Chapter 3

Measurement of Be-7 in Environmental Materials

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3.1 Overview of Digital Gamma Spectrometry Systems

Gamma-ray spectrometry is the only ‘routine’ method for measuring the natural cosmogenic radionuclide ^7Be . Activity concentrations (Bq kg^{-1}) of ^7Be are determined by analysing the 477.6 keV gamma energy emitted by the first excited state of ^7Li (branching ratio 10.44%) (DDEP 2017) as it achieves the ground state. A typical gamma-spectrometric system consists of a semiconductor crystal detector, liquid nitrogen or mechanical cooling system, preamplifier, detector bias supply, linear amplifier, analogue-to-digital converter (ADC), multi-channel analyser (MCA) of the spectrum, and output data devices (ANSI 1999). An example of a typical gamma detector is given in Fig. 3.1.

High purity germanium (HPGe) detectors currently represent the most widely used gamma-detector systems. The semiconductor crystal is manufactured from ultrapure germanium (impurity level 10^9 atoms cm^{-3}) in various shapes and in a range of sizes for a wide range of applications (Debertin and Helmer 1988). The specification of a HPGe detector is defined by (i) the energy resolution, (ii) the detection efficiency and (iii) the peak-to-Compton ratio (see Glossary). When purchasing a HPGe semiconductor detector, these key parameters need to be considered together with the detector material and configuration, detector volume, window material and its thickness. Excepting the ultra-low energy configurations, all detector types commercially available are suitable for the measurement of ^7Be activity concentrations.

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Fig. 3.1 **a** Example of a HPGe gamma detector showing the detector head, the cartridge containing electronic management system and the dip-stick which is immersed in liquid nitrogen to cool down the detector to 77 K; **b** the detector encased in a lead shield with the dip stick immersed in a liquid nitrogen tank. *NB: The liquid nitrogen tank should be filled on a regular basis (every 7–10 days) to maintain a fully operational detector*

3.2 Preparation of Samples

3.2.1 Sample Management

A fundamental consideration for the analyst when managing a set of soil, sediment or rainwater samples is the relatively short half-life of ^7Be and its low activity in the majority of environmental materials. It is important, therefore, that the laboratory is well organised and ready to process the samples for ^7Be analysis as soon as possible after collection so that a relatively high emission of gamma rays can be taken advantage of. Additionally, maintenance of a high standard of cleanliness in order

to prevent contamination of the detector end cap or potential inter-contamination between samples should be prioritized.

3.2.2 Soil Samples

Samples of soil will normally consist of a range of particle sizes. Gamma spectrometric analysis of fallout radionuclides (FRN) in bulk soils should typically be performed on dried, homogenised material that has been sieved to less than 2 mm (Pennock and Appleby 2002) noting that some applications may require sieving to a smaller particle size. Typically, soil samples are oven-dried at 105 °C until a constant weight is reached (commonly for 24 h). A practical alternative is sample freeze-drying, which disaggregates the solids and makes subsequent sample processing more effective. Furthermore, oven-dried samples are disaggregated using a mortar and pestle and size fractionation of the dried solid can then be carried out on the ground sample using a metal or plastic sieve with a defined mesh size. Appendix 3.1 summarises the sample preparation and the associated packing protocol.

3.2.3 Samples of Rainwater

Samples of rainwater from the study site are required to assess the atmospheric deposition flux of ^7Be during a period, encompassing the sampling campaign (see Chap. 2). The protocol for extraction of ^7Be from rainwater is presented in Appendix 2.1. ^7Be can be pre-concentrated from solution by co-precipitation with MnO_2 following the method detailed by Short et al. (2007). The precipitate is recovered on glass fibre filters which are placed in a Petri dish of suitable diameter and allowed to dry before counting on an HPGe gamma detector.

