Sections 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 137Cs, 134Cs, 103Ru, 106Ru and 131I 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

1. Maintain and review the method QA documentation related to the method.
2. Assist in investigations related to the method.

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.E). 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; v1.3 and 1.4
2. The Genie-2000 Operations Manual; 9233652E v3.0*
3. Lynx Digital Signal Analyzer User's Manual; 9240227E
4. Cryo-Cycle Hybrid Cryostat; 9239789C
5. Genie-2000 Customization Tools Manual; 9233653E v3.0*
6. Canberra Germanium Detector Manual
7. 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 along with legacy power supply and electronic signal processing manuals.

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

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

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 the instrument room. 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 1 kg 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 400 mL fill line is etched on the container based on a calculated fill height. The height is measured using a height caliper which is verified immediately before and after using with height gauges. See SOP-000420.

D. Height caliper - Verified before and after height measurement on each day of measurements with traceable height gauge blocks.

E. 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.

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, and 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), record these specifics in the Gamma Worksheet section of 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. Identify the subs represented in the composite and document the procedure used to homogenize the edible portion in the Gamma Worksheet section of the Analysis Database in the designated section on p. 2.

3. Sample Weighing, Counting Preparation

a. Ensure that the counting container can be referenced to a verification of the fill line marking. The height verification identification number is etched on the bottom of the container. Record the identification number as the “Container Id”.

b. 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).

c. Tare container with lid to zero, then pack the homogenized composite into the container up to the fill line. Cover with the lid and reweigh the filled container. Record the mass of the analytical portion to the nearest 0.1g. Data may be recorded on a raw data attachment or recorded directly into the database. When a raw data sheet is used, scan the sheet for inclusion in the worksheet.

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

e. 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. <u>Routine Operation</u> –

a. If the LCS has not been run for the current week, place the LCS standard on the platform of the detector, count, and obtain the

LCS report as described in Section 6.3.B.3. Enter the reported results into the LCS Results page of Attachment A - Gamma Analysis Report (WEAC-TMPL.132), which may be accessed via the Data Entry button of the Gamma QC Menu of the Analysis Database or directly from QMIS.

b. While in the spreadsheet, click “save to pdf” to create the report in the appropriate file and click “transfer to data base”. Attach the spectrometer report to the Excel report that was saved in H:\Analytical Branch\Radiochemistry \Gamma LCS Attachment copies\Fiscal Year\Week Starting *mmddyy*. 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.

2. 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. Enter the shorthand standard identification (e.g. 14V) for the sample Id. In the sample description, include analyst’s initials and a sample description (e.g. Vegetation).

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

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 using Genie 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. In the sample description, enter the analyst’s initials and the sample description. For total diet study samples, include the sub number (e.g., KMG: 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 an IT approved 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.

   Note: Sample reprocessing should adhere to the instructions listed in WEAC-AB-RN.12.0. No changes should be made to the sample or LCS Analysis Sequence File (ASF).

4. Enter the reported results into WEAC-TMPL.132 Gamma Analysis Spreadsheet. While in the spreadsheet, click “save to pdf” to create

the report and click “transfer to database”. Attach the sample spectrometer report to the sample Excel report that is generated.

5. For regulatory samples, assemble the worksheet and attachment pages either as a .pdf or physical worksheet as per program guidance.

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. High abundance background gamma-emissions from K-40, Th-232 and progeny and U-238 and progeny should not be misidentified as radionuclide contaminants if the spectrometer is properly energy calibrated. Keyline selections in the library and the confidence threshold value in the analysis sequence file have been selected so that background radionuclides at typical activity levels will not be misidentified (with 95% confidence). Due to the small percentage chance of a false positive, especially to due noise peaks or atypically elevated background, every analysis resulting in an activity at levels that are above both the MDC and the 2-sigma uncertainty for a radionuclide other than K-40 requires a confirmatory count (typically performed by the same analyst). Note that this evaluation must be made using only lines that are detected, which is indicated on the spectrometer report with an * next to the line energy.

