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Standard Operating Procedure for
48-Hour Gross Alpha Test, Total, Evaporation

Method ECLS-R-GA
Radioanalytical Services

Division of Public Health Infrastructure, Laboratories and Emergency Preparedness
Public Health and Environmental Laboratories
Environmental and Chemical Laboratory Services

NJDEP Lab ID#: 11036
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Double Count 48-hour Gross Alpha, Total, Evaporation

1. Identification of the Test Method

1.1 This procedure illustrates a method for determination of total gross alpha activity in water samples analyzed within 48 hours from sample collection time.

2. Applicable Matrix or Matrices

2.1 This method is for use in aqueous matrices such as drinking water (including New Jersey Private Well Testing Act samples), surface water, ground water, domestic and industrial wastewater, and monitoring well water.

3. Detection Limits

3.1 For Safe Drinking Water Act (SDWA) compliance monitoring samples, the required detection limit for gross alpha activity is 3 pCi/L.

3.2 The SDWA Detection Limit (DL) is calculated for each sample in the batch of analysis using the background data, as well as the sample aliquot, count time, and counting efficiency. Refer to SDWA DL equation in page 22 of the SOP. A typical SDWA DL for gross alpha determination is 2.15 pCi/L, assuming:

0.100 L of sample
60 minute background count time
400 minute sample count time
15% detection efficiency for α
0.06 cpm α background count rate

3.3 See Section 5.2.2.3 in the Radioanalytical Services Laboratory Quality Manual (RSLQM) for more details.

4. Scope and Application

4.1 Parameters: Gross alpha activity

4.2 Range of volume aliquot for analysis: Normally 100-200 ml

4.3 For SDWA compliance monitoring samples, mass thickness of the plancheted residue should not be more than 5 mg/cm^2 (100 mg on a 2-in diameter planchet) for gross alpha.

4.4 Radionuclides that are volatile under the sample preparation conditions of this method may be lost.

4.5 Maximum Contaminant Level (MCL) for gross alpha activity (including Ra-224 & Ra-226 but excluding radon & uranium) for compliance monitoring samples is

15 pCi/L. Refer to Section 5.5 of RSLQM when the measured gross alpha activity exceeds MCL.

5. Summary of Test Method

5.1 A measured aliquot of water sample is carefully evaporated to 10 ml or less and the residue is transferred to a pre-weighed 2-in stainless steel planchet for counting. During evaporation, the sample must not be allowed to boil violently ("bump") or come to complete dryness in the beaker. The residue transferred to the planchet is evaporated to dryness under a heat lamp, flamed on a Bunsen burner, and reweighed to determine residue mass. The sample is then counted in a low-background thin-window gas-flow proportional counter.

5.2 This SOP is based on a modification of EPA Method 900.0 where the required modifications are specified in the NJDEP Office of Quality Assurance regulations, The Regulations Governing the Certification of Laboratories and Environmental Measurements, N.J.A.C. 7:18 at 6.4(a) 3. Sections i and iii remain unchanged and ii and iv are further clarified with the following paragraphs, 5.3 and 5.4.

5.3 The first count of the prepared sample should start no sooner than 36 hours from sample collection time and must be completed before 48 hours from sample collection time.

5.4 If the measured gross alpha activity is over 5 pCi/L, the same plancheted sample must be recounted between 20 to 28 hours after the midpoint of the first counting time. At this time, the Rn-222 progenies have undergone completed decay and any impact of unsupported Pb-212 on gross alpha measurement is reduced by 80%.

6. Definitions

6.1 "Picocurie (pCi)" means that quantity of radioactive material producing 2.22 nuclear transformations per minute.

6.2 "Gross alpha particle activity" means the radioactivity due to alpha particle emission as inferred from measurements on a sample.

6.3 "Gross beta particle activity" means the radioactivity due to beta particle emission as inferred from measurements on a sample.

6.4 "RSLQM" is the abbreviation for the "Radioanalytical Services Laboratory Quality Manual".

7. Interferences

7.1 Non-uniformity in sample source preparation for gross alpha/beta assay and its geometry difference from the standard prepared for detector calibration interferes with the accuracy and precision of the method.

7.2 The matrix difference between the salt solids used to prepare the calibration curve and the salts in the sample is another potential source of error.

7.3 Alpha energy difference between alpha-emitting radionuclides in the sample and the standards used (Th-230 for our laboratory) for the detection efficiency calibration of detectors could lead to bias in determinations.

7.4 In cases where samples contain short-lived radionuclides, e.g., unsupported Ra-224 and Pb-212, the timing of sample count relative to preparation and sampling may lead to comparatively different values in results.

7.5 If volatile species, such as I-131, are present, flaming of planchet residue may tend to vaporize these radionuclides.

8. Safety

8.1 Lab coats and safety glasses (or face shields) must be worn in the laboratory at all times.

8.2 Protective gloves must be worn in handling of acids, solvents, and chemicals.

8.3 All chemical work must be carried out under the hood.

8.4 Refer to Chapter Nine in the RSLQM and the Radiation Safety Manual for further information.

8.5 For general safety issues, refer to Chapter 11 (Health and Safety) in the ECLS QM.

9. Equipment , Supplies, and Maintenance

9.1 Analytical balance, 0.1 mg sensitivity, Mettler Model AE 260
Model AE 260, SN P32823 (BAL-21)
Model AE 160, SN 73740 (BAL-20)

9.2 Top loader balance, 0.1 g sensitivity, Mettler Model SB 16001
Model SB16001, SN 1121493698 (BAL-22)
Model SB16001, SN 1125412036 (BAL-24)
Note: See Section 3.4.4 of the RSLQM and Chapter Four in the ECLS Quality manual for the operation and maintenance procedures of balances.

