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colour inside

Nuclear instrumentation – Measurement of activity or emission rate of gamma-ray emitting radionuclides – Calibration and use of germanium-based spectrometers

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NUCLEAR INSTRUMENTATION – MEASUREMENT OF ACTIVITY OR EMISSION RATE OF GAMMA-RAY EMITTING RADIONUCLIDES – CALIBRATION AND USE OF GERMANIUM-BASED SPECTROMETERS

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IEC 61452 has been prepared by IEC technical committee 45: Nuclear instrumentation. It is an International Standard.

This second edition cancels and replaces the first edition published in 1995. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

a) Title modified;
b) Additional information on digital electronics;
c) Information on Monte Carlo simulations;
d) Reference to detection limits calculations.

The text of this International Standard is based on the following documents:

FDIS	Report on voting
45/921/FDIS	45/925/RVD

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this International Standard is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/standardsdev/publications.

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- withdrawn,
- replaced by a revised edition, or
- amended.

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INTRODUCTION

A typical gamma-ray spectrometer consists of a high purity germanium (HPGe) detector with its liquid nitrogen or mechanically refrigerated cryostat and preamplifier, associated to either analog or digital electronic modules including the detector biasing and signal processing (amplification, multichannel conversion and storage) and data-readout devices. The spectrometers include or are associated with computers and their acquisition software. A radiation shield often surrounds the detector to reduce the counting rate from room background radiation for shield construction guidelines). Primary interactions of the photons (X- and gamma-rays) in the HPGe crystal (by photoelectric absorption, Compton scattering or pair production) impart energy to electrons whose energy is finally released by creation of electron-hole pairs. These electrons and holes are collected to produce a pulse whose amplitude is proportional to the energy deposited in the active volume of the HPGe crystal. These pulses are amplified, shaped and sorted according to pulse height to produce a histogram showing, as a function of energy, the number of photons absorbed by the detector. After the accumulation of a sufficient number of pulses the histogram will display a spectrum with one or more peaks with an approximately normal (Gaussian) distribution corresponding to photons that transferred their entire energy to the detector. These are superimposed on continuum constituted by the events related to the partial deposition of energy.

The recorded peak area depends on the emission rate of the gamma-ray and on the detection efficiency of the detector, which is energy dependent. The emission rate, $R(E)$, for a gamma-ray of energy E is determined by dividing the net area, $N(E)$, in the full-energy peak by the measurement live time, T_{L}, and full-energy-peak efficiency, $\varepsilon(E)$, of the detector for the counting geometry used. A curve or functional representation of the full-energy-peak efficiency permits interpolation between available calibration points. Corrections may be needed for:

a) decay of the source during sampling (e.g., with air filters) and counting and/or ingrowth;

b) decay of the source from a previous time to the counting period and/or ingrowth;

c) attenuation of photons within and/or external to the source that is not accounted for by the full-energy-peak efficiency calibration;

d) solid angle correction that is not accounted for by the full-energy-peak efficiency calibration;

e) true coincidence (cascade) summing;

f) loss of pulses due to pulse pile-up (at high counting rates).

NUCLEAR INSTRUMENTATION – MEASUREMENT OF ACTIVITY OR EMISSION RATE OF GAMMA-RAY EMITTING RADIONUCLIDES – CALIBRATION AND USE OF GERMANIUM-BASED SPECTROMETERS

1 Scope

This document establishes methods for the calibration and use of high purity germanium spectrometers for the measurement of photon energies and emission rates over the energy range from 45 keV to approximately 3 000 keV and the calculation of radionuclide activities from these measurements. Minimum requirements for automated peak finding are stated. This document establishes methods for measuring the full-energy peak efficiency with calibrated sources.

