

GAMMA-EMITTING RADIONUCLIDES

Procedure for gamma-ray spectrometry measurements for activity calibration and impurity measurements using High Purity Germanium (HPGe) detectors

1. Purpose

This procedure describes the activity calibration of gamma-ray emitting sources (single or mixed radionuclides in solution, solid, or gas form, and in special geometries) using the Radiation Physics Division (RPD) High Purity Germanium (HPGe) detectors. It also describes the procedure for measurements of gamma-ray emitting impurities as required for corrections to other radionuclide calibration techniques.

2. Scope

The HPGe detectors are used for activity (as Bq), emission rates (as gammas/sec) and impurity measurements of gamma-ray emitting radionuclides. Measurements include source calibrations for different organizations including nuclear power plants, radiopharmaceutical companies, universities, national laboratories, as well as for the National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) and radionuclide metrology research programs.

This calibration service is relevant for radionuclides emitting gamma-rays with energies between 35 keV and 3.5 MeV and an activity range of 1 kBq to 10 MBq. The nominal relative uncertainty range is 0.8 percent to 2 percent (corresponding to 1 standard deviation). The calibration of the HPGe system is performed by obtaining efficiency curves for different source geometries and source-to-detector distances using NIST SRMs and NIST calibrated sources.

3. Definitions

Terms used in this procedure are listed in IEC 61452 [1] and only a few relevant terms are listed here.

Activity (A): the expected number of spontaneous nuclear decays (transformations) in unit time from a specified energy state (excluding prompt decays from lower nuclear levels) for a given amount of a radionuclide. Its SI unit is the becquerel (Bq) where one Bq equals one decay per second.

Background: spectral data, including peaks, not caused by decay attributable to the measured source but occurring in the surrounding environment or resulting from cosmic-ray interactions in or adjacent to the detector.

Impurities: radionuclides found in the measured sources that are not the radionuclide of interest.

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	1 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

4. Equipment

The HPGe detectors are setup in accordance with the manufacturer-provided manual in combination with the methods described in the IEC 61452 standard. Additional information can be found in reference [8].

Gamma-ray measurements of radioactive sources (for activity or impurity analysis) are performed using the HPGe Gamma-Ray Spectrometry System located in Building 245, Room C015 (basement). This system consists of the following components:

- 5 closed-end coaxial HPGe detectors (known as B-detector, G-detector, N-detector, T-detector, X-detector) and 1 Si-drifted detector (known as S-detector), see Table 1 and Annex B for detectors specifications
- In addition, a Compton suppression system (known as the R-detector) composed of a HPGe detector and a NaI(Tl) detector is also available.
- Most of the detectors are shielded by lead, internally surrounded by cadmium and copper, see Table 1
- All detectors are connected to digital electronic units (that include the power supply, amplifier, and MCA)
- The digital electronics are connected to a personal computer that is connected to the NIST network. The computer clock is set to Eastern Standard Time all year around and it is periodically synchronized with the NIST atomic clock.
- Gamma-Vision-32 software package for data acquisition [2]
- Genie-2000 [3] and FitzPeaks [4] software packages for data analysis
- Calibrated ^{54}Mn , ^{56}Co , ^{57}Co , ^{60}Co , ^{85}Kr , ^{85}Sr , ^{88}Y , ^{99}Mo , ^{109}Cd , ^{113}Sn , ^{125}I , ^{125}Sb , ^{133}Ba , ^{133}Xe , ^{134}Cs , ^{137}Cs , ^{139}Ce , ^{152}Eu , ^{154}Eu , ^{155}Eu , ^{153}Gd , ^{203}Hg , ^{207}Bi , ^{210}Pb , ^{226}Ra , ^{228}Th , ^{232}U , ^{241}Am sources are used for efficiency and energy calibration. The ^{57}Co and ^{60}Co sources are also used for periodic constancy check of the energy and efficiency calibration. These sources are produced from NIST SRMs, which are validated through intercomparisons with other National Metrology Institutions and the BIPM, and the periodic submission of measured sources to the International Reference System (SIR) for gamma-ray emitting sources at the BIPM [5].

The detectors are calibrated through the development of efficiency curves for different source geometries and various source-to-detector distances. These efficiency curves are verified monthly or more frequently if needed (such as when there has been a change in the instrument configuration), using previously calibrated ^{57}Co and ^{60}Co point sources for the HPGe detectors and ^{57}Co point sources for the Si-drifted detector to determine the efficiency value and energy calibration reproducibility, see Annex E for additional information.

The efficiency curves for the HPGe detectors are developed for point sources (solids), 5 mL ampoules (liquids), Marinelli beakers, plastic vials (different volumes) and 33 cm³ spheres (gas) for gamma-ray emitting sources for the energy range 14 keV < E < 3.6 MeV. For additional information about the measurement geometries see Annex B.2.

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	2 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

The efficiency curves for the Li-drifted Si detector are developed for point sources (solids), and 5 mL ampoules (liquids).

Table 1: Details of HPGe detectors¹

	B-detector	G-detector	N-detector	T-detector	X-detector	S-detector
Detector	HPGe	HPGe	HPGe	HPGe	HPGe	Li-drifted Si
Detector type	n-type closed-end coaxial	n-type closed-end coaxial	p-type closed-end coaxial	p-type closed-end coaxial	p-type closed-end coaxial	Planar
Detector dimensions	Diameter: 54.9 mm Length: 54.2 mm	Diameter: 58 mm Length: 57.6 mm	Diameter: 70.4 mm Length: 86.4 mm	Diameter: 55 mm Length: 49 mm	Diameter: 49.7 mm Length: 22.1 mm	Diameter: 10 mm Length: 5 mm
Shielding	Pb: 10 cm Cd: 3 mm Cu: 0.635 mm	Pb: 10 cm Cd: 3 mm Cu: 0.635 mm	Pb: 4.5 cm	Pb: 10 cm Cd: 3 mm Cu: 0.635 mm	Pb: 10 cm Cd: 3 mm Cu: 0.635 mm	None
End-cap window	Beryllium	Beryllium	Aluminum	Aluminum	Carbon fiber	Beryllium
High voltage	-3.3 kV	-4.4 kV	-4.0 kV	+3.5 kV	+3.0 kV	-600 V
Amplifier model	ORTEC 927	ORTEC DSPEC-Pro	ORTEC 927	ORTEC 927	ORTEC	ORTEC DSPEC jr. 2.0
Shaping time	6 μ s	6 μ s	6 μ s	6 μ s	6 μ s	10 μ s
Energy range	30 keV – 3.6 MeV	14 keV – 1.9 MeV	30 keV – 1.9 MeV	30 keV – 1.9 MeV	14 keV – 1.9 MeV	5 keV – 200 keV
Number of channels	16384	16384	16384	16384	16384	4096
Electronics model	Lynx	ORTEC DSPEC-Pro	Lynx	Lynx	ORTEC DSPEC 502/502A	ORTEC DSPEC jr. 2.0
Electronics [†]	Digital	Digital	Digital	Digital	Digital	Digital
Source geometries*	ps, amp, gs, mb	ps, amp	ps, amp, gs	ps, amp, gs, mb	ps, amp	ps, amp
* ps: point source; amp: 5mL glass ampoule; mb: Marinelli beaker; gs: gas sphere. For additional information about the measurement geometries see Annex B.2						
[†] Settings are described in Annex D						

5. Health and Safety Precautions

Radiation Safety

Sources used for calibration purposes are of low activity. If high activity sources are measured, they are transported inside a lead pig to the edge of the detector in order to minimize exposure time when mounting the sources on the source holder. Sources not in use are kept in a separate room or are kept behind lead bricks when left inside the counting room. Calibrations are performed using Ionizing Radiation Safety Committee-approved

¹ Certain commercial equipment, instruments, or materials are identified to foster understanding. Such identification does not imply recommendation by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	3 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

protocols and non-radiological activities (e.g. moving lead pigs) are performed using management-approved procedures.

