Sections included in this document

1. Purpose

This procedure describes the sample counting and processing related steps for the determination of gamma-ray emitting radionuclides in food samples using high-purity germanium spectrometers. The procedure for the determination of sample-specific efficiencies to be used for this method is described in WEAC-AB-TM.003 “Gamma Efficiency Calibration”.

2. Scope

This document details the methods for preparing food samples for gamma counting, for using the high-purity germanium spectrometers, for using the associated spectrometers and spectroscopy software, and for using the Analysis Database to analyze the sample for gamma-emitting radionuclides. The procedure is suitable for sample collections that provide a minimum of 400mL of the edible portion of the sample. The method is designed to measure ^{137}Cs, ^{134}Cs, ^{103}Ru, ^{106}Ru and ^{131}I with an inaccuracy of <10% and a 1-σ imprecision of <5% at the corresponding derived intervention levels (DILs) for each radionuclide. These DILs are specified in the FDA Compliance Policy Guidance, Sec. 560.750 "Guidance Levels for Radionuclides in Domestic and Imported Foods" (July 2004). The method is also intended to detect and measure other gamma-ray emitting radionuclides. The spectral analysis parameters are set so that each of the radionuclides contained in the spectral library may be identified in a spectrum for quantification. The procedure is reliable and reproducible over the range of typical food densities (approximately 0.4g/mL to 1.4g/mL).

3. Responsibility

A. Supervisors

1. Ensure this procedure is properly implemented.
2. Ensure that the appropriate personnel are trained to perform the analysis using this SOP.
3. Ensure that the analysts are capable of providing acceptable analytical results through proficiency evaluation.

B. Method Monitors

Maintain and review the method QA logbook.

C. Analysts

1. Adhere to this SOP.
2. Perform and document required function verification and preventive maintenance on the spectrometer used for the analysis.
3. Ensure all analytical results are fully supported by acceptable quality control data.
4. Inform their supervisor when problems arise that could negatively impact timely sample analysis or the quality of sample results.
5. Document sample analyses on appropriate worksheet.

4. Background

A cylindrical polypropylene container with 400mL of sample is used for counting. Gamma-ray spectrometers are configured to accumulate counts for gamma emissions of 40 to 2000 keV in 4096 channels (0.5 keV per channel). A system background is established using a container identical to the sample container filled with 400mL of lab grade water and placed in the center of the detector platform. The system background is subtracted from the sample and laboratory control sample (LCS) results. The system background is applied as a paired observation (see Section 7.I). The spectrometer is energy calibrated whenever alignment is performed or fine tuning is desired. The energy calibration must be

performed prior to efficiency calibration using a mixed-gamma standard. The spectrometer is efficiency calibrated with four mixed gamma standards having matrices of different densities using WEAC-AB-TM.003 (see Sect 9.D). From these efficiency measurements, the dependence of efficiency on sample density is determined for each energy, and a correction factor calculated from these determinations is incorporated into an Excel spreadsheet. This spreadsheet calculates the activity concentration, 2-σ uncertainty, MDC, and LOQ of each radionuclide in the sample when the respective sample weight, the activity, and 1-σ uncertainty (uncorrected for density as reported by the spectrometer) are input.

5. References

A. Canberra Manuals:

1. Basic User Reference The APEX Lab Productivity Suite User's Manual; 9235237B v1.1*
2. Basic User Reference The APEX Lab Productivity Suite User's Manual; 923527C v1.3
3. The Genie-2000 Operations Manual; 9233652E v3.0*
4. DSA-2000 Digital Spectrum Analyzer User's Manual; 9231280H, 4/00*
5. Lynx Digital Signal Analyzer User's Manual; 9240227E
6. Cryo-Cycle Hybrid Cryostat; 9239789C
7. Genie-2000 Customization Tools Manual; 9233653E v3.0*
8. Model 9600 AIM/ICB System Setup Manual
9. Model 2100 NIM Bin/Power Supply User Manual
10. Model 9633 ADC User Manual
11. Model 9615 Amplifier User Manual
12. Model 9645 High Voltage Power Supply User Manual
13. Model 556A Acquisition Interface Module User Manual
14. Canberra Germanium Detector Manual
15. EG&G Ortec Solid-State Photon Detector Operator Manual

* These manuals are found on the instrument server computer in the C:\Genie2K\pdfs\docs\ folder.

Also see instructions found on each screen and/or function in the Analysis Database (H:\Analytical Branch\Radiochemistry\Analysis Database.accde).