9.3 Low-background thin-window gas-flow proportional counter
Canberra Tennelec LB4100,SN# 76812, (LB4100-BLUE)
Canberra Tennelec LB4100,SN# 84890 (LB4100-RED)
Tennelec Series 5 XLB, SN# 0400557 (5XLB)

9.4 Hot plate

9.5 Heat lamps

9.6 Fisher burner

9.7 Beakers: various sizes

9.8 Stainless steel planchets, 2" diameter (1/sample)

9.9 Rainin pipets, 20 µL- 20 mL

9.10 NIST traceable "S" Class weight, Ainsworth, Denver, CO.

9.11 NIST traceable "S" Class weight, 1000 g, Denver Instrument, Denver, CO.

9.12 Forceps and/or tongs.

9.13 Desiccator

9.14 Trays

There is no required routine maintenance of the equipment used in this procedure.

10. Reagents and Standards

10.1 The reagents and standards ordering process follow the ECLS regulations as prescribed in the ECLS Quality Manual.

10.1.1 The Radiochemistry Lab supervisor is responsible for checking the accuracy of the order(s) received and labeling the bottles with his/her initial and date. He/she then assigns each a unique identification number, consisting of receipt date, lot #, and bottle #, per instructions in Section 3.3.1 of the RSLQM.

10.1.2 The original packing slip is kept at the warehouse and copies are maintained in the purchasing section and room L185.

10.1.3 The reagents are stored in rooms L185 and L185A, and the radioactive standards are stored, either in room L185, L185A, or L180B.

10.2 Reagents

10.2.1 Distilled or deionized water

10.2.2 Nitric acid, concentrated (16N)

10.2.3 Nitric acid, 1 N: mix 62.5 ml 16 N HNO_3 with DI (or distilled) water to 1L

10.2.4 For additional information regarding reagents and their preparation procedure, see Section 3.3.1 of RSLQM.

10.2.5 Refer to Section 4.4 (Traceability of Reference Standards) in the ECLS QM for further information about reagents and standard solutions.

10.3 Standards

10.3.1 NIST traceable commercially produced Sr-90 and Th-230 check sources for instrument plateau determination and daily performance check.

10.3.2 Th-230: NIST traceable standard solutions used for self-absorption determinations and detector efficiency calibration, as well as spiking the laboratory fortified samples.

10.3.3 See Sections 3.3.3 and 3.3.4 in the RSLQM for more information about the radioactive standards.

11. Sample Collection, Preservation, Shipment and Storage

11.1 Water samples are collected in 1-gal cubitainers or 2-gal polyethylene containers.

11.1.1 All bottles must be thoroughly cleaned, rinsed with DI water, and finally acid rinsed to remove soluble material from containers.

11.2 Maximum volume for gross alpha/beta testing is one liter.

11.2.1 Based on the results of gross alpha/beta analysis, other tests, such as Ra-226, Ra-228, and uranium, may be required. A total volume of one gallon

should be provided as a sufficient quantity to accommodate possible follow-up analyses.

11.2.2 If additional tests, such as Ra-224 or unsupported Pb-212 is also requested, 2 gallons of sample will be needed. In case the sample is the only one in the batch for these tests, then a 3-gallon supply should be supplied to ensure performance of the required laboratory QC practices.

11.3 Immediately upon sample collection, water samples are preserved by addition of HNO_3 to a pH of <2.

11.3.1 If not preserved at the time of collection, the samples are preserved at the lab, and a minimum 16-hour holding time should be observed before start of analysis.

11.3.2 In situations where field preservation is not possible, about 5-15 ml of conc. HNO_3 will be added to the containers sent to the clients, to ensure completion of analysis within the 48-hour turn around time and to comply with the sample preservation requirement.

11.4 The analyzed samples will be stored in the Radioanalytical Services Temporary Storage area (Room L185B). Samples will be discarded after the completion of all analyses, unless instructed otherwise by the client. See Section 17.1, Sample Disposal.

12. Quality Control

Refer to Chapter 6 of the RSLQM for comprehensive discussion of this topic.

12.1 Check sources, consisting of NIST traceable, commercially produced, and certificated Th-230 and Sr-90 sources are run daily on each detector prior to sample counting and the results are stored electronically on each system's computer. Each counting system typically has some form of charting ability or quality assessment indicators, but this varies by manufacturer. The Berthold LB770 and LB780 alpha/beta counting systems have the ability to give the analyst immediate indicators regarding acceptance; currently the acceptance criteria is set at ±3%. (This is a conservative value compared to the actual limits calculated using the running, 20-point method required by the EPA). Refer to the RSLQM, Section 6.3, for further information.

12.2 Background checks, consisting of clean planchets, are run daily prior to sample counting. Detectors showing elevated background counts (i.e. exceeding 3 sigma variation from the daily, 20-point, running mean background value from the current chart) are re-counted after cleaning the planchet and planchet carrier. The results for each detector are stored electronically on each system's computer. Each counting system typically has some form of charting ability or quality assessment indicators, but this varies by manufacturer. Refer to RSLQM, section 6.3 for additional information.

12.3 Duplicate samples are prepared and run with each preparation batch with a frequency of no less than one per 10 samples. The criteria of acceptance for duplicate results are defined in Section 7.4.3.b) of the RSLQM.

12.4 A laboratory reagent blank (LRB), also referred to as method blank, sample is submitted with each preparation batch of samples. The criteria of acceptance for LRBs are defined in Section 7.4.1 of the RSLQM.

12.5 A laboratory fortified blank (LFB) sample, also referred to as laboratory control sample (LCS), or spiked blank (BS), is included with each preparation batch of samples. The criteria of acceptance for LFBs are defined in Section 7.4.2.b) of the RSLQM.

12.6 Laboratory fortified matrix (LFM), also referred to as matrix spike (MS), samples are regularly conducted, one with each preparation batch of samples. The criteria of acceptance for LFMs are defined in Section 7.4.3.a) of RSLQM.