6. Procedures

Preliminary

- Customer contact: Customer must be informed of the need for sources to comply with the given specifications of geometry and activity limits.
- NIST paperwork and acceptance procedure: submit completed NIST 364 (or NIST 365), Radioactive Material Request, for approval before arrival of materials. Specifically notify the Chief of Radiation Safety Division (RSD) or designate of the arrival and departure of all isotopes of plutonium and uranium. Complete any other necessary forms if necessary (i.e, NIST 64, Order No; NIST 77, Calibration and Test Fee Computing Form; NIST 796A, Shipper's Declaration for Radioactive Materials, etc.).
- E-Commerce: Note dates of material received (and returned) in the E-commerce system

Acceptance of Sources

- Sources are shipped to NIST RSD for documentation and contamination check upon arrival. Sources are kept in custody of NIST personnel responsible for the source measurements.
- Sources to be measured by HPGe are labeled as to the source number, radionuclide, approximate activity and date (of arrival or, in the case of sources prepared at NIST, of preparation).
- NIST personnel fill out a calibration form (see Annex A) indicating the radionuclide, approximate activity, reference time, type of measurement required (e.g., activity, impurity measurement) and source type (e.g., point source, 5 mL ampoule, gas sphere, stainless steel disks or other type as requested by customer).
- Labeled sources are placed in the IN box, placed on top of the source cabinet in Room C019 Building 245. If sources are to be measured the same day they are received or prepared, they are kept temporarily behind lead bricks in C015.

Measurement Setup

- Based on activity and radionuclide(s) of interest, select the detector and source-to-detector distance.
- Source-to-detector distance should be such that the count rate is less than 2000 counts per second (less than 5 % dead time) to minimize corrections due to pileup, dead time and/or summing. In some cases, this cannot be achieved, and the source is run with a higher dead time, in this case additional corrections are applied, see Annex C.
- Install the required source holder according to source type (different geometries require different holders) and source-to-detector distance. There are several

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	4 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

different source holders to accommodate solids, liquids, gas, and other possible geometries of the source to be measured (the holders are labeled).

Measurement Sequence

Gamma-ray spectra are acquired using Genie 2000, Maestro, and GammaVision™. Genie 2000 is commercially available software developed and marketed by Mirion/Canberra and Maestro and GammaVision™ are developed and marketed by AMETEK/ORTEC (see software manuals for additional information).

- Place the source inside or on top of the correct source holder on the selected detector.
- Open GammaVision™ or Genie 2000 [2, 3].
- Choose the selected detector from the detector list.
- Clear old spectra if needed.
- Select counting time (preset the live time in seconds, or data taking can be stopped manually).
- Enter description of the source name, counting geometry and date.
- Start taking data by clicking “start.”
- Note in the logbook for the selected detector: the starting acquisition time, radionuclide, source number, source type, source-to-detector distance, live time, and filename under which the spectrum is going to be saved.
- Once the data acquisition time is completed, or the counting time manually stopped (by clicking “stop”), save the data in the directory assigned to the selected detector (note that files are saved in subdirectories that reflect the month and year in which the source was measured). The filename should include radionuclide, source number, detector name, source geometry, month, and date.
- For impurity measurements the source is run only once (additional measurements may be required).
- For activity or gammas/sec measurements, the source is run 3 times in the same geometry of a given detector and in at least 3 other geometries in different detectors.

Data Analysis

Data analysis is performed by hand (with the use of spreadsheets) or by using Genie-2000 or FitzPeaks (additional information is found in the operational manuals located in Room C015 or 300WSAE). The choice of the data analysis software packages was based on in-house comparisons of the results obtained by several [3] different software packages and by hand (using spread sheets). In addition, the data analysis software used by customers was taken into account. Such commercial products are identified in order to specify these procedures accurately.

- Copy the spectrum to be analyzed to another personal computer outside of the data acquisition room to reduce personnel radiation exposure to any sources remaining in the room.
- Copy the background to be used (generally the most recently run) in same folder where the measurement spectrum was copied.

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	5 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

- Use Genie-2000 [3] or FitzPeaks [4] software depending on type of measurement [activity, emission rates or impurity].
- Open the file of interest

Impurity Measurements

- Use Genie-2000.
- Report results in Bq.
- Load energy/shape calibration (only) for given detector located in Genie-2000 *CALFILES* directory (if necessary).
- Load efficiency calibration (only) for given detector and source-to-detector distance located in Genie-2000 *CALFILES* directory.
- Perform a peak search.
- Obtain the peak area.
- Check the fit for each peak using *interactive peak fit*.
- Subtract the background normalized to the live time.
- Run the efficiency correction.
- If a radionuclide library entry exists for the particular radionuclides in the source, run *Nuclide Identification with Interference Correction*. If such an entry does not exist for the particular radionuclides in the source, then create a library using the library editor before running the *Nuclide Identification with Interference Correction*.
- Run *Nuclide Identification* (without Interference Correction) or *Nuclide Identification with Interference Correction* using the Detection Limits library.
- Run *Detection Limits (Currie MDA)*.
- Verify that all the peaks listed are identified. If necessary, manually identify the unidentified peaks.
- Apply all corrections needed (source decay, pulse pile-up, cascade summing, sample attenuation, see Annex C)

Activity Measurements

- Use Spreadsheets (such as Microsoft Excel), Genie-2000 or FitzPeaks for these types of measurements.
- Create a spreadsheet (can use templates already created using Microsoft Excel, see Annex A for an example) that includes: list of radionuclides in the source, half-lives for each radionuclide (and associated uncertainty), gamma-ray energies (and associated uncertainty), gamma-ray emission probabilities (and associated uncertainty), efficiency for each gamma-ray energy (and associated uncertainty), live time and run time for the particular measurement, peak area (and associated uncertainty) and reference time for reporting the results. Gamma-ray decay data should be obtained from ENSDF Nuclear Data Sheets [6] or from LNE-LNHB [5] websites.
- Perform a peak search using Genie-2000 or FitzPeaks.
- Obtain the peak area using Genie-2000 or FitzPeaks.

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	6 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

- Check the fit for each peak using interactive peak fit and extract the peak area for each peak of interest using Genie-2000 or FitzPeaks.
- Use the net peak area (and associated uncertainty) to calculate the source activity using the spreadsheet, Genie-2000 or FitzPeaks.
- Subtract background from the peak area if there is a background peak at the energy of the radionuclide of interest. The background spectrum should be analyzed as the sample spectrum to obtain the net peak area for each peak of interest.
- Apply all corrections needed (source decay, pulse pile-up, cascade summing, sample attenuation, see Annex C) as described in the references [1, 8].
- Use Genie-2000 to check for impurities and detection limits as described above if requested by the customer.

