

## 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 measuring gamma-ray-emitting impurities, as required for corrections to other radionuclide calibration techniques.

### 2. Scope

The HPGe detectors are used to measure activity (in Bq), emission rates (in gamma/sec), and impurity levels for gamma-ray-emitting radionuclides. Measurements include source calibrations for different organizations, including nuclear power plants, radiopharmaceutical companies, universities, and national laboratories. This procedure may be used in other applicable procedures, such as RPD-P-1, RPD-P-15, RPD-P-16, and RPD-P-22.

This calibration service is relevant for radionuclides that emit gamma-rays with energies between 35 keV and 3.5 MeV and activities ranging from 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 Standard Reference Materials (SRMs) and NIST calibrated sources.

### 3. Definitions

Most terms used in this procedure are listed in IEC 61452 [1], so 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.

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## 4. Equipment

The HPGe detectors are set up in accordance with the manufacturer-provided manual in combination with the methods described in the IEC 61452 standard [1]. Additional information can be found in reference [9].

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, C1-detector, T-detector, X-detector) and one Li-drifted Si 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 and internally surrounded by cadmium and copper; see Table 1.
- All detectors are connected to digital electronic units (that include the power supply, amplifier, and multi-channel analyzer (MCA)).
- The digital electronics are connected to a personal computer that is connected to the NIST network. The computer clock is always set to Eastern Standard Time and periodically synchronized with the NIST atomic clock.
- The Genie 2000 and GammaVision-32 software packages are used for data acquisition [2, 3].
- Genie 2000 [3] and FitzPeaks [4] software packages are used for data analysis.
- Calibrated sources are used for efficiency calibration, these sources include some of the following radionuclides:  $^{54}\text{Mn}$ ,  $^{56}\text{Co}$ ,  $^{57}\text{Co}$ ,  $^{60}\text{Co}$ ,  $^{85}\text{Kr}$ ,  $^{85}\text{Sr}$ ,  $^{88}\text{Y}$ ,  $^{99}\text{Mo}$ ,  $^{109}\text{Cd}$ ,  $^{113}\text{Sn}$ ,  $^{125}\text{I}$ ,  $^{125}\text{Sb}$ ,  $^{133}\text{Ba}$ ,  $^{133}\text{Xe}$ ,  $^{134}\text{Cs}$ ,  $^{137}\text{Cs}$ ,  $^{139}\text{Ce}$ ,  $^{152}\text{Eu}$ ,  $^{154}\text{Eu}$ ,  $^{155}\text{Eu}$ ,  $^{153}\text{Gd}$ ,  $^{203}\text{Hg}$ ,  $^{207}\text{Bi}$ ,  $^{210}\text{Pb}$ ,  $^{226}\text{Ra}$ ,  $^{228}\text{Th}$ ,  $^{232}\text{U}$ ,  $^{241}\text{Am}$ . Which sources are used depends on the source geometry and available sources. The  $^{57}\text{Co}$  and  $^{60}\text{Co}$  sources are used for periodic constancy check of the energy and efficiency calibration, in addition to the peak shape. These sources are produced and calibrated at NIST or produced from NIST SRMs, which are validated through intercomparisons with other National Metrology Institutes and the BIPM, and the periodic submission of measured sources to the International Reference System (SIR) for gamma-ray emitting sources at the BIPM [7].

The detectors are calibrated by developing efficiency curves for various source geometries and 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  $^{57}\text{Co}$  and  $^{60}\text{Co}$  point sources for the HPGe detectors and  $^{57}\text{Co}$  point sources for the Li-drifted Si detector to determine the efficiency value and energy calibration reproducibility, as well as the peak shape; see Annex E for additional information.

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The efficiency curves for the HPGe detectors are developed mainly for point sources (solids) and 5 mL NIST ampoules (liquids) for gamma-ray-emitting sources in the energy range  $14 \text{ keV} < E < 3.6 \text{ MeV}$ . For additional information about the measurement geometries, see Annex B.2.