3.2.4 Sample Geometry

Subsequent to physical processing samples, for analysis, can have masses in the range from a few grams to several kilograms, according to the aims of the project and its sample collection requirements. There are a range of measurement geometries, from Marinelli beakers (0.25–1 L) to small vials ($\sim 5\text{ cm}^3$) capable of holding a wide range of solid weights (from about 3 g of material to 4 kg) (Fig. 3.2). The choice of the container geometry for ^7Be analysis depends on the matrix and on the available amount of material. It is essential that the weights of both empty and filled sample holders are registered on a calibrated laboratory balance to at least 2 decimal places (or at least 4 decimal places for volume calibration standards).

Fig. 3.2 Several examples of containers used to perform gamma spectrometric analysis of solid phases. From left to right: 1 L Marinelli beaker; 300 mL Marinelli beaker; 90 mm diameter plastic Petri dish; 50 mm diameter Petri dish; 4 mL plastic vial (commonly used for conducting analysis using well detectors). To comply with laboratory safety, the radioactive standards are bound in red tape

Prior to purchasing any sample/standard container (especially for Marinelli beaker geometries), the analyst should determine the diameter of the detector end cap so that the container with an appropriate internal diameter will fit as close as possible on the detector end cap. For cylindrical and vial containers, it is recommended to place the samples on a holder (e.g. of light polycarbonate matrix) to assure the reproducibility of the sample-detector arrangement for all samples, including the measurement of the standard. The use of such holders will prevent contamination of the detector end cap, which can be further protected with a thin sheet of cling film or, in the case of well detectors, the sample vial covered in film. Care should be also taken to ensure that the sample/standard container external surface is free of particulate matter.

It is recommended that the geometries of the standard and the soil samples are identical; any differences between them, such as in the sample filling height (e.g. because of insufficient sample amount) or in soil chemical composition, will imply the need for geometry corrections (see Glossary). It is important to note that if the containers are being re-used, the analyst needs to carry out acid cleaning and drying procedures before refilling them. The packing date, nominal weight and an identification code should be indicated on each sample container after packing. As regards filter-mounted samples, these should be measured after placing them in cylindrical Petri dishes of appropriate diameter. Similar contamination precautions are to be taken for the filter geometry as for the other container types discussed above.

3.3 Calibration Approaches

An accurate and precise calibration of a gamma detector is essential for high quality results in the determination of ^7Be activity concentrations. The calibration of a gamma spectrometric system involves two major steps:

- (1) *Energy calibration* (includes energy calculation as a function of the number of the channel and Full-Width-Half-Maximum (FWHM) as a function of energy);
- (2) *Efficiency calibration* (the efficiency as a function of the energy correlated with the source geometry).

Energy calibration is commonly performed using point sources (e.g. ^{152}Eu , ^{137}Cs) or multinuclide standards covering the energy range of interest (Fig. 3.3). The construction of the full energy peak efficiency curve is performed following (1) a *semi-empirical approach* (based on measurement of standard point sources or multinuclide standard sources of soil or liquid matrix), or (2) *Monte Carlo methods* (using a detailed description of the detector and the source). In both cases, the efficiency calibration procedure involves (1) true photopeak efficiency determination for the gamma energies of the radionuclides included in the certified standard source, and (2) the construction of the efficiency curve (within a range of energies given by the radionuclides present in the source) by polynomial fitting. Discrepancies between simulated (Monte Carlo) and experimental efficiency values should be determined and included as an uncertainty component in the uncertainty budget of the ^7Be activity value. Note that different detectors and different system-sample configurations will result in different values of the true photopeak efficiencies, which should finally produce the same result of the radionuclide activity value for the actual sample. Coincidence summing corrections should be performed for relevant nuclides present in the calibration source and decaying through a cascade of successive photon emissions (e.g. ^{60}Co , ^{88}Y , ^{139}Ce). These can be determined through general or dedicated codes (Vidmar et al. 2010). Coincidence summing correction factors depend on the nuclide decay scheme, sample geometry and composition, and on detector parameters. The factor is equal to unity in cases where the radionuclide has no cascade of gamma-rays.