The source activity is obtained from equation (1).

$$A = \frac{N(E)}{T \times \epsilon(E) \times P(E)} \prod_i C_i \quad (1)$$

where $N(E)$ is the net number of counts in the full-energy peak, T is the live time, $\epsilon(E)$ is the full-energy-peak efficiency, $P(E)$ is the gamma-ray emission probability at the energy E , A is the source activity and $\prod C_i$ is the product of the correction factors, C_i , applied to the measurement. The correction factors that are applied to these measurements may include correction due to the self-attenuation due to difference in the source density, and the decay correction of the radionuclide during the measurement time.

NOTE – On some occasions the source activity per unit mass is required. In those cases, the activity in equation (1) needs to be divided by the mass of the source and the uncertainty in the mass needs to be propagated as the other variables in equation (2) (see section 7 “Evaluation of Measurement Uncertainties”).

Reporting Results

Impurity Measurements

The Report should include (see Annex A):

- Radionuclide measured
- Source number
- Reference date and time (e.g., measurement date and time)
- Detector and source-to-detector distances used for the measurement
- File name of the spectrum analyzed
- Impurities found including activity and uncertainty
- Activity of the main radionuclide in the source and its uncertainty
- Detection limits

Activity or Emission Rate Measurements

The Report should include (see Annex A):

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	7 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

- Radionuclides measured
- Source number
- Reference date and time
- Detectors and source-to-detector distances used for the measurements
- Activity or emission rates of the main radionuclides in the source and the associated uncertainty budget
- Any gamma-ray emitting impurities (with activity and associated uncertainty budget)
- Detection limits

These reports are then used to create the Report of Test or Report of Calibration as specified in section 8.

Validation

The gamma-ray spectrometry measurements are validated by the following techniques:

- Comparison of results achieved with other validated methods.
- Evaluation of measurement uncertainty of the results based on an understanding of the theoretical principles of the method and practical experience of the performance of the method.

7. Evaluation of Measurement Uncertainties

Uncertainties on gamma-ray spectrometry measurements are determined based on the “Guidelines for Evaluation and Expressing Uncertainty of NIST Measurement Results” [7]. The Type B components are detailed below. The combined standard uncertainty of the activity (or emission rate) measurements is equal to the square root of the quadratic sum of the Type A and Type B uncertainties, with a final reported expanded uncertainty obtained by multiplying the combined standard uncertainty by a coverage factor of two, representing an interval having a level of confidence of approximately 95 %.

Type B uncertainty components:

- Decay (half-life) of the radionuclide
- Live and run times (dead time corrections)
- Peak area fit
- Background peak area variations
- Source-detector geometry
- Pulse pile-up and cascade summing (if corrections are needed)
- Variations in sample attenuation (if attenuators are used in the measurements or if measured source density is different from that of the calibration sources)
- Gamma-ray emission probability
- Full-energy-peak efficiency

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	8 of 28	Procedure23v100

The uncertainty on the impurity measurement is obtained by:

$$u_{imp/source} = \sqrt{u_{eff}^2 + u_{measure}^2}$$

where u_{eff} is the uncertainty of the efficiency for the measured gamma-ray energies for the impurity and/or source, and $u_{measure}$ is the uncertainty calculated based on the gamma-ray lines used to determine the activity of the measured impurity and/or source. Only the u_{eff} and $u_{measure}$ uncertainty components are used to estimate the expanded uncertainty, as they are the biggest contribution. As impurity levels are generally much lower than the nuclide being calibrated, the uncertainty on the impurity measurement is often on the order of 5 % to 20 % (at 1 sigma, $k = 1$). The measurement report specifies the coverage factor (i.e., k value) used to report the measurement uncertainty.

The uncertainty on the source activity (or emission rate) is obtained using uncertainty propagation, assuming that all measured quantities are independent. Therefore, when there is no correlation, the uncertainty for the source activity, A , is given by:

$$u_A = \sqrt{\left(\frac{\partial A}{\partial N}\right)^2 u_N^2 + \left(\frac{\partial A}{\partial T}\right)^2 u_T^2 + \left(\frac{\partial A}{\partial \epsilon}\right)^2 u_\epsilon^2 + \left(\frac{\partial A}{\partial P}\right)^2 u_P^2 + \sum_i \left(\frac{\partial A}{\partial C_i}\right)^2 u_{C_i}^2} \quad (2)$$

where u_N , u_T , u_ϵ , u_P , and u_{C_i} are the uncertainties associated with the quantity $\epsilon N(E)\epsilon T$, $\epsilon(E)$, $P(E)$, and C_i , respectively. The nominal uncertainty range is approximately between 1 – 3 percent (expanded uncertainties, $k = 2$). Additional information is provided in Annex C.

If there is correlation, the uncertainty is evaluated using the technique described in section 5.2 of the “Guide to the expression of uncertainty in measurement (GUM)” [13]. A tool created by the NIST Statistical Engineering Division is used to compute the correlation matrix as described in the GUM [14]. This tool can run in Excel or in R [14] and can be downloaded from the NIST website [15].

8. Report of Test or Calibration and Technical Review

The NIST Report of Test or Calibration contains information about the composition, the properties, and additional required information as stated in NIST-QM-I.

Prior to issuance, the report undergoes a final technical review. The technical review must be approved (and signed) by the principal investigator, Radioactivity Group leader, and the RPD chief.

Reports of Test or Calibration, at a minimum, should contain or address the following, unless there are valid reasons for not doing so. Additional information may be provided

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	9 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

- a title (e.g., “Report of Test”, “Calibration Report” or “Report of Special Test”).
- the first page shall be on NIST letterhead or be equivalently identified as originating from NIST.
- the name and address of the laboratory.
- the NIST Calibration Services order number, and service ID number.
- page numbers, with the last page designated as the end of the report.
- the name and address of the customer (refer to USPS Publication 28, Postal Addressing Standards).
- a description, unambiguous identification, and, when necessary, the condition of the item(s) tested or calibrated.
- the principal radionuclide
- reference time and date
- equipment used and method of calibration (traceability to primary standard); a description of the calibration method; additions to, deviations, or exclusions from the method, if applicable.
- certified values for activity with the units of measurement
- decay-scheme assumptions, if applicable
- assessment of radionuclidic purity
- overall uncertainty determinations for calibration
- the date of receipt of the test or calibration item(s), where this is critical to the validity and application of the results.
- the conditions (e.g., environmental) under which the calibrations were made that have an influence on the measurement results, if applicable
- the date(s) of performance of the laboratory activity.
- the date of issue of the report.
- a statement to the effect that the results relate only to the items tested, calibrated, or sampled; evidence or a statement that the measurement results are traceable.
- the name of the person(s) performing the measurements and analyses; the name, function, and signature of the person(s) authorizing the report.
- an explanation of the proper use and interpretation of the reported results as necessary (this can be in the form of an addendum).