B. "Standard Methods for the Examination of Water and Waste-water", 20th edition, editors Lenore S. Clesceri, Arnold E. Greenberg, and Andrew D. Eaton.

C. Compliance Policy Guidance, Sec. 560.750 "Guidance Levels for Radionuclides in Domestic and Imported Foods" (July 2004)

D. ISO/IEC 17025: 1999. "General Requirements for the Competence of Testing and Calibration and Laboratories".

E. E 181-98, Standard Test Methods for Detector Calibration and Analysis of Radionuclides ASTM International Publication (2003)

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

G. "Validation of Excel Spreadsheet for the Calculations Set Forth by the Method SOP-WEAC.RN.Method.3.0", Zhichao Lin and Zhongyu Wu.

H. "Validation of Excel Spreadsheet Form.WEAC.RN.Attachment A Gamma Analysis.xls version 2.0 and 2.1 for the Calculations from WEAC.RN.Method.3.0", Modified by Kelly Garnick; Original Validation by Zhichao Lin and Zhongyu Wu

I. "Validation of Excel Spreadsheet Form.WEAC.AB.RN.Gamma. LCS.ControlChart.xls version 1.0 for the QC Tracking of Gamma Analysis of Laboratory Control Samples", Kelly Garnick.

J. ORA-LAB.4.9 Control of Non-Conforming Work

6. Procedure

6.1. Instrumentation, Equipment, and Supplies

A. Gamma-Ray spectrometer: A high-resolution germanium spectrometer. Specific gamma spectrometer descriptions are provided in the spectrometer QA logbook (located in Room 116). The spectrometers are operated with APEX/ GENIE-2000 Canberra Spectroscopy Software. Nuclide identification is

dependent on energy calibration, spectral analysis parameters, and the library used.

B. Balance: A calibrated balance capable of weighing samples up to 1kg with a readability of one-tenth of one gram.

C. Sample counting container: SMC Stoesser 500mL polypropylene (2 5/8" x 4") container with lid (part numbers 4258 and 4000, respectively). The 400mL fill line is etched on the container based on a calculated fill height and verified using a calibrated ruler.

D. Utensils: Mortar and pestle, food processors, and common food utensils such as spoons, cutting knives, spatulas.

6.2. Reagents and Standards

A. Laboratory Grade Water (from Milli-Q system or equivalent water purification system)

B. Laboratory Control Sample (LCS) - A NIST-traceable mixed-gamma reference standard prepared in the same geometry as that used for the samples. The standard must contain radionuclides with photons that represent the low, middle, and high energy range of 40 – 2000 keV. An LCS is counted under the same conditions as a sample (i.e., counting time) once per calendar week (i.e. a batch starts on Sunday and ends after Saturday) for each detector used to count samples. The same LCS should be used for a given spectrometer week-to-week. Each current LCS standard is maintained in the radioactive materials cabinet in the counting room and is identified with the spectrometers it's currently associated with.

6.3. Analytical Procedure

A. Sample Preparation

1. Sample Preservation

Samples are typically thawed and maintained at refrigeration temperatures until composited.

a. Samples should be obtained and secured according to WEAC-QMS.5.8, WEAC Sample Handling Procedures.

b. Maintain sample in accordance with its labeled instructions for preservation and storage. Ensure that all

refrigerators and freezers used meet the QC requirements found in WEAC-AB.3.0, Monitoring of Freezers, Incubators, Ovens, Waterbaths & Refrigerators.

c. When no labeling or storage instruction is indicated, take appropriate measures (usually refrigerating or freezing) to maintain the sample's quality until it's composited.

d. To minimize uncertainties due to composite layer separation and settling, ensure counting proceeds as soon after compositing as possible. When preservation and storage procedures are atypical (e.g., a sample is refrozen or preserved with formaldehyde), document these specifics on the appropriate worksheet as follows:

   i. For regulatory samples:

      1) For routine samples use WEAC-TMPL.074, Gamma and Strontium-90 Worksheet.

      2) For emergency samples, use WEAC-TMPL.140 Gamma Worksheet for Emergency Samples.

   ii. For non-regulatory samples: enter sample information into the Analysis Database.

2. Sample Compositing

   a. Remove the inedible portion of the sample from all portions that will be used for analysis. Ensure utensils used for sample preparation are clean. To prevent cross-contamination, **do not reuse** utensils **until** they've been cleaned in accordance with WEAC-LAB.23.0, Laboratory Glassware Washing and General Maintenance.

   b. Combine the edible portions of sample subs in accordance with WEAC-AB.8.0, Laboratory Sub Sampling Procedure (usually using a food processor or blender) to create a homogenous composite.

      i. For regulatory samples:

         1) Identify the subs represented in the composite and document the procedure used to homogenize the edible portion on the WEAC-TMPL.074, Gamma and Strontium-90 Worksheet for routine samples, or

2) WEAC-TMPL.140 Gamma Worksheet for Emergency Sample for emergency samples.

ii. For non-regulatory samples: enter sample information for Total Diet Samples and other non-regulatory samples into the Analysis Database.