3.3.1 Semi-empirical Calibration Method

For routine environmental sample analyses, efficiency calibration of gamma detectors can be performed using (a) multiple standard point sources or standard volume sources of mixed radionuclides (for soils measurements), or (b) filters spiked with standard solution (for ^7Be precipitate analysis). Considering the short half-life of ^7Be , standard sources containing ^7Be are not commercially available. When purchasing a standard for efficiency calibration, it is essential that these should carry a certificate of calibration from a metrological labora-

Fig. 3.3 Print screen capture of the gamma detector energy calibration performed using a multi-radionuclide standard and the GammaVision commercial software. Note the interpolated 477.6 keV gamma energy of ^7Be between 391.7 keV (^{113}Sn) and 661.7 (^{137}Cs). The upper box is the energy table; the middle box is a plot of the gamma-ray energy against the channel number; the lower box shows the ^7Be peak

tory accredited for the preparation of standards. An appropriate calibration standard should have low overall uncertainties of each radionuclide in the source (up to 2–3%), as this uncertainty is further propagated into the uncertainty of ^7Be activity. The analyst should be aware that specific international regulations apply for the import of radioactive sources. After purchasing, the calibration standard solution (commonly delivered in sealed glass vial) must be gravimetrically diluted for subsequent use. Furthermore, the analyst shall choose how to use the diluted standard solution for the detector efficiency calibration. Bear in mind that special safety procedures and authorisations apply to the laboratories handling radioactive sources as regards to the risk of radioactive contamination. Appendix 3.2 details a recommended step-by-step approach for the preparation of a multi-nuclide calibration standard of soil matrix.

An example of a multi-nuclide standard solution suitable for ^7Be measurements will comprise artificial radionuclides such as ^{241}Am , ^{109}Cd , ^{57}Co , ^{139}Ce , ^{203}Hg , ^{113}Sn , ^{85}Sr , ^{137}Cs , ^{60}Co and ^{88}Y , with energy lines from 59 to 1836 keV. Such multi-nuclide standard can be used to calibrate gamma detectors for efficiency in the vicinity of the gamma emission from ^7Be (477.6 keV) which lies between that of ^{113}Sn at 391.7 keV, ^{85}Sr at 514.0 keV (difficult to determine accurately due to the overlapping with the 511 keV annihilation peak) and ^{137}Cs at 661.7 keV (DDEP 2017), as presented in Fig. 3.4. A disadvantage in the short half-lives of some nuclides (e.g. ^{203}Hg , ^{113}Sn)

Fig. 3.4 The efficiency calibration curve of a HPGe detector using a multi-nuclide standard solution containing 10 artificial radionuclides (plotted in Microsoft Excel spreadsheet). Note the ^7Be gamma emission is interpolated between the gamma energies of ^{113}Sn and ^{85}Sr . After the decay of short-lived radionuclides i.e. ^{85}Sr , ^{113}Sn and ^{203}Hg , the closest lower energy for interpolation will be ^{139}Ce . An interpolation of ^7Be energy between ^{139}Ce and ^{137}Cs will lead to an increased efficiency value for ^7Be

present in the standard must be recognized, thus making the source useful over the whole spectra region only for a short period of time (i.e. a few months).

(i) *Preparation of a secondary soil calibration standard*

A soil sample should be collected, preferably from an area where fallout radionuclides are minimal (e.g. subsoil) and prepared as in Sect. 3.2.2. The detection and quantification of the background gamma radiation in the soil sample is required. Then the prepared soil fraction below 2 mm particle size is spiked with an aliquot of the liquid standard. It is highly important to ensure that the soil-liquid standard mixing is complete, ensuring the spiked material is homogeneous (see step-by-step instruction in Appendix 3.2). The spiked soil is dried and an appropriate sample holder (selected from those in Fig. 3.2), with a similar geometry to the samples, is then selected and filled with the dried spiked soil. The secondary soil standard can then be used to calibrate the detector for efficiency (Fig. 3.5). If there are differences in geometry (different containers and/or filling height) and/or matrix (e.g. different chemical composition) between the soil samples and the calibration source, an efficiency transfer factor should be determined to correct this difference, as ratio between the efficiency of the sample and the efficiency of the standard.