Performance tests are described that ascertain if the spectrometer is functioning within acceptable limits. These tests evaluate the limitations of the algorithms used for locating and fitting single and multiplet peaks. Methods for the measurement of and the correction for pulse pile-up are suggested. A test to ascertain the approximate magnitude of true coincidence summing is described. Techniques are recommended for the inspection of spectral analysis results for large errors resulting from true coincidence summing of cascade gamma-rays in the detector. Suggestions are provided for the establishment of data libraries for radionuclide identification, decay corrections, the conversion of gamma-ray emission rates to decay rates and Monte Carlo simulations.

The measurement of X-ray emission rates is not included because different functional fits are required for X-ray peaks, which have intrinsically different peak shapes than gamma-ray peaks. Further, X-ray peaks are complex multiplets (e.g., the K X-rays of Tl include 10 individual components that form four partially resolved peaks). This document does not address the measurement of emission rates of annihilation radiation peaks or single- and double-escape peaks resulting from partial energy deposition in the detector from pair production. Escape peaks may require different fitting functions than comparable full-energy peaks. Further, annihilation radiation and single-escape peaks have a different and larger width than a gamma-ray peak of similar energy. Discussion of acceptable methods for measuring the lower limits of detection as they relate to specific radionuclides is beyond the scope of this document.

The object of this document is to provide a basis for the routine calibration and use of germanium (HPGe) semiconductor detectors for the measurement of gamma-ray emission rates and thereby the activities of the radionuclides in a sample. It is intended for use by persons who have an understanding of the principles of HPGe gamma-ray spectrometry and are responsible for the development of correct procedures for the calibration and use of such detectors. This document is primarily intended for routine analytical measurements. Related documents are IEC 60973 and ISO 20042.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60050-395:2014, *International Electrotechnical Vocabulary (IEV) – Part 395: Nuclear instrumentation – Physical phenomena, basic concepts, instruments, systems, equipment and detectors*
IEC 60050-395:2014/AMD1:2016
IEC 60050-395:2014/AMD2:2020

IEC 60973, *Test procedures for germanium gamma-ray detectors*

ISO 11929 (all parts), *Determination of the characteristic limits (decision threshold, detection limit and limits of the confidence interval) for measurements of ionizing radiation – Fundamentals and application*

ISO 20042, *Measurement of radioactivity – Gamma-ray emitting radionuclides – Generic test method using gamma-ray spectrometry*

JCGM 100:2008, *Evaluation of measurement data – Guide to the expression of uncertainty in measurement (GUM)*

JCGM 200:2012*, International vocabulary of metrology – Basic and general concepts and associated terms (VIM), 3rd edition 2008 version with minor corrections*

3 Terms, definitions and symbols

For the purposes of this document, the following terms and definitions apply, as well as those given in IEC 60050-395 and JCGM 200.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia available at: http://www.electropedia.org/ and IEC Glossary available at: http://std.iec.ch/glossary
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1 Terms and definitions

3.1.1
accuracy
closeness of agreement between a measured quantity value and a true quantity value of a measurand

[SOURCE: JCGM 200:2012]

3.1.2
activity
A
number dN of spontaneous nuclear transitions or nuclear disintegrations for a radionuclide of amount N produced during a short time interval dt, divided by this time interval

Note 1 to entry: The unit is becquerel (Bq).

[SOURCE: IEC 60050-395:2014, 395-01-05]

3.1.3
analog-to-digital converter
ADC
electronic device used to convert the amplitude of a voltage pulse from analog to digital format

3.1.4
ADC conversion gain
number of channels over which the full amplitude span can be spread

Note 1 to entry: Usually 4 096 to 16 384 channels are used for HPGe gamma-ray spectrometry.