3. Sample Weighing, Counting Preparations

a. Ensure that the balance meets QC requirements in accordance with WEAC-LAB.6.0, Operation and Maintenance of Laboratory Balances.

Note: The fill line is etched on the sample container at the 400mL level (based on the height measurement).

b. Tare container with lid to zero, then pack the homogenized composite into the container up to the fill line. Don't over pack. Cover with a lid and reweigh the filled container. Record the mass of the analytical portion to the nearest 0.1g on the worksheet.

c. Verify that the spectrometer QC calibration check was performed according to WEAC-AB-RN.12.0 and that the detector meets QC specifications.

d. Verify that the daily background/method blank was counted and that the QC specifications were met. Check the online QA report to determine if QC specifications were met. (See instrument SOP for instructions).

B. Laboratory Control Sample Analysis

1. Routine Operation – Check the QA logbook to see if the LCS had been counted on the spectrometer during the week.

a. If not, place the LCS standard on the platform of the detector, count, and obtain the LSC report as described in Section 6.3.D.3. Enter the reported results into the Gamma Analysis Spreadsheet (H:\QMS\WEAC SOP's\FORMS\Radionuclide Forms\Form.WEAC.RN. Attachment A-LCS Gamma Analysis).

b. While in the spreadsheet, click "save to pdf" to create the report in the appropriate file and click "transfer to data base" (ensure that you don't have the database open at this time which will prevent macro operation). Attach the

spectrometer report to the Excel report that was saved in H:\Analytical Branch\Radiochemistry \Gamma LCS Attachment copies*Fiscal Year*\Week Starting xx/xx/xx. Open the Analysis Database, go to Gamma QC button and verify that the results meet specifications as described in the Quality Control Section of this document and print a copy of the control charts.

2. Check the Form.WEAC.AB.RN.2.1, Function Verification/ Preventative Maintenance (FV/PM) Chart for High-Purity Germanium Gamma-Ray Spectrometers to ensure the LCS count was collected.

3. To count an LCS using APEX software:
   a. Go to the “Samples” tab and the “Samples” page (or you can go to the Start button in the upper right corner of the detector MCA) and choose “LCS” from the “Sample Type” pull down menu. Type “LCS” followed by the standard’s identification number as the sample identification.
   b. Click on the plus sign in the box to the left of the procedure name “WEAC-RN-Method.3.0”.
   c. Select the procedure that corresponds to the sample identification and click “Next”.
   d. Enter the standard reference date and time and the standard weight in kilograms and click “Save”.
   e. Go to the “Main” tab and drag and drop the LCS sample information that’s now listed in the sample list to the left of the spectral window in the appropriate spectrometer window.

Note: If the count is started using the MCA start button, the count will start automatically, so make sure that the LCS is in place in the spectrometer ***before*** clicking the start button.

C. Sample Analysis

Note: Before starting your count, check the nitrogen Cryostat under the germanium detector to ensure it’s not empty, which is indicated with a red fault light.

1. Place the sample container at the center of the sample holder on the face of the shielded high purity germanium (HPGe) detector. Close the shield and count the sample as described below.
2. To count the sample usingGenie-2000/APEX software:
   a. Go to the “Samples” tab and the “Samples” page. (Again, this can also be done from the MCA window “Start” button, if the sample has already been placed in the spectrometer.)
   b. Select the appropriate sample type from the pulldown menu. Enter the sample FACTS number as the sample identification. Enter the sample description as it is stated on the sample worksheet. For total diet study samples, add the sub number to the end of the sample description (e.g., Whole milk, sub001).
   c. Go to the “Procedure Selection” tab, check “WEAC RN Method 3.0”. Select the procedure (e.g. “Routine Food – decay Corrected” or “Routine Food –not decay Corrected”) procedure based on the sample type. Click “Next”.
   d. Input sample weight and sample collection or reference time, if necessary.
   e. Go to the “Main” tab and drag and drop the sample information now listed in the sample list to the left of the spectral window in the appropriate spectrometer window.

Note: The count will start automatically if initiated via the MCA window “Start” button, so make sure that the sample is already in place in the spectrometer).