(ii) *Preparation of a secondary liquid calibration standard*

Monte Carlo approaches for automatic efficiency calibration (e.g. LabSOCS—Laboratory SOURCEless Calibration Software) (Bronson and Wang 1996) are commonly integrated into some commercial software of gamma detectors. These codes include several options for detector type and configuration, sample shape and volume (e.g. point, disc, and cylinder), sample/source matrix, source-to-detector distance and for the type of fit used to describe the efficiency-energy dependency (linear, quadratic, polynomial) (Jovanovic et al. 1997; Bronson and Wang 1996). For each particular sample, the analyst needs to create a specific sample geometry based on the holder dimensions (diameter and wall thickness), sample filling height, chemical composition of the sample matrix and density. It is also important for the analyst to know the precise details of the technical characteristics of the gamma detector or to obtain a ‘characterisation file’ of the detector from its manufacturer in order to run the codes. However, no programming knowledge is necessary to use the available commercial codes. The efficiencies generated for a specific sample can be saved and stored by the analysis software and used to automatically derive the radionuclide activity without performing an experimental calibration. However, the experimentally obtained efficiencies can be as well included in the analysis software, in case these data are available.

The automatic calibration by the Monte Carlo approach eliminates the cost of purchasing, tracking, and disposing of radioactive standards. However, the analytical results still need to be validated using certified reference materials in similar matrices. The main constraints in the application of Monte Carlo approaches are related to uncertainties of the detector configuration (e.g. shape and size of the effective crystal volume, photons and electron interaction probability and angular distributions) and measurement traceability to primary standards.

3.4 Data Handling and ^7Be Analysis

Analyses of ^7Be can be challenging because of low activity concentrations and, therefore, high counting uncertainties. Sample measurements are completed after performing the energy and efficiency calibrations. Data evaluation is commonly realised using specialised gamma software, provided by the detector manufacturer or made ‘in-house’. Prior to spectrum analysis, ^7Be nuclear data (gamma energy, half-life and emission probability) should be included in the software library. The stability of the spectrometer should be checked during the measurement and during routine quality assurance procedures. Despite the apparent simplicity of gamma-ray measurements, bear in mind that there are a number of correction factors to the spectrum counting data that must be considered for the determination of ^7Be activity concentration e.g. decay correction to the sampling date, efficiency transfer factor, background correction, instrument dead time correction (IAEA 2004), while a correction for soil moisture content is applied to the dried soil sample mass.

A flow chart of ^7Be measurement by gamma-ray spectrometry is presented in Fig. 3.6. Sample counting times need to be long enough (typically between 86,000

Fig. 3.6 Schematic protocol for ^7Be analysis by gamma-ray spectrometry (MDA = Minimum Detectable Activity)

and 259,000 s) to meet laboratory standard statistical uncertainty of the measurement result (commonly between 10 and 30% for ^7Be in environmental samples).

Based on the information provided in the library, the instrument-specific gamma evaluation software will identify the ^7Be peak and analyse the number of counts within its peak area (Fig. 3.7). Furthermore, following default automated sequences; the software is able to report the activity concentration (Bq kg^{-1}), combined standard uncertainty and minimum detectable activity of the radionuclide found by the peak-search routine (Debertin and Helmer 1988). However, to achieve reliable results using the library approach, any shift effect in the spectrum should be corrected and energy calibration should be precise enough to achieve no more than one or two channels deviation between ^7Be reference energy and the one calculated from the calibration curve (IAEA 2002).