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 detectors and the Li-drifted Si detector<sup>1</sup>

	B-detector	G-detector	C1-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 (i.e., crystal dimensions)	Diameter: 54.9 mm Length: 54.2 mm	Diameter: 58 mm Length: 57.6 mm	Diameter: 62.0 mm Length: 40.0 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	+3.3 kV	+3.5 kV	+3.0 kV	-600 V
Energy range	30 keV – 3.6 MeV	14 keV – 2.6 MeV	30 keV – 2.6 MeV	30 keV – 2.6 MeV	14 keV – 2.6 MeV	5 keV – 200 keV
Electronics model <sup>†</sup>	Mirion Lynx	ORTEC DSPEC-Pro	Mirion Lynx	Mirion Lynx	ORTEC DSPEC 502/502A	ORTEC DSPEC jr. 2.0
Source geometries*	ps, amp	ps, amp	ps, amp	ps, amp	ps, amp	ps, amp
* ps: point source; amp: 5mL glass ampoule. For additional information about the measurement geometries, see Annex B.2						
<sup>†</sup> Settings are described in Annex D						

## 5. Health and Safety Precautions

### Radiation Safety

Sources used for calibration purposes are of low activity ( $\leq 1 \text{ MBq}$ ). If high-activity sources ( $> 1 \text{ MBq}$ ) are measured, they are transported inside a lead pig to the edge of the detector to minimize exposure time when mounting the sources on the source holder. Sources not in use are kept in a separate room (i.e., Building 245, Room C019) or are kept inside a container behind lead bricks when left in the counting room. Calibrations are performed using Ionizing Radiation Safety Committee-approved protocols, and non-radiological activities (e.g., moving lead pigs) are performed using management-approved procedures.

<sup>1</sup> 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.

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## 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 (with attention to the source custodian) for documentation and contamination check upon arrival. One of the NIST personnel responsible for the source measurements serves as the source custodian.
- Sources to be measured by HPGe are labeled with 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), source type (e.g., point source, 5 mL ampoule, gas sphere, stainless steel disks, or other type as requested by customer), source mass, and chemical composition.
- Labeled sources are placed in the IN box 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 these cases, 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 different source holders to accommodate solids, liquids, gases, and other possible geometries of the source to be measured (the holders are labeled).

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*Measurement Sequence*

Gamma-ray spectra are acquired using Genie 2000 and GammaVision™. Genie 2000 is commercially available software developed and marketed by Mirion/Canberra, and GammaVision™ is 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.
- Save and clear old spectra if needed.
- Select counting time (preset the live time in seconds, or data acquisition can be stopped manually).
- Enter the description of the source measurement that includes the radionuclide name, source name/number, detector name, counting geometry, and date.
- Start taking data by clicking “start” if the measurement is manually acquired. If a measurement sequence is performed as a batch (so several spectra can be automatically acquired without moving the source), the setup is as follows for the different types of data acquisition systems:
  - For Genie 2000, go to the “Edit” tab and select “Analysis sequence”. Click on “Acquisition” and “Save Datasource” to add them to the sequence as many times as the number of runs that are needed. Click on the first “Acquisition” instance and change the live time to the required time (this time will be the same for all runs). Click on “Sequence preferences” and make sure that all selection buttons are unchecked. Click on “Execute.” The program will prompt the user to enter the filename under which each spectrum is going to be saved; check that the correct folder where the files are going to be saved is selected. The measurements will start.
  - For GammaVision™, a “\*.job” file is needed to run a batch job. The file is created or edited from a previous file; see Annex F for additional information and an example. After creating the file click on the “Service” tab and select “Job control” select the file that was created, verify that the file has the correct parameters; check that the SET\_DETECTOR has the correct detector number, check that the SET\_PRESET\_LIVE is the desired measurement time in seconds, check that the LOOP is set to the number of runs needed, check that the DESCRIBE\_SAMPLE has the desired measurement description for the source being measured, check that the SAVE has the correct destination folder name and filename under which the files are going to be saved, if changes are made save and close the file, if no changes were made click open and the measurements will start. If changes

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were made, click on the “\*.job” file that was saved, click open, and the measurements will start.

