C (8 hrs) 575 °C (16 hrs)	All foods	~250 g/dish
B	250 °C (4 hrs) 315 °C (4 hrs) 480 °C (8 hrs) 575 °C (16 hrs)	Dry foods, oil, butter, & salad dressing	~250 g/dish
		Honey, syrup, & sugar	~50 g/dish (5 dishes/sample)

C	575 °C (16 hrs)	Re-ashing*	N/A
D	120 °C (48 hrs)	Water	N/A

*Only required when ash contains large amount of carbon soot.

7. After completion of sample ashing, wait for the furnace to reach ambient temperature.
8. Remove sample ash from furnace and inspect the ash quality visually.

   Note: When ash appears to contain large amount of carbon soot, it should be re-ashed using the ashing method C listed in the table above.

9. Transfer all sample ash into a clean and labeled 100-mL FlipMate digestion cup or 100-mL DigiTube as described below.

   Note: Examine 100-mL FlipMate digestion cup or 100-mL DigiTube used for sample analysis for cracks and damage prior to digestion.

   Caution: A dust mask can be used to avoid inhaling contaminants during ash transfer.

Attribute of Ash	**Transfer Method**
Copious amount	Use spoon or brush to transfer ash into digestion cup
Miniscule or invisible	Rinse dish interior surface with 20 mL of conc. HNO_3 and then pour solution into digestion cup
Bonded to ashing dish	Digest ash in dish using 20 mL conc. HNO_3 and then pour solution into digestion cup

10. Cap the digestion cup and record its ID on analytical worksheet.

C. Sample Digestion

1. Weigh LCS ash equivalent to 250 g of food into a clean and labeled 100-mL FlipMate digestion cup or 100-mL DigiTube followed by addition of 1 mL of ^{90}Sr LCS spike. Record its ID and LCS ash weight on analytical worksheet. Process the LCS in the same manner as described below.

2. Prepare a reagent blank with a clean and labeled 100-mL FlipMate digestion cup or 100-mL DigiTube in the same manner as described below.
3. Set the HotBlock digestion system to 150 °C.
4. Slowly add 50 mL of conc. HNO_3 to each sample under a fume hood.

   Note: If the digestion cup already contains 20 mL of acid, adjust the sample volume to 50 mL with conc. HNO_3

5. Add 1 mL of 1 mg/mL Y carrier standard to each sample using a QC qualified pipette.
6. Mix each sample thoroughly.
7. Wait for the HotBlock to reach 150 °C.
8. Place transfer rack loaded with samples onto the HotBlock digestion system.
9. Cover each digestion cup with a plastic watch glass.
10. Heat each sample for 30 minutes.

    Note: Brown fumes may be given off by the sample.

    Caution: The digestion cup may delaminate if heated for more than 30 minutes. If delamination appears in the digestion cup, break up the blisters or bubbles resulted from delamination so the trapped sample digest can be released (sometimes it is just gas trapped inside). The broken flakes will be filtered out during sample filtration.

11. Remove each sample from the HotBlock and wait for sample to reach room temperature.
12. Rinse the droplets on the back of the watch glass into its sample digestion cup with ~3 mL of H_2O.
13. Label a clean 100-mL FlipMate digestion cup with sample ID for each sample and then draw a black line at 75- and 100-mL volume marks, respectively.

    Caution: Do not use 100-mL DigiTube for filtration as it is incompatible with filtration apparatus.

14. Make a filter apparatus for each sample by attaching a filter assembly on to its respective FlipMate digestion cup.
15. Attach each filter apparatus onto a 12-port FlipMate vacuum manifold connected to vacuum source.
16. First turn on vacuum source and then open the valve on the vacuum manifold for each sample.
17. Slowly pour each sample digest into its respective filter assembly.

    Note: If filtration is too slow, gently rub the filter surface with a plastic transfer pipette to re-suspend the precipitate.

    Note: In case of analyzing low yttrium recovery samples, allow the precipitate portion of the digest to settle and decant the liquid portion to the filtering apparatus.

18. Allow to drain completely.
19. Close the valve on the vacuum manifold for each sample.
20. Rinse each digestion cup with ~5 mL of H_2O and then pour the rinse into its respective filter assembly.

    Note: Ensure that the H_2O rinse is fully mixed with sample residue to allow total dissolution of $Sr(NO_3)_2$ precipitate that may be collected by sample residue and filter assembly.

    Note: The sample digest trapped in the groove around rim of the filter assembly should be thoroughly rinsed out.

    Note: In case of analyzing low yttrium recovery samples, add each H_2O rinse to the solid portion in the digestion tube and shake vigorously.

21. Open the valve on the vacuum manifold for each sample.
22. Allow to drain completely.
23. Repeat steps 19 – 22 until each sample filtrate volume reaches the 75-mL line mark.

    Note: In case of analyzing low yttrium recovery samples, add 25 mL of concentrated HNO_3 to the digestion tube, shake vigorously

and pour through the filter assembly. Allow the sample to drain completely and skip step 26.

24. Turn off the vacuum source and then disconnect each filter apparatus from the FlipMate vacuum manifold.
25. Remove filter assembly from each FlipMate digestion cup.
26. Adjust sample volume in each FlipMate digestion cup up to the 100-mL line mark with conc. HNO_3.

    Note: While adding conc. HNO_3, wash droplets on side of the digestion cup into the sample filtrate.

27. Cap each FlipMate digestion cup and then record its ID on analytical worksheet.

D. **Extraction of ^{90}Y**

1. Drop a Teflon stir bar into each -FlipMate digestion cup.
2. Place each FlipMate digestion cup on a stir plate then adjust stir plate speed (~500 rpm) to create a vortex capable of pulling resin vertically into the solution.
3. For each sample, shake a vial containing wetted DGA resin a few times and then pour the resin slurry into its respective FlipMate digestion cup.

   Note: The DGA resin wetted in 10 mL of 3M HNO_3 should be made available ahead of sample analysis.

   Note: It is normal to leave a small amount of DGA resin in the vial after transfer.

4. Let stir for ~15 minutes.
5. For each sample, prepare a funnel assembly by affixing a 145-mL plastic funnel on a 10-cm plastic column.
6. Label each column with sample ID.
7. Set up a vacuum box as shown in Attachment B with top-cutout 1-L high density polypropylene square bottles inside the vacuum box to collect sample filtrate from the columns.

Caution: Inspect Flipmate vacuum manifold and vacuum tube prior to use. To ensure safety, each column/funnel assembly must be firmly connected onto the vacuum box and secured by column/funnel assembly plate holder.

8. Pour the resin slurry into its respective column.
9. Apply vacuum to the vacuum box.
10. Allow to drain completely.
11. Record $^{90}Sr/^{90}Y$ separation time for each sample.
12. Shut off the vacuum and vent the vacuum box by disconnecting the vacuum tubing.
13. Add 10 mL of 0.05M HNO_3 to each FlipMate digestion cup and rinse the remaining resin on the wall into the solution.
14. Use the solution to wash all resin stuck on the funnel into the column.

    Note: If the column drains too slowly, gently stir the resin bed and scrape the top surface of the frit using a long-stem plastic transfer pipette.

15. Allow to drain completely.
16. Repeat steps 13 - 15 twice.
17. Detach each column assembly from the vacuum box.

    Note: The non-rad sample filtrates collected in polypropylene square bottles should be transferred into a waste bottle labeled “58% conc. HNO_3+18% H_2O+23% 0.05M HNO_3+1% 1M HNO_3+Y”.

    The LCS sample filtrate collected in polypropylene square bottle should be transferred into a rad waste bottle labeled “58% conc. HNO_3+18% H_2O+23% 0.05M HNO_3+$^{90}Sr/^{90}Y$”.

18. Remove funnel from each column then place the column on a column rack.
19. To reclaim Y from DGA resin, place a clean and labeled 250-mL glass beaker under each column.
20. Slowly add 10 mL of 0.5M HCl on to each column.
21. Allow to drain completely.

22. Repeat steps 20 - 21 twice.

    Note: To ensure higher Y recovery, the column must be allowed to drain completely before each addition.

23. Lift and drop each column on the rack a few times to collect as much of the eluate as possible.
24. Place each beaker containing sample solution on a hotplate.
25. Set hotplate temperature at ~300 °C to evaporate each sample solution to dryness.

    Caution: Lower the temperature to ~200 °C near the end of evaporation to avoid splattering. Ensure that no condensation remains on beaker wall.

26. Add 2 mL of conc. HNO_3 to each beaker.
27. Swirl each beaker to dissolve the residue and then evaporate it to dryness.

    Note: To avoid color quench during Cerenkov counting of ^{90}Y, the residue must be colorless. The residue can be discolored by evaporating it repeatedly with 1 mL of conc. HNO_3 and 1 mL of conc. H_2O_2.

E. **Purification of ^{90}Y**

1. Obtain a vial of prepared TRU resin for each sample.
2. For each sample, label an empty 2-mL Eichrom plastic column. Ensure a 2-mL column frit is inside the bottom of the column. If the frit is missing, use a clean polytetrafluorethylene (PTFE) rod to insert the frit inside the column gently to avoid breaking.
3. Set each labeled column on a column rack and place a plastic specimen cup under each column.
4. Transfer the TRU resin slurry into each labeled column and then rinse the residual resin into the column using 2-3 mL of 1M HNO_3.

   Note: An alternative way for preparing the TRU columns can be done by adding 1 mL of water to each 2-mL Eichrom plastic column and then draw a fill line at the top of the water level. Snap the

tip off each column then add enough TRU resin slurry to each column to yield a uniform resin bed up to the fill line.

5. Allow to drain until liquid is just at the top of the resin bed and then remove each plastic specimen cup from under each column. Columns can be capped to keep resin wet if not ready to add sample right away. 1M HNO_3 can be added if needed to keep the resin wet.

   Note: To yield a uniform resin bed, remove any air pockets trapped in the resin bed. If needed, add more 1M HNO_3 to the column and then re-suspend the resin to expel the air bubbles.

6. For each sample, Label and weigh a clean 20-mL polyethylene LSC vial to the nearest 0.00001 grams and record weight, date vials were received, and vial ID on analytical worksheet.

   Caution: Since the 20-mL polyethylene LSC vials tend to yellow over time, they must be used for sample analysis within 2 years from the date received in laboratory. Extended use beyond expiration date is only acceptable after the suitability of the vials has been demonstrated by successful analysis of LCS using the vial in question.

   Caution: Any writing, mark, or dust on the exterior wall of LSC vial will obstruct photon transmission. Tissue paper damped with ethanol can be used to clean the vial's exterior surface.

7. Place each vial under its respective column.

   Note: LSC vial holder block can be used to prevent vial tipping over while collecting eluate from column.

   Note: To avoid misidentification, pair each LSC vial under the column with its vial cap showing sample ID.

8. At room temperature, add 2 mL of 1M HNO_3 to each beaker saved in the step 27 of Section 6.3.E and gently swirl the beaker to dissolve the sample.

   Note: Ensure the reagent bottle contains sufficient 1M HNO_3 for processing the entire sample batch as the density for 1M HNO_3 needs to be the same in calculation of result.

9. Load each sample solution dropwise onto its respective column using a fine-tip 2-mL plastic transfer pipette.

   Note: The sample solution should be added in a manner to minimize resuspension of the resin bed.

10. Wait for drain to complete.
11. Add 2 mL of 1M HNO_3 to rinse each beaker and then load the solution dropwise onto its respective column.
12. Allow to drain completely.
13. Repeat steps 11 - 12 once.
14. Allow to drain completely.
15. Add 4 mL of 1M HNO_3 to rinse each beaker and then load the solution dropwise onto its respective column.
16. Allow to drain completely then lift and drop each column on the column rack a few times to collect the last few drops.
17. Firmly cap each vial and then mix each sample by gently swirling while keeping the sample solution away from the cap.

    Note: The additions of 1M HNO_3 should be done to maintain the final sample volume as close to 10 mL (or 10 grams) as possible.

18. Weigh each vial to the nearest 0.00001 grams and record weight on analytical worksheet.
19. Prepare an instrument background sample by adding 10 mL of 1M HNO_3 into a 20-mL polyethylene LSC vial to determine Cerenkov background.
20. Check each vial to make sure that there is no droplet hanging on the interior wall.