9. Records

A record of all measurements performed is kept in a laboratory notebook located in Building 245 Room C015 where the detection system is located. Software manuals, and binders with the detailed dimensions of all the detectors are also kept in Room C015.

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	10 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

Electronic files of all the sources measured are kept in the personal computer in the same location. Electronic files of the data analysis, the list of the sources used for efficiency and energy calibration containing the RS# and the source numbers with activities and reference dates along with any equipment maintenance and calibrations are kept in a personal computer in building 245 room C300WSAE. Electronic files are backed up daily into an external hard drive. Calibration reports and data files will be stored in 68_PML/682/internal/682_Shared/CalData/682.04/43090S.

10. Filing and Retention

- Calibration printouts are stored in folders identified by company, radionuclide, date, and service number. If the source is returned, the date that the source is shipped back to the customer should be entered into the folder.
- For customer calibration, prepare calibration report and obtain required signatures. Make a copy for customer file and send original. Reports created and signed digitally are downloaded to the calibration record in the E-commerce system.
- Calibration Records are maintained in 245/C300-3 for at least five years. The RPD Quality Manager shall maintain the original and all past versions of this RPD Procedure.

11. References

1. IEC 61452, Nuclear Instrumentation – Measurement of gamma-ray emission rates of radionuclides – Calibration and use of Germanium spectrometers.
2. Gamma Vision: GammaVision™ (latest version), HPGe Gamma-ray spectrum analysis and MCA emulation, ORTEC.
3. Genie-2000: Genie-2000 Software Suite (latest version), Canberra Industries, Inc.
4. FitzPeaks: FitzPeaks32 Version 3.40, Gamma Analysis and Calibration software, Jim Fitzgerald.
5. Decay Data Evaluation Project (DDEP). The recommended data are available from this website hosted and updated by the Laboratoire National Henri Becquerel, <http://www.lnhb.fr/nuclear-data/>
<http://www.bipm.org/en/scientific/ionizing/radionuclides/sir/>.
6. National Nuclear Data Center (NNDC), Evaluated Nuclear Structure Data File (ENSDF), <http://www.nndc.bnl.gov/ensdf/index.jsp>.
7. Taylor, Barry N., and Kuyatt, Chris E., Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results, *National Institute of Standards and Technology Technical Note 1297* (Sep. 1994).
8. Debertin K. and Helmer R.G. Gamma- and x-ray spectrometry with semiconductor detector, Elsevier Science, North-Holland, 1988.

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	11 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

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10. GESPECOR®, Version 4.2, CID Media GmbH.
11. XCOM: Photon Cross Sections Database. NIST Standard Reference Database 8 (XGAM). M.J. Berger, J.H. Hubbell, S.M. Seltzer, J. Chang, J.S. Coursey, R. Sukumar, D.S. Zucker, and K. Olsen.
12. Eri Jay Cohen, Live Time and Pile-up Correction for Multichannel Analyzer Spectra. Nuclear Instruments and Methods 121 pp. 25-32, 1974.
13. Evaluation of measurement data — Guide to the expression of uncertainty in measurement (GUM), JCGM 100 (latest version).
14. The R Project for Statistical Computing, <http://www.r-project.org/>, (<http://cran.rstudio.com/>) and (<https://www.rstudio.com/ide/download/desktop>).
15. <http://www.nist.gov/itl/sed/gsg/metrology-for-microsoft-excel.cfm>.

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	12 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

Annex A:**A.1. Calibration form – Request for calibration**

SOURCE MEASUREMENT REQUEST FORM – HPGe SYSTEM		
RADIONUCLIDE	COMPANY	SOURCE ID #
		RS #
APPROXIMATE ACTIVITY	REFERENCE TIME	DATE RECEIVED
REQUESTER NAME:		
SOURCE TYPE		
POINT SOURCE <input type="checkbox"/> AMPOULE <input type="checkbox"/> GAS SPHERE <input type="checkbox"/> GAS AMPOULE <input type="checkbox"/>		
OTHER <input type="checkbox"/> – DESCRIBE:		
PROGRAM		MEASUREMENT QUANTITY REQUIRED
<input type="checkbox"/> PHARMACEUTICAL		<input type="checkbox"/> IMPURITY
<input type="checkbox"/> UTILITY		<input type="checkbox"/> ACTIVITY
<input type="checkbox"/> OTHER – DESCRIBE:		<input type="checkbox"/> OTHER – DESCRIBE:
DETECTOR GEOMETRY USED FOR MEASUREMENTS		
COMMENTS		RESULTS
WORK COMPLETED	SIGNATURE	DATE

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	13 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

A.2. Example of data analysis template

This example shows the data analysis performed using both software packages (Genie-2000 and Fitzpeaks). The first sheet shows the data analysis for one detector geometry.

live time	74777 sec		mass	0.105858 grams							
run time	75900 sec										
start	11/25/13 11:53 AM EST										
T1/2 Sr-82	25.35 days +/-	0.03 days	T1/2 Sr-85	64.85 days +/-	0.007 days						
decay factor	0.988061763 Unc +/-	2.8426E-05	decay factor	0.99531624 Unc +/-	1.0135E-06						
Sr-82 (keV)	error energy	emis prob	err em prob	efficiency	err eff	err eff %	err emprob %				
776.52	0.003	0.1508	0.0016	5.18E-05	4.74E-08	0.092	1.061				
1395.14	0.03	0.00529	0.00007	3.44E-05	5.02E-08	0.146	1.323				
Sr-85 (keV)	error energy	emis prob	err em prob	efficiency	err eff	err eff %	err emprob %				
514.0048	0.0022	0.985	0.004	7.10E-05	1.31E-07	0.184	0.406				
Reference Time =	10/31/13 12:00 PM EST		DF Sr-82=	1.98068785	DF Sr-85=	1.30625					
	Time to reference time =		-24.99514 days								
Genie2k											
Sr-82 (keV)	area	err A	count rate	err c rate	DF corr	Activity	error act	act/g	error act/g	Act ref T	err Act ref T
776.52	2.1971E+05	5.0533E+02	2.9382E+00	6.7578E-03	2.9737E+00	3.8093E+05	4.1502E+03	3.5985E+06	3.9206E+04	7.5451E+05	8.2203E+03
1395.14	4.9500E+03	1.0445E+02	6.6197E-02	1.3968E-03	6.6997E-02	3.6778E+05	9.1757E+03	3.4743E+06	8.6679E+04	7.2847E+05	1.8174E+04
Sr-85 (keV)	area	err A	count rate	err c rate	DF corr	Activity	error act	act/g	error act/g	Act ref T	err Act ref T
514.0048	3.1397E+06	1.5699E+03	4.1988E+01	2.0994E-02	4.2185E+01	6.03E+05	8.04E+03	5.70E+06	7.59E+04	7.8802E+05	1.0498E+04
Fitzpeaks											
Sr-82 (keV)	area	err A	count rate	err c rate	DF corr	Activity	error act	act/g	error act/g	Act ref T	err Act ref T
776.52	2.1990E+05	8.7962E+02	2.9407973	0.011763189	2.97632943	3.81E+05	4.34E+03	3.60E+06	4.10E+04	7.5517E+05	8.5908E+03
1395.14	5.0260E+03	1.7088E+02	0.06721318	0.002285248	0.06802528	3.73E+05	1.36E+04	3.53E+06	1.29E+05	7.3965E+05	2.7007E+04
Sr-85 (keV)	area	err A	count rate	err c rate	DF corr	Activity	error act	act/g	error act/g	Act ref T	err Act ref T
514.0048	2.6459E+06	2.3813E+04	35.3835136	0.318451623	35.5500213	5.08E+05	5.11E+03	4.80E+06	4.82E+04	6.6408E+05	6.6701E+03