- If the R-detector is used to perform a measurement, see Annex B.3 for information on how to start/stop a measurement and save a spectrum file.
- Note in the logbook for the selected detector: the starting acquisition time, radionuclide, source number, source type, source-to-detector distance, live time, and the filename under which the spectrum will be saved.
- Once the data acquisition time is completed, or the counting time is 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 usually measured only once (additional measurements may be required).
- For activity or emission rate (gammas/sec) measurements, the source is measured 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 (using spreadsheets) or automatically using Genie 2000 or FitzPeaks (additional information is available in the operational manuals located in Room C015 or 300WSAE). The choice of data analysis software packages is based on in-house comparisons of results obtained by several [3] software packages and by hand calculations (using spreadsheets). The data analysis is carried out following the following steps:

- Copy the spectrum to be analyzed to another personal computer outside of the data acquisition room to reduce personnel radiation exposure from any sources remaining in the room.
- Copy the background to be used (generally the most recently run) into the same folder where the measurement spectrum was copied.
- Use Genie 2000 [3] or FitzPeaks [4] software depending on the type of measurement [activity, emission rates, or impurity].
- Open the file of interest.

### **Impurity Measurements**

- Use Genie 2000.
- Report results in Bq.

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- Load energy/shape calibration (only) for the given detector located in the Genie 2000 *CALFILES* directory (if necessary).
- Load efficiency calibration (only) for the given detector and source-to-detector distance located in the 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 radionuclides in the source, run *Nuclide Identification with Interference Correction*. If such an entry does not exist for the radionuclides in the source, then create a library using the library editor before running the *Nuclide Identification with Interference Correction*. If MDAs are needed, run *Detection Limits (Currie MDA)*.
- 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.2 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 intensities (and associated uncertainty), efficiency for each gamma-ray energy (and associated uncertainty), live time and run time for the particular measurement, net 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.

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- 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 the background from the net 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, 9].
- 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 intensity 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 include the correction due to self-attenuation, due to the difference in the source density (if applicable), the decay correction to the reference time, and the decay correction of the radionuclide during the measurement time (additional corrections may be needed, see Annex C).

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 – Internally within NIST***

#### 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 (the filename of the report is the same as the filename of the spectrum).

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- Impurities found, including their activities and uncertainties.
- Activity of the main radionuclide in the source and its uncertainty.
- Detection limits and minimum detectable activity (MDA), if applicable.

Activity or Emission Rate Measurements

The Report should include (see Annex A):

- 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 (each with activity and associated uncertainty budget).
- Detection limits and minimum detectable activity (MDA), if applicable.

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 in 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” [8]. 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.

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- 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 intensity.
- Full-energy-peak efficiency.

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 main 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 must specify 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 between uncertainties, 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  $u_N$ ,  $u_T$ ,  $u_\epsilon$ ,  $u_P$ , and  $u_{C_i}$  are the uncertainties associated with the quantity  $N(E)$ ,  $T$ ,  $\epsilon(E)$ ,  $P(E)$ , 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.

The uncertainty is evaluated using the technique described in section 5.2 of the “Guide to the expression of uncertainty in measurement (GUM)” [14]. A tool created by the NIST Statistical Engineering Division is used to compute the correlation matrix as described in the GUM [15]. This tool can run in Excel or in R [15] and can be downloaded from the NIST website [16].

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## 8. Report of Test or Calibration and Technical Review

The NIST Report of Test or Calibration contains information about the chemical composition, the physical 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.

- 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,

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- 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. Electronic files containing data for 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 on a personal computer in building 245, room C300WSAE. Electronic files are backed up daily into an external hard drive. Calibration reports and data files, upon completion, will be stored on the central network storage system “elwood” 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 it is shipped back to the customer should be entered in the folder.
- For customer calibration, prepare a calibration report and obtain the required signatures. Make a copy for the customer file and send the 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 activity or emission rate of gamma-ray emitting radionuclides – Calibration and use of germanium-based spectrometers.

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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/>
6. National Nuclear Data Center (NNDC), Evaluated Nuclear Structure Data File (ENSDF), <http://www.nndc.bnl.gov/ensdf/index.jsp>.
7. International Reference System, <https://www.bipm.org/en/radionuclide-metrology-sir>
8. 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).
9. Debertin K. and Helmer R.G. Gamma- and x-ray spectrometry with semiconductor detector, Elsevier Science, North-Holland, 1988.
10. ETNA, Efficiency Transfer Nuclide Activity Measurement, Laboratoire National Henri Becquerel, 2004.
11. GESPECOR®, Version 4.2, CID Media GmbH.
12. 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.
13. Eri Jay Cohen, Live Time and Pile-up Correction for Multichannel Analyzer Spectra. *Nuclear Instruments and Methods* 121 pp. 25-32, 1974.
14. Evaluation of measurement data — Guide to the expression of uncertainty in measurement (GUM), JCGM 100 (latest version).
15. The R Project for Statistical Computing, <http://www.r-project.org/>, (<http://cran.rstudio.com/>) and (<https://www.rstudio.com/ide/download/desktop>).
16. <http://www.nist.gov/itl/sed/gsg/metrology-for-microsoft-excel.cfm>.