6.4. Liquid Scintillation Counting

A. Counting with Quantulus GCT 6220

1. Perform time-of-use instrument performance assessment (IPA) as described in Section 6.10. A.2 if it hasn't already been done that day.
2. Load each sample, LCS, reagent blank, and instrument background sample into a sample cassette.

3. Attach appropriate flag for TDSSR protocol to the sample cassette and then push flag tab out to the left.
4. Place the cassette on the right side of the sample changer deck then close the deck cover.
5. Open the counting protocol “TDSSR” then click <Worklist> tab.
6. Enter appropriate flag number in the <PID#> field and sample ID in the <Sample Name> field
7. Click <OK> to save the protocol.
8. Close the counter lid and then dark adapt the samples for at least 30 minutes.
9. Click the green <Start> button in the upper left corner of the screen.
10. Wait for sample counting to finish.

Note: The sample counting report is saved in C:\Packard\Tricarb\Results\TDSSR\YYYYMMDD_HHMM\

B. **Counting with Quantulus 1220**

1. If WinQ isn’t activated, click the “WinQ” icon on the Windows desktop.
2. Choose an available counter
3. Perform time-of-use instrument performance assessment (IPA) as described in Section 6.10. A.2 if it hasn’t already been done that day.
4. Load each sample, LCS, reagent blank, and instrument background sample into a sample tray and note its sample position number as shown in Attachment E.
5. Click <Users> from WinQ window.
6. Select folder “TDSSR” from the <Users> list.
7. Select the protocol corresponding to the counter to be used, for example, Cerenkov Counter 1, and then click <Edit>.
8. Click <Sample Parameters> to enter sample ID and position number for each sample according to its actual location in the sample tray.
9. Click <OK> to save the protocol.
10. Go to the Queue pane, select the counter tab for the corresponding counter to be used.
11. Click <Queue> to send the counting protocol to the Queue pane.

12. Close the counter front door and then dark adapt the samples for at least 30 minutes.

    Note: One of the following existing counting protocols can be added before a sample counting protocol to provide 30 min wait time:

    For CTR1: 30 Min Delay CTR1
    For CTR2: 30 Min Delay CTR2
    For CTR3: 30 Min Delay CTR3

13. Click<Counters> and then "►" to start sample counting.
14. Wait for sample counting to finish.

    Note: Depending on which counter is used, the sample counting report can be found in the folder located in C:\D\TDSSR\CTRx\Sxxx.

    For example: C:\D\TDSSR\CTR1\S005

6.5. Determination of ^{90}Y Recovery

A. Preparation of XRF Sample

1. For each sample, assemble an XRF sample cup as shown in Attachment C.

   Note: The film must be mounted onto XRF sample cup flat, tight, and wrinkle free.

2. Write sample ID on cup cap then record it in analytical worksheet.
3. Transfer sample solution from scintillation vial into the cup.

   Note: The sample transfer can be carried out by pouring or using a disposable transfer pipette, given that bulk of the sample solution is transferred from scintillation vial into XRF sample cup.

4. Prepare a Y batch standard by assembling an XRF sample cup and then adding 10 mL of 100 µg/mL Y standard solution into the cup.
5. Use an inspection mirror to check each filled XRF sample cup for air bubbles and ensure there are no air bubbles appearing on cup film.

6. Cap each XRF sample firmly and then place it on a piece of leak-test paper for leakage test.

   Note: Any wet spots shown on leak-test paper indicate leakage.

   Note: To minimize change in Y concentration resulting from evaporation of sample solution, the XRF analysis should be run as soon as possible after the sample is transferred into XRF sample cup.

   Note: If a sample cup leaks inside the XRF analyzer, remove the leaking sample from the instrument, use paper towel to soak up any visible sample solution, and notify instrument monitor immediately.

B. **Determination of Sample Y Concentration**

1. Perform time-of-use instrument QC as described in Section 6.10. A.3 to ensure that XRF analyzer meets the QC requirements.
2. Open <Acquisition Manager>.
3. Click File>New>Quantitative Tray.
4. Enter sample ID in <Sample> column.
5. Click the file icon in <Method file> column.
6. Select valid Y method file then click <Open>.

   Note: The valid Y method is the one that has a file name with the most recent date.

7. Click the empty cell in <Sample> column then enter next sample ID.
8. Repeat step 7 for the rest of samples and Y batch standard.
9. Click the bottom empty cell in <Sample> column.
10. Open sample chamber lid.
11. Place samples and Y batch standard in the sample tray according to their IDs and position numbers on screen.

    Note: To avoid damaging the motors that turn the carousel inside the XRF and could cause misalignment between sample and detector, do not move or adjust XRF carousel by hand even

though sample position 1 is not at the start position from the previous run.

12. Ensure that the sample position in the sample tray matches the entry on screen.
13. Close sample chamber lid and then click <GO>.
14. Wait for analysis to finish.
15. Click File>Save Report…
16. Click ►QUANT'X shown in the path tab located on top of the screen.
17. Double click TDS folder.
18. Enter file name "MMDDYY_XRFx_Initial"

    For example: 050417_XRF1_JP

19. Click <Save>.

    Note: The saved sample analysis report is located in C:\QUANT'X\TDS

    Caution: To avoid changing original file structure, do not edit or save sample analysis report using MS Word.

20. Click File>Exit.
21. Click <No>
22. Remove all samples from sample tray after sample analysis is completed.

    Caution: Do not keep XRF samples inside instrument chamber after sample analysis. Acid fumes can diffuse out of the sample cups and corrode instrument.

23. Transfer each sample solution from XRF cup into a waste bottle labeled with "58% conc. HNO_3+18% H_2O+23% 0.05M HNO_3+1% 1M HNO_3+Y".

6.6. Calculation of Analytical Results

The validated Excel spreadsheet named "FORM-001190 Excel Spreadsheet - Calculation of ^{90}Sr Activity Concentration" must be used for calculation of analytical results. As directed by the spreadsheet,

A. Enter the analytical data collected throughout sample preparation into the spreadsheet.

B. Enter the counting data shown in liquid scintillation counting report into the spreadsheet.

C. Enter the Y analysis results shown in XRF analysis report into the spreadsheet.

6.7. Instrument Calibration

A. Determination of ^{90}Y Cerenkov Counting Efficiency

In general, a valid counting efficiency has already been determined and made available for routine use. If the efficiency needs to be updated, see Attachment F.

B. Calibration of XRF Analyzer

In general, a valid Y calibration curve has been already established and made available for routine use. If a new curve needs to be generated, see Attachment G.

6.8. Calculations

A. Sample ^{90}Sr Activity Concentration

The net sample ^{90}Sr activity concentration, C_{Sr}, at a given reference time, T_2, is calculated as:

$$C_{Sr} = \frac{A_s - A_{rb}}{W_s} \times D_{Sr} \quad (1)$$

$$D_{Sr} = e^{\lambda_{Sr} \times (T_1 - T_2)} \quad (2)$$

$$W_s = W_2 - W_1 \quad (3)$$

Where,

A_s = Instrument background corrected sample ^{90}Sr activity, Bq

A_{rb} = Instrument background corrected reagent blank ^{90}Sr activity, Bq

W_s = Net sample weight, g

D_{Sr} = ^{90}Sr decay correction factor

λ_{Sr} = 6.592 x 10^{-5}, decay constant of ^{90}Sr, day^{-1}

T_1 = Time of ^{90}Sr/^{90}Y separation for sample

T_2 = Sample reference time

W_1 = Weight of empty ashing dish, g

W_2 = Weight of filled ashing dish, g

A_s and A_{rb} are related to their respective count rates, ^{90}Y counting efficiencies, and Y recoveries as follows:

$$A_s = \frac{R_s}{E_y \times Y_s \times 60} \times D_{Y_s} \quad (4)$$

$$A_{rb} = \frac{R_{rb}}{E_y \times Y_{rb} \times 60} \times D_{Y_{rb}} \quad (5)$$

$$D_{Ys} = e^{\lambda_Y \times (T_3 - T_1)} \quad (6)$$

$$D_{Yrb} = e^{\lambda_Y \times (T_4 - T_7)} \quad (7)$$

Where,

R_s = Instrument background corrected sample ^{90}Y count rate, cpm

R_{rb} = Instrument background corrected reagent blank ^{90}Y count rate, cpm

R_i = Instrument background count rate, cpm

E_y = ^{90}Y Cerenkov counting efficiency, %

Y_s = Sample Y recovery, %

Y_{rb} = Reagent blank Y recovery, %

λ_Y = 2.599 x 10^{-1}, decay constant of ^{90}Y, day^{-1}

D_{Ys} = Sample ^{90}Y decay correction factor

D_{Yrb} = Reagent blank ^{90}Y decay correction factor

T_3 = Time at sample mid-count, i.e., $T_3 = T_5 - \frac{T_s}{2}$

T_4 = Time at reagent blank mid-count, i.e., $T_4 = T_6 - \frac{T_{mb}}{2}$

T_5 = Time at end of sample count

T_6 = Time at end of reagent blank count

T_7 = Time of $^{90}Sr/^{90}Y$ separation for reagent blank

T_s = Sample count time, min

T_{rb} = Reagent blank count time, min

Since ^{90}Y decay correction is only applicable to the ^{90}Ycount rates contributed by sample and reagent blank, the instrument background is subtracted from the observed sample ^{90}Y count rate and the observed reagent blank ^{90}Y count rate, respectively, as follows:

$$R_s = R_{os} - R_i \quad (8)$$

$$R_{rb} = R_{orb} - R_i \quad (9)$$

Where,

R_{os} = Observed sample ^{90}Y count rate, cpm

R_{orb} = Observed reagent blank ^{90}Y count rate, cpm

R_i = Instrument background count rate, cpm

After Cerenkov counting, Y concentrations for each sample and reagent blank are measured using XRF analyzer for determination of Y recovery.

$$Y_s = \frac{C_s \times \Delta M_s}{D \times C_y \times V_{Ys} \times 1000} \times F \times 100 \quad (10)$$

$$Y_{rb} = \frac{C_{rb} \times \Delta M_{rb}}{D \times C_y \times V_{Yrb} \times 1000} \times F \times 100 \quad (11)$$

As the laboratory control sample (LCS) analyzed along with sample batch is often prepared using a ^{90}Sr spike standard containing tens of micrograms of Y, the Y recovery for LCS sample needs to be calculated by taking the extra Y from the ^{90}Sr spike standard into account:

$$Y_{lcs} = \frac{C_{lcs} \times \Delta M_{lcs}}{D \times \mathrm{Y}_{total}} \times F \times 100 \quad (12)$$

$$Y_{total} = C_y \times V_{Ylcs} \times 1000 + C_{YSr90} \times V_{Sr90} \quad (13)$$

Furthermore,

$$\Delta M_s = M_{s2} - M_{s1} \quad (14)$$

$$\Delta M_{rb} = M_{rb2} - M_{rb1} \quad (15)$$

$$\Delta M_{lcs} = M_{lcs2} - M_{lcs1} \quad (16)$$

$$F = \frac{C_{std}}{C_{XRF}} \quad (17)$$

Where,

Y_s = Y recovery for sample, %

Y_{rb} = Y recovery for reagent blank, %

Y_{lcs} = Y recovery for LCS sample, %

Y_{total} = Total amount of Y added to LCS sample, µg

C_s = Sample Y concentration measured by XRF analyzer, ppm (µg/mL)

C_{rb} = Reagent blank Y concentration measured by XRF analyzer, ppm (µg/mL)

C_{lcs} = LCS sample Y concentration measured by XRF analyzer, ppm (µg/mL)