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	14 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

A.3. Impurity measurement results form – Summary of impurity measurementsReport of MeasurementRadionuclide: (radionuclide name)Source Number: (source number)Measurement Geometry: (detector, distance, geometry)Live Time: xxxxx secondsReal Time: yyyyy secondsStart Time: xx/yy/zzzz xx:xx:xx EST (Date and Time)Impurities Observed:

(list of impurities, radionuclides, activities and uncertainties)

Approximate source Activity:

(source, radionuclide, activity and uncertainty)

All uncertainties are k=1

Detection Limits:

keV < E < keV	xx	gammas/s
keV < E < keV	yy	gammas/s
keV < E < keV	xx	gammas/s
keV < E < keV	yy	gammas/s

Report Date/Year

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	15 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

A.4. Activity measurement results

Activity measurements results are normally reported in a excel file. The information contained in this file depends on the corrections performed for the measurements and the required information requested by the person/manufacturer of the source. An example is shown here.

Radionuclide	Energy (keV)	Ampoule Activity (Bq/g)	Std dev (%)	Geom (%)	Decay factor (%)	Peak Fit (%)	Eff (%)	Unc distance (%)	pile-up corr unc (%)	Emis Prob (%)	Comb (%)	Expand (%) k=2
Bi-211	351.03	7.481E+05	0.074	0.320	0.050	0.1	0.46	0.345	0.282	1.13	1.348	2.695
Pb-211	404.834	7.497E+05	0.186	0.101	0.050	0.2	0.50	0.345	0.282	1.23	1.437	2.873
	427.15	7.604E+05	0.309	0.341	0.050	0.2	0.54	0.345	0.282	1.22	1.495	2.989
	704.675	7.680E+05	0.450	0.558	0.050	0.5	0.41	0.345	0.282	1.21	1.616	3.231
	766.68	7.581E+05	0.237	0.403	0.050	0.4	0.42	0.345	0.282	1.30	1.568	3.137
	831.984	7.614E+05	0.274	0.383	0.050	0.2	0.37	0.345	0.282	1.16	1.395	2.791
Rn-219	271.228	7.454E+05	0.085	0.338	0.050	0.1	0.75	0.345	0.282	1.29	1.599	3.198
	401.81	7.514E+05	0.110	0.299	0.050	0.2	0.35	0.345	0.282	1.36	1.524	3.048
Ra-223	122.319	7.519E+05	0.253	0.514	0.050	0.5	0.40	0.345	0.282	1.45	1.743	3.486
	144.27	7.613E+05	0.031	0.368	0.050	0.4	0.50	0.345	0.282	1.43	1.672	3.345
	154.208	7.618E+05	0.176	0.314	0.050	0.2	0.76	0.345	0.282	1.49	1.779	3.559
	269.463	7.469E+05	0.051	0.330	0.050	0.1	0.69	0.345	0.282	1.19	1.491	2.981
	323.871	7.442E+05	0.135	0.261	0.050	0.2	0.45	0.345	0.282	1.36	1.539	3.077
	338.282	7.489E+05	0.182	0.257	0.050	0.2	0.48	0.345	0.282	1.53	1.705	3.409
	445.033	7.523E+05	0.171	0.349	0.050	0.2	0.45	0.345	0.282	1.31	1.518	3.036

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	16 of 28	Procedure23v100

Annex B: Information about detectors and sources

B.1. List of parameters for HPGe detectors

The list of detailed dimensions for each of the HPGe detectors is kept in binders stored in building 245 Room C015

B.2. Information about measurement source geometries

Below is a picture showing the different types of sources measured in this system, see Figure 7. The HPGe detectors are calibrated to measure point sources, 5 mL ampoules, disks, Marinelli beakers, gas spheres and plastic vials. For all other measurement geometries corrections to the measured efficiency curves need to be made to account for the geometrical differences. Detailed information about the dimension of the 5 mL ampoule is shown in Figure 8. Table 2 summarizes all the source-to-detector distances for the different measurement geometries.



Figure 1: Picture showing the different source types measured in the HPGe detectors

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	17 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