3. Once the count is complete:
   a. Go to the Data Review tab and search for the count in the review list.
   b. Highlight the count of interest and click “Next”.
   c. Review the count report.
   d. Place a USB portable device into the computer and click on the export file icon just above the report (right side).
   e. Save the .pdf report file and transfer to an FDA intranet computer.

4. Enter the reported results into the Gamma Analysis Spreadsheet.
   a. Use WEAC-TMPL.132, Attachment A – 100 min Gamma Analysis Spreadsheet for routine samples that have been counted for 100 minutes.
   b. Use WEAC-TMPL.139, Attachment A – 10 min Count Gamma Analysis Spreadsheet for samples that have been counted for 10 minutes.
   c. Use WEAC-TMPL.133, Attachment A – LCS Gamma Analysis Spreadsheet for LCS counts.

6.4. Interferences

A. Chemical Interferences: Chemical interference in gamma-ray analysis can be seen in the attenuation of the gamma activity by the sample matrix. This attenuation is most significant when the sample density in the container is high and the energy of the gamma emission being evaluated is low.

B. Radiological Interferences:

1. Misidentification of naturally occurring radionuclide peaks should not be misidentified as radionuclide contaminants if the spectrometer is properly energy calibrated and the library acceptance criteria is set appropriately. However, occasionally, small noise peaks may produce this type of error. For this reason, every analysis resulting in reportable activity for a radionuclide other than K-40 requires a duplicate analysis.
2. Errors may also occur when a radionuclide is present in the sample at a high level. The summation of characteristic gamma emissions may result in falsely identified summation peaks. Carefully examine the spectrum for the presence of these peaks when a sample is found to contain reportable amounts of contaminant radionuclides.

6.5. Calculations

The Canberra spectroscopy package provides vendor-validated algorithms that allow for the detection and integration of peaks in the spectrum given the proper peak processing parameters. The

calculations described in this section are embedded in the Gamma Analysis spreadsheets (WEAC-TMPL.132, WEAC-TMPL-139, and WEAC-TMPL.139).

A. Radionuclide Determination

To determine whether a radionuclide is present in the sample, the spectrometer compares the measured energy of the radionuclide emissions with the nuclide library. If a peak is detected within the user defined energy tolerance range, a peak match is declared. If more than one peak is found within the energy tolerance range, the closest match is chosen.

The following equation is used to quantify the activity concentration of a radionuclide in the sample:

$$A_d = \frac{P}{q \times \varepsilon_d \times b \times E_l} \times e^{\lambda T_s}$$

B. Efficiency Value

The efficiency value, ε_d, is dependent on sample density. It's calculated by determining efficiency curves for four standards of varying density. These determinations are accomplished using the method WEAC-AB-TM.003, Determination of Food-Specific Efficiency Calibrations for Gamma-Emitting Radionuclides in High-Purity Germanium Detectors.

C. Density Correction Factor

The equation used to calculate the density correction factor is shown below:

$$dcf = \frac{\varepsilon_u}{\varepsilon_d}$$

To quantify the 1-sigma uncertainty associated with the activity measurement prior to density correction, the following equation is used. (The contribution due to half-life uncertainty is not included because it is extremely small):

$$\Delta A_u = A_u \sqrt{\left[\frac{\Delta P}{P}\right]^2 + \left[\frac{\Delta b}{b}\right]^2 + \left[\frac{\Delta \varepsilon_u}{\varepsilon_u}\right]^2 + \left[\frac{\Delta q}{q}\right]^2}$$

The density corrected activity uncertainty is reported as 2-sigma. The following calculation is performed in the Excel spreadsheet to account for the uncertainty in the density correction factor and the uncertainty due to the volume measurement.

$$\Delta A_d = 2A_u \varepsilon_d \sqrt{\left[\frac{\Delta A_u}{A_u}\right]^2 + \left[\frac{\Delta \varepsilon_d}{\varepsilon_d}\right]^2 + \left[\frac{\Delta V}{V}\right]^2}$$

D. Minimum Detectable Activity Concentration

To calculate the minimum detectable activity concentration (MDC) the following equation is used by the Genie-2000/APEX software:

$$MDC_u = \frac{\left(2.71 + 4.65 \times \sqrt{B}\right)}{q \times \varepsilon_u \times b \times E_l} \times e^{\lambda T_s}$$

The spectral report provides the MDC value prior to the density correction. The uncorrected MDC value from the spectral report is entered into the validated Excel spreadsheet and multiplied by the density correction factor to complete the calculation.