C_y = Concentration of Y carrier standard, mg/mL

C_{YSr90} = Concentration of Y in ^{90}Sr LCS spike standard, µg/mL

V_{Sr90} = Volume of ^{90}Sr spike standard added to LCS sample, mL

C_{XRF} = Y concentration measured for XRF normalization standard, µg/mL

D = Density of 1M HNO_3 solution used for Cerenkov LSC counting, g/mL

ΔM_s = Net amount of 1M HNO_3 in sample vial, g

ΔM_{rb} = Net amount of 1M HNO_3 in reagent blank vial, g

ΔM_{lcs} = Net amount of 1M HNO_3 in LCS sample vial, g

V_{Ys} = Volume of Y carrier standard added to sample, mL

V_{Yrb} = Volume of Y carrier standard added to reagent blank, mL

V_{Ylcs} = Volume of Y carrier standard added to LCS sample, mL

F = XRF normalization factor

M_{s2} = Weight of filled sample LSC vial, g

M_{s1} = Weight of empty sample LSC vial, g

M_{rb2} = Weight of filled reagent blank LSC vial, g

M_{rb1} = Weight of empty reagent blank LSC vial, g

M_{lcs2} = Weight of filled LCS sample LSC vial, g

M_{lcs1} = Weight of empty LCS sample LSC vial, g

C_{std} = Y concentration for XRF normalization standard, µg/mL

B. **Measurement Uncertainty**

The combined standard uncertainty for net sample ^{90}Sr activity concentration is calculated by assuming that all variables are independent, the uncertainty associated with decay correction is negligible, and all type B uncertainties have uniform distribution.

$$u_{C_{Sr}} = |C_{Sr}| \times \sqrt{\left(\frac{u_{A_s}}{A_s - A_{rb}}\right)^2 + \left(\frac{u_{A_{rb}}}{A_s - A_{rb}}\right)^2 + \left(\frac{u_{W_s}}{W_s}\right)^2} \quad (18)$$

Furthermore,

$$u_{A_s} = |A_s| \times \sqrt{\left(\frac{u_{R_s}}{R_s}\right)^2 + \left(\frac{u_{E_Y}}{E_Y}\right)^2 + \left(\frac{u_{Y_s}}{Y_s}\right)^2} \quad (19)$$

$$u_{A_{rb}} = |A_{rb}| \times \sqrt{\left(\frac{u_{R_{rb}}}{R_{rb}}\right)^2 + \left(\frac{u_{E_Y}}{E_Y}\right)^2 + \left(\frac{u_{Y_{rb}}}{Y_{rb}}\right)^2} \quad (20)$$

$$u_{W_s} = \sqrt{u_{W_1}^2 + u_{W_2}^2} \quad (21)$$

$$u_{Y_s} = Y_s \times \sqrt{\left(\frac{u_{C_s}}{C_s}\right)^2 + \left(ru_{xrf}\right)^2 + \left(\frac{u_{C_y}}{\sqrt{3} \times C_y}\right)^2 + \left(\frac{u_{\Delta M_s}}{\Delta M_s}\right)^2 + \left(\frac{u_{V_{Ys}}}{V_{Ys}}\right)^2 + \left(\frac{u_F}{F}\right)^2 + \left(\frac{u_D}{D}\right)^2} \quad (22)$$

$$u_{Y_{rb}} = Y_{rb} \times \sqrt{\left(\frac{u_{C_{rb}}}{C_{rb}}\right)^2 + \left(ru_{xrf}\right)^2 + \left(\frac{u_{C_y}}{\sqrt{3} \times C_y}\right)^2 + \left(\frac{u_{\Delta M_{rb}}}{\Delta M_{rb}}\right)^2 + \left(\frac{u_{V_{Yrb}}}{V_{Yrb}}\right)^2 + \left(\frac{u_F}{F}\right)^2 + \left(\frac{u_D}{D}\right)^2} \quad (23)$$

$$u_{Y_{lcs}} = Y_{lcs} \times \sqrt{\left(\frac{u_{C_{lcs}}}{C_{lcs}}\right)^2 + \left(ru_{xrf}\right)^2 + \left(\frac{u_{Y_{total}}}{Y_{total}}\right)^2 + \left(\frac{u_{\Delta M_{lcs}}}{\Delta M_{lcs}}\right)^2 + \left(\frac{u_F}{F}\right)^2 + \left(\frac{u_D}{D}\right)^2} \quad (24)$$

$$u_{Y_{total}} = \sqrt{\mathrm{V}_{Ylcs} \times 1000 \times \left(\frac{u_{C_y}}{\sqrt{3}}\right)^2 + c_y \times 1000 \times u^2_{\mathrm{V}_{Ylcs}} + V_{Sr90} \times u^2_{c_{YSr90}} + c_{YSr90} \times u^2_{\mathrm{V}_{Sr90}}} \quad (25)$$

$$u_{\Delta M_s} = \sqrt{u^2_{M_{s2}} + u^2_{M_{s1}}} \quad (26)$$

$$u_{\Delta M_{rb}} = \sqrt{u^2_{M_{rb2}} + u^2_{M_{rb1}}} \quad (27)$$

$$u_{\Delta M_{lcs}} = \sqrt{u^2_{M_{lcs2}} + u^2_{M_{lcs1}}} \quad (28)$$

$$u_F = F \times \sqrt{\left(\frac{u_{C_{XRF}}}{C_{XRF}}\right)^2 + \left(ru_{xrf}\right)^2 + \left(\frac{u_{C_{std}}}{\sqrt{3}C_{std}}\right)^2} \quad (29)$$

Where,

u_{A_s} = Standard uncertainty of instrument background corrected sample activity, Bq

$u_{A_{rb}}$ = Standard uncertainty of instrument background corrected reagent blank activity, Bq

u_{W_s} = Standard uncertainty of net sample weight, g

u_{R_s} = Standard uncertainty of instrument background corrected sample count rate, cpm

u_{E_Y} = Standard uncertainty of ^{90}Y Cerenkov counting efficiency, %

u_{Y_s} = Standard uncertainty of Y recovery for sample, %

$u_{Y_{rb}}$ = Standard uncertainty of Y recovery for reagent blank, %

$u_{Y_{lcs}}$ = Standard uncertainty of Y recovery for LCS, %

ru_{xrf} = Typical variability of XRF analyzer assessed to be 1.5%, fractional

$u_{Y_{total}}$ = Standard uncertainty of total amount of Y added to LCS sample, µg

u_{W_1} = Standard uncertainty of empty ashing dish weight, g

u_{W_2} = Standard uncertainty of filled ashing dish weight, g

u_{C_s} = Standard uncertainty of sample Y concentration measured by XRF analyzer, ppm (µg/mL)

$u_{C_{rb}}$ = Standard uncertainty of reagent blank Y concentration measured by XRF analyzer, ppm (µg/mL)

u_{C_y} = Standard uncertainty of Y carrier standard concentration (type B uncertainty), ppm (µg/mL)

$u_{C_{YSr90}}$ = Standard uncertainty of Y concentration for ^{90}Sr LCS spike standard, µg/mL

$u_{\Delta M_s}$ = Standard uncertainty of net amount of 1M HNO_3 in sample vial, g

$u_{\Delta M_{rb}}$ = Standard uncertainty of net amount of 1M HNO_3 in reagent blank vial, g

$u_{\Delta M_{lcs}}$ = Standard uncertainty of net amount of 1M HNO_3 in LCS sample vial, g

$u_{M_{s2}}$ = Standard uncertainty of filled sample LSC vial weight, g

$u_{M_{s1}}$ = Standard uncertainty of empty sample LSC vial weight, g

$u_{M_{rb2}}$ = Standard uncertainty of filled reagent blank LSC vial weight, g

$u_{M_{rb1}}$ = Standard uncertainty of empty reagent blank LSC vial weight, g

$u_{M_{lcs2}}$ = Standard uncertainty of filled LCS sample LSC vial weight, g

$u_{M_{lcs1}}$ = Standard uncertainty of empty LCS sample LSC vial weight, g

$u_{V_{Ys}}$ = Standard uncertainty for volume of Y carrier standard added to sample, mL

$u_{V_{Yrb}}$ = Standard uncertainty for volume of Y carrier standard added to reagent blank, mL

$u_{V_{Ylcs}}$ = Standard uncertainty for volume of Y carrier standard added to LCS sample, mL

$u_{V_{Sr90}}$ = Standard uncertainty for volume of ^{90}Sr spike standard added to LCS sample, mL

u_F = Standard uncertainty for XRF normalization factor

u_D = Standard uncertainty for density of 1M HNO_3 used for Cerenkov counting, g/mL

$u_{C_{std}}$ = Standard uncertainty of Y concentration for XRF normalization standard (type B uncertainty), ppm (µg/mL)

$u_{C_{XRF}}$ = Standard uncertainty of Y concentration determined by XRF for XRF normalization standard, ppm (µg/mL)

The expanded uncertainty for the net sample ^{90}Sr activity concentration, $U_{C_{Sr}}$, at 95% confidence level is calculated by multiplying the combined standard uncertainty, $u_{C_{Sr}}$, by a coverage factor of *k*=2.

$$U_{C_{Sr}} = 2 \times u_{C_{Sr}} \tag{30}$$

C. **Method Detectability**

1. **Minimum Detectable Concentration (MDC)**

The method detectability expressed as minimum detectable concentration (MDC) is estimated according to the typical sample weight, counting efficiency, Y recovery, and reagent blank as follows:

$$MDC = \frac{2.71+3.29\times\sqrt{R_{orb}\times T_S\times\left(1+\frac{T_S}{T_{rb}}\right)}}{E_Y\times W_S\times Y_S\times T_S\times 60} \tag{31}$$

Where,

R_{orb} = Reagent blank count rate, cpm

T_{rb} = Reagent blank count time, min

T_s = Sample count time, min

60 = Conversion factor from minutes to seconds

As no evidence shows that the observed reagent blank count rate follows ^{90}Y decay, the MDC is estimated without taking decay correction into consideration.

2. **Limit of Quantification (LOQ)**

The method quantification capability expressed as the limit of quantification (LOQ) is estimated according to the typical sample weight, counting efficiency, Y recovery, and reagent blank as follows:

$$LOQ = \frac{50\times\left(1+\sqrt{1+\left(\frac{R_{orb}\times T_S\times\left(1+\frac{T_S}{T_{rb}}\right)}{25}\right)}\right)}{E_Y\times W_S\times Y_S\times T_S\times 60} \tag{32}$$

As no evidence indicates that the observed reagent blank count rate follows ^{90}Y decay, the LOQ is estimated without taking decay correction into consideration.

6.9. Performance Characteristics

A. Accuracy and Precision

This method was validated using a variety of foods spiked with a known amount of ^{90}Sr in a single laboratory study. The typical method accuracy and precision are found to be better than ±7.2% and 7.2%, respectively.

B. MDC and LOQ

Based on a sample size of 0.25 kg, Y recovery of 80%, counting efficiency of 53%, reagent blank count rate of 1.08 cpm, and count time of 100 min, the typical method MDC and LOQ are estimated to be 0.08 Bq/kg and 0.32 Bq/kg, respectively.

6.10. Quality Control

A. Instrument Calibration and Acceptance Criteria

1. Requirements of Instrument Calibrations

To qualify a LSC counter and an XRF analyzer for sample analysis, they must be calibrated according to Attachments F and G, respectively. The calibrations for both must be performed when necessary, such as routine biennial calibration, unscheduled calibration after instrument repair, or recalibration warranted by nonconformance investigation.

LSC Counter

After calibration of liquid scintillation counter, three laboratory control samples (LCS) must be analyzed to assess the acceptability of ^{90}Y Cerenkov counting efficiency. The *Z*-scores of the LCS results must meet the QC criteria as described in Section 6.10. B. 2.

Note: The results from at least 4 efficiency standards are required for calculation of average LSC counting efficiency.

XRF Analyzer

After calibration of XRF analyzer, three standard solutions with concentrations of 50, 75, and 100 µg/mL Y must be analyzed to assess the acceptability of Y calibration curve. The differences between the measured value and its respective known value must be ≤ ±3% for all

three standards, and the square of correlation coefficient (R^2) for the Y calibration curve must be ≥0.99.

2. **Time-of-Use LSC Counter QC Check and Acceptance Criteria**

   Before using LSC counter for sample counting, time-of-use IPA must be performed.

   Quantulus 1220

   a. From the Windows desktop, open "WinQ" if it isn't already activated.
   b. Ensure that ^{3}H, ^{14}C, and blank standards are in positions 1, 2, and 5 respectively.
   c. Click the <Users> tab.
   d. Select the IPA folder in respect to the counter to be used

      For example**:** Ipac1 for counter 1
      Ipac2 for counter 2
      Ipac3 for counter 3

   e. Click the <Queue> button to send the protocol to Queue pane.
   f. Select the corresponding counter from the counter list shown on the bottom of the Queue pane
   g. Click the <Counters> tab to switch from <Users> menu to <Counters> menu.
   h. Start QC measurement by clicking the "►" corresponding to the counter to be used.
   i. Wait for QC measurement to finish.
   j. Go to the Windows desktop.
   k. Double click on the Excel IPA processing file corresponding to the counter it was run on.