   Due to the potential for systematic uncertainties in levels of naturally occurring background (U-238 and Th-232 and progeny) due to environmental factors in which dust may be created (e.g. nearby construction), we have included an assessment of the average MDC’s for Ra-226, Bi-214 and Pb-214. In order to assure that variations in background levels either during the background or sample count do not lead to false reporting of these naturally occurring background radionuclides (Ra-226, Bi-214 and Pb-214), analysts should identify samples with reportable activities (as defined in the previous paragraph) of any of these radionuclides that are below the following average method MDC values as not detected.

Average Method MDC's for common natural progeny based on primary energy line

Ra-226 (186keV)	29 Bq/kg
Bi-214 (609keV)	14 Bq/kg
Pb-214 (351keV)	29 Bq/kg

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 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 spreadsheet (WEAC-TMPL.132).

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:

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

B. Efficiency Value

The efficiency value, ε_d, is dependent on sample density. It is 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 Value

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 paired sample-background approach by Currie is applied as follows:

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

For scope radionuclides with no net peak for the energy in the system background spectrum, B is the gross signal in the energy region. 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. Limits of Quantification

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

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

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.

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

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):	x2
				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-131 Activity 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 (DP, Db, Dq 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 efficincies from DET1 and 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)	% Uncertainty (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

Table 3: Uncertainty in the volume measurement

Volume (mL)	±	1-sigma uncertainty of volume (mL)	Geometry entry D1.1 - container bottom (cm)	±	1-sigma uncertainty in D1.1 (cm)	Geometry entry D1.3 - container diameter (cm)	±	1-sigma uncertainty in D1.3 (cm)	Geometry entry D2.1 + D1.1 = height of fill line (cm)	±	1-sigma uncertainty in D2.1 (cm)
400.0	±	5.6	2.00	±	1.00	98.10	±	0.50	54.92	±	0.50

5% systematic uncertainty is propagated for volume uncertainty, which includes uncertainties in the fill line and in filling the container to volume.

6.6. Quality Control

A time-of-use background is counted in accordance with the spectrometer SOP.

A. Method Blank (also called systematic background)

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 1460 keV 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.

   When background values are elevated, the analyst should inform the monitor of all warning and control limit results so that trends may be observed as soon as possible. If an “action” flag appears for an elevated background total counts, an analyst may obtain a clean counting container filled to the line with Milli-Q water and recount the background using the new counting container. If the subsequent count is in acceptable limits, the analyst may use the detector for sample analysis. When background issues occur, it is necessary to evaluate the impact on samples analyzed since the last acceptable background was obtained.

   Note that for emergency operation (i.e. when 10-minute counts are in use), method blanks or 100-min systematic backgrounds are performed at least once a week. During this time of operation, the analyst may perform either a 100-min systematic background or a 10-min background check to satisfy the background requirement for the day.

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. Criteria for LCS: The z-score of the activity of each radio-nuclide 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.

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 near a high voltage power supply.

7. Glossary/Definitions

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-□ 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 Lloyd 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)

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

E. Gamma Control Charts – (found in H:\Analytical Branch\Radiochemistry\WEAC.AB.Log.3.0)

9. Supporting Documents

A. SOP-000420 Marking Gamma Container Fill Line

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

C. WEAC-AB.8.0 Laboratory Sub Sampling

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

E. WEAC-AB-RN.022 Digital Signatures

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

G. WEAC-LAB.6.0 Laboratory Balances

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

I. WEAC-QMS.5.9 WEAC Proficiency Testing

10. Document History

Revision #	Status* (D, I, R)	Date	Author Name and Title	Approving Official Name and Title
1.0	I	12/3/04	KELLY GARNICK, CHEMIST 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
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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

Revision #	Status* (D, I, R)	Date	Author Name and Title	Approving Official Name and Title
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
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9.3	R	8/2/2016	KELLY GARNICK, CHEMIST	PATRICK REGAN ANALYTICAL BRANCH DIRECTOR
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10	R	11/8/2019	KELLY GARNICK, CHEMIST	PATRICK REGAN ANALYTICAL BRANCH DIRECTOR
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12	R	See InfoCard	KELLY GARNICK, CHEMIST	PATRICK REGAN ANALYTICAL BRANCH DIRECTOR

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

11. Change History

Revision #	Change
12	Updated manuals and references and removed site-specific information. Indicated that raw data sheets are optional. Added note about background QC during emergency operation. Added references to SOP-000420.

12. Attachments

None