3.1.5
attenuation
net loss at the detector of primary photons of a given energy resulting from their interaction with matter either due to the occurrence of scattering or absorption in the sample or in material between the sample and the active volume of the detector

3.1.6
background
spectral data including peaks not caused by the source but rather resulting from radioactive decay occurring in the surrounding environment or from cosmic ray interactions in or adjacent to the detector (see 3.1.11)

3.1.7
calibration
determination of a value that converts a measured number into a desired physical quantity (e.g., pulse-height into photon energy, or counts per second into emission rate)

3.1.8
cascade transitions
gamma-rays in the radioactive decay of a single radionuclide that are emitted sequentially and within the resolving time of the spectrometer

3.1.9
true coincidence summing
simultaneous detection of two or more photons originating from a single radioactive decay that results in only one observed (summed) pulse (also cascade summing)

3.1.10
combined standard uncertainty
standard uncertainty of the result of a measurement when that result is obtained from the values of a number of other quantities, equal to the positive square root of a sum of terms, the terms being the variances or covariances of these other quantities weighted according to how the measurement result varies with changes in these quantities

Note 1 to entry: See JCGM 100:2008 GUM 1995 with minor corrections.

3.1.11
continuum
part of the pulse-height distribution lying underneath a peak including contributions associated with the source, detector, and measuring conditions that affect the spectral shape

3.1.12
conventional true value <of a quantity>
commonly accepted best estimate of the value of that quantity

Note 1 to entry: This and its associated uncertainty will normally be determined by a national or international transfer standard, or by a reference instrument that has been calibrated against a national or international transfer standard.

3.1.13
counting rate
number of pulses registered by the detector per unit of time being registered in a selected voltage or energy interval (expressed in s^{-1})

3.1.14
crossover gamma-ray
gamma-ray occurring between two non-adjacent nuclear levels

3.1.15
dead time
time during which a counting system is unable to process an input pulse

3.1.16
direct current (DC) level
input or output voltage level on a DC-coupled instrument when there are no pulses present

3.1.17
direct current (DC) offset
difference between a current or voltage level and a reference level

3.1.18
emission intensity per decay
emission intensity and yield
$P(E)$ or $P_\gamma(E)$
probability that a radioactive decay will be followed by the emission of the specified radiation

Note 1 to entry: Gamma-ray emission intensities are often expressed per 100 decays.

3.1.19
energy resolution, full width at half maximum
FWHM
width of a peak at half of the maximum peak height where the baseline is measured from the continuum

3.1.20
energy resolution, full width at tenth maximum
FWTM
width of a peak at one tenth of the maximum peak height where the baseline is measured from the continuum

Note 1 to entry: For a normal (Gaussian) distribution, FWTM is 1,823 times its FWHM.

3.1.21
error
measurement error
measured quantity value minus a reference quantity value

Note 1 to entry: The concept of 'measurement error' can be used both:

a) when there is a single reference quantity value to refer to, which occurs if a calibration is made by means of a measurement standard with a measured quantity value having a negligible measurement uncertainty or if a conventional quantity value is given, in which case the measurement error is known, and

b) if a measurand is supposed to be represented by a unique true quantity value or a set of true quantity values of negligible range, in which case the measurement error is not known.

Note 2 to entry: Measurement error should not be confused with production error or mistake.

[SOURCE: JCGM 200:2012]

3.1.22
full-energy peak
photopeak
FEP
peak in the spectrum resulting from the complete (total) absorption of the energy of a photon in the active volume of the germanium crystal and collection of all of the resulting charge

3.1.23
full-energy-peak efficiency
$\varepsilon(E)$
ratio between the number of counts in the net area of the full-energy peak to the number of photons of that energy emitted by a source with specified characteristics for a specified measurement geometry (i.e., source-to-detector distance, source type)

3.1.24
gamma-ray branching ratio
$f(E)$
for a given excited nuclear state, ratio of the emission rate of a particular gamma-ray to the total transition rate from that state

3.1.25
gamma-ray emission rate
$R(E)$
rate at which a gamma-ray of a given energy from the decay of a particular radionuclide is emitted from a given source

Note 1 to entry: The gamma-ray emission rate is the activity times the gamma-ray emission intensity.