      For example: Counter 1 Daily IPA for counter 1
      Counter 2 Daily IPA for counter 2
      Counter 3 Daily IPA for counter 3

l. At the prompt, open the IPA-D folder with the latest sequence number.

m. Open the “Registry” file to generate time-of-use IPA report.

n. Review the flag in the report’s “IPA Status” column for QC acceptance.

o. Enter analyst initials at the top right of the sheet and click the button to save to PDF.

Note: Depending on which counter it was run on, the QC Report is located in:

For counter 1: C:\D\IPAC1 Report

For counter 2: C:\D\IPAC2 Report

For counter 3: C:\D\IPAC3 Report

Quantulus GCT 6220

a. From the Windows desktop, open “QuantaSmart” if it isn’t already activated.

b. Ensure that ^{14}C standard, empty vial, ^{3}H standard, and background vial are in positions 1, 2, 3, and 4, respectively.

Note: The empty vial must be the same type of LSC vial used for sample analysis.

c. Plush the flag out to the left on the SNC plug.

d. Click start IPA counting.

e. Wait for QC measurement to complete.

f. Go to the Windows desktop.

g. Double click on Excel IPA processing file “GCT_Daily IPA Report”.

h. At the prompt, open the folder with the latest date and time stamp.

i. Open the “Registry” file to generate time-of-use IPA report.

j. Review the flag in the report’s “IPA Status” column for QC acceptance.

k. Enter analyst initials at the top right of the sheet and click the button to save to PDF.

Note: The QC Report is located in C:\IPA Report. The file will save with the date and time of the run as the filename.

All QC results shown in instrument IPA report must meet their acceptance criteria. When a performance indicator listed in the IPA report is flagged as "Pass with Warning", it is considered acceptable if the same performance indicator has not been flagged as "Pass with Warning" more than three consecutive times.

3. **Time-of-Use XRF Analyzer QC and Acceptance Criteria**

Before using XRF analyzer for Y recovery determination, time-of-use QC check must be performed with a standard containing 10 mL of 100 µg/mL Y standard solution and a blank containing 10 mL of 1M HNO_3.

a. Open <Acquisition Manager>

b. Click File>Open

c. Select the file "Time-of-Use QC Method" found in the directory of C/Quant'X/Master Methods

d. Double click on <Time-of-Use QC Method>

e. Click the file icon to select the Y method with a valid Y calibration curve.

Note: The method file with a valid Y calibration curve is the one that has a file name with the latest date.

f. Double click on the first cell in the last row to accept the file selection.

g. Load the sample containing 1M HNO_3 in position 1 and the sample containing 100 µg/mL Y standard solution in position 2

Note: To avoid damaging the motors that turn the carousel inside the XRF and could cause misalignment between sample and detector, do not move or adjust XRF carousel by hand even though sample position 1 is not at the start position from the previous run.

h. Click <Go>

i. Wait for QC measurement to finish

j. Click File>Save report…

k. Click ► QUANT'X shown in the path tab located on top of the screen

l. Double click <Time-of-Use QC Report> folder

m. Enter file name "QCMMDDYY-Initial"

For example: QC050417-JP

n. Click <Save>

o. Click File>Exit

Note: Do NOT open the Time-of-Use QC Report. It will corrupt the file.

p. Open Excel file "XRF QC Check" to evaluate instrument QC acceptance

q. Double click the XRF Time-of-Use QC report saved from the instrument QC run.

r. Enter initial in cell H6.

s. Click File>Save to update the Excel file

Note: The QC Report is located in C:\QUANT'X\Time-of-Use QC Report

To qualify XRF analyzer, the result of blank must show undetectable Y and the result of standard must meet the *Z*-score acceptance criteria. When a result shown in XRF QC report is flagged as "Investigate", it is considered acceptable if the same performance indicator has not been flagged as "Investigate" more than three consecutive times. The *Z*-score for the standard result is calculated as below:

$$Z = \frac{V_{obs} - V_{known}}{\sqrt{S^2_{V_{obs}} + S^2_{xrf} + S^2_{V_{known}}}} \quad (33)$$

Where,

V_{obs} = Observed Y standard concentration, ppm (µg/mL)

V_{known} = Known concentration of Y standard, ppm (µg/mL)

S_{Vobs} = Uncertainty for the measured standard, ppm (µg/mL), 1s

S_{Vknown} = Uncertainty for Y standard, ppm (µg/mL), 1s

S_{xrf} = Additional uncertainty for V_{obs} caused by variability of XRF analyzer, $S_{xrf} = V_{obs}$ x 1.5/100. The typical variability of XRF analyzer (ru_{xrf}) is assessed to be 1.5%

The acceptance criteria for the calculated *Z*-score are as follows:

Pass	If |Z-score| ≤ 2
Investigate	If 2 < |Z-score| ≤ 3
Action	If |Z-score| > 3

B. **Method QC and Acceptance Criteria**

1. **Reagent Blank**

For each sample batch to be analyzed, a reagent blank must be processed and analyzed in the same manner as a test sample. A numerical QC indicator, *i.e., Z*-score, as expressed below must be used to determine acceptance of reagent blank result.

$$Z = \frac{V_{rb} - V_{mean}}{\sqrt{S_{rb}^2 + S_{mean}^2}} \quad (34)$$

Where,

V_{rb} = Observed reagent blank value, Bq

V_{mean} = Typical reagent blank value, Bq

S_{rb} = Combined standard uncertainty for the observed reagent blank value, Bq

S_{mean} = One standard deviation for the typical reagent blank value, Bq

The criteria for reagent blank *Z*-score are as follows:

Pass	If Z-score ≤ 2
Investigate	If 2 < Z-score ≤ 3
Action	If Z-score > 3

A result flagged for "Investigate" for more than three consecutive times is considered unacceptable.

The *Z*-score for reagent blank is calculated, recorded, and displayed on a control chart for acceptance evaluation.

A reagent blank result flagged for "Action" invalidates its accompanying sample analysis and appropriate corrective actions must be taken to address the nonconformance.

2. **Laboratory Control Sample (LCS)**

The food matrix blank used for preparation of LCS must be ashed and qualified per Section 6.3.C of this SOP as appropriate. The LCS documentation should state the amount of ash needed for LCS preparation, which must be equivalent to 250 g of its original food product. The LCS food matrix blank preparation documentation must be filed in the method QA logbook.

For sample analysis, a LCS must be analyzed along with each batch of samples. Each sample batch is limited to 12 samples not including the QC samples. A numerical QC indicator, i.e., *Z*-score, as expressed below must be used to determine acceptance of LCS result.

$$Z = \frac{V_{obs} - V_{known}}{\sqrt{S_{obs}^2 + S_{known}^2}} \quad (35)$$

Where,

Z = *Z*-score value

V_{obs} = Observed LCS value, Bq/mL

V_{known} = Known LCS value, Bq/mL

S_{obs} = Combined standard uncertainty for the observed LCS value, Bq/mL

S_{known} = One standard deviation for the known LCS value, Bq/mL

The criteria for LCS *Z*-score are as follows:

Pass	If |Z-score| ≤ 2
Investigate	If 2 < |Z-score| ≤ 3
Action	If |Z-score| > 3

A result flagged for "Investigate" for more than three consecutive times is considered unacceptable.

The *Z*-score for the laboratory control samples is calculated, recorded, and displayed on a control chart for acceptance evaluation.

A LCS result flagged for "Action" invalidates its accompanying sample analysis and appropriate corrective actions must be taken to address the nonconformance.

3. **MDC Criteria for Sample Analysis**

To ensure that the calculated sample-specific MDC will not exceed the typical method MDC (0.12 Bq/kg), Y recovery for each sample can't be lower than an expected value depending on the LSC counter used. Since the counting efficiency varies from counter to counter, it can be cumbersome to implement different Y recovery criteria for different LSC counters. Instead, a more practical approach is used to ensure the acceptability of sample Y recovery, i.e., directly evaluate the calculated sample MDC against the typical method MDC. Considering the reagent blank is used for contamination monitoring, no calculation and evaluation of MDC for reagent blank will be needed.

The criteria for MDC are as follows:

Pass	MDC ≤ 0.12 Bq/kg
Action	MDC > 0.12 Bq/kg

4. **Measurement Accuracy**

Percent difference between the observed and known LCS values, D, as expressed below must be used to determine acceptance of measurement accuracy.

$$D = \frac{V_{obs} - V_{known}}{V_{known}} \times 100 \quad (36)$$

Where,

D = Percent difference between observed and known LCS values, %

V_{obs} = Observed LCS value, Bq/mL

V_{known} = Known LCS value, Bq/mL

The criteria for measurement accuracy are as follows:

Pass	If |D| ≤ 10%
Investigate	If 10% < |D| ≤ 20%
Action	If |D| > 20%

The percent difference between the observed and known LCS values is calculated using data processing spreadsheet displayed on a control chart for acceptance evaluation.

When the percent difference between the observed and known LCS values is outside of the 20% range, appropriate corrective actions must be taken to address the nonconformance.

5. **Measurement Precision**

To evaluate measurement precision, the relative standard deviations of LCS results observed over time and different analysts are calculated using the equation below and monitored using a control chart.

$$RS_{batch} = \frac{1}{\bar{V}} \times \sqrt{\frac{\sum(V_{batch} - \bar{V})^2}{2}} \times 100 \quad (37)$$

Where,

RS_{batch} = Relative standard deviation calculated with 3 consecutive LCS results including one from the current sample batch

V_{batch} = LCS value obtained for the current sample batch

$\bar{V}$ = Average of three consecutive LCS results including one from the current sample batch

Note: LCS values that are already identified as “Action” should not be entered into this control chart to avoid misidentification of RS_{batch} failures on subsequent batches.

Each relative standard deviation (RS_{batch}) calculated for the current sample batch is evaluated against a precision control limit established at 20%.

When the value of RS_{batch} for the current sample batch is >20%, appropriate corrective actions must be taken to address the nonconformance.

6. **Contamination Control**

   Whenever possible, disposable laboratory ware should be used for sample analysis. When reusable laboratory ware is used, it must be properly cleaned to avoid cross contamination.

7. **Maintaining Standard/Reagent Records**

   Information associated with purchased and in-house prepared standards and reagents must be recorded and archived. Form WEAC-TMPL.001 must be used while preparing in-house standards and reagents.

C. **Analytical Worksheet Review**

1. Analyst must verify QC compliance related to the sample analysis.
2. Analyst must verify that all data on analytical worksheet and instrument reports are correctly transferred into data processing spreadsheet.

D. **Corrective Action**

Analyst must take necessary corrective actions following the procedure as described in SOP WEAC-QMS.4.11 in case of nonconformance. The investigative actions should include but are not limited to:

1. Verify that the correct counting protocol was used for sample analysis.
2. All instrument parameters were set correctly. If not, correct the parameters according to Attachment D.
3. Re-rerun IPA to see whether the instrument is functioning properly.
4. Check the data entries for typos.
5. Inspect standards, reagents, and laboratory ware for contaminations.
6. Review facility maintenance records for indications of a momentary power interruption and abnormal environmental conditions
7. Recount the same samples.
8. Check instrument calibrations.

6.11. Safety and Hazardous Waste Management

To ensure personnel safety, the analyst must be properly trained and always wear safety glasses, gloves, and a protective coat while performing this analysis. Full face shield is recommended. A dust mask may be worn to avoid inhaling contaminants when transferring the ash between different containers. It also may be worn when handling DGA resin, TRU resin and Sr resin. Be familiar with safety data sheet of the three resins before use. In addition, sample digestion, extraction, filtration, and evaporation must be carried out cautiously in a well-ventilated fume hood. Having acid spill kit nearby is recommended.

All glassware, 100-mL FlipMate digestion cup, and 100-mL DigiTube should be examined for cracks and damage prior to use.

Fume hood should be cleared out after each use.

Analyst must handle, store, and dispose of wastes safely. Regular chemical wastes and radioactive wastes must be disposed of separately. Return labeled waste containers to their proper (labeled) storage area after use. Consult the RSO, IH, or supervisor concerning the safety and waste disposal procedures.