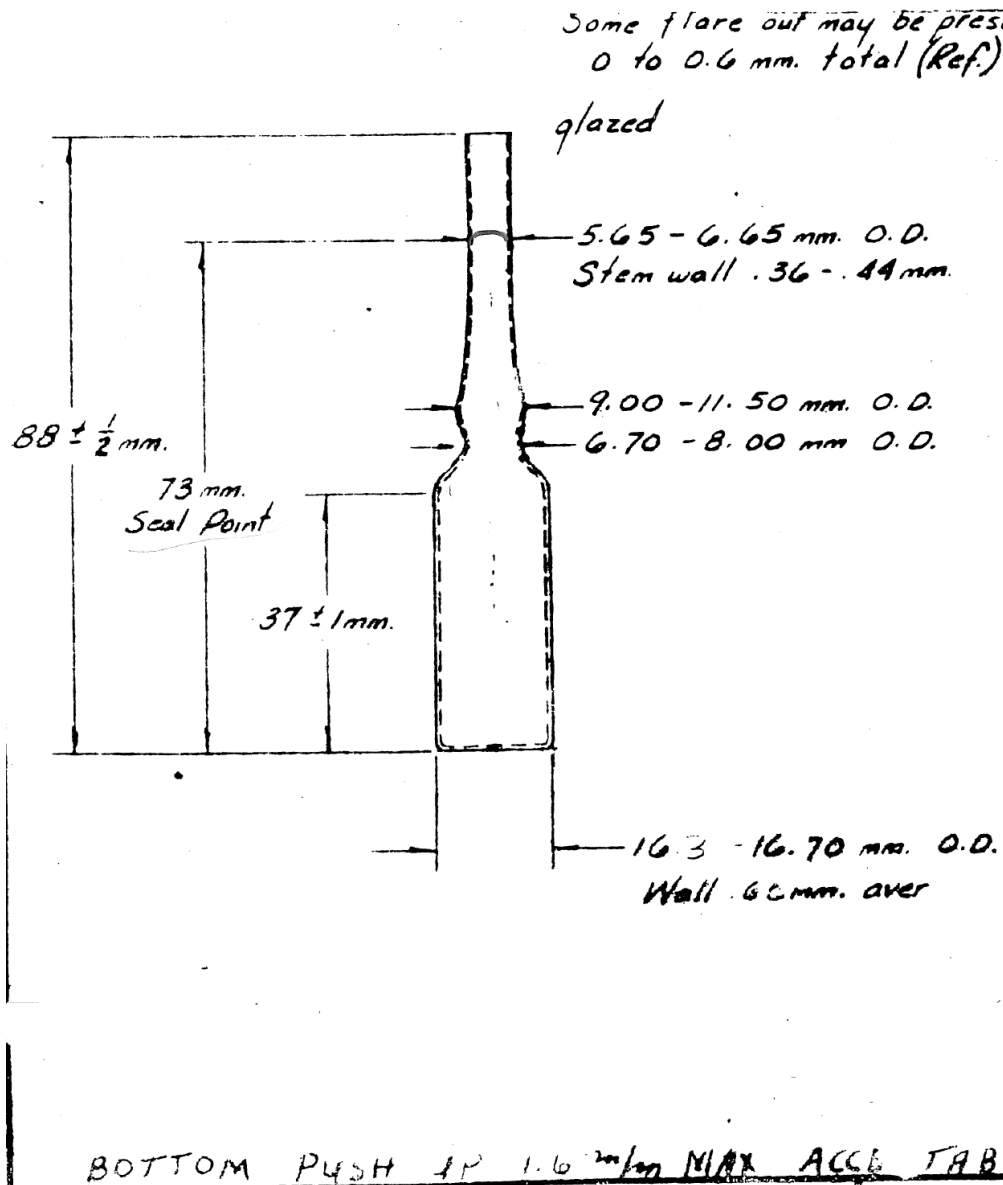


Figure 2: Dimensions of 5 mL ampoules

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	18 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

Table 2: List of measurement source-to-detector distances

Geometry name	Point source	5 mL ampoule	Steel disks	Gas sphere	Marinelli beaker
B-detector source-to-detector distances (cm)					
1 m	90.04	88.78	90.04		
35 cm	36.24	34.98	36.24	40.49	
25 cm	26.14	24.88	26.14		
MB					0.36
X-detector source-to-detector distances (cm)					
40cm	38.23	37.30	38.23		
25cm	25.08	24.15	25.08	29.34	
10cm	10.05	9.12	10.05		
5cm	5.00	4.07	5.00		
T-detector source-to-detector distances (cm)					
90cm	82.31	81.12	82.31		
45cm	42.31	41.12	42.31		
35cm	32.46	31.27	32.46		
25cm	22.62	21.43	22.62		
MB					0.32
G-detector source-to-detector distances (cm)					
90cm	90.89	89.70			
40cm	40.89	39.70			
20cm	20.89	19.70			
10cm	10.64	9.70			
N-detector source-to-detector distances (cm)					
1.5 m	150	150			
50 cm	50				
40 cm		41.48		32.59	
S-detector source-to-detector distances (cm)					
5 cm	5.00	5.00			

Annex C: Detailed calculations for activity measurements

The source activity is obtained from equation (1).

$$A = \frac{N(E)}{T_l \times \epsilon(E) \times P(E)} \prod_i C_i \quad (\text{C.1})$$

where $N(E)$ is the number of counts in the full-energy peak (or net peak area), T_l is the live time, $\epsilon(E)$ is the full-energy-peak efficiency, $P(E)$ is the gamma-ray emission probability at the energy E , A is the source activity and $\prod C_i$ is the product of the correction factors, C_i , applied to the measurements. The correction factors that are applied to these measurements may include correction due to the self-attenuation, to difference in the source density, and the decay correction of the radionuclide during the measurement time. The uncertainty on the source activity is obtained using uncertainty propagation, assuming that all measured quantities are independent. The uncertainty for the source activity, A , is given by

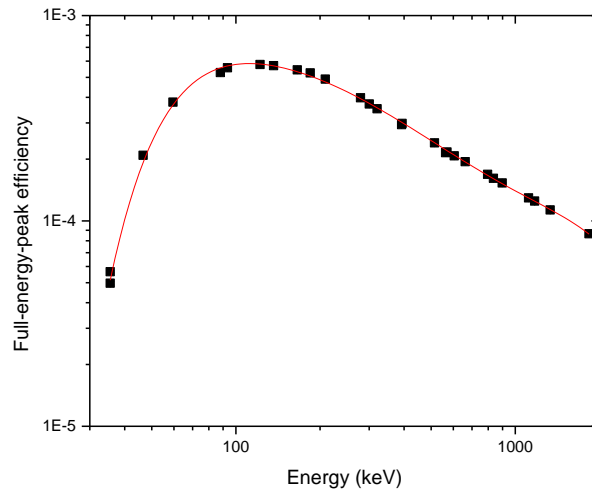
$$u_A = \sqrt{\left(\frac{\partial A}{\partial N}\right)^2 u_N^2 + \left(\frac{\partial A}{\partial T_l}\right)^2 u_{T_l}^2 + \left(\frac{\partial A}{\partial \epsilon}\right)^2 u_{\epsilon}^2 + \left(\frac{\partial A}{\partial P}\right)^2 u_P^2 + \left(\frac{\partial A}{\partial C_i}\right)^2 u_{C_i}^2} \quad (\text{C.2})$$

where u_N , u_{T_l} , $u_{\epsilon(E)}$, u_P , and u_{C_i} are the uncertainties associated with the quantities $N(E)$, T_l , $\epsilon(E)$, $P(E)$, and C_i , respectively [5, 7, 8].

The detectors are calibrated using the full-energy-peak efficiency, $\epsilon(E)$. Several full-energy-peak efficiency curves are obtained for different source geometries and various source-to-detector distances. The full-energy-peak efficiency curves for the high purity germanium (HPGe) detectors are developed using different types of gamma-ray emitting sources (e.g., point sources, 5 mL liquid ampoules, Marinelli beakers, plastic vials, 33 cm³ gas spheres) for energies ranging from 14 keV to 3.6 MeV. In most cases the full-energy-peak efficiency to be used in the activity calculation comes from a fit to the measured efficiencies, in this case should be taken to the function used to fit this curve. Some of the most commonly used fitting functions are a polynomial or a spline, an example using a polynomial fit is shown in the figure below (error bars are smaller than the size of the symbols).

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	20 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