3.1.26
live time
T_L
time interval of a count during which a counting system is capable of processing input pulses

3.1.27
multichannel pulse-height analyser
MCA
electronic device that records and stores pulses according to their amplitude. It consists of three function segments:

- an ADC to provide a means of measuring pulse amplitude;
- memory registers (one for each channel of the spectrum) to tally the number of pulses having an amplitude within a given voltage increment; and
- an input/output section that permits transfer of the spectral information to other devices such as a computer, or other display or permanent storage media.

3.1.28
peak-to-Compton ratio for the 1 332 keV ^{60}Co peak
ratio of the full-energy-peak height, for ^{60}Co measured at 1 332 keV, to the average height of the corresponding Compton plateau between 1 040 keV and 1 096 keV

3.1.29
pole-zero cancellation
pole-zero adjustment on the shaping amplifier adjusts the zero location of the pole-zero network to exactly cancel the preamplifier output pole and thus provide single-pole (i.e., no under or overshoot) response of the signal pulse at the amplifier output

Note 1 to entry: This operation converts the long-tailed preamplifier pulse to a short-tailed pulse suitable for signal optimization and subsequent pulse-height analysis.

3.1.30
pulse baseline
average of the level from which a pulse departs and to which it returns in the absence of a following overlapping pulse

3.1.31
pulse pile-up
random summing
occurrence of two successive pulses closely associated in time but from separate decays such that they contribute to each other's pulse height and shape

Note 1 to entry: Usually, the system processes the two inputs as a composite single pulse which is stored in a spectral channel different from that at which either of the component pulses would have been stored.

Note 2 to entry: Pulse pile-up is a function of the square of the counting rate and of the amplifier pulse width.

3.1.32
real time counting period
T_r
counting time uncompensated for periods in which an instrument might be unable to respond

Note 1 to entry: Real time of a count equals live time plus dead time.

3.1.33
relative full-energy-peak efficiency <for detector specification>
ε_r
ratio of full-energy-peak efficiency for a point source of ^{60}Co (1 332 keV photons) to that of a NaI(Tl) crystal of 7,6 cm diameter × 7,6 cm high for a source-to-detector distance of 25 cm (that is 1,2 × 10^{-3}).

$$\varepsilon_r = 0{,}833 \times 10^3 \times \frac{n(1332)}{R(1332)} \qquad (1)$$

where

$n(1332)$	is the measured full-energy peak counting rate;
$R(1332)$	is the 1 332 keV gamma-ray emission rate from the ^{60}Co source;
$0{,}833 \times 10^3$	is the reciprocal of the full-energy-peak efficiency of the NaI(Tl) detector at that energy and distance.

3.1.34
sample
radioactive material measured using the HPGe detector to determine the activity and/or emission rate

3.1.35
shaping-time constant
index
indicator of shaped pulse width (width of a shaped pulse at 50 % of its peak height). Unless otherwise specified, that width is $f_{1/2}$

3.1.36
source
calibrated radioactive material used to determine the HPGe detector response

3.1.37
standardization coefficient
$S_c(i,E)$
factor used for the direct conversion of the counts contained under a net full energy peak of a given energy, E, and from a specific radionuclide, i, to the activity of the radionuclide

3.1.38
total efficiency

$\varepsilon_t(E)$

ratio of the number of pulses recorded in the entire energy spectrum and the number of photons emitted by the source of a given energy, E, for a specified source-to-detector distance

3.1.39
uncertainty

non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used

[SOURCE: JCGM 200:2012]