The following waste containers labeled as follows must be used to collect their respective wastes:

A. **Non-radioactive Wastes**:

1. 58% conc. HNO_3+18% H_2O+23% 0.05M HNO_3+1% 1M HNO_3+Y
2. Used DGA resin
3. Used TRU resin

B. **Radioactive Wastes**:

1. 58% conc. HNO_3+19%H_2O+23% 0.05M HNO_3+$^{90}Sr/^{90}Y$
2. Sr resin+$^{90}Sr/^{90}Y$
3. 100% 1M HNO_3+^{90}Y (Decay to non-radioactive waste after 30 days)

For any questions regarding waste disposal, the analyst should consult with the industrial hygienist and the radiation safety officer.

7. Glossary/Definitions

A. **Cerenkov Radiation**: Electromagnetic radiation emitted when beta particles pass through water at a speed faster than light.

B. **Limit of Quantification (LOQ)**: The lowest activity concentration in a sample that can be quantified with a precision of 10%.

C. **Reagent Blank**: For this method, a reagent blank is a matrix-free sample that contains all reagents and goes through all steps used in sample analysis.

D. **Minimum Detectable Concentration (MDC)**: The lowest net activity concentration that has 95% chance of being detected and it is the detection limit expressed as an activity concentration.

E. **Radioactive Equilibrium**: A situation when all radionuclides decay at the same rate for a given decay series.

F. **Analytical Batch**: An analytical batch consists of samples, standards, and blanks which are analyzed together with the same method sequence and same lots of reagents and with the manipulations common to each sample within the same time period (usually within one day) or in continuous sequential time periods.

8. Records

A. TDSAnalysesReporting.xlsm (Reside on H drive)

B. Analytical Worksheet from FORM-001508 TDS Rad Reporting Database – Sample Preparation; Batch Preparation (Database Version)

C. Analytical Worksheet – Sample Preparation; Batch Preparation (Paperwork Version)

D. Excel Spreadsheet from FORM-001508 TDS Rad Reporting Database – Calculation of ^{90}Sr Activity Concentration (Database Version)

E. Excel Spreadsheets – Calculation of ^{90}Sr Activity Concentrations (Paperwork Version)

F. Excel Spreadsheet – Calculation of Liquid Scintillation Counter Efficiency (Paperwork Version)

G. Analytical Worksheet – Liquid Scintillation Counter Efficiency Calibration (Paperwork Version)

H. Analytical Worksheet – XRF Analyzer Y Calibration Curve Worksheet (Paperwork Version)

I. Liquid Scintillation Counter Time-of-Use QC Chart and Report (Reside on instrument computer)
J. XRF Analyzer Time-of-Use QC Chart and Report (Reside on instrument computer)
K. Method QC Charts (Reside on H drive)
L. Validation Report – Calculation of ^{90}Sr Activity Concentration
M. Validation Report – Calculation of Liquid Scintillation Counter Efficiency
N. Validation Report – Liquid Scintillation Counter Time-of-Use QC Chart and Report
O. Validation Report – XRF Analyzer Time-of-Use QC Chart and Report
P. Validation Report – Method QC Charts
Q. Method QA Logbook (Reside on H drive)
R. Reagent Preparation Sheet (Reside on H drive)
S. Method Development and Validation Report (Reside on H drive)
T. WEAC-TMPL.112, Analytical Branch Laboratory Balances Function Verification/Preventive Maintenance Sheet

9. Supporting Documents

A. FORM-001178 Eichrom Resins Sign-Out Sheet
B. FORM-001185 Calculation of Liquid Scintillation Counter Efficiency – Sr90
C. FORM-001186 XRF Analyzer Time-of-Use QC Chart and Report
D. FORM-001187 Sr-90 by LSC Analytical Worksheet – Sample and Batch Preparation
E. FORM-001190 Excel Spreadsheets – Calculation of 90Sr Activity Concentrations
F. FORM-001223 Sr-90 by LSC Method QC Charts
G. FORM-001246 Analytical Worksheet – XRF Analyzer Y Calibration Curve Worksheet
H. SOP-000281 PerkinElmer Quantulus GCT 6220 Liquid Scintillation Counter
I. SOP-000741 Mettler Toledo Automated Powder Dispenser QS30
J. WEAC-AB-RN.15.0 Quantulus 1220 Liquid Scintillation Counter
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.4.11 WEAC Corrective Action Procedure

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

10. Document History

Revision #	Status* (D, I, R)	Date	Author Name and Title	Approving Official Name and Title
00	I	2/24/2020	JINGJING PAN, CHEMIST KATHRYN EMANUELE, CHEMIST EILEEN MAHER, CHEMIST JENNIFER SZYMANSKI, CHEMIST ZHICHAO LIN, CHEMIST STEPHANIE HEALEY, CHEMIST	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR
01	R	11/18/2020	ZHICHAO LIN, CHEMIST JINGJING PAN, CHEMIST KATHRYN EMANUELE, CHEMIST EILEEN MAHER, CHEMIST JENNIFER SZYMANSKI, CHEMIST STEPHANIE HEALEY, CHEMIST	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR
02	R	7/15/2021	ZHICHAO LIN, CHEMIST JINGJING PAN, CHEMIST KATHRYN EMANUELE, CHEMIST EILEEN MAHER, CHEMIST JENNIFER SZYMANSKI, CHEMIST STEPHANIE HEALEY, CHEMIST	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR
03	R	3/7/2022	ZHICHAO LIN, CHEMIST JINGJING PAN, CHEMIST KATHRYN EMANUELE, CHEMIST EILEEN MAHER, CHEMIST JENNIFER SZYMANSKI, CHEMIST STEPHANIE HEALEY, CHEMIST	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR
04	R	8/1/2022	ZHICHAO LIN, CHEMIST JINGJING PAN, CHEMIST KATHRYN EMANUELE, CHEMIST EILEEN MAHER, CHEMIST JENNIFER SZYMANSKI, CHEMIST STEPHANIE HEALEY, CHEMIST KELLY GARNICK, CHEMIST	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR

Revision #	Status* (D, I, R)	Date	Author Name and Title	Approving Official Name and Title
05	R	10/25/2022	ZHICHAO LIN, CHEMIST JINGJING PAN, CHEMIST KATHRYN EMANUELE, CHEMIST EILEEN MAHER, CHEMIST JENNIFER SZYMANSKI, CHEMIST STEPHANIE HEALEY, CHEMIST	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR
06	R	SEE INFOCARD	ZHICHAO LIN, CHEMIST JINGJING PAN, CHEMIST KATHRYN EMANUELE, CHEMIST EILEEN MAHER, CHEMIST JENNIFER SZYMANSKI, CHEMIST STEPHANIE HEALEY, CHEMIST	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR

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

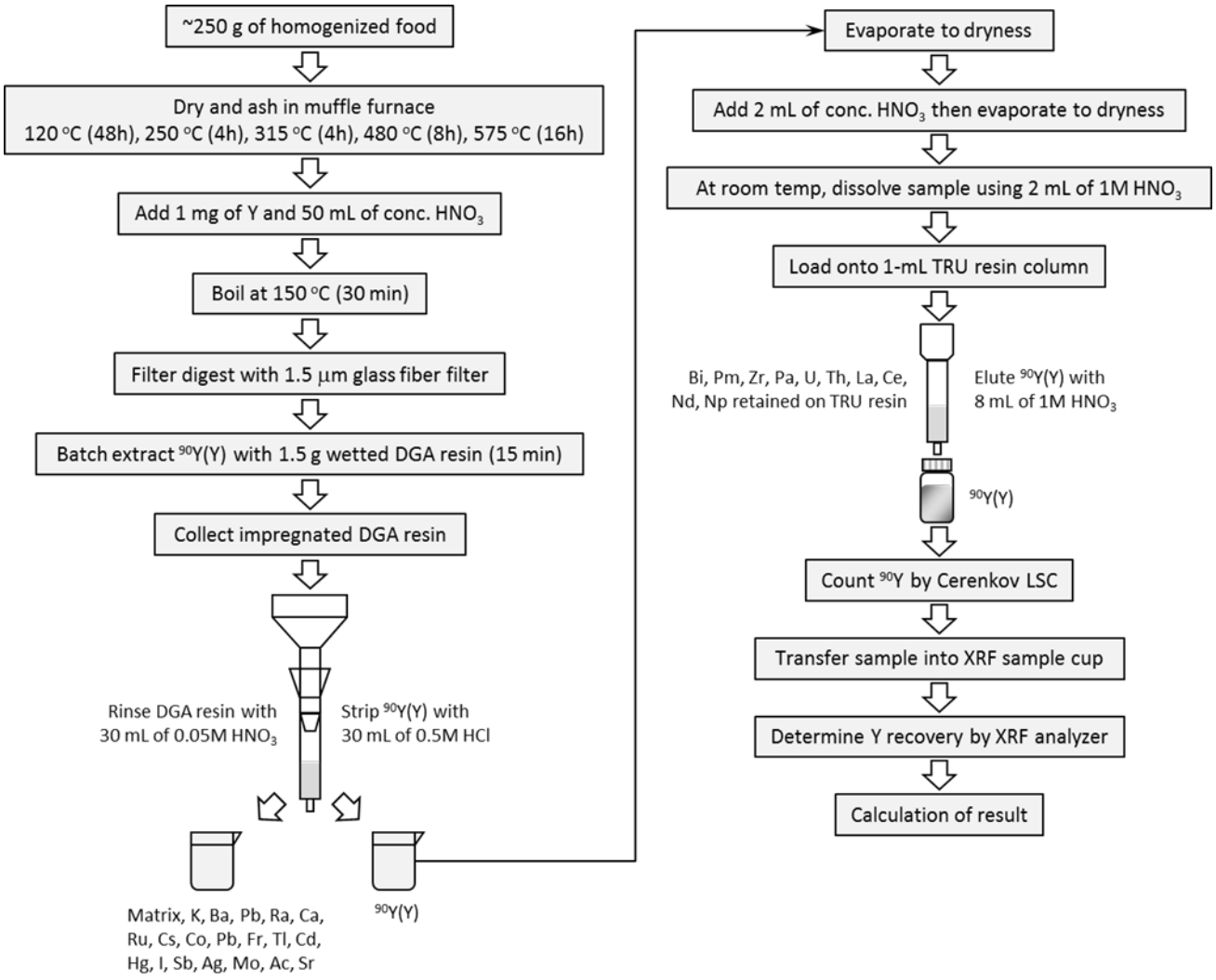
11. Change History

Revision #	Change
06	Minor editorial and formatting changes throughout the document; 6.1 added notes and removed part and catalog #s; 6.10 changed “investigate” to “pass with warning”, updated acceptance criteria and changed “Y Recovery” to “MDC Criteria for Sample Analysis” and revised the section; Att F removed part and catalog #s and removed section on Energy Alignment of XRF Analyzers; and Att G removed catalog #s.