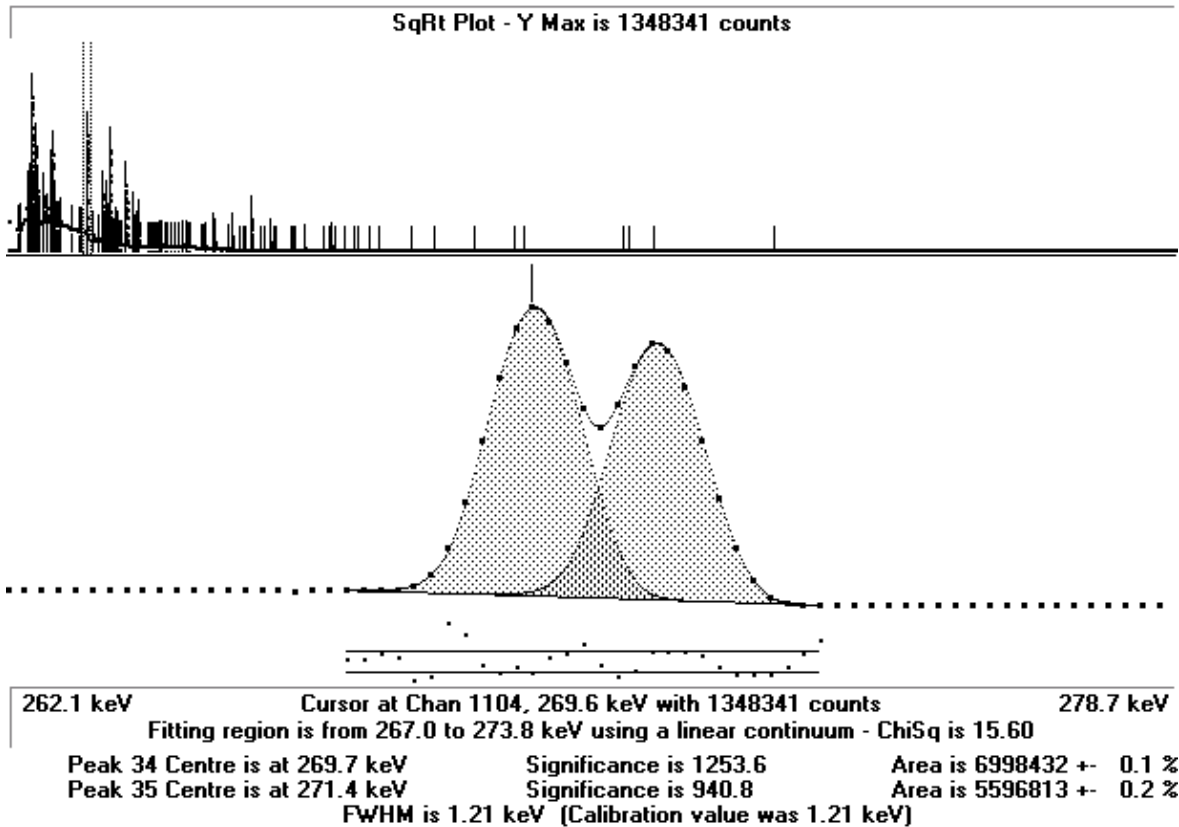
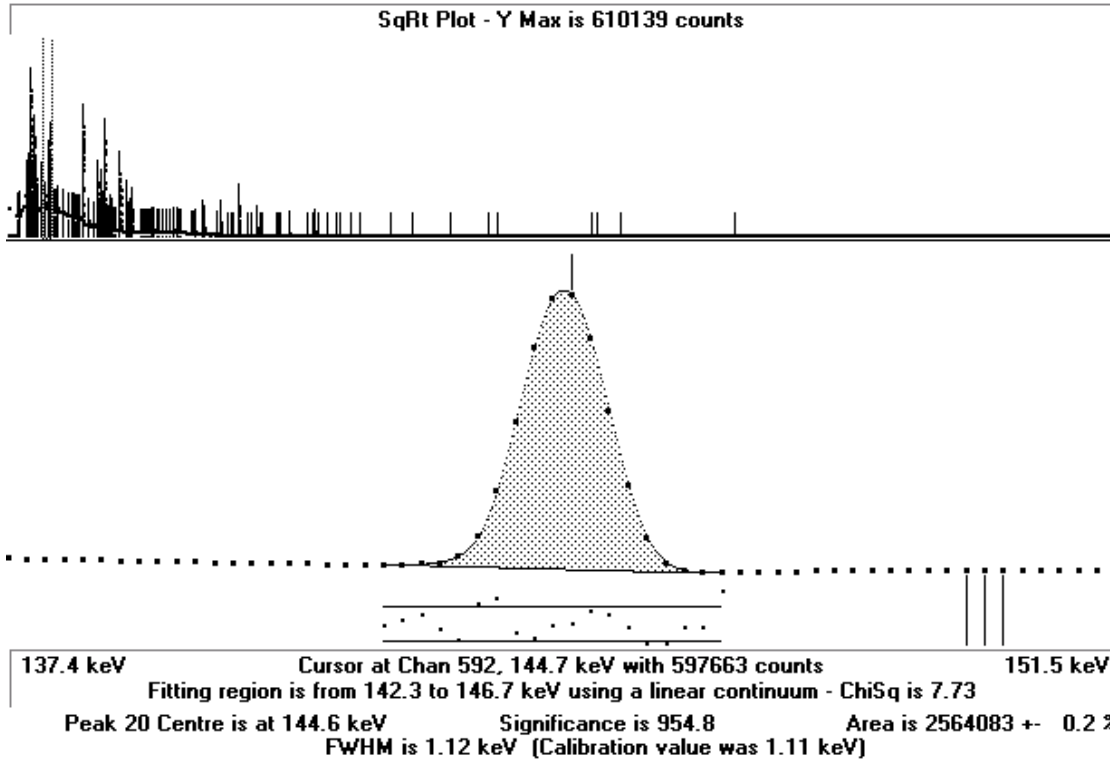


For better accuracy in the measurements, it is recommended to do the measurements using the same measurement geometry (i.e., source type, source-to-detector distance) used to develop the full-energy-peak efficiency curves. In the case that the source type or the source-to-detector distance used for the measurements is different than the one used to the efficiency calibration, corrections should be made to account for the changes in the source measurement geometry.

The determination of the net peak area is normally performed by fitting the gamma-ray peak with a Gaussian function with additional low-energy and high-energy tails. When performing this fit, care should be taken to select the function used to subtract the Compton background; in general, a linear or a step function is used (depending on the spectral shape). There are several commercially available programs that can be used to perform this fit (e.g., Genie 2000, GammaVision™, Maestro, Fitzpeak), examples are shown below.

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	21 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES



Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	22 of 28	Procedure23v100

C.1 Possible corrections

The most common corrections used for different gamma-ray spectrometry measurements using HPGe detectors are listed below. Additional corrections may be needed depending on the type of measurements performed [8].

C.1.1. Decay correction during measurement

If we have to determine the source activity for radionuclides with half-lives that are short compared to the measurement time, we need to account for the decay during the counting interval. It is recommended to correct at the midpoint of the counting interval, so the correction factor is

$$\frac{1}{c_d} = K_d = \frac{T_{1/2}}{\ln(2)t_r} \left(1 - e^{-\ln(2) t_r/T_{1/2}}\right) \quad (\text{C.3})$$

where $T_{1/2}$ is the half-life, and t_r is the time interval over which the source decays, corresponding to the run (or real) time of the measurement.

The associated uncertainty is given by:

$$u_{K_d} = \frac{1}{\ln(2)t_r} \left(1 - e^{-\ln(2)t_r/T_{1/2}}\right) - \frac{1}{T_{1/2}} e^{-\ln(2)t_r/T_{1/2}} \quad (\text{C.4})$$

The uncertainty in the measurement time t is considered to be equal to zero as the acquisition system provides a measurement time value to the milli-second compared to source acquisition times used of approximately 6 to 24 hours.

C.1.2. Decay factor correction to a reference time

If several measurements for a given source are performed at different times and the results need to be combined and reported at a given reference time then the activity needs to be corrected, and the correction factor is

$$C_{df} = e^{-\lambda t} \quad (\text{C.5})$$

where λ is the decay constant $\ln(2)/T_{1/2}$, $T_{1/2}$ is the half-life, and t is the time interval over which the source decays between the reference time and the measurement time, see [8].