Fig. 3.7 Print screen capture of a gamma spectrum registered with the GammaVision commercial software and showing the ^7Be peak at 477.56 keV and its net area and gross/net count rate (peak data box), measurement starting time and live time (upper right part of the screen)

Measurement results are commonly reported with associated combined uncertainty at the 95% confidence level (JCGM 2008). For detailed information on the determination of each uncertainty component, readers can refer to Lépy et al. (2015). The detection limit (LD) provides the detection capabilities of a measurement system.

3.4.1 Quality Assurance

In routine laboratory practice, it is necessary to validate the measurement results for certain working conditions. Both internal and external validation methods are an important part of ‘good laboratory practice’ and also a requirement of ISO/IEC 17025 for ‘in-house’ methods (ISO/IEC 17043 2010; ISO/IEC 17025 2017). The internal validation procedure can be employed using distinctive approaches:

- Various types of blanks;
- Replicate analysis to check changes in precision;
- Use of certified reference materials with similar matrix and geometry (e.g. provided by well-recognized international providers such as the IAEA);
- Standards and point sources to check the stability of the response;
- Use of long-term control charts of standards and point source measurements to check the stability of the detector response;
- Proficiency tests participation.

Note that because of its short half-life, there are no reference materials available which contain ^7Be . However, it is a common practice to check the accuracy of the laboratory procedure for radionuclides emitting gamma rays in the vicinity of ^7Be energy (e.g. ^{137}Cs) in samples of similar matrix and geometry.

In addition, external validation can be achieved through: (i) inter-laboratory comparison exercises and/or (ii) participation in international proficiency tests for radionuclide determination in soil and water samples such as the ones organized regularly by IAEA and its Member States.

Glossary

The energy resolution (FWHM)

The full width of the peak at one-half of the maximum height above any underlying continuous spectral background.¹

Absolute efficiency

The ratio between the number of pulses recorded and the number of radiation quanta emitted by source.

Relative efficiency

Detector efficiency relative to that of a NaI(Tl) detector.²

Peak-to-Compton ratio

The ratio of the number of counts in the biggest channel of the 1332.50 keV ^{60}Co energy peak to the average channel count in the Compton continuum between 1040 and 1096 keV in the same spectrum.

Coincidence summing correction factor

The ratio between the apparent efficiency for the line with energy E of the nuclide having cascading radiations, to the full energy peak efficiency at the same energy obtained from the energy curve measured with single-photon emitting radionuclides. The apparent activity (corrected for background) must be multiplied by the coincidence summing correction factor to

¹Debertin and Helmer (1988).

²Knoll (2000).

	obtain the true activity value for the radionuclides affected by cascade effect. ³
Geometry and self-attenuation correction factor	The ratio of the full energy peak efficiency for the sample and the full energy peak efficiency for the calibration standard. The correction is commonly performed through an efficiency transfer approach, by multiplying the correction factor with the ⁷ Be full energy peak efficiency.
Efficiency transfer	The “transfer” of the efficiency of a standard to another sample, which can be in the same geometry or not, and acting as corrective factor for both geometry and self-absorption effect.
Minimum Detectable Activity (MDA)	The lowest activity value that can be detected with a measuring system at 95% confidence. ⁴
Detection Limit (LD)	The lowest expectation value of the counting rate of a net peak area that can be detected with a measuring system.

Appendix 3.1: Soil Sample Preparation Prior to Gamma Analysis

- Weigh the total wet soil sample in grams to 2 decimal places. Record weight as total wet weight. In case of a soil core, section core into desired slice increments and further follow the same procedure as previously described.
- Freeze dry each soil sample (or oven dry at 105 °C).
- Weight total dry sample (g) to 2 decimal places. Record weight as total dry weight.
- Homogenize each sample with a pestle and mortar to ensure soil material is fully mixed and disaggregated.
- Choose the container size according to the available sample volume to obtain a full (or near full) container after sample packing. Pack a suitable pre-weighed container (Fig. 3.2) with a subsample of the homogenised soil material:

³ANSI (1999).