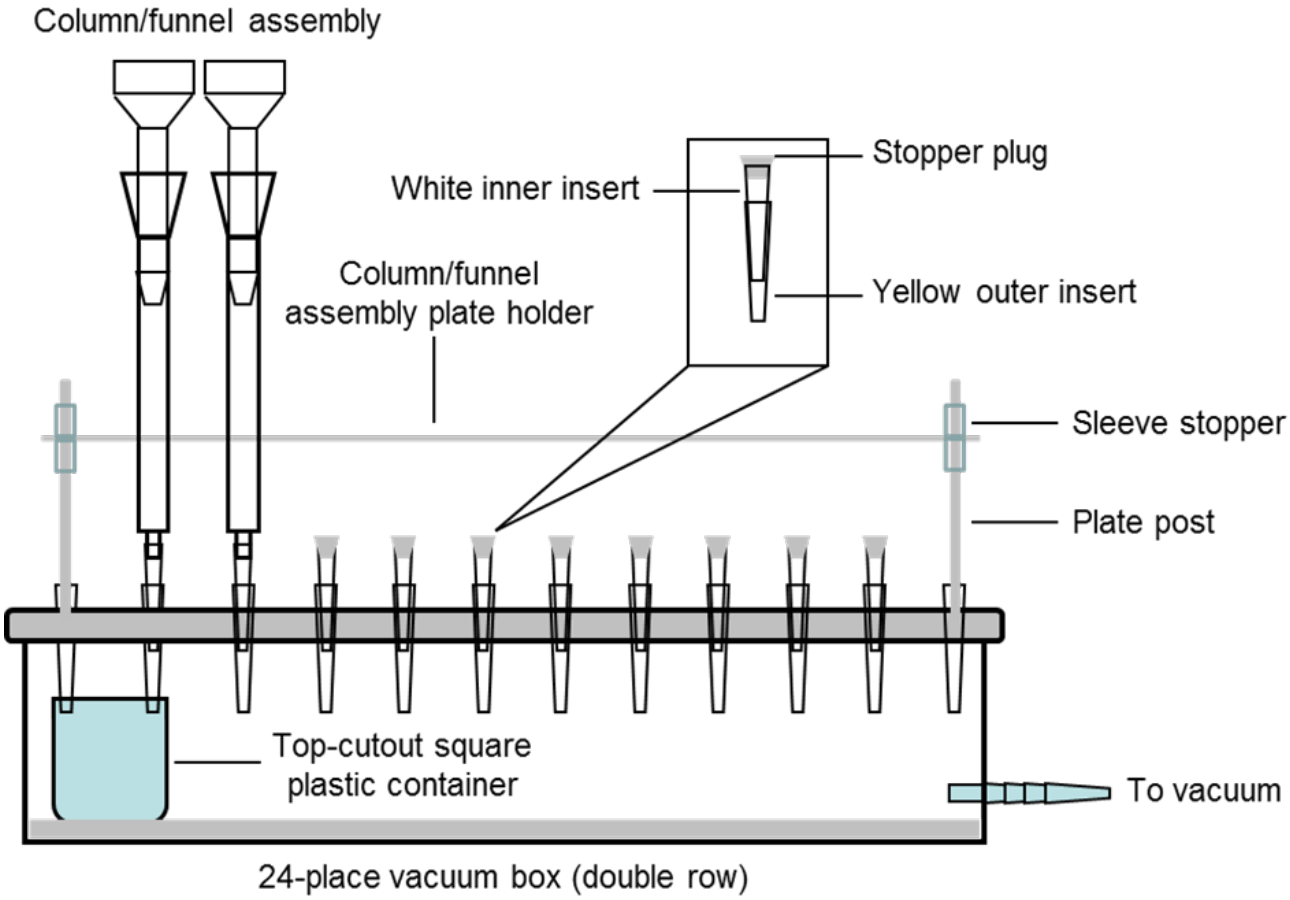
12. Attachments

List of Attachments

Attachment A - **Procedure Flowchart**



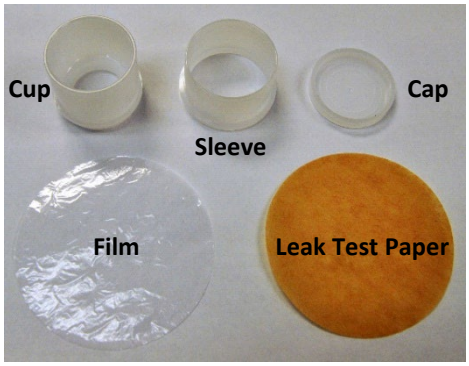
Attachment B - **Filtration Vacuum Box Setup**



24-place vacuum box (double row)

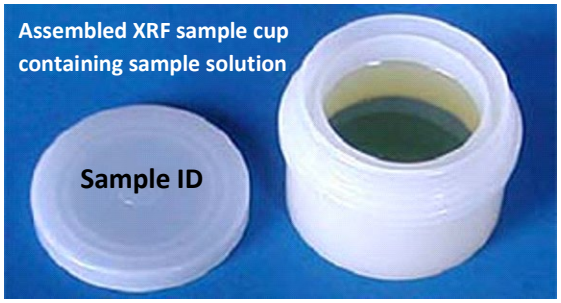
Attachment C - **XRF Sample Cup Assembly for Quantification of Y Recovery**

Assembly Parts



Assemble Sample Cup

Filled Sample Cup



Leak Test Sample Cup

Attachment D - **Quantulus 1220 Settings and Sample Counting Protocol**

General Parameters:
Save Path: C:\D\TDSSR\CTRx\Sxxx
Protocol Name: Cerenkov Counter x
Number of Cycles: 1
Parameter Listing: Checked

MCA & Window Settings:
Configuration: Special
Send Spectra: 11, 12
Coincidence Bias: Low
PAC: 200
PSA: N/A

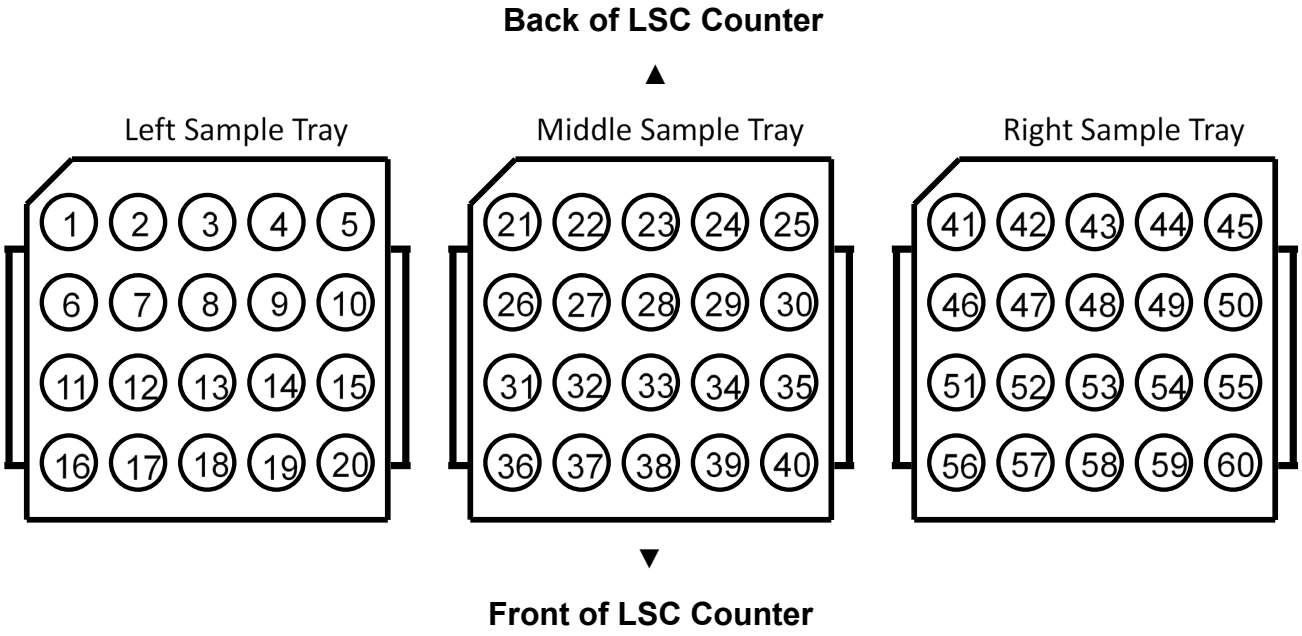
MCA & Window Settings

Window	MCA	Half	Channels	Window	MCA	Half	Channels
1	1	1	1-1024	5	2	1	1-1024
2	1	2	20-400	6	2	1	1-1024
3	1	2	20-400	7	2	2	1-1024
4	1	2	1-1024	8	2	2	1-1024

Sample Parameters

ORD	POS	ID	CTIME	COUNTS	COUNTS	MCW	REP	ST	STMS	STME
1	21	RBxxx	100	No Lim	No Lim	1	1	N		
2	22	LCSxx	100	No Lim	No Lim	1	1	N		
3	23	Sxxxx	100	No Lim	No Lim	1	1	N		

Attachment E - **Diagram of Position Number for Quantulus 1220 Sample Tray**



Caution: When load a sample tray into the counter, the side of the tray with a cut-out corner must face toward the back of the counter. Also, all trays must be flush against the front of the instrument.

Attachment F - **Determination of ^{90}Y Cerenkov Counting Efficiency**

1. Procedure

1.1 Equipment

- Fume hoods with utilities: vacuum, water, and power outlets
- Analytical balance with 0.00001 g readabilities
- Milli-Q water purification system
- Digital hotplates
- Ultra-low background liquid scintillation counters
- X-ray fluorescence analyzer (ARL QUANT'X EDXRF, Thermo Scientific)
- Handheld density meter (Mettler-Toledo, LLC)
- XRF sample cups
- XRF prolene® thin film
- Test paper for leak test of XRF sample cups
- Plastic trays for XRF samples
- LSC vial holder block
- 50-mL glass beakers
- 20-mL Teflon-coated, anti-static polyethylene LSC vials
- Vial racks
- 2-mL plastic columns
- Column racks
- 120-mL specimen cups with caps
- 2-mL plastic transfer pipettes
- 1-mL calibrated automatic pipette
- 5-mL and 10-mL adjustable pipettes and tips
- Paper towels
- Gloves, safety glasses, and laboratory coat
- Fine-tip and ultra-fine tip permanent markers
- Digital clock

- Polypropylene vented caps (Performance Systematix Inc)
- Wax-coated glass waste bottles
- Chemical waste labels
- Radioactive labels
- Inspection mirrors

1.2 Reagents and Standards

- Copper XRF energy adjustment disk
- Sr resin (100–150 µm, Part # SR-B50-A or # SR-B100-A, Eichrom)

  Note: Although Sr resin come with one-year manufacturer warranty, the manufacturer has instructed that the warranty begins with the time of purchase, not the time of manufacture. If the resin is kept dry in original bottle, the expiration day can be up to 2 years.

- Laboratory grade water
- Concentrated HNO_3 (15.8M), ACS reagent grade or equivalent
- Concentrated hydrogen peroxide (~30%), reagent grade
- 3M HNO_3: Dilute 190 mL of 15.8M HNO_3 to 1 L with laboratory grade water
- 1M HNO_3: Dilute 63 mL of 15.8M HNO_3 to 1 L with laboratory grade water

  Note: Density of the prepared 1M HNO_3 solution and standard uncertainty must be determined and recorded.

- NIST traceable Y carrier standard, 1 mg/mL in 1M HNO_3, with known solution density, g/mL
- NIST traceable Y batch standard, 100 µg/mL in 1M HNO_3
- XRF standards for Y calibration curve (See Attachment G)
- NIST traceable ^{90}Sr standard, ~150 Bq/g

  Note: The ^{90}Sr standard must be stored in an air-tight container. If it contains Y carrier, the bias in Y recovery caused by the ^{90}Sr standard must less than 0.1%. Otherwise, the bias must be corrected.

1.3 Efficiency Determination

Efficiency determination should be performed biennially and after each instrument repair whichever comes first. In this method, ^{90}Y Cerenkov counting efficiency is determined using ^{90}Y calibration standards prepared from a NIST traceable ^{90}Sr standard as follows:

A. Weighing ^{90}Sr Standards and Blank

1. Label 6 clean 50-mL glass beakers, 5 for standards and one for blank.
2. Add known amount of NIST traceable ^{90}Sr standard solution into each beaker labeled for standard. Record weighing data on instrument calibration worksheet.

   Note: The addition of ^{90}Sr standard into each beaker can be done by differential weighting using a pycnometer. If a ^{90}Sr standard solution of ~150 Bq/g is used, ~1 g of solution should be used for each standard.

3. Add known amount of NIST traceable Y carrier solution into each beaker containing ^{90}Sr standard. Record weighing data on instrument calibration worksheet.

   Note: The amount of Y carrier solution added to each beaker can be gravimetrically determined via differential weighing. To deliver replicable amount of Y carrier with ease, a 1-mL pipette can be used to assist the gravimetric determination.

4. Prepare a blank by adding a known amount of the same Y carrier solution into a beaker labeled for blank.
5. Swirl each beaker to mix the solution thoroughly.
6. Evaporate each standard and blank to dryness on a hotplate.
7. Cool each beaker to room temperature.

B. Preparation of Sr Resin Column

1. Place 6 empty 2-mL Eichrom plastic columns on a column rack.
2. Add 2 mL of laboratory grade water to each column.
3. Draw a fill line on each column at the top of the water level.
4. Place a 120-mL plastic specimen cup under each column then snap the tip off each column.