3.2 Symbols

A	activity, or decays per second, of a nuclide (Bq)
$B(E)$	net counting rate of an interfering background peak at energy E
C_i	multiplicative correction factor, where i is: a (attenuation), c (true coincidence summing), d (decay), or r (pile-up)
e	2,71828... base of natural or Naperian (or Napierian) logarithms, ln
$f(E)$	fraction of transitions from a level which produce gamma-rays with energy E
$N(E)$	net counts in a full-energy peak due to a gamma-ray of energy E
$n(E)$	net counting rate in a full-energy peak due to a gamma-ray of energy E
$P(E)$	probability per decay or emission intensity per decay for a gamma-ray with energy E
$R(E)$	emission rate of gamma-rays with energy E by nuclei in a sample
$S_c(i, E)$	fraction of decays of radionuclide, i, in a sample which lead to counts in a full-energy peak corresponding to a gamma-ray of energy E
$f_{1/2}$	shaping-time constant (width of a shaped pulse at 50 % of its peak height)
$T_{1/2}$	half life
λ	decay constant, $\lambda = \ln(2)/T_{1/2}$
T_L	live-time counting period
T_r	real-time counting period
T_d	decay time from a reference time to the start of the counting period
$\alpha_K(E)$	K-shell internal conversion coefficient: the ratio of transitions of energy E that occur by K internal conversion to those that emit gamma-rays
$\alpha_t(E)$	total internal conversion coefficient: the ratio of transitions of energy E that occur by internal conversion to those that emit gamma-rays
$\varepsilon(E)$	full-energy-peak efficiency for gamma-rays of energy E
ε_r	relative full-energy-peak efficiency for detector specification
$\varepsilon_t(E)$	total efficiency for detecting a gamma-ray of energy E and producing a pulse with a height greater than the noise level of the amplifier
ω_K	K-shell fluorescence yield: the fraction of atomic K vacancies which lead to K X-rays
Π	operator that takes the product of the correction factors

4 Installation of instrumentation

Locate the HPGe spectrometer where stable, clean power is available or provide it via a power conditioner or uninterruptable power supply. Assure that the ambient temperature is stable and complies with the operating conditions specified by the manufacturer. Shield the spectrometer from interfering background radiation, including natural radiation emitted from the walls, floor, and ceiling, or resulting from cosmic-rays. The detector should be isolated from vibration producing equipment (see also Annex D).

Precaution – When filling the detector dewar with liquid nitrogen (LN_2), avoid spilling it in the vicinity of the cryostat or preamplifier which could lead to cracks in the vacuum seal rings of the cryostat or damage to the preamplifier. To avoid distortion of the measured spectrum by microphonics, data should not be acquired during the filling process.

Recommendation – Often the instrumentation associated with a HPGe spectrometer is located in more than one equipment rack or in two or more locations so that all components are not plugged into the same circuit or do not share the same ground. This can result in ground loops that adversely affect the detector resolution. To avoid such loops, a single connector box should be used into which all the instruments can be plugged. This box, in turn, should be plugged into a convenient main receptacle. Also, it is desirable to bundle together all the cables leading to the preamplifier, such as the cables from the detector-bias supply, the signal- and test-pulse cables, etc.

Follow the manufacturer's instructions and the procedures in Annex A for installation and preliminary testing of the individual components. Measure the energy resolution (i.e., FWHM, FWTM, FWFM) at 122 keV (^{57}Co) and at 1 332 keV (^{60}Co), and the peak-to-Compton ratio at 1 332 keV as described by the detector manufacturer's user manual. IEC 60973 and Annex A of this document may be useful in performing the above adjustments and measurements.

The experimental conditions recommended by the manufacturer for initial installation of the instrumentation are usually intended to assure that the HPGe spectrometer meets the various specifications (e.g., energy resolution) claimed by the manufacturer. These conditions are often not optimum for routine analyses. Therefore, the user should adjust the various spectrometer settings, including the amplifier shaping-time constant, baseline restorer, pole-zero cancellation, etc., for optimal performance under routine counting conditions before conducting the performance tests in this document. For example, timing of any blanking signals for reset preamplifiers should be checked.

If the measured values do not meet the manufacturer's specifications, the calibration should not proceed until the problem has been investigated and corrected where possible. All settings and experimental conditions shall be documented in a logbook for each specific HPGe spectrometer, and any changes or maintenance shall be so indicated at the time they occur.