E. Limit of Quantification

The Limit of Quantification in activity concentration (LOQ) is calculated using the following equation:

$$LOQ_d = \frac{50\left\{1 + \left[1 + \frac{B}{12.5}\right]^{\frac{1}{2}}\right\}}{q \times \varepsilon_d \times b \times E_l} \times e^{\lambda T_s}$$

The following equation has been derived to calculate the density corrected LOQ using the uncorrected MDC provided by the spectral report. This equation is used to calculate the corrected LOQ in the validated Excel spreadsheet.

$$LOQ_d = \frac{50 \times \left[1 + \sqrt{1 + \frac{\left(\frac{\left(MDC_d \times q \times \varepsilon_d \times b \times 6000\right) - 2.71}{4.65}\right)^2}{12.5}}\right]}{q \times \varepsilon_d \times b \times 6000} \times e^{\lambda T_s}$$

A_u=Activity concentration (Bq/kg) prior to density correction

A_d=Activity concentration (Bq/kg) corrected for sample density

$P=N_S-N_B$; Net Peak Area in sample after subtraction of environmental background

N_S=Sample Net Peak Area (counts)

N_B=Net Peak Area (counts) in background subtraction spectrum

q=Sample quantity (kg)

d=Sample packing density (kg/L); d=q/V

V=container fill volume; V=400mL or 0.4L

ε_u=Uncorrected counting efficiency

ε_d= Density adjusted counting efficiency; the uncorrected counting efficiency /dcf

b=gamma-ray abundance

E_l=Elapsed live time (seconds)

λ=decay constant ($ln2/T_{1/2}$); seconds^{-1}

$T_{1/2}$=half-life of radionuclide; seconds

T_s=sample date - acquisition date; seconds

$B=B_{Sc}+N_B$; The background counts used for the MDC and LOQ calculations in the region of the radionuclide key-line energy

B_{Sc}=The continuum counts in the region of the radionuclide key-line energy in the sample spectrum

dcf=density correction factor

MDC_u= Minimum Detectable Activity Concentration with density correction

MDC_d= MDC_u x dcf

LOQ_d=Limit of Quantification with density correction

ΔA_u=The uncertainty of the activity concentration prior to density correction

ΔA_d=The uncertainty of the density corrected activity concentration

ΔP=The uncertainty of the Net Peak Area in sample after subtraction of environmental background

Δb=The uncertainty in the gamma-ray abundance, which is obtained from the radionuclide library.

$\Delta\varepsilon_u$=The uncertainty in the efficiency prior to density correction.

$\Delta\varepsilon_d$=The uncertainty in the density corrected efficiency, which is determined by propagating the uncertainties from the library information (i.e. half-life and abundance), the uncertainty of the calibration standard and the uncertainty of the curve fitting.

Δq=The uncertainty in the sample quantity, which is determined by the balance readability.

ΔV=The uncertainty in the container fill volume; 400mL±20mL (5%).

Table 1. Estimates of the Uncertainty in Activity Concentration for Radionuclides of Interest for the Method at the LOQ and Target Level

Description of Source of Standard Uncertainty	Typical Value of Uncertainty Source	Standard Uncertainty in value	Unit	Evaluation Method for the Standard Uncertainty (A) statistical method (B) other method	Relative Standard Uncertainty
Sample Weight, W_s [4]	0.4000	0.0001	kg	σW_s, estimated (B)	0.03%
Net Area in Peak, $C_{s\ at\ LOQ}$	100	10	counts	σC_s, estimated (A)	10.00%
Net Area in Peak Ru-103, C_s at target level[1]	118674	344	counts	σC_s, estimated (A)	0.29%
Net Area in Peak Ru-106, C_s at target level[1]	851	29	counts	σC_s, estimated (A)	3.43%
Net Area in Peak I-131, C_s at target level[1]	3680	61	counts	σC_s, estimated (A)	1.65%
Net Area in Peak Cs-134, C_s at target level[1]	18045	134	counts	σC_s, estimated (A)	0.74%
Net Area in Peak Cs-137, C_s at target level[1]	31414	177	counts	σC_s, estimated (A)	0.56%
Counting Efficiency, E_s [3]	various	various	fractional	σE_s, estimated (B)	2.56%
Volume, V	400	20	mL	σV, estimated (B)	5.00%
Abundance, b_{Ru103} [2]	91.00	0.00	%	σ_b, estimated (B)	0.50%
Abundance, b_{Ru106} [2]	9.93	0.00	%	σ_b, estimated (B)	0.50%
Abundance, b_{I131} [2]	81.70	0.00	%	σ_b, estimated (B)	0.08%
Abundance, b_{Cs134} [2]	85.40	0.00	%	σ_b, estimated (B)	0.40%
Abundance, b_{Cs137} [2]	85.10	0.00	%	σ_b, estimated (B)	0.23%