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

The form can be downloaded from 682 ([\elwood.nist.gov](http://elwood.nist.gov)) L:\internal\PL846\_04\HPGe measurement request form

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:		
SAMPLE CHEMICAL COMPOSITION:		
SAMPLE MASS:		
SAMPLE DENSITY:		
PROGRAM		MEASUREMENT QUANTITY REQUIRED
<input type="checkbox"/> CALIBRATION SERVICE		<input type="checkbox"/> IMPURITY
<input type="checkbox"/> OTHER – DESCRIBE:		<input type="checkbox"/> ACTIVITY
		<input type="checkbox"/> OTHER – DESCRIBE:
DETECTOR GEOMETRY USED FOR MEASUREMENTS		
COMMENTS		RESULTS
WORK COMPLETED	SIGNATURE	DATE

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A.2. Example of data analysis template

This example shows the data analysis performed using both software packages (Genie 2000 and Fitzpeaks). A different format may be used depending on the type of data analysis performed.

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															
<b>Genie2k</b>																					
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										
<b>Fitzpeaks</b>																					
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										

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**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)Filename: zzzzz.docxImpurities 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

MDA (if applicable):

Report Date/Year

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**A.4. Activity measurement results**

Activity measurements results are normally reported in an 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. A different format may be used depending on data being reported.

Radionuclide	Energy (keV)	Ampoule Activity (Bq/g)	Std dev (%)	Geometry (%)	Decay factor (%)	Peak Fit (%)	Eff (%)	Unc distance (%)	pile-up corr unc (%)	Emission Intensity (%)	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

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## 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 that can be measured in this system, see Figure 1. The HPGe detectors are mainly calibrated to measure point sources and 5 mL ampoules (NIST-1 and NIST-3), but calibrations can be extended to disks, glass vials, Marinelli beakers, gas spheres, plastic bottles, and plastic vials. For some measurement geometries, corrections to the measured efficiency curves (i.e., detector calibration measurements) are needed to account for geometric differences. Detailed information about the dimensions of the NIST-1 and NIST-3 5 mL ampoules is shown in Figure 2 and Figure 3, respectively. The 5 mL ampoules are expected to contain a certain volume, based on the efficiency calibration geometry; corrections are made if this volume is different from the expected value. The detectors are calibrated at different source-to-detector distances for the different types of sources. These distances may change if the detector is sent for repair or modifications are made to the source holders. In these cases, the detector will be recalibrated at the new distance.