5 Peak analysis and calibration procedures

5.1 Energy calibration

The energy calibration (gamma-ray energy as a function of channel number) of the HPGe spectrometer system shall be established by measuring the positions of selected full-energy peaks with large peak-height-to-background ratios and whose energies are known precisely. The energies of the selected peaks should cover the energy range of interest. Laboratories may have detectors that can cover different energy ranges so the ADC zeros and discriminators along with the gains should be adjusted as needed. A procedure to set the ADC zero and gain is described in A.3.3. If all detectors in a laboratory need to cover the same energy range, then it is useful to adjust the ADC zeros and discriminators along with the gains to give the same gain for all spectrometers. This allows the direct comparison of spectra from different detectors.

The calibration source should be counted for a duration that gives sufficient counts (i.e., at least 20 000) in the peaks to permit measurement of the peak positions with a precision of <0,2 keV. The peak positions at the calibration energies should be used to specify the coefficients of the fitting function (linear or quadratic) which are stored for subsequent evaluation of the energies of the measured peaks in the sample spectra. Differences of the order of 0,5 keV in the peak positions of the sample spectra are usually sufficient for correct radionuclide identification. If a linear or quadratic function does not adequately fit the entire energy range, it may be necessary to correct further for the pulse height nonlinearity. Sources that are useful for measuring the pulse-height nonlinearity are ^{152}Eu or ^{110m}Ag and ^{56}Co or ^{88}Y or ^{144}Ce/^{144}Pr or a mixed gamma source (see A.8.2).

Recommendation – The calibration of the FWHM and low-energy tail of the peaks is useful for automatic peak finding routines. This calibration can be performed simultaneously with the energy calibration.

5.2 Energy resolution calibration

The energy resolution is an important parameter used in the peak-finding algorithms, and in the peak area determination. The peak width is affected by different factors, such as electronic noise or counting rate, and depends on the deposited energy. The energy resolution calibration shall be established in the same way as the energy calibration, by determining the full-width at half maximum (FWHM) of selected full-energy peaks in the energy range of interest. The experimental conditions should follow the same criteria as for the energy calibration (both calibrations can be performed simultaneously). In a first approximation, the square of the FWHM should be a linear function of the energy.

5.3 Peak-finding algorithm

If an automated peak-finding routine is used in the spectral analysis, it should be able to find small, well-formed single peaks whose areas are statistically significant (above continuum). The peak-fitting routine should be able to find multiplet peaks (and discern if there is a single or multiple peaks) that meet the peak-area criterion for a singlet, are of the same intensity and are separated by one full-width-at-half-maximum (see Clause 8 for the test procedures). Optimization of the peak-search parameters of the peak-finding algorithm is left to the user or software vendor (see Annex B). These should be adjusted so that statistically significant peaks are found with a minimum number of false peaks being reported (see performance tests in Clause 8). Alternatively, it is possible to use a library to search for peaks at particular energies in the spectrum.

5.4 Peak position and area measurement

Any method that consistently produces satisfactory results for the measuring conditions and analysis requirements may be used to determine the peak position and area. Nonlinear and linear peak-fitting methods are primarily useful for complex spectra containing multiplet peaks. In general, more experience is required of the person using these more sophisticated peak-fitting methods. Gamma-ray spectroscopists should be thoroughly trained before using nonlinear fitting functions containing both a Gaussian and a tailing function. In all cases the same peak-area measuring technique should be used for the analysis of unknown samples as is used to measure the detector efficiency. Analyses of single gamma-ray peaks (i.e., >3 FWHM separation) are recommended over those with overlapping interferences (i.e., <3 FWHM separation) whenever there is a choice.

Warning – The peak-fitting algorithm may influence the energy dependence of pile-up corrections (see 7.6 and Clause A.7).

Recommendation – It is very helpful to be able to evaluate the quality of any peak fit visually through a display of the spectral counts in the peak region with the fitted function and continuum superimposed. This capability may permit resolution of questionable results. A display of the residuals between the fitted function and data in units of the estimated standard deviation of the data is also recommended.