⁴ISO 11929 (2010).

- a. Weigh empty container and its lid (g) to 2 decimal places;
 - b. Pack container with soil and assure that (1) the material is firmly patted down inside the container, so that no void spaces are left within the sample container; (2) the soil surface is flat;
 - c. Weigh packed container and its lid (g) to 2 decimal places;
 - d. Record total packed subsample weight (g) to 2 decimal places;
 - e. Seal container with gas-tight insulating tape;
 - f. Label container with the pre-arranged sample ID and note date of packing;
- Record all weighed data and sample ID onto supplied spreadsheet.

Appendix 3.2: Preparation of a Secondary Soil Calibration Standard

(A) Opening and Diluting the Mixed Traceable Standard Solution

- A mixed traceable standard source is typically purchased from an accredited metrological institute in the form of acid liquid solution inside a flame-sealed glass ampoule. The vial will be opened under fume-hood conditions following the radiation protection laboratory procedures.
- Caution should be taken to avoid any spillage of the solution when the 'swan neck' is broken (along the scratch mark).
- A clean, acid-washed 100 mL Grade A glass volumetric flask is weighed (4 decimal places) and fitted with a small funnel. Most of the traceable reference source is transferred to the volumetric flask using a disposable Pasteur pipette.
- The Pasteur pipette is then filled with 1–2 mL of 4 M HCl which is used to wash the remaining traceable solution into the volumetric flask via the funnel. This should be repeated several times to ensure that all the traceable solution is transferred to the volumetric flask. When all the liquid has been transferred from the vial the liquid in the flask should be filled up to the mark with 4 M HCl and weighed again for 3 times. The mean arithmetic weight of the flask with the standard solution is recorded to 4 decimal places.
- The liquid in the volumetric flask is then transferred to a clean plastic bottle and labelled as 'Stock Solution' of the mixed radionuclide reference source and clearly marked with trefoil.

(B) Dosing a Soil Sample with the Mixed Standard Solution

- A soil sample, typically about 100 g, with the grain size <2 mm is prepared as mentioned in Sect. 3.3.1(i) and placed in a suitable beaker. The soil is moistened with a small volume of distilled water until the water level is above the soil surface, to enable the mixing of the slurry with the liquid standard.
- An appropriate volume of the diluted mixed traceable reference solution is added to the slurry using a calibrated pipette. The amount added should be sufficient to

achieve <1% uncertainty of the counting statistics of the peaks of interest within a reasonable counting time when measuring the calibration standard.

- The slurry is thoroughly mixed using a glass rod.
- The slurry is dried in an oven at 105 °C or, preferably, in a freeze-drier until constant weight is achieved (commonly over 24–48 h).
- The dried solid is then carefully transferred to a rotating ball mill to ensure that the final dried solid is completely mixed. The final product is a fine powder that can be allocated to appropriate sample holders, such as 50 mm diameter, dried, pre-weighed Petri dishes.

For safety, any particle transfer process should be completed in a fume hood and the operator should be protected with a mask.

- The filled Petri dish, and any other containers, should be re-weighed and sample weights recorded so the activity concentrations of each of the radionuclides in the standard can be evaluated. Afterwards, the standard should be bound with adhesive tape in distinct colour (e.g. red) for safety reasons. Its sample number, or reference data, should be written on the lid in indelible ink.
- The Petri dish should then be wiped with a moist cloth to remove any extraneous particles. It should be kept in a sealable plastic bag when out of use.
- These will be further used as secondary standards, in different geometries, for the efficiency calibration of the gamma detectors. A record should be kept on the radionuclide inventory: (i) present in the secondary standard, and (ii) left in the ‘Stock Solution’.

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