Relative Total Combined Standard Uncertainty of Activity Result at LOQ (%): <	11.48%
Coverage factor, k (k=2):	x 2
Relative Expanded Unceratinty of Activity Result at LOQ(%): <	22.96%
Relative Expanded Uncertainty of Ru-103 Activity at Target Level (%): <	11.29%
Relative Expanded Uncertainty of Ru-106 Activity at Target Level (%): <	13.20%
Relative Expanded Uncertainty of I-131Activity at Target Level (%): <	11.71%
Relative Expanded Uncertainty of Cs-134 Activity at Target Level (%): <	11.36%
Relative Expanded Uncertainty of Cs-137 Activity at Target Level (%): <	11.30%

[1] The counts at the target level are calculated by multiplying the area at the LOQ by the ratio of the act. at the target level to the act. at the LOQ level

LOQ values are from DET12, which has the lowest relative efficiency provides maximum values, assuming a typical density of 1.0g/mL.

[2] Abundance uncertainties are obtained from Brookhaven National Library

[3] Counting Efficiency uncertainty includes the uncertainty of the fits and the uncertainty due to spectral factors ($\Delta P, \Delta b, \Delta q$ and uncertainty in standard activities). The uncertainty due to the fits is estimated over a range of densities (0.4-1.4g/mL) using absolute efficiencies from DET1and calculating an average uncertainty from low and high energy extremes (See Table 2). The estimated uncertainty due to spectral factors is 1.46%.

Table 2. Estimation of Typical Uncertainty in Calculated Efficiency Value

				95% Prediction Limits		95% Confident Limits						
Standard Identification	Energy	X (logE)	Y (logEff)	LPL (logEff)	UPL (logEff)	LCL (logEff)	UCL (logEff)	LCL (Eff)	UCL (Eff)	Mean (Eff)	% Uncertainty (Eff)	(Avg)
SRS70622-260	59.54	1.77481	-1.86329	-1.8848	-1.84178	-1.8766	-1.8500	0.0133	0.0141	0.0137	3.06%	3.05%
Paper	1836.06	3.26389	-1.93753	-1.9590	-1.91607	-1.9507	-1.9243	0.0112	0.0119	0.0116	3.04%	
SRS70623-260	59.54	1.77481	-1.93309	-1.96592	-1.90026	-1.9572	-1.9090	0.0110	0.0123	0.0117	5.55%	5.54%
Coffee Grounds	1836.06	3.26389	-1.96202	-1.9948	-1.92925	-1.9861	-1.9380	0.0103	0.0115	0.0109	5.54%	
SRS70624-260	59.54	1.77481	-2.0297	-2.05997	-1.99942	-2.0520	-2.0074	0.0089	0.0098	0.0094	5.12%	5.11%
Aqueous equivalent	1836.06	3.26389	-2.00317	-2.0334	-1.97295	-2.0254	-1.9810	0.0094	0.0104	0.0099	5.10%	
SRS70625A-260	59.54	1.77481	-2.06961	-2.0877	-2.05152	-2.0829	-2.0563	0.0083	0.0088	0.0085	3.06%	3.06%
Honey	1836.06	3.26389	-2.02711	-2.04517	-2.00905	-2.0404	-2.0139	0.0091	0.0097	0.0094	3.05%	
										Grand Avg (1σ%)		2.1%
										Grand Avg (2σ%)		4.2%

[1] Uncertainties are estimated using the measured efficiency values from DET1

[2] 95% prediction limits and 95% confidence limits for the log of the efficiency are determined by STATGRAPHICS software.

[3] % uncertainty includes the uncertainties in the sample specific efficiencies at standard gamma-ray energies and the uncertainty in the curving fitting

6.6. Quality Control

A daily background is counted in accordance with the spectrometer SOP. As part of this previous QC, a report is generated containing the peak search results for the daily background count. The values of the limits and their supporting data are documented in the method QA logbook.

A. Method Blank

1. Method Blank Count: The method blank QC specifications are established based on historical data or standard reference values and are applied to the previously counted daily background count results. The net area in the 1460keV peak due to K-40 (prior to the subtraction of the typical background) and the total counts in the spectrum are

automatically transferred to a quality assurance file at the time of collection.