5. Prepare Sr resin slurry by mixing ~6 grams of Sr resin with 20 mL of laboratory grade water.
6. Transfer enough prepared Sr resin slurry to each column to establish a resin bed up to the fill line.
7. Ensure that each resin bed is free of air bubbles.
8. Condition each resin bed with 12 mL of 3M HNO_3 in three 4-mL increments.
9. Dispose the solution in each specimen cup to waste.

C. **Separation of ^{90}Y from ^{90}Sr**

1. Place a clean, labeled 100-mL glass beaker under each column.
2. Add 0.5 mL of 3M HNO_3 to each beaker then gently swirl each beaker to solubilize the residue.
3. Load the solution in each beaker onto its corresponding column with a 2-mL plastic transfer pipette then wait for drain to complete.
4. Repeat steps 2 - 3 twice.
5. Add 2 mL of 3M HNO_3 to each column then wait for drain to complete.
6. Repeat step 5 three times.
7. Wait for the last addition to drain completely.
8. Record $^{90}Sr/^{90}Y$ separation time for each standard.
9. Evaporate each collected eluent to dryness on a hotplate.
10. Remove each beaker from the hotplate.
11. At room temperature, add 1 mL of conc, HNO_3 and 10 drops of conc. H_2O_2 to each beaker.
12. Heat each beaker to dryness on a hotplate.
13. Repeat steps 11 – 12 until the residue in each beaker is colorless.

    Note: It is critical to eliminate yellow color from the residue because the blue/violet Cerenkov light emitted by the sample can be reduced as a result of cancellation effect between the complementary colors of yellow and blue.

14. For each prepared standard, weigh a clean and labeled 20-mL polyethylene LSC vial to the nearest 0.00001 grams then record the exact weight on instrument calibration worksheet.

15. At room temperature, add 2 mL of 1M HNO_3 to each beaker.
16. Gently swirl each beaker then transfer the solution to its weighted 20-mL polyethylene LSC vial.
17. Repeat steps 15 - 16 four times.
18. Mix the solution in each vial thoroughly.
19. Weigh each filled polyethylene LSC vial to the nearest 0.00001 grams then record the exact weight on instrument calibration worksheet.

D. **Counting ^{90}Y Standard and Blank**

Using Quantulus GCT 6220

1. Qualify the instrument by performing daily IPA.
2. Load the prepared ^{90}Y standards and blank into a sample cassette.
3. Attach protocol flag plug# 17 to the sample cassette then push out flag plug.
4. Place the cassette on the right side of the sample changer deck then close the deck cover.
5. Click TDSSR>Worklist.
6. Enter 17 in the <PID#> field.
7. Enter the standard and blank IDs in the <Sample Name> field.
8. Ensure that the standard and blank IDs listed in the counting protocol matches their positions in the cassette.
9. Click <OK> to save the protocol.
10. Start counting by clicking on the green button in the upper left corner of the screen.
11. Wait for count to finish.

Note: The counting report is saved in C:\Packard\TriCarb\Result\TDSSR

Using Quantulus 1220

1. Qualify the counter by performing daily IPA.
2. Load the prepared ^{90}Y standards and blank into the available sample holders.

3. Click <Users> button from WinQ window.
4. Select “TDSSR” from <Users> list.
5. Select the protocol for the counter to be calibrated, such as Cerenkov Counter 1.
6. Click Edit>Sample Parameters.
7. Enter the standard and blank IDs and position numbers according to their sample holder position number as shown in Attachment E.
8. Click <OK> to save the protocol.
9. Click <Queue> to send the protocol to queue pane.
10. At the bottom of the queue pane, select the counter to be used.
11. Close counter’s front door.
12. Allow dark adaption of standards and blank for at least 30 minutes.
13. Click<Counters> and then “►” to start count.
14. Wait for count to finish.

Note: Depending on which counter is used, the counting report is saved in C:\D\TDSSR\CTRx\Sxxx

1.4 Determination of Y Recovery

A. Preparation of XRF Samples

1. Make an XRF sample cup for each standard and blank as shown in Attachment C.
2. For each standard and blank, pour the entire volume of sample solution from scintillation vial into an XRF sample cup.
3. Ensure that no air bubbles appear on the bottom of the XRF sample cup.
4. Firmly place a cap on the sample cup.
5. Check each sample cup for leakage using a piece of leak-test paper.
6. Write sample ID on the cap and analytical worksheet.

B. QC Check of XRF Analyzer

1. Prepare a QC check sample using 10 mL of 100 µg/mL Y batch standard solution and a blank using 10 mL of 1M HNO_3.
2. Click File>New>Quantitative Tray.

3. Place the standards in position 1 of the sample changer.
4. Enter the standard ID into the cell below column title “Sample”.
5. Click the file icon to select a valid Y method file.

   Note: The valid method file is the one that has a file name with the latest date.

6. Click Open.
7. Double click on the first cell in the next row to activate <GO> icon.
8. Click green <Go> icon to begin analysis.
9. Wait for acquisition to finish.
10. Click File>Save Report.
11. Click on QUANT’X on the path tab.
12. Double click on Time-of-Use XRF QC Check folder.
13. Enter file name “QC-MMDDYY”.
14. Click <Save>.
15. Click File>Exit then click <No>.

C. **Determination of Y Concentration**

1. Open <Acquisition Manager>.
2. Click File>New>Quantitative Tray.
3. Enter standard ID in <Sample> column.
4. Click the file icon in <Method file> column.
5. Select valid Y method file then click <Open>.

   Note: Valid Y method has a file name with the latest expiration date.

6. Click the empty cell in <Sample> column then enter next standard ID.
7. Repeat step 6 for the rest of the standards and blank as well as the Y batch standard and blank used for XRF analyzer QC check.
8. Click the bottom empty cell in <Sample> column to accept the final entries.
9. Open sample chamber lid.

10. Place all samples in the sample tray according to their IDs and position numbers shown on the acquisition list.
11. Close sample chamber lid and then click <GO>.
12. Wait for analysis to finish.
13. Click File>Save Report…
14. Click QUANT'X shown in the path tab located on top of the screen.
15. Double click TDS folder and then enter file name "MMDDYY_XRFx_Initial"

    For example: 050417_XRF1_JP

16. Click <Save>.

    Note: The sample analysis report is saved in C:\QUANT'X\TDS

17. Click File>Exit>No.
18. Remove all samples from sample tray.

    Caution: Keeping samples inside instrument chamber for an extended period will cause acid fume to diffuse out of the sample cups and corrode instrument.

1.5 Calculation of ^{90}Y Cerenkov Counting Efficiency

A. Efficiency Calculation

1. Transfer the data recorded on instrument calibration worksheet, LSC counting report and XRF analysis report into efficiency calculation spreadsheet.
2. Save the completed spreadsheet.

For a given LSC counter, its average ^{90}Y Cerenkov counting efficiency $\bar{E}_y$ is calculated as:

$$\bar{E}_y = \frac{\sum E_{Y_i}}{N} \qquad \text{F1}$$

$$E_{Y_i} = \frac{R_{rc}^i}{A_{dc}^i} \times 100 \qquad \text{F2}$$

$$R_{rc}^{i} = \frac{R_{dc}^{i}}{Y_{std}^{i} \times \frac{1}{100}} \quad \text{F3}$$

$$R_{dc}^{i} = \frac{R_{bc}^{i}}{D_{Y}^{i}} \quad \text{F4}$$

$$R_{bc}^{i} = R_{std}^{i} - R_{b} \quad \text{F5}$$

$$D_{Y}^{i} = e^{-\lambda_{Y} \times (T_{3}^{i} - T_{1}^{i})} \quad \text{F6}$$

$$Y_{std}^{i} = \frac{M_{XRF}^{i}}{M_{Y}^{i}} \times F \times 100 \quad \text{F7}$$

$$M_{XRF}^{i} = \frac{C_{XRF}^{i} \times W_{XRF}^{i}}{D} \quad \text{F8}$$

$$M_{Y}^{i} = \frac{C_{std}^{Y} \times W_{Y}^{i}}{D_{yc}} \times 1000 \quad \text{F9}$$

$$F = \frac{C_{NOR}^{Y}}{C_{XRF}^{Y}} \quad \text{F10}$$

$$A_{dc}^{i} = C_{std}^{Sr} \times W_{Sr}^{i} \times D_{Sr}^{i} \times 60 \quad \text{F11}$$

$$D_{Sr}^{i} = e^{-\lambda_{Sr} \times (T_{1}^{i} - T_{Ref})} \quad \text{F12}$$

Where,

$\bar{E}_{y}$ = Average ^{90}Y Cerenkov counting efficiency, %
E_{Y_i} = ^{90}Y Cerenkov counting efficiency determined with the *i* th standard, %
R_{rc}^{i} = Recovery corrected ^{90}Y count rate for the *i th* standard, cpm
A_{dc}^{i} = Known ^{90}Sr activity for the *i th* standard decay corrected to the date and time of ^{90}Sr/^{90}Y separation, dpm
R_{dc}^{i} = Decay corrected ^{90}Y count rate for the *i th* standard, cpm
Y_{std}^{i} = ^{90}Y recovery for the *i th* standard, %
R_{bc}^{i} = Blank corrected ^{90}Y count rate for the *i th* standard, cpm
D_{Y}^{i} = ^{90}Y decay correction factor for the *i th* standard
λ_{Y} = ^{90}Y decay constant, ln(2)/(64/24) = 2.599x10^{-1}, day^{-1}

T_3^i = Date and time at the mid-count of the *i th* standard
$T_3^i = T_2^i - T_4^i/60/24/2$
T_2^i = Date and time at the end of count time of the i *th* standard
T_1^i = Date and time of $^{90}Sr/^{90}Y$ separation for the *i th* standard
T_4^i = Count time for the *i th* standard, min
R_{std}^i = Gross count rate for the *i th* standard, cpm
R_b = Gross count rate for the blank, cpm
M_{XRF}^i = Mass of Y in the *i th* standard determined by XRF, µg
C_{XRF}^i = Y concentration for the *i th* standard determined by XRF, µg/mL (ppm)
W_{XRF}^i = Solution weight of the *i th* standard, g
D = Density of 1M HNO_3 used for preparation of Cerenkov counting efficiency standard, g/mL
M_Y^i = Mass of Y added to the *i th* standard, µg
C_{std}^Y = Concentration of Y carrier solution, mg/mL
W_Y^i = Weight of Y carrier solution used for preparation of the *i th* standard, g
D_{yc} = Density of Y carrier solution, g/mL
F = XRF normalization factor
C_{NOR}^Y = Y concentration of XRF normalization standard, µg/mL (ppm)
C_{XRF}^Y = Y concentration of XRF normalization standard determined by XRF, µg/mL (ppm)
C_{std}^{Sr} = Concentration of ^{90}Sr calibration standard solution at the reference time, Bq/g
W_{Sr}^i = Weight of ^{90}Sr calibration standard solution used for preparing the *i th* standard, g
D_{Sr}^i = ^{90}Sr decay correction factor for the *i th* standard
λ_{Sr} = ^{90}Sr decay constant, ln(2)/(28.79x365.24) = 6.592x10^{-5}, day^{-1}
T_{Ref} = Reference time of ^{90}Sr calibration standard solution
N = Number of standards used for ^{90}Y Cerenkov efficiency calibration

B. **Uncertainty Calculation**

The standard uncertainty ($u\bar{E}_y$) for the average ^{90}Y Cerenkov counting efficiency is propagated to include the uncertainties for concentration of ^{90}Sr calibration standard solution, concentration of Y carrier solution, density of Y carrier solution, density of 1M HNO_3 solution used for preparing LSC calibration standard, and XRF normalization factor. All type B uncertainties are assumed to have uniform distributions.

$$u\bar{E}_y = \bar{E}_y \times \sqrt{(rs_{\bar{E}_y})^2 + (rs_{C_{std}^{Sr}})^2 + (\frac{rs_{C_{std}^{Y}}}{\sqrt{3}})^2 + (rs_{D_{yc}})^2 + (rs_D)^2 + (rs_F)^2}$$ F13