12.7 Control charts are maintained for every quality parameter using the software program ChartRunner. Refer to the RSLQM, Section 6.3 for location and usage information.

13. Calibration and Standardization

13.1 Initial Calibration Process

13.1.1 For gross alpha and gross beta measurements, the detectors must be calibrated to obtain the ratio of count rate to disintegration rate.

13.1.2 Thorium-230 and Cs-137 are used as the calibration standards in gross alpha and gross beta activity determinations, respectively. Standards should be prepared in the geometry and weight ranges to be encountered in these gross analyses.

13.1.3 For each counting instrument to be used, the analyst should prepare separate alpha and beta particle self-absorption graphs showing water sample residue weight (mg) vs the efficiency factor (cpm/dpm), using Th-230 (for gross α) and Cs-137 (for gross β) standards.

13.1.4 For the alpha graph, Th-230 is added to varying size aliquots of tap water, such that the aliquot residue weight is varied between 0 and 120 mg. A similar graph is prepared for the beta graph, using Cs-137 and tap water aliquots, varying the residue weight between 0 to 200 mg.

13.1.5 Procedure for Efficiency Calibration in Gross Alpha/Gross Beta Measurements

13.1.5.1 With a 5-g "S" class weight, verify the proper operation of the balances. Record this on the Balance Log Book.

13.1.5.2 Label (use a magic marker) and pre-weigh stainless steel planchets to 0.1 mg. In Element, create a batch/bench sheet for "Gross Alpha/Beta ECLS-R-GA and/or EPA 900.0". For each calibration standard that will be prepared, add a "Laboratory Control Sample." Instructions for batching and creating bench sheets can be found in Sections 5 & 6 of the Radioanalytical Services Element Guidelines. Record all data on this bench sheet.

13.1.5.3 Determine the mg/L of dissolved material in the water to be used as the absorbing matrix. This is done by weighing out a known quantity of the water (0.1 kg) in a preweighed beaker and completely evaporating the liquid to dryness. Reweigh the beaker and determine the final weight of dissolved matter. The amount of dissolved solids per kilogram of liquid is determined as follows:

Dissolved solids per kilogram = (Dissolved Material in mg)/0.1 kg

13.1.5.4 Add the required amount of water containing dissolved solids to each pre-labeled beaker, so you will end up with weights ranging from nearly 0 to 120 mg, in 5-mg increments, for Th-230 standards and from 0 to 200 mg for Cs-137 standards. Make duplicate samples for 0-30 mg samples (Th- 230 standard).

13.1.5.5 Tare an empty 20-ml weighing boat

13.1.5.6 Put on a pair of protective rubber gloves. With a Rainin pipet, measure out 1.00 ml of radioactive spike (Th-230 or Cs-137) into the weighing boat. Record the mass of the spike.

13.1.5.7 Transfer the spike from the weighing boat to the appropriate beaker.

13.1.5.8 Repeat steps 13.1.5.6 and 13.1.5.7 until all spikes have been completed. Remove the protective gloves.

13.1.5.9 Determine the total dpm by multiplying the spike transferred by the expected dpm/g of spike.

13.1.5.10 Place the beakers on the hot plate and evaporate each sample to a point where there is approximately 5-10 ml left in the beaker.

13.1.5.11 Place the preweighed and labeled planchets on the heat lamp platform and with a magic marker, write the planchet ID# on the platform.

13.1.5.12 Transfer the remainder of the residue from the beaker to the pre-weighed stainless steel planchets. It may take two attempts to complete the transfer of the residue..

13.1.5.13 Rinse each beaker with about 5 ml of 1 N HNO_3 from a wash bottle, carefully washing down the sides of the beaker.

13.1.5.14 Transfer the rinsed residue from each beaker to their respective pre-weighed planchets.

13.1.5.15 Repeat steps 13.1.5.13 and 13.1.5.14.

13.1.5.16 Place the heat lamp at its most elevated point and turn on for 45 minutes. This is just to start the samples warming up. Don't take samples to dryness under the heat lamp. Turn off heat lamp before drying is completed.

13.1.5.17 Allow samples to come to almost complete dryness by air-drying (until all visible moisture is evaporated). Turn on heat lamp for 45 minutes to take out the rest of the moisture. Turn off heat lamp.

13.1.5.18 Flame the Th-230 and the Cs-137 planchets for 3 minutes on a Fisher burner.

13.1.5.19 Cool and reweigh planchet to determine actual sample weight.

13.1.5.20 Count each planchet in the gas proportional alpha/beta counter until a minimum of 20,000 counts are accumulated (record results on the counting data worksheet). Step by step instructions for creating a fitted efficiency calibration procedure are outlined on pages 35-39 in Chapter 3 of the Apex Alpha/Beta User's Manual. The section is titled "Fitted Efficiency Calibration."

13.1.5.21 After the standards have been cycled through all detectors, the data can be reviewed. Choose the Data Review button on the main toolbar and then select the batch for review. Click on the Fitted Efficiency tab of the Data Review window to choose the best fit model. Choose the best fit for the curve, which will be the fit that has the highest correlation coefficient.

13.1.5.22 In this Data Review, any point can be reviewed, approved or rejected. See See Apex Alpha/Beta User's Manual Chapter 8: Data Review View for instructions regarding reviewing and approving efficiency curves.

13.2 Efficiency calibrations are verified every year. At least three of the original solids absorption standards made to produce the current solids absorption curve are selected for re-measurement. The original measurement of the solids standard should occur within the range defined by the uncertainty of the reverification measurement calculated at the 95 percent confidence level. If any of the verification checks are not within this range, all absorption standards will be re-measured for activity and weight. If more than one standard's weight is not

within 5 percent of its previously recorded weight, then reverification will be attempted using at least 3 newly prepared solids absorption standards, each representative of the low, medium, and high weight ranges. If the measurement of the 3 newly prepared standards occurs within the range defined by the uncertainty of the reverification measurement calculated at the 95 percent confidence level, then the curve is verified. If not, then a new complete set of solids absorption standards is produced and implemented.