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

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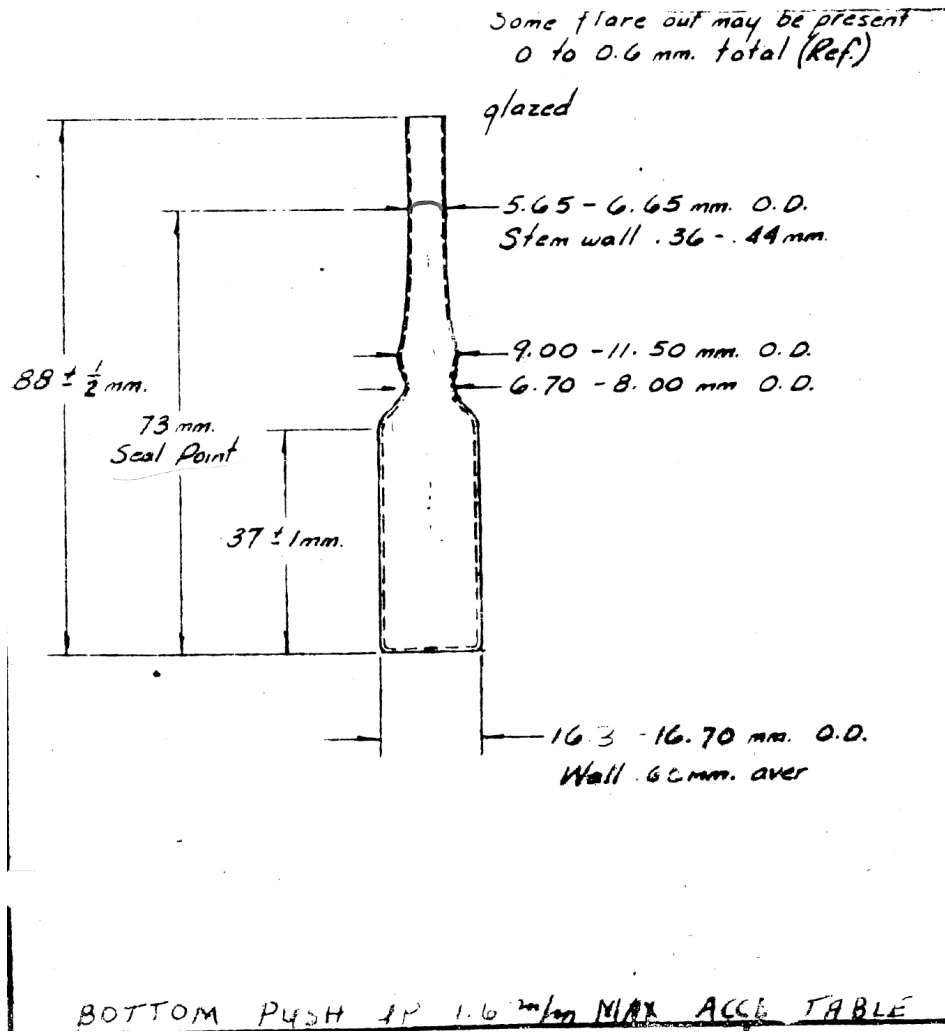


Figure 2: Dimensions of NIST-1 5 mL ampoules

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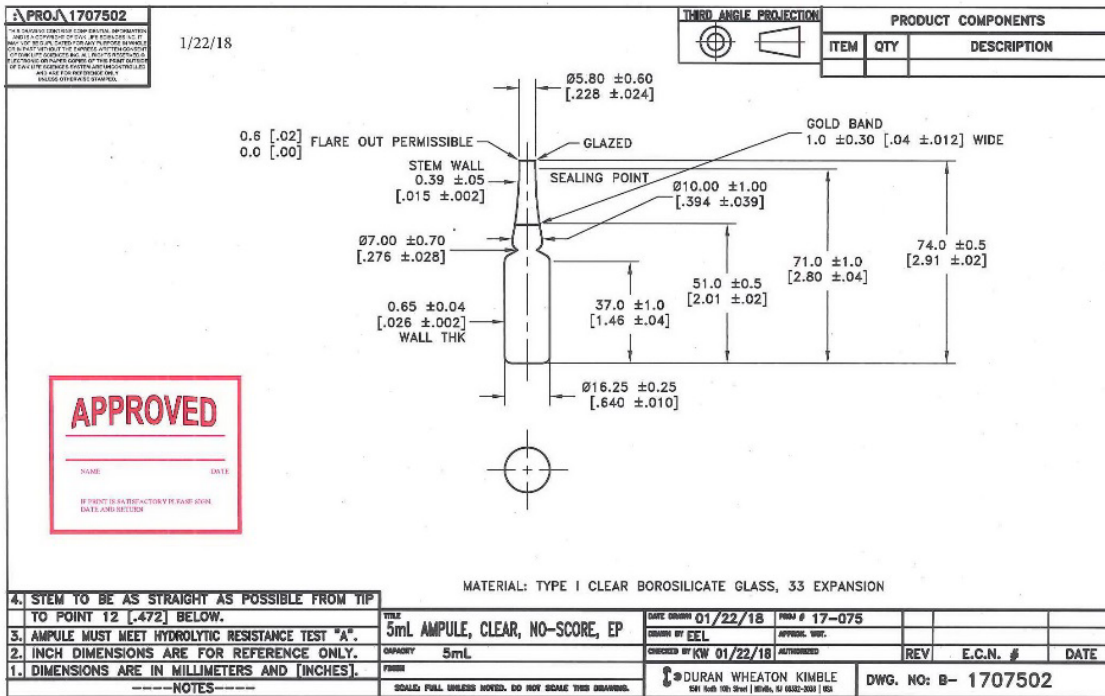