Furthermore,

$$rS_{\bar{E}_y} = \frac{S_{\bar{E}_y}}{\bar{E}_y}$$ F14

$$S_{\bar{E}_y} = \sqrt{\frac{\sum\left(E_{Y_i} - \bar{E}_y\right)^2}{N-1}}$$ F15

$$rs_F = \sqrt{\left(\frac{rs_{C_{NOR}^{Y}}}{\sqrt{3}}\right)^2 + \left(ru_{xrf}\right)^2 + \left(rs_{C_{XRF}^{Y}}\right)^2} \times 100$$ F16

Where,

$S_{\bar{E}_y}$ = Standard deviation for average ^{90}Y Cerenkov counting efficiency

$rs_{\bar{E}_y}$ = Relative standard deviation of average ^{90}Y Cerenkov counting efficiency, fractional

$rs_{C_{std}^{Sr}}$ = Relative standard uncertainty for concentration of ^{90}Sr calibration standard solution, fractional

$rs_{C_{std}^{Y}}$ = Relative standard uncertainty for concentration of Y carrier solution, fractional

$rs_{D_{yc}}$ = Relative standard uncertainty for density of Y carrier solution, fractional

rs_D = Relative standard uncertainty for density of 1M HNO_3 used for preparation of Cerenkov counting efficiency standard, fractional

rs_F = Relative standard uncertainty for XRF normalization factor, fractional

$rs_{C_{NOR}^{Y}}$ = Relative standard uncertainty for Y concentration of XRF normalization standard, fractional

$rs_{C_{XRF}^{Y}}$ = Relative standard uncertainty for Y concentration of XRF normalization standard determined by XRF, fractional

ru_{xrf} = Typical variability of XRF analyzer assessed to be 1.5%, fractional

1.6 Verification of ^{90}Y Cerenkov Counting Efficiency

After determination of ^{90}Y Cerenkov counting efficiency for each LSC counter, the counting efficiency must be verified using food-based LCS samples spiked with known ^{90}Sr activity. Three LCS samples and a reagent blank should be analyzed following the method procedure applied for routine sample analysis and the results must meet their QC acceptance criteria as described in Section 6.10.B.2 and Section 6.10.B.3. The analytical data and LCS results for efficiency verification should be included in efficiency calibration report.

Attachment G - **Calibration of XRF Analyzer**

1. Procedure

1.1 Equipment

- Milli-Q water purification system
- X-ray fluorescence analyzer (ARL QUANT'X EDXRF, Thermo Scientific)
- XRF sample cups
- XRF prolene® thin film
- Test paper for leak test of XRF sample cups
- Plastic trays for XRF samples
- 10-mL adjustable pipette and tips
- Paper towels
- Gloves, safety glasses, and laboratory coat
- Fine-tip and ultra-fine tip permanent markers
- Polypropylene vented caps (Performance Systematix Inc)
- Wax-coated glass waste bottles
- Chemical waste labels
- Inspection mirrors

1.2 Reagents and Standards

- Copper XRF energy adjustment disk
- Laboratory grade water
- Concentrated HNO_3 (15.8M), reagent grade
- 1M HNO_3 Blank: Dilute 63 mL of 15.8M HNO_3 to 1 L with laboratory grade water
- NIST traceable Y standards in 1M HNO_3 at concentration of 25, 50, 75, 100, 125, and 150 µg/mL, respectively

1.3 Generation of Y Calibration Curve

Y calibration curve should be established biennially and after each instrument repair whichever comes first. In this method, the Y calibration curve to be used for quantification of sample Y concentration is generated using a set of NIST

traceable Y standard solutions purchased from a commercial reference material provider.

A. **Preparation of XRF Standards**

1. Assemble 7 XRF sample cups as shown in Attachment C.
2. Label one cap for blank and six for standards.
3. Pipette 10 mL of 1M HNO_3 into the sample cup labeled for blank and record data on XRF Analyzer Calibration Worksheet.
4. Pipette 10 mL of 25, 50, 75, 100, 125, and 150 µg/mL standard solutions into 6 XRF sample cups assigned to their respective standards and record data on XRF Analyzer Calibration Worksheet.
5. Ensure there are no air bubbles visible at the bottom of each sample cup. Remove air bubbles as necessary.
6. Firmly cap each sample cup.
7. Check each sample cup for leakage using a piece of leak-test paper.

B. **Optimization of XRF Analyzer**

1. If <Acquisition Manger> isn't opened, double click <Acquisition Manger> icon on the Windows desktop.
2. Place a Cu XRF energy adjustment disk in the position 1 of sample tray.
3. Click File>New>Qualitative Tray List.
4. Click Analyze>Energy Adjustment.
5. Click on the box next to <Acquire for all count rates> then click <OK>.
6. Wait for energy adjustment to finish then click <OK>.
7. Click File>Save Report.
8. Click <QUANT'X> shown on the path tab located on top of the screen then open <EA> folder.
9. Enter file name as "EA-MMDDYY-Initial" then click on <Save>.
10. Click File>Exit.
11. Remove Cu XRF energy adjustment disk from sample tray.

C. **Creation of Standard Library**

1. If a standard library file for the current Y standard set has been created, proceed to Section D. Otherwise,

2. Exit <Acquisition Manger>.
3. Double click <Standards Library> icon on the Windows desktop.
4. Click File>New.
5. Click Standard>New>Bulk Standard.
6. Enter standard ID in the cell in <Standard Name> column.

   For example: VHG-ZYN-1M-25-100

7. Click the cell under <Component> column then enter a capital letter Y as element symbol for yttrium.

   Note: The 1st letter for element symbol must be in uppercase. If the element symbol carries second letter, a lowercase letter must be used.

8. Press <Tab> key then enter the Y concentration value.
9. Press <Tab> key then select “ppm” from <Unit> dropdown list.
10. Press <Tab> key then select “Certified” from <Certification> dropdown list.
11. Press <Tab> key to accept the entries.
12. Repeat steps 6 - 11 for all other standards.
13. Click File>Save As.
14. Click <QUANT’X> shown on the path tab located on top of the screen.
15. Open <Standards> folder.
16. Enter file name as “Y_STD_Exp_MMYYYY”.

    For example**:** Y_STD_Exp_042018

17. Click <Save>.
18. Click File>Exit.

D. **Generation of Y Calibration Curve**

1. Double click <Method Explorer> icon on the Windows desktop.
2. Click File>Open.
3. Select file “Y Method” then click <Open>.

4. Click File>Settings.
5. Click <Standards Library> dropdown list.
6. Select the valid standard library file from the list then click <Open>.

   Note: Valid standard library carries a file name with the latest expiration date.

7. Click <OK>.
8. Click <Calibration> on the left side bar.
9. Click <Calibration> on the top menu bar.
10. Click <Add Standard…>.

    Note: All existing standards must be deleted before adding new standards.

11. Hold down <Shift> and <Arrow Down> keys to select all standards from the list then click <OK>.
12. Click <Calibration> on the top menu bar then select Calibrate.
13. Select <Collect All> then click <OK>.
14. Open sample chamber lid.
15. Place each prepared standard and blank in the sample tray according to their position numbers displayed in <Position> column.
16. Close sample chamber lid and then click green color <GO> icon.
17. Wait for measurement to finish.
18. Click <Continue> then <OK>.
19. Click File>Save As.

    Caution: To avoid overwriting the original method template file, user must always use <Save As> option to save newly created method file.

20. Enter file name as “Y Method_XRFx_MMYYYY” using the date of calibration.

    For example: Y Method_XRF1_042018

21. Click <Save>.

22. Exit Method Explorer.

1.4 Verification of Y Calibration Curve

A new Y calibration curve should be generated when new standards are going to be used. Before Y calibration curve can be used for routine sample analysis, it must be validated by analyzing standards with Y concentrations of 50, 75, and 100 µg/mL, respectively. The accuracy for the results of verification measurements must be better than ±3%.

To perform verification sample analysis, analyst should follow the procedure below:

1. Double click <Acquisition Manger> icon on the Windows desktop.
2. Click File>New>Quantitative Try List.
3. Enter the ID for 1M HNO_3 blank in <Sample> column.
4. Click the file icon in <Method file> column.
5. Select the newly created Y method file then click <Open>.
6. Click the empty cell in <Sample> column.
7. Enter the ID for the standard containing 50 ppm of Y.
8. Click the empty cell in <Sample> column.
9. Enter the ID for the standard containing 75 ppm of Y.
10. Click the empty cell in <Sample> column.
11. Enter the ID for the standard containing 100 ppm of Y.
12. Click the empty cell in <Sample> column.
13. Open sample chamber lid.
14. Place the 1M HNO_3 blank, 50 ppm Y standard, 75 ppm standard, and 100 ppm Y standard in the sample tray according to their position numbers displayed in <Position> column.
15. Close sample chamber lid and then click green color <GO> icon.
16. Wait for analysis to complete.
17. Evaluate the results per the acceptance criteria listed in Section 6.10. A.1.
18. Click File>Save Report…
19. Click QUANT'X shown on the path tab located on top of the screen.
20. Double click <Y Curve Verification> folder.

21. Enter file name “CURVEMMDDYY” then click <Save>.
22. Click File>Exit>No.

Note: Make sure “Time of Use” method and QC chart is updated with the current calibration curve and standard information.

Attachment H - **Instruction for Operating Automated Powder Dispenser**

The automated powder dispenser is an integrated system consists of an analytical balance, a powder dosing assembly, a revolving turntable, an antistatic unit, and a label printer. To dispense DGA resin, operate the system as follows:

A. **Power on the system**

1. Press button on the antistatic unit to turn on the antistatic unit.
2. Press key on the keypad to turn on the analytical balance.
3. Power on label printer.

B. **Perform time-of-use QC**

1. Press key on the keypad to return the system to Home position.
2. Press key on the keypad to open the glass door.
3. Ensure that the bubble in the spirit level is centered.
4. Replace the stainless-steel cover located at home position with weighing pan located in accessories drawer next to balance. Ensure the pan fits snugly in the tray.
5. Press →0← key on the keypad to zero the balance.
6. Place the standard weight onto the weighing pan.
7. Press key on the keypad to close the glass door.
8. Record the weight reading onto FV/PM sheet.
9. Ensure that the system meets the QC criteria.
10. Press key on the keypad to open the glass door.
11. Remove the standard weight.
12. Replace the weighing pan located at home position with the stainless-steel cover.
13. Press key on the keypad to close the glass door.

C. **Dispense DGA resin**

1. Press key on the keypad.
2. Select application by pressing <Dosing> icon.

3. Mount a dosing head on DGA resin bottle as shown below.

   Note: Make sure that the bottle contains enough amount of DGA resin to be dispensed.

4. Press  key on the keypad to open the door. Slide the dosing head mounted with DGA resin bottle onto the dosing head support until it comes to full stop.
5. Press the dosing head down slightly until it is properly seated in the holding pins.
6. Press  key on the keypad to return the system to Home position.
7. Press  icon located on the lower right corner of the keypad screen twice.
8. Select the setup  icon shown on the bottom of the screen.
9. Ensure “Auto sampler” and “SafePos” are checked.
10. Press <Start adjustments> icon.
11. Follow the instructions on the keypad screen to complete the alignment between the dosing head and vessel.

    Note: Move the dosing head upward by turning the manual adjustment wheel if needed.

Note: Make sure that the tip of the dosing head just slightly dips into the vial without interfering with balance reset and turntable movement.

Note: If alignment of sample vial is needed, make sure the opening of the sample vial is located exactly below the dosing head. The alignment should be centered.

12. For dispensing DGA resin, ensure target quantity is 1500 mg. If not, press the Quantity icon and enter 1500 mg.

    For dispensing TRU resin, ensure target quantity is 370 mg. If not, press the Quantity icon and enter 370 mg.

13. Load the desired number of vessels onto the revolving turntable.

    Note: To move turntable toward left or right, press  first and then press  or  icon on the keypad screen. When finished, press →|←(home) icon to return to home position.

14. Press  icon on the keypad to close the glass door.
15. Press  icon and then <Start> icon.
16. Enter the number of samples to be dispensed.
17. Press <OK> to dispense.
18. Upon completion of dosing, press  key to unlock the dosing head.

    If the glass door is closed, press  icon.

19. Remove the dosing head and DGA resin bottle. Detach the dispensing head assembly from the QS30 and then store it in the dosing head container.
20. Retrieve each sample from the revolving turntable.
21. Place the respective label on the vessel.

    Note: The label for each sample will be printed automatically.

22. Press  key on the keypad to close the glass door.
23. Turn the system off.