14. Analytical Procedure

14.1 Sample pick-up and batching

14.1.1 Take a cart and pickup samples requested for gross alpha/beta testing and their respective chain of custody forms from sample receiving (Room L176).

14.1.2 Check to make sure date and time of sample collection are recorded and the field number for each sample matches its Element-assigned Sample Number.

14.1.3 If error is found or sample collection date/time will prevent test completion within the 48-hour turn-around time, notify ECLS sample receiving personnel.

14.1.4 Place the samples on the cart and proceed to the Radiochemistry Laboratory (Room L185).

14.1.5 Samples are stored in Room L185B until a complete batch for gross alpha/beta analysis is achieved.

14.1.6 Within Element, create a batch and bench sheet for the samples to be included in the batch. Instructions for batching and creating bench sheets can be found in Sections 5 & 6 of the Radioanalytical Services Element Guidelines.

14.2 Sample Preparation

14.2.1 Record the sample number on a 400 ml beaker. Place on a top-loader balance and tare it.

14.2.2 Shake each sample for 10 seconds and then weigh 100-200 g (or a predetermined aliquot established based on sample's prior history of residue weights) into the appropriate beaker.

14.2.3 For duplicate and laboratory fortified samples repeat steps 14.2.1 and 14.2.2.

14.2.4 Place the beakers containing the samples on the hot plate set at approximately 100° C and evaporate to about 2-5 ml. Do not allow samples to go completely dry or boil violently. If water samples are known or suspected to contain chloride salts, those chloride salts should be converted to nitrate salts before the sample residue is transferred to a stainless steel planchet. Chloride salts can be converted to nitrate salts by

adding 2 ml of 16 N HNO_3 to the sample residue and evaporating to near dryness. One treatment is usually sufficient.

14.2.5 Transfer the residue quantitatively to a pre-weighed (recorded to the nearest 0.1 mg on the Gross Alpha/Beta Evap. Bench sheet) and labeled stainless steel planchet.

14.2.6 Rinse the beaker with 4 to 5 ml of 1 N HNO_3 from a wash bottle, carefully washing down the sides of the beaker. Transfer the residue to the planchet.

14.2.7 Evaporate to dryness under a heat lamp.

14.2.8 Place the planchet over a Fisher burner and flame for 3 minutes.

14.2.9 Weigh a 5-g and a 10-g "S" class weights and record their values in the Balance Log Book.

14.2.10 Weigh the plancheted sample (to 0.1 mg) and record on the Gross Alpha/Beta, Evap. Bench sheet. **Note:** For samples with residue mass exceeding the 100 mg limit, repeat sample preparation by taking a proportionately lower aliquot to ensure method requirements are met.

14.2.11 Deliver the plancheted samples residue along with their respective Request for Analysis and Chain of Custody form (RAD-4 Form), dated and signed. Also submit a copy of the completed "Gross Alpha/Beta, Evap." bench sheet for the batch of prepared samples to the Radiation Instrumentation room (L180) for counting.

14.3 Instrumental Analysis

14.3.1 General System Description

14.3.1.1 Each sample or background planchet is placed on a carrier, and counted in 1 of 2 Tennelec 4100 16-Channel PC-Controlled low- background gas-flow alpha/beta counting systems or the Tennelec Series 5 XLB single detector counting system. The Tennelec system utilizes 16 (57mm-diameter) detectors total with four drawers, each having four detectors. Each detector has individual replaceable gold-plated Mylar (80 $\mu g/cm^2$) windows. Once the cam elevates the samples, the source-to-detector distance is about 4 mm between the actual sample and the detector window. The Tennelec Series 5 XLB also has an ultra-thin 80 $\mu g/cm^2$ entrance window for high counting efficiency. The detectors in both counting systems approach an optimal 2π counting geometry.

14.3.1.2 The Canberra Tennelec Series 5 XLB alpha/beta counting system consists of a single detector, gas-flow proportional counter interfaced with a Pentium class computer. The

detector has an automatic sample changer and can analyze up to 50 samples in one procedure. The computer operating system is Windows XP and the counting software is Eclipse LB. Refer to the S550 Eclipse LB User's Manual for further detail.

14.3.1.3 The Tennelec 4100 alpha/beta counting system consists of a 16-detector, gas flow, proportional counter interfaced with a pentium-class computer that interface with 2 system controllers each of which communicates with 2 drawers. The computer operating system is Windows 98SE utilizing counting and control software written by Canberra based on Excel v5.0. Refer to the LB4110 Low Background Operating System, Users' Manual for further detail.

Before operating the system, the operator should be familiar with the Tennelec Series 5 XLB and Tennelec 4100 operating manuals.

14.3.2 Pre-Counting Operations

14.3.2.1 Turn on the gas supply to the detectors to the pressure of 10 psi which equates to a flow rate of 0.2 ft^3/hr as indicated by the flowmeter.

14.3.2.2 Make sure the detector bias supply is ON.

14.3.2.3 Make sure the supply of printing paper is adequate.

14.3.3 Sample Insertion

Tennelec 4100

14.3.3.1 Pull the slide-plate drawer forward (toward the operator) to open drawer.

14.3.3.2 To count check sources, remove the carriers, as the check sources are individually mounted to their own carriers. Place the source in its appropriate position in the drawer.

14.3.3.3 To count sample or background planchets, place the planchets into sample carriers that are in the drawer. Record their positions in the Sample Submission and Counting cover form and the respective alpha/beta counting logbook.

14.3.3.4 Push the slide-plate drawer in.

Tennelec Series 5 XLB

14.3.3.5 Prior to loading samples in the sample changer, an End Plate and 2 empty carrier plates should always be arranged in the following manner. Place an End plate under the receive (left) stack reader, a blank carrier under the send (right) stack reader and another blank carrier in the center, insertion position

14.3.3.6 After having these 3 plates in place, the GROUP carrier (A,B,C,D,etc) that will be used is placed in the bottom position of the send (right) stack. Any samples are then loaded on top of the GROUP plate into any numbered carrier containing an insert.