Figure 3: Dimensions of NIST-3 5 mL ampoules

**B.3. Information about how to start/stop a measurement and save a spectrum file using the R-detector**

**Setup in the R-detector acquisition program:**

There are 2 data sources (1:2) and (2:2):

- Data source (1:2) is the coincidence channel
- Data source (2:2) is the non-coincidence channel

**Start the measurements:**

Make sure that the (1:2) data source is selected before starting the measurement.

Enter the description of the source measurement that includes radionuclide name, source name/number, detector name, counting geometry, and date.

After selecting the acquisition time, press the start button.

**Save the measurement:**

Make sure that the (1:2) data source is selected, manually stop the measurement, or wait until the measurement preset time is completed.

Make sure that the (1:2) data source is selected and save the file using the source description name.

Switch to the (2:2) data source, click on “Datasource”, then click on Next source.

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Save the file using the source description name and add “NC” at the end of the filename (this denotes the non-coincidence file).

Make sure that both files are saved.

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## 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 intensity 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 corrections due to self-attenuation, to differences in the source density or source volume, the decay correction of the radionuclide during the measurement time, and decay correction to the reference time (among others). 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, 8, 9]

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 cm<sup>3</sup> 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; care 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 Figure 4 (error bars are smaller than the size of the symbols).

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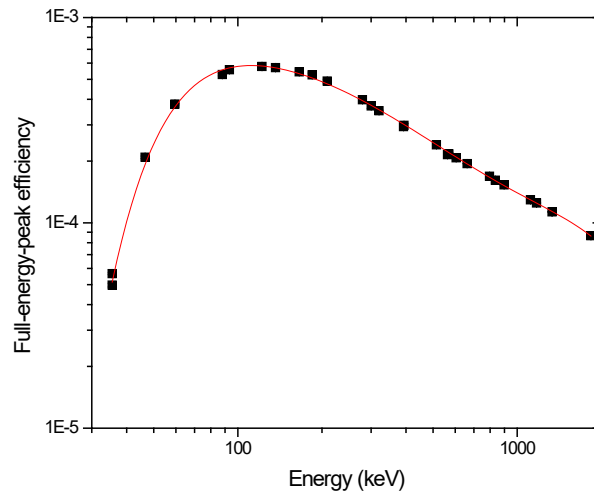