The associated uncertainty is given by:

$$u_{C_{df}} = \frac{\ln(2) t}{T_{1/2}^2} e^{-\ln(2)t/T_{1/2}} \quad (\text{C.6})$$

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	23 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

The uncertainty in the measurement time t is considered to be equal to zero as the acquisition system provides a measurement time value to the milli-second compared to source acquisition times used of approximately 6 to 24 hours.

C.1.3. Pile-up correction

If the count rate in the detector is high, pile-up may occur due to dead-time. To correct for this effect it is necessary to determine the loss in photopeak rate as a function of increasing total count rate in the detector. This needs to be verified for different gamma-ray energies. There are two techniques that can be used to perform this correction [8].

- a. This can be accomplished by measurements with a set of sources containing known relative amounts of different radionuclides emitting photons with energies in the region of interest. Such a set of sources contains: ^{57}Co , ^{133}Ba , ^{54}Mn and ^{65}Zn with activity ratios of approximately 1:3:6:10:15. If no pile-up losses occur the ratios of the measured peak area N_i ($i=1,2,\dots$) and the activity A_i should be independent of the total count rate, provided that the same analyzer live-time interval T_l is chosen each time. For each energy, the ratios $N_i/(A_i T_l)$ can be fitted by a linear function of the total count rate n_t . This rate is defined as the total number of counts stored in the entire spectrum, N_t , divided by the live-time; that is $n_t = N_t / T_l$.
- b. This can also be accomplished by using two sources whose activities need not be known. One source needs to be weak with one photon energy E_1 (e.g., ^{137}Cs) and it needs to be kept at a fixed position. The second source needs to be stronger and have gamma-rays with energies lower than E_1 (e.g., ^{57}Co), and this source is moved at different distances from the detector in order to change the total measured count rate. The count rate of the peak corresponding to the energy E_1 is plotted as a function of $n_t = N_t / T_l$. The data are fitted by a straight line. The correction $C_p = 1/e^{-\tau N_t}$, $e^{-\tau N_t} \approx 1 - \tau N_t$ where τ is the slope of the linear fit [12].

C.1.4. Coincidence summing correction

The correction depends on the decay scheme of the radionuclide under measurement [8]. There are several ways to compute the magnitude of this correction. There are several software packages that can perform these calculations (e.g., ETNA, GESPECOR). These corrections are not needed if the measured radionuclides are the same as the calibration radionuclides and if the source-to-detector distance and source geometry are kept the same for the calibration and the source measurements. These corrections are a function of the total and full-energy-peak efficiencies; the general functional form is $C_s = \frac{1}{1 \pm \epsilon_i}$ (the exact formula depends on the number of gamma-rays with different energies that are emitted within the resolving time of the spectrometer). If the efficiency values are of the order of 10^{-3} then the correction will be smaller than 0.1 %. These efficiency values can be achieved by measuring at a faraway distance or by using small detectors.

C.1.5. Source geometry

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	24 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

If the source geometry used for the measurements is the same as the one used for the HPGe detector calibration, then no correction is needed. These corrections take into account the differences in solid angle between the source and the detector as well as the differences in the source-to-detector distance, attenuation through air, self-attenuation in the source and attenuation through any changes in possible shielding surrounding the source [9]. There are several software packages that can perform these calculations (e.g., ETNA, GESPECOR [9, 10]).

The attenuation through material (i.e., air, self-attenuation, shielding) can be corrected using the mass attenuation coefficients [8] as follows:

$$C_{Att} = \frac{1}{e^{-[(\mu/\rho)x\rho]}} \quad (C.7)$$

where μ/ρ is the mass attenuation coefficient (in cm^2/g), x is the material thickness (in cm) and ρ is the material density (in g/cm^3). Mass attenuation coefficients and material densities can be obtained from XCOM [11].

For additional information about the different corrections see reference [8].

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	25 of 28	Procedure23v100

Annex D: Electronics settings

D.1. N-detector electronics

The Lynx electronics from Mirion/Canberra are used for the N-detector. The instrument settings are adjusted to optimize the detector performance. The energy calibration is performed after the electronics setup and regularly when found out of range. The Genie 2000 software is used for the data acquisition for this detector.

D.2. X-detector electronics

The ORTEC DSPEC 502/502A Digital Signal Processing Gamma Spectrometers digital electronics are connected to the X-detector. The electronics are adjusted to optimize the detector performance. The energy calibration is performed after the electronics setup and regularly when found out of range. The GammaVision software is used for the data acquisition for this detector.

D.3. T-detector electronics

The Lynx electronics from Mirion/Canberra are used for the T-detector. The instrument settings are adjusted to optimize the detector performance. The energy calibration is performed after the electronics setup and regularly when found out of range. The Genie 2000 software is used for the data acquisition for this detector.

D.4. B-detector electronics

The Lynx electronics from Mirion/Canberra are used for the B-detector. The instrument settings are adjusted to optimize the detector performance. The energy calibration is performed after the electronics setup and regularly when found out of range. The Genie 2000 software is used for the data acquisition for this detector.

D.5. G-detector electronics

The ORTEC digital electronics DSPEC-Pro are connected to the G-detector. The electronics are adjusted to optimize the detector performance. The energy calibration is performed after the electronics setup and regularly when found out of range. The GammaVision software is used for the data acquisition for this detector.

D.6. S-detector electronics

The ORTEC digital electronics DSPEC jr. 2.0 are connected to the S-detector. The electronics are adjusted to optimize the detector performance. The energy calibration is performed after the electronics setup and regularly when found out of range. The GammaVision™ or Maestro software is used for the data acquisition for this detector.

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	26 of 28	Procedure23v100

Annex E: System checks

A set of ^{57}Co and ^{60}Co point sources are used to check the system to ensure that the system performance is not changing over time. These measurements are performed monthly and when there is a change to the system (e.g., electronics, detector).

The full-energy peak (FEP) efficiency, the full-width-half maximum (FWHM) and the energy calibration is measured and recorded during this system check. If differences from the initial calibration measurements are observed different actions are taken depending on the observed difference. For example:

- 1) If there is a difference in the energy for the measured gamma-ray peaks, then a new energy calibration is performed.
- 2) If there is a change in the FWHM or FEP efficiency this is a sign that either the electronics or detector are not working properly, so the reason for the change is determined and fixed depending on what is found. When this occurs either the electronics or detector usually needs to be sent for repair.

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	27 of 28	Procedure23v100

GAMMA-EMITTING RADIONUCLIDES

Annex F: Procedure revisions**Procedure for gamma-ray spectrometry measurements for activity calibration and impurity measurements using High Purity Germanium (HPGe) detectors**

Document version number	Date revised	Filename	Reviewed by	Approved by
0.00	June 2004	ProcedureGammaSpecV0.00	Leticia Pibida	Leticia Pibida
1.00	May 2014	ProcedureGammaSpecV1.00	Leticia Pibida	Leticia Pibida
2.00	June 2015	ProcedureGammaSpecV2.00	Leticia Pibida	Leticia Pibida
3.00	March 2020	ProcedureGammaSpecV3.00	Leticia Pibida	Leticia Pibida
1.00	February 2024	Procedure 23 v1.00	Leticia Pibida	Alan Thompson

Version	Date	Author	Approval	Pages	Filename
1.00	1/19/24	LP	AKT	28 of 28	Procedure23v100