14.3.4 Daily Check Sources

14.3.4.1 Tennelec Series 5 XLB - Daily Check and Background QC Sequence

14.3.4.1.1 A QC sequence can be created in Eclipse in order to define a series of procedures to be run in a specified in order. Sequences can be set up according to Chapter 10 of the S550 Eclipse User Manual. The daily efficiency and background check is initiated using an auto QC sequence.

14.3.4.1.2 A Th-230 (07-064-26) and a Cs-137 (07-064-27), NIST traceable, commercially produced source is used for daily efficiency checks. The sources are located near the instrument.

14.3.4.1.3 Load the sample changer

14.3.4.1.3.1 End plate and 2 empty carrier plates arranged in the usual manner according to (14.3.3.5 and 14.3.3.6).

14.3.4.1.3.2 Place the QC plate at the bottom position of the send (right) stack.

14.3.4.1.3.3 Enter the Th-230 source in carrier # 1 and place on top of QC plate

14.3.4.1.3.4 Enter the Cs-137 source in carrier # 2 and place on top of Carrier #1.

14.3.4.1.3.5 Enter an empty planchet in carrier # 3 and place on top of Carrier # 2.

14.3.4.1.3.6 Place an End plate on top of Carrier # 3. (NOTE: It's important to put the sources and planchet in the designated carrier numbers because the auto QC sequence is set up to recognize the sources in these specific carriers.)

14.3.4.1.4 Click the GO button (Green hand).

14.3.4.1.5 Highlight "th-230 and **sr-90 (needs to change to Cs-137)** and ab bkg checks" and Click OK

14.3.4.1.6 Results for both sources and the background will print upon completion of counting

14.3.4.1.7 To view control charts for any of the check sources or the backgrounds, click on the **QC** drop down menu, and select **Create Charts**. Select the desired date range (or select all data points) and then choose the appropriate QC profile. After highlighting the QC Profile (e.g "Daily Alpha Eff") click **Show Chart**. Use the control chart to determine acceptance. Refer to your supervisor if a detector repeatedly fails.. Refer to Chapter Six (Quality Control) in the RSLQM for further guidance.

14.3.4.2 LB4100 System

14.3.4.2.1 Separate sets of eight Th-230 (#833 through #840) and eight Sr-90 (#841 through #848), NIST traceable, commercially produced sources are maintained for the Tennelec LB4100 system. These check sources are retained in room L180.

14.3.4.2.2 Open the drawers A through D of the system and temporarily remove the stainless steel inserts and any planchets. Store the carrier inserts (and any sample or background planchets).

14.3.4.2.3 Place the Th230 sources in their respective detectors, (i.e. Th#833 in detector A1, Th#834 in A2, through Th#840 in B4; and Sr#841 in detector C1, and Sr#842 in C2, through Sr#848 in D4. Orient them in the six o'clock position. Push in the drawer. Make sure to turn

the sample cam (the black knob directly beneath the drawer handle). It should point upward to raise the sample carriers against the detector face.

14.3.4.2.4 To initiate counting of the check sources, go to the "Sample Assigner" screen. Select "All Detectors" from the Current Display Group drop down menu. Highlight the row for Detector Red A1. Hold down Shift, and then click on Red B4. All Red Detectors from A1 – B4 should now be highlighted

14.3.4.2.5 Click the "Alpha Source Check" button on the right side of the screen. Count time should be set for 180 seconds. Click "OK." .

14.3.4.2.6 Highlight Detector Red C1. Hold Shift and click Red D4. Then click "Beta Source Check." Count time should be set for 180 minutes. Click "OK."

14.3.4.2.7 As soon as the sources complete counting (3 minutes), open the drawers (A & B) with the Th-230 sources. This will eliminate the daughter progenies from contaminating the detector window area and therefore elevating subsequent background counting. The older sources have enough ingrowth of progeny to cause transient raise in detector background.

14.3.4.2.8 After the first cycle is complete, switch the check sources to the other drawers (i.e. – move sources Th#833-836 to drawer C, Th#837-840 to drawer D, Sr#841-844 drawer A, and finally Sr#845-848 to drawer B).

14.3.4.2.9 To initiate counting, highlight Detectors Red A1-Red B4, and click "Beta Source Check." Click "OK."

14.3.4.2.10 Highlight Detectors Red C1 – Red D4 and click "Alpha Source Check." Click "OK."

14.3.4.2.11 Steps 14.3.4.1.3 – 14.3.4.1.10 can be repeated using the Blue tower, but instead highlight the respective Blue detectors (e.g. Blue A1, Blue D1, etc.)

14.3.4.2.12 To view the counting of the sources, click on the "Main" screen. Use the "Current Display Group" drop down menu to cycle through detectors.

14.3.4.2.13 To review the results of check source run, click the "QA" tab. Click "Red-A & B." Then click "+Red A1." Highlight "Alpha Efficiency. " Then select a date range for the data you want to review. Select "Daily Source Check" in the Calibration drop down menu. Then click "View." If the most recent data point is within the control limits in the chart, proceed with your review. Then highlight "Beta Efficiency" for Red A1. Then click "View." If the most recent data point is within the control limits for beta efficiency, the detector passes both daily efficiency checks.

14.3.4.2.14 Repeat step14.3.4.1.13 for each detector that daily checks were run.

14.3.4.2.15 If an efficiency check for a particular detector fails, repeat the efficiency check on that detector. If the check fails repeatedly, refer to your supervisor and refer to Chapter six (Quality Control) of the RSLQM for further guidance.