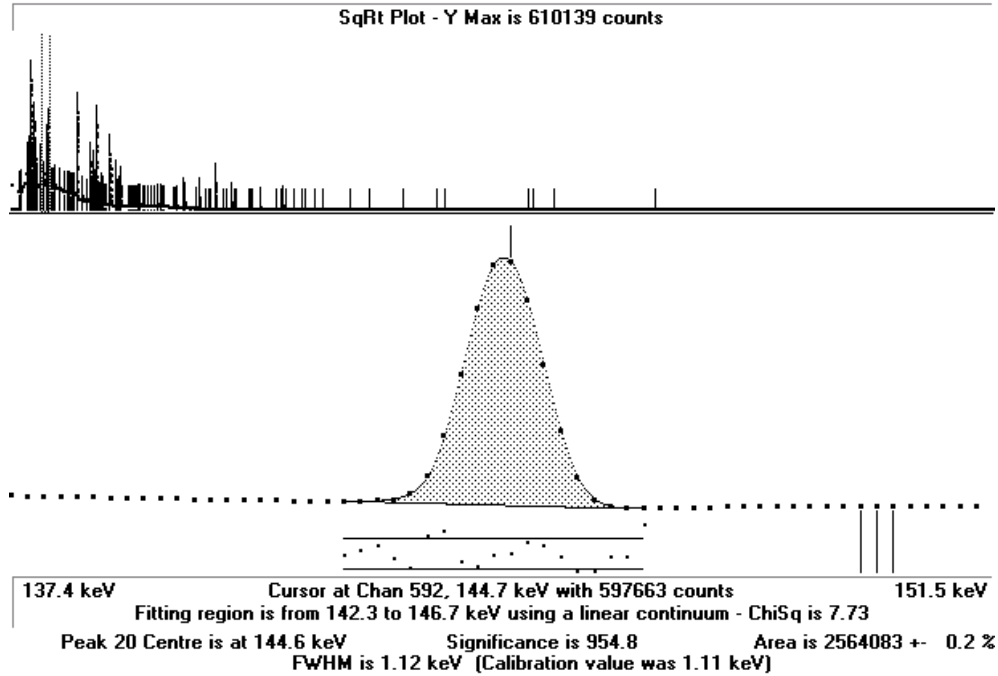
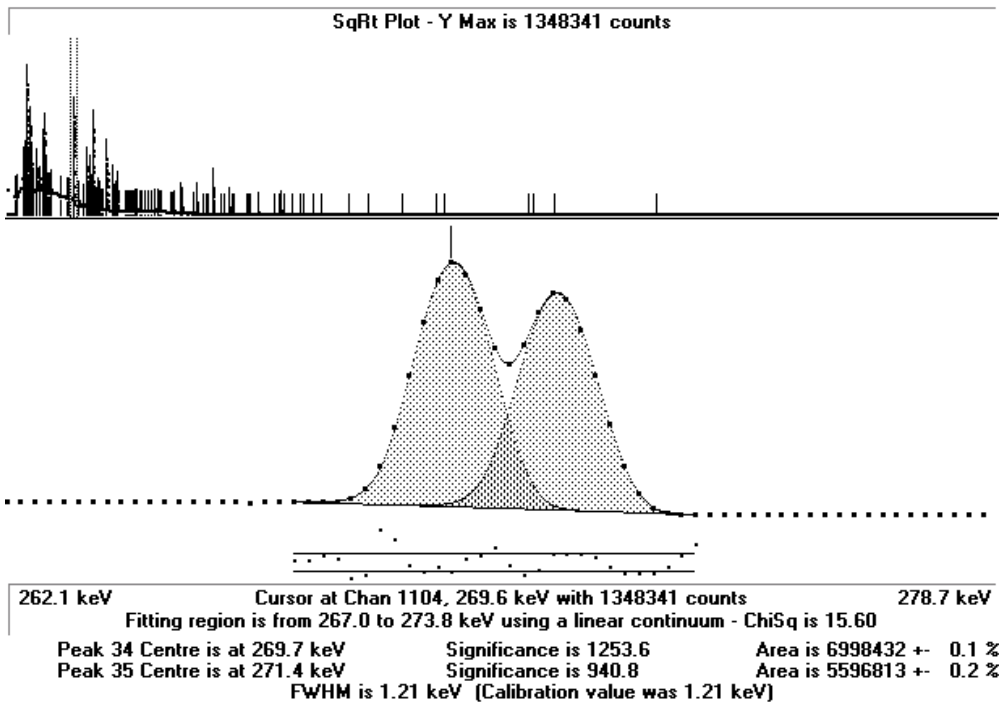
Figure 4: Example of full-energy-peak efficiency as a function of gamma-ray energy

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. If the source type or the source-to-detector distance used for the measurements differs from that used for 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™, FitzPeaks). Examples of the fit using FitzPeaks are shown in Figures 5 and 6.

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Figure 5: Example of fit to a single gamma-ray line using [FitzPeaks](#)Figure 6: Example of fit to an overlapping gamma-ray lines using [FitzPeaks](#)

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## 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 [9].

### C.1.1. Decay correction during measurement

When determining the source activity for radionuclides with short half-lives relative to the measurement time, we need to account for 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:

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

The uncertainty in the measurement time  $t$  is considered to be equal to zero, since the acquisition system provides measurement times with millisecond accuracy, while source acquisition times are typically 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  $\lambda$  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 [9].

The associated uncertainty is given by:

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

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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 millisecond 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 [9].

- a. This can be accomplished by measuring a set of sources containing known relative amounts of different radionuclides that emit photons with energies in the region of interest. Such a set of sources contains:  $^{57}\text{Co}$ ,  $^{133}\text{Ba}$ ,  $^{54}\text{Mn}$ , and  $^{65}\text{Zn}$  with activity ratios of approximately 1:3:6:10:15 (for each radionuclide used in the measurement). 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  $T_i$  is chosen each time. For each energy, the ratios  $N_i/(A_i T_i)$  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  $n_t = N_t / T_t$ .
- 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.,  $^{137}\text{Cs}$ ), 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.,  $^{57}\text{Co}$ ), 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  $n_t = N_t / T_t$ . 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  $\tau$  is the slope of the linear fit [13].