2. Method Blank Acceptance Criteria: The total counts must be evaluated against pre-established warning and control limits, 2σ and 3σ, respectively. The method blank is acceptable if the total count value is within the warning limits or between the warning and control limits not more than three consecutive times. The method blank pre-established limits should be reevaluated following spectrometer repair or shielding reconfiguration. The K-40 value is presented to provide the analyst with additional information and for trending purposes. This value is not subject to acceptance criteria.

B. Laboratory Control Sample (LCS):

1. Routine operation: The LCS must be analyzed by the sample method with each batch of samples. A batch is defined as all the samples that are analyzed on a detector in one calendar week. The LCS is counted under the same conditions as the sample, the same analysis routines are performed by the spectrometer, and the results are also density-corrected. The LCS analysis results are entered into the Analysis Database for evaluation of QC criteria. Quality control criteria are applied to Ba-133, Cs-137, and Co-60, which represent the low, middle, and high regions of the energy range.

2. Surge Capacity Operations: The QC calibration check source should be counted as an LCS once per twenty samples using the procedure described in WEAC-AB-RN. 12.0.

3. Criteria for LCS: The z-score of the activity of each radionuclide must be evaluated against warning and control limits that are set to be ±2 and ±3, respectively. The LCS result is acceptable only if its z-score is within ±2, or between ±2 and ±3 not more than three consecutive times. Otherwise, the entire batch analysis is invalid.

4. Corrective Action for Out-of Control Results

   a. When an "**Action**" flag appears, the spectrometer isn't considered suitable for analysis until an investigation into

the cause of the problem is completed, action is taken to correct the problem, and a repeat analysis meets all acceptance criteria.

b. When an "**Investigate**" flag appears: the analyst must examine the LCS quality control chart.

   i. If they find three consecutive flags for the same result, the spectrometer isn't considered suitable for analysis and the flag is treated as if it were an "**Action**" flag.

   ii. If recent historical data shows no other flags for the same result, the spectrometer is considered suitable for analysis.

6.7. Method Performance

Proficiency Sample Analysis: The WEAC laboratory must participate in proficiency studies that adhere to the requirements of WEAC-QMS.5.9, WEAC Proficiency Testing. Whenever possible, studies should contain multiple radionuclides, possible radiological interference (i.e., naturally occurring radionuclides or fission products), and non-aqueous matrices.

6.8. Safety, Contamination Control and Waste Management

Refer to the Chemical Hygiene Plan and Hazardous Waste Management Program (Sect 9.G and H) and the WEAC Radiation Safety Manual and Radioactive Waste Handling Procedure (Sect 9.J and K) for guidance in handling and disposing of radioactive standards. The WEAC Radiation Safety Manual also presents procedures for decontamination of surfaces if necessary. Warnings and safety procedures associated specifically with spectrometer use are provided in the manuals (Section 5.A. 1-15). The primary safety considerations while using the spectrometer are 1) safely handling liquid nitrogen and 2) using appropriate precautions in the vicinity of a high voltage power supply.

7. Definitions/ Glossary

A. **Density**: The mass per unit volume of the sample.

B. **DIL**: Derived intervention level

C. **Edible portion**: The portion of a sample that is routinely eaten or routinely used as an ingredient in food.

D. **Empty shield**: The detector shield chamber is empty except for the detector itself.

E. **Inedible portion**: The portion of a sample that is not routinely consumed.

F. **Limit of Quantitation (LOQ)**: The activity concentration level at which an imprecision of less than 10% is expected (1-σ based on counting statistics alone).

G. **Minimum Detectable Activity Concentration (MDC)**: The minimum detectable activity (MDA), as calculated by the Currie limit, corrected for sample weight and expressed in Bq/kg.

H. **NIST** – National Institute of Standards and Technology

I. **Paired observation**: A term used by Llyod Currie to describe an analytical background subtraction when both the sample and the background are counted for the same length of time.

J. **Z-score**: The difference between the measured and the expected value divided by the root sum square of their respective standard uncertainties.

8. Records

A. Spectrometer reports – method blank, sample and LCS

B. Density-corrected Excel report – sample and LCS

C. Analytical worksheet (in Analysis Database for TDS or 431 for regulatory samples)

D. Background Control Charts – (found on the spectrometer software)

E. LCS Control Charts – (found in the Analysis Database)

9. Supporting documents

A. WEAC-AB.3.0 Monitoring of Freezers, Incubators, Ovens, Water Baths and Refrigerators

B. WEAC-AB.8.0 Laboratory Sub Sampling

C. WEAC-AB-RN.12.0 The Canberra APEX Operated High-Purity Germanium Gamma Spectrometers

D. WEAC-AB-TM.003 Determination of Food-Specific Efficiency Calibrations for Gamma-Emitting Radionuclides in High-Purity Germanium Spectrometry