14.3.4.2.16 Remove the sources and return them to their original location.

14.3.4.3 Assessing Instrument Drift

14.3.4.3.1 In order to check the instrument drift in detector efficiency, check sources are run within 24 hours before and after each measurement. See Section 6.1.2 in the Radioanalytical Services Laboratory Quality Manual (RSLQM) for further information.

14.3.4.4 Alpha/beta cross-talk corrections

14.3.4.4.1 Cross talk corrections are made according to the operations manual for each system. For Canberra LB4100 system, use the instruction in Canberra's APEX alpha/beta Counting Productivity software (pages 25 and 81). For Canberra 5XLB, use the instructions in Canberra's ECLIPSE software V3.2, page 53.

14.3.5 Daily Background Measurement

14.3.5.1 Tennelec 5 XLB.

14.3.5.1.1 The background is run with the daily checks as part of the daily QC sequence as described in 14.3.4.1.

14.3.5.1.2 Upon completion of the background measurement, an automatic printout of the results will occur. These results should then be transcribed into the logbook. Typically, alpha background values range from 0 to 0.1 counts per minute (cpm) and beta ranges from 0.3 to 1.0 cpm.

14.3.5.2 LB4100 System

14.3.5.2.1 Place the background planchets on the carrier inserts of each detector.

14.3.5.2.2 To initiate background counting, go to the Sample Assigner screen, and highlight the appropriate detectors. (using Ctrl or Shift Keys to highlight multiple detectors if needed) Then click "Background Check" on the right side of the screen. Select "Gross Alpha/Beta Evap" as the Calibration. Count time should be 3600 seconds. Click "OK."

14.3.5.2.3 "START COUNT"

14.3.5.2.4 After counting completes, review background data by clicking the "Data Review" button..

14.3.5.2.5 To conduct a search for background data, select "Completed" in the "State:" field. For "Type:" select "Background Cal Check." Check "All Procedures." For Date/Time Range select "No Date/Time Filter." Click "Search Now."

14.3.5.2.6 Search results will appear in the right side of the screen. Check the appropriate boxes that correspond to the background checks that you have just run. It is easiest to use the date and time as a reference.

14.3.5.2.7 If background checks are run on all 32 detectors, there will be 8 batches that have to be selected (1 for each drawer of detectors). Each batch will consist of data for 4 detectors. (e.g. A1, A2, A3, A4)

14.3.5.2.8 Click the box next to the appropriate batches and then click

"Next."

14.3.5.2.9 Click the Report tab, and then click "Print." This will print the background data for one tray (e.g Red A1-Red A4). In the Available Batches drop down menu, select any remaining batches you wish to review. Print all remaining batches until all background data is printed.

14.3.5.2.10 Typically, alpha background values range from 0 to 0.1 counts per minute (cpm) and beta ranges from 0.3 to 1.0 cpm.

14.3.6 Sample Counting

14.3.6.1 Tennelec Series 5 XLB

14.3.6.1.1 To initiate sample counting, click **Manage, then Samples…**

14.3.6.1.2 Click Show Batches (uncounted). Then, highlight <<New>> and click OK and the Sample Manager window opens.

14.3.6.1.3 Enter a batch ID. Use the Element Batch ID.

14.3.6.1.4 Select Simultaneous Gross AB for the Procedure

14.3.6.1.5 Enter the sample's Element number into the LABID field that will be the first sample analyzed based on the setup of the automatic sample changer.

14.3.6.1.6 Select a Sample Type from the drop down menu. (Unknown for all samples, Blank for the blank sample)

14.3.6.1.7 Click on the appropriate Sample State (Liquid).

14.3.6.1.8 Enter the sample Quantity in Liters.

14.3.6.1.9 Enter the Residue Mass in milligrams

14.3.6.1.10 Click Update Sample

14.3.6.1.11 A new window with empty fields will appear. Repeat steps 14.3.6.1.6 – 14.3.6.10 for every sample that is part of the

batch and click Save

14.3.6.1.12 The duration of each counting is set to be 200 minutes. To change the count time (or any other aspect of the Simultaneous Gross AB procedure) click Manage, Procedures.., and Select Unknown Procedures. Click Next and select Simultaneous Gross AB from the drop down menu. Edit the count time field to change the count time. Refer to the XLB5 User Guide's Manual Make if needed. Make sure the gas supply in the tank is adequate for the counting period; replace the tank if it is not.

14.3.6.1.13 The application software will automatically print the final report sheet. The report will provide final activities in pCi/L. Activity units can be changed within the Simultaneous Gross AB procedure manager window as described in 14.3.6.13.

14.3.6.2 LB4110 System

14.3.6.2.1 To create a counting batch, click on the "Batches" button at the top.

14.3.6.2.2 Click "New." When prompted to use Sample Helper, click "No." (Sample Helper can be used if analyst chooses to do so).

14.3.6.2.3 Fill in Batch Name, Batch Description, and select "Gross Alpha/Beta 400min" as the Procedure. Make sure Unknown Batch is selected below. If it is not, "Gross Alpha/Beta 400min" will not appear.

14.3.6.2.4 Click "New Sample." Enter the Element Laboratory ID number in the "Sample Name" field. Sample Type will be Unknown for samples, and other identifiers are available for laboratory control samples. The "Recovery Factor" for this method will be 1. Highlight "Liquid" for Sample State. Highlight "Use Acquisition Date" for Sample Date. For Quantity enter the appropriate volume in liters. Enter the mass in milligrams in the Residual mass field. Enter 0.1mg as the uncertainty of the residual mass.

14.3.6.2.5 Click "Update Sample."

14.3.6.2.6 Repeat step 14.3.6.4 for remaining samples in the batch. When

the last sample is entered, hit "Save."

14.3.6.2.7 The batch isn't ready to be counted until the batch is "Committed for Counting." Make sure every piece of data is accurate in the batch before highlighting the "Commit for Counting" Box. Then click "Save."