### C.1.4. Coincidence summing correction

The correction depends on the decay scheme of the radionuclide under measurement [9]. 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

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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, source volume, 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 [10, 11]).

The attenuation through material (i.e., air, self-attenuation, shielding) can be corrected using the mass attenuation coefficients [9] as follows:

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

where  $\mu/\rho$  is the mass attenuation coefficient (in  $\text{cm}^2/\text{g}$ ),  $x$  is the material thickness (in cm) and  $\rho$  is the material density (in  $\text{g}/\text{cm}^3$ ). Mass attenuation coefficients and material densities can be obtained from XCOM [12].

For additional information about the different corrections see reference [9].

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## **Annex D: Electronics settings**

### **D.1. C1-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 (as described in Annex E). 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.

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## Annex E: System checks

A set of  $^{57}\text{Co}$  and  $^{60}\text{Co}$  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 are 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 the detector is 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.

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**Annex F: GammaVision™ Job Control file**

The extension of the filename for a job control file is “.job”. The user can select the filename used; it is normally related to the detector name.

The GammaVision™ user manual provides information about how to set up a job control file. Below is an example of a job control file:

```
SET_DETECTOR 00004
SET_PRESET_CLEAR
SET_PRESET_LIVE 28800
CLEAR
START
LOOP 3
WAIT
FILL_BUFFER
CLEAR
START
SET_DETECTOR 0
DESCRIBE_SAMPLE “Am-241 #PS4 G-det half-sph ps 40cm ???”
SAVE “C:\User\G Detector\June 2025\Am241PS4G40hsps???.Spc”
SET_DETECTOR 00004
END_LOOP
```

NOTE - The “???” symbols in the filename will be automatically replaced by a number sequence starting in “000” once the acquisition of each spectrum is completed and saved.

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**Annex G: 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
1.01	April 2024	Procedure 23 v110	LP/LLP/BZ/AL/CMO	Alan Thompson
1.02	April 2026	Procedure 23 v102	LP/BEZ/LLP/AKT	

**List of changes for different versions of the document:**

Changes are tracked starting with version 1.10.

1. Changes to version 1.10
  - a. *Scope*: added the procedures that make use of this RPD-23 procedure.
  - b. *Equipment*: clarify the sources that are used for calibration, changed the N-detector to the C1-detector (as the N-detector is no longer used). Changed Table 1 to reflect the changes in the detectors and geometries.
  - c. *Radiation Safety*: clarified what is low and high activity values. Specified that sources are kept in a container.
  - d. *Acceptance of Sources*: clarified that sources are shipped with the attention to the source custodian.
  - e. *Measurement Sequence*: Add the description of performing measurements using a batch job or measurement sequence. Clarified the source description name. Clarified that the measurement can stop automatically or manually.
  - f. *Data Analysis – Activity Measurement*: Changed Annex A to A.2.
  - g. *Data Analysis – Reporting results*: Add the possibility of reporting MDAs

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- h. *Evaluation of Measurement Uncertainty*: Fixed some typos under equation 2.
- i. *Annex A.1 and A.3*: Updated the examples of the forms.
- j. *Annex A.2 and A.4*: Noted that these are examples and these might change depending on the analysis performed.
- k. *Annex B.2*: Edited the information to describe the status of the calibration service. Added a figure for the NIST-3 ampoules. Deleted Table 2 as it is no longer applicable.
- l. *Added Annex B.3*. Information about how to start/stop a measurement and save a spectrum file using the R-detector.
- m. *Annex C*: Added figure captions and references to the figure numbers. Under C1.1. edited equations C.4 and C.6. In C.1.3 made is clear that the ratios are for each radionuclide. In C.1.5 clarified that source volume is part of the source geometry.
- n. Added a new Annex F that describes how to edit or create a job control file in GammaVision™.
- o. Annex F was changed to Annex G. In Annex G, the list of changes was added. The filename for version V1.01 was mislabeled.

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