E. WEAC-LAB.6.0 Laboratory Balances

F. WEAC-LAB.8.0 Quality Control for Sample Storage Areas

G. WEAC-LAB.12.0 Chemical Hygiene Plan

H. WEAC-LAB.14.0 Hazardous Waste Management Program

I. WEAC-LAB.23.0 Laboratory Glassware Washing and General Maintenance

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

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

L. WEAC-QMS.4.11 WEAC Corrective Action Procedure

M. WEAC-QMS.5.4 Method Verification and Validation

N. WEAC-QMS.5.9 WEAC Proficiency Testing

O. Form.WEAC.AB.RN.2.1/ WEAC-TMPL.037 Function Verification/ Preventative Maintenance Chart for High-Purity Germanium Gamma-Ray Spectrometers

P. Form.WEAC.AB.RN.2.2/ WEAC-TMPL.038 Liquid Nitrogen Fill Log for High-Purity Germanium Gamma-Ray Spectrometers

Q. Form.WEAC.AB.RN.2.3/ WEAC-TMPL.039 Modification/ Investigation for High-Purity Germanium Gamma-Ray Spectrometers

R. WEAC-TMPL.074 Gamma and Strontium-90 Worksheet

S. WEAC-TMPL.132 Attachment A - Gamma Analysis Report

T. WEAC-TMPL.133 Attachment A - LCS Gamma Analysis Report

U. WEAC-TMPL.139 Attachment A - 10 min Count Gamma Analysis Spreadsheet

V. Form.WEAC.RN.Method.3.2/ WEAC-TMPL.140 Gamma Worksheet for Emergency Samples

10. Attachments

None

Document History

Version #	Status* (D,I, R, C)	Date	Author Name and Title	Approving Official Name and Title
1.0	I	12/3/04	KELLY GARNICK, CHEMIST MIKE CASEY, CHEMIST	PAMELA MACKILL ANALYTICAL BRANCH DIRECTOR
2.0	R	5/24/05	KELLY GARNICK, CHEMIST MIKE CASEY, CHEMIST	PAMELA MACKILL ANALYTICAL BRANCH DIRECTOR
3.0	R	3/9/06	KELLY GARNICK, CHEMIST MIKE CASEY, CHEMIST	PAMELA MACKILL ANALYTICAL BRANCH DIRECTOR
4.0	R	3/31/06	KELLY GARNICK, CHEMIST MIKE CASEY, CHEMIST	PAMELA MACKILL ANALYTICAL BRANCH DIRECTOR
5.0	R	1/9/07	KELLY GARNICK, CHEMIST MIKE CASEY, CHEMIST	PAMELA MACKILL ANALYTICAL BRANCH DIRECTOR
5.1	R	2/2/07	KELLY GARNICK, CHEMIST MIKE CASEY, CHEMIST	PAMELA MACKILL ANALYTICAL BRANCH DIRECTOR
6.0	R	5/14/07	KELLY GARNICK, CHEMIST MIKE CASEY, CHEMIST	PAMELA MACKILL ANALYTICAL BRANCH DIRECTOR
6.1	R	7/30/08	KELLY GARNICK, CHEMIST MIKE CASEY, CHEMIST	PAMELA MACKILL ANALYTICAL BRANCH DIRECTOR
7.0	R	5/13/2010	KELLY GARNICK, CHEMIST MIKE CASEY, CHEMIST	CONG WEI, ACTING ANALYTICAL BRANCH DIRECTOR
7.1	R	7/29/2010	KELLY GARNICK, CHEMIST STEPHANIE HEALEY, CHEMIST	PAMELA MACKILL ANALYTICAL BRANCH DIRECTOR
8.0	R	3/24/2011	KELLY GARNICK, CHEMIST THOMAS SCOTT, CHEMIST	PAMELA MACKILL ANALYTICAL BRANCH DIRECTOR
8.1	R	1/30/2013	KELLY GARNICK, CHEMIST THOMAS SCOTT, CHEMIST	PATRICK REGAN ANALYTICAL BRANCH DIRECTOR

9.0	R	9/2/2014	KELLY GARNICK, CHEMIST	PATRICK REGAN ANALYTICAL BRANCH DIRECTOR
9.1	R	12/12/2014	ZHICHAO LIN, CHEMIST	PATRICK REGAN ANALYTICAL BRANCH DIRECTOR

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

Approving Official's signature: _____**In QMiS**_____

Change History

Version 9.1 - LOQ equation was corrected in section 6.5.E.