14.3.6.2.8 Counting is ready to be initiated. Go to the "Sample Assigner" screen.

14.3.6.2.9 In the left window, click the plus sign icon next to "Unknown." Batches should branch out underneath. Click the plus sign next to the batch name that is to be counted. The samples assigned to that batch should now appear below the batch name.

14.3.6.2.10 Highlight the first sample on the list, and drag and drop the sample into its assigned detector. Repeat this step until all samples have been assigned to a detector.

14.3.6.2.11 After all samples are assigned, click "Load Samples."

14.3.6.2.12 An "Assignment Summary" window will pop up summarizing the counting batch. Verify your sample assignment, and click "Start Count."

14.3.6.2.13 The "Gross Alpha/Beta 400min" procedure in Apex Alpha/Beta will count the samples for a repeated 400 minute cycle to comply with Sections 5.3 and 5.4 of this SOP.

14.3.6.2.14 When counting completes, the data can be reviewed and printed by going to the Data Review Screen.

14.3.6.2.15 Select "Completed" for State and "Unknown" for Type. Make sure that "Gross Alpha/Beta 400min" is highlighted in the Procedure box. Highlight "No Date/Time Filter" and then click "Search Now." The appropriate batch should appear in the Search Results. Highlight this batch, and click "Next."

14.3.6.2.16 Click on the "Report" Tab. Then Click the Print icon. Then click OK. A report summarizing the counting batch will be printed.

15. Data Reduction

Important Note: The data reduction of the sample initial counting data must be completed, at the latest, before 12:00 pm the next day.

15.1 The "Unknown Batch Report" generated and printed by the Apex Alpha/Beta software contains the final calculated activities of all the samples in the counting batch. Data reduction is only necessary for the recovery calculations of the batch LFB and LFM samples. The recoveries are calculated using "Apex Alpha Beta QC calcs.xls. One can access this spreadsheet on the V: drive in the "ALPHA BETA calc sheets" folder. The path to this folder is PHEL317/Rads/Rad. Excel Data Reduction/ALPHA BETA calc sheets. See Section 7.1 in RSLQM for further information.

15.2 The computer will prompt the user through the program by asking a series of questions about the LFB and LFM in each batch. The user must enter the appropriate data in the designated cells, and hit "print" when finished. In addition, the spreadsheet must then also be saved to the "REPORTS" folder in the "Alpha-Beta Reports" sub folder. The path to this is on the V: drive, PHEL317/Rads/REPORTS/Alpha-Beta Reports. The file associated with each batch report sheet is then saved under the name "xxxxx.xls", where xxxxx refers to the batch number. All QC results (LRB's, LFB's, LFM's, duplicates) also must be entered into a QC folder in the appropriate test folder/ spreadsheet. (See Section 6.3.1 of RSLQM for more details). The path to this is on the V: drive, PHEL317/Rads/QC for Radiation/Gails from C.

15.3 The gross alpha and gross beta activities are given on the printout in units of picoCuries per liter (pCi/L). The minimum detectable concentration (MDC) is also given in pCi/L. The SDWA Detection Limit (DL) should be calculated separately in accordance with the equation presented in page 22 of this SOP. If the SDWA DL exceeds 3.0 pCi/L for gross alpha (which is noted at the bottom of the printout), inform the supervisor so a proper plan of action is devised.

15.4 Once the counting report has printed out and the recoveries have all been calculated, the results are entered into Element under Data Entry/Review. Steps for entering results into Element are outlined in Section 7 of the Radioanalytical Services Element Guidelines.

15.5 When the sample has a duplicate, both values are recorded into Element as well as the average activity. Instructions for where to put these values are in the Radioanalytical Services Element Guidelines, Section 7.1 (Data Entry Detailed Instructions).

$$Average\ activity = \frac{\text{sample activity} + \text{dup activity}}{2}$$

The uncertainty associated with average concentration is obtained as follows:

$$\frac{1.65\sigma \text{ sample} + 1.65\sigma \text{ duplicate}}{2}$$

15.6 If a second count is needed for a sample, that result is also entered into Element.

15.7 If the computer is inoperable, results must be calculated as follows:

$$\text{Gross Sample Count Rate, } R_G = \frac{\text{Sample Counts}}{\text{Sample Count Time (min.)}}$$

$$\text{Background Count Rate, } R_B = \frac{\text{Background Counts}}{\text{Background Count Time (min.)}}$$

Net Count Rate = Gross Count Rate -Background Count Rate

$$\text{Activity (pCi/L)} = \frac{\text{Net Count Rate}}{(\text{Efficiency})(\text{Volume})(2.22)}$$

Uncertainty associated with activity (pCi/L) = 1.65 σ

$$= 1.65 \text{ x } \frac{\sqrt{\dfrac{\text{Sample Gross Count Rate (cpm)}}{\text{Sample Count Time (min.)}} + \dfrac{\text{Background Count Rate (cpm)}}{\text{Background Count Time (min.)}}}}{(\text{Efficiency}) \text{ x (Volume) x } 2.22}$$

$$\text{SDWA DL (pCi/L)} = \frac{1.96^2}{2t_G} x \frac{\left[1 + \sqrt{1 + \dfrac{4t_G^2}{1.96^2} x R_B x \left(\dfrac{1}{t_G} + \dfrac{1}{t_B}\right)}\right]}{(\text{Efficiency})(\text{Volume})(\text{Chemical Recovery})(2.22)}$$

Where, t_G and t_B are the count times for sample and background, respectively.

Note that the above SDWA DL equation is applicable to all analyses. For gross alpha counting, Chemical Recovery = 1, assumed. Note that depending on the type of the analysis, the factor in the denominator may include other components, such as decay or ingrowth expressions.

If calculation of the Minimum Detectable Concentration (MDC) is also desirable, use the following equation:

$$\text{MDC (pCi/L)} = \frac{2.71/t_B + 4.65\sqrt{\text{Background Count Rate (cpm)}/t_B}}{(\text{Efficiency})(\text{Volume})(2.22)}$$

15.8 The results are checked by at least another analyst, before being reviewed by the Instrumentation Section Supervisor. The supervisor reviews the batch in Element according to Section 8 of the Radioanalytical Services Element Guidelines. His/Her digital signature will be on the final report.

15.9 At this stage, the gross alpha/gross beta analyses is considered to be complete and the Instrumentation Section Supervisor will discard the planchet. The date/time of discard will be noted on the chain of custody form (RAD-4 Form) and initialed.

15.10 If the gross alpha/beta results exceed the mandated limits, further analyses could be required, based on US EPA or New Jersey Department of Environmental Protection regulations. See Section 5.5.2 of RSLQM. The Instrumentation Section Supervisor will proceed to make the appropriate Sample "Change of Status" Request. (See Section 4.7 of RSLQM, Sample Change of Status). A copy of the addition in sample work analysis is distributed to the Radiochemistry Laboratory and one copy is filed in the sample folder. Any additional analyses needed for a sample will also be entered into Element. Steps for adding an analysis to a sample can be found in Section 4.3.3, "Corrections, Additions, or Cancellations of Logged Samples", of the Radioanalytical Services Element Guidelines.

16. Method Performance

16.1 Demonstration of Capability

16.1.1 Before beginning the analysis of compliance samples, the analyst must demonstrate acceptable results for each method by analyzing a set of samples as an Initial Demonstration of Capability (IDC). Refer to Section 5.3 in the RSLQM for further details.

16.1.2 Continuing demonstration of capability (CDC) is performed annually.

16.1.3 The results of the IDC and CDC will be documented with supporting records, calculations, and instrument output to establish its technical validity.

16.1.4 Documentation for IDC and CDC will be maintained in the analyst's training file in Room L180.

17. Pollution Prevention

17.1 Sample Disposal

17.1.1 After gross alpha/beta analysis has been performed, results reviewed and reported, in case no further analyses are required and there is no client requirement, then the samples can be discarded by the Laboratory staff. Only designated samples will be returned to sample receiving (Room L176).

17.1.2 Samples to be discarded, designated by their respective samples numbers, are listed in the sample discard list log book (located in Room L180), and sample containers are taken out from the Radioanalytical Services Temporary Storage area (Room L185B) to a discard cart by the Laboratory personnel. The samples in the discard list are then checked again by a Radiochemistry Lab personnel, and if the sample constituent radioactivity is below the regulatory levels, it will be poured down the sink. The discard date/time and personnel's signature will be recorded in the Request for Analysis and Sample Chain of Custody Record form (RAD-4 Form).

17.2 Reagent Disposal

17.2.1 Never pour corrosive materials or flammable liquid compounds that give off toxic vapors down the drain.

17.2.2 Acids, cyanides and phenols should be placed in separate appropriately marked containers for proper disposal.

17.2.3 Segregate chlorinated and nonchlorinated wastes into glass containers.

17.2.4 Label each container with type of waste, initial and final date of collection.

17.2.5 When bottles are full, contact the Laboratory Safety Officer to arrange for their disposal.

17.2.6 Transfer the bottles to the PHEAL loading dock and discard the waste into the appropriate 55-Gal waste drum for later pickup by the contracted waste disposal agency. The amount of disposed waste and the date/time of the discard into the waste drum are documented by the Laboratory Safety Officer.

17.3 Spill Cleanup

17.3.1 Chemical Spill

17.3.1.1 Use appropriate kit to neutralize and absorb inorganic acids and bases. Collect residue, place in container, and dispose as chemical waste.

17.3.1.2 For other chemicals, use appropriate kit or absorb spill with Vermiculite, dry sand, or diatomaceous earth. Collect residue, place in container and dispose as chemical waste.

17.3.1.3 Clean spill area with water.

17.3.1.4

17.3.2 Radiation Spill

17.3.2.1 Place absorbent paper towels over liquid spill. Place towels dampened with water over spills of solid materials.

17.3.2.2 Using forceps, place towels in plastic bag. Dispose in radiation waste container.

17.3.2.3 Wash the area of spill with decontaminating solution. Use sponges, mop, and bucket, as necessary. Again, retain all wash water as radioactive waste. Treat mop and sponges, once used, as radioactive waste.

17.3.2.4 Monitor area, hands, and shoes for contamination with an appropriate survey meter or method. Repeat cleanup until contamination is no longer detected.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

18.1 Refer to Section 7.4 in the RSLQM for the data acceptance /rejection criteria.

19. Corrective Action for Out of Control Analyses

19.1 Section 7.5 in the RSLQM describes the procedures that should be taken for the out of control analyses.

20. Contingencies for Handling Out of Control or Unacceptable Data

20.1 If time permits, sample will be re-prepared for the analysis within the 48-hour turn around time protocol. Otherwise, the sample will be reported with qualifier statements. See Section 7.1 of the Radioanalytical Services Element Guidelines for details on the use of qualifiers.

20.2 The qualified status of analysis will be communicated to the client and re-sampling will be requested.

20.3 See Data Handling in Chapter 7 of RSLQM and Chapter 9 of ECLS QM for further details.

21. Waste Management

21.1 For procedures to handle the waste, refer to ECLS Quality Manual.

22. References

22.1 Prescribed Procedures for Measurement of Radioactivity in Drinking Water; EPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio (EPA-600/4-80-032, August 1980).

22.2 Standard Methods for the Examination of Water and Wastewater, 21st Edition. Prepared by APHA -A WWA -WPCF. Published by American Public Health Association, 2005.

22.3 Tennelec 4110 Operating Instruction Manual.

22.4 Tennelec 5 XLB Operating Instruction Mannual

22.5 Radioanalytical Services Laboratory Quality Manual, Revision 4, New Jersey Department of Health, June 1, 2013.