5.5 Efficiency calibration measurement

5.5.1 General

Three separate approaches are considered for the calibration of the detector efficiency:

a) measurement of a standardization coefficient for a specific gamma-ray and radionuclide by direct comparison with a standard source of known activity,

b) measurement of the full-energy-peak efficiency as a function of energy, and

c) calculations or modelling. These can be used in place of measurements to deduce efficiencies, or to infer efficiencies for one counting geometry from those measured for another; however, satisfactory agreement shall be demonstrated by direct measurement for at least one typical geometry. Monte Carlo simulations may be used to determine the detector efficiency. This will require knowledge of all the detector dimensions that shall be provided by the detector manufacturer. Validation measurements shall be performed to assess the accuracy of the simulations.

For approach b) two cases are considered:

1) source-to-detector distances for which essentially no need exists for true coincidence summing corrections, that is, greater than 10 cm, and
2) small source-to-detector distances that are required for sensitivity (low-activity samples) but may compromise the accuracy of the source configuration geometry and add the requirement for true coincidence summing corrections.

Clause A.8 contains efficiency measuring procedures for each of these experimental conditions.

5.5.2 Standardization coefficient for specific radionuclides

When the activity or gamma-ray emission rates of a limited number of radionuclides are to be measured, it is most accurate to measure them relative to a standardized sample of the same radionuclide in exactly the same source geometry and matrix. From measurements on the standardized samples, a standardization coefficient can be calculated for a selected gamma-ray emitted from each specific radionuclide. The standardization coefficient converts the net peak area divided by the live time to the activity of the radionuclide being measured. The need to interpolate between measured efficiency values, to correct for true coincidence summing, and to account for the uncertainty in the gamma-ray emission intensity is eliminated with this method. If the counting rates of the samples are low or closely approximate those of the standard, pulse pile-up corrections can also be neglected.

Severe true coincidence summing of cascade gamma-rays or of X-rays from electron capture or from electron conversion with coincident gamma-rays can occur, especially when counting samples located inside the well of a HPGe well detector (also see first precaution in 6.2.5). Therefore, the standardization coefficient method is the efficiency calibration technique recommended for these or similar experimental conditions.

5.5.3 Detector efficiency as a function of energy

5.5.3.1 General

The general method for measuring the emission rates of gamma-rays is by establishing the detector efficiency curve as a function of energy for a defined geometry and energy range. The efficiency values at intermediate energies are determined by interpolation between measured values. With this method, corrections may be required for pulse pile-up and true coincidence summing of gamma-rays. The corresponding radionuclide activities can be calculated from the gamma-ray emission rates when the gamma-ray emission intensities are known.

5.5.3.2 Large source-to-detector distances

At large sample-to-detector distances (typically, a source-to-detector distance >10 cm) the uncertainty in the replication of the sample-detector geometry – and in the correction for true coincidence summing of photon pulses in the detector – can usually be neglected for routine measurements. For these reasons, measurements at large sample-to-detector distances are preferred over measurements close to the detector, provided the source activity is high enough to achieve an acceptable accuracy within the allotted counting time.

5.5.3.3 Small source-to-detector distances

The analysis of low-activity-level samples is performed at many different types of nuclear facilities. Effluent and environmental samples typically require small sample-to-detector distances and long counting times. These experimental conditions can result in large uncertainties/errors due to large background contributions, non-reproducibility of sample-to-detector distance, and large true coincidence summing effects resulting from two or more coincident gamma-rays from the same radionuclide. For example, an uncertainty of ±1,0 mm in the source-to-detector distance reproducibility at 5 cm results in an additional relative uncertainty of approximately 4 % in the net peak area for a point source and a 5 cm diameter HPGe detector. A similar size or larger uncertainty can result from the true coincidence summing of gamma-rays at a 5 cm source-to-detector distance for a point source depending on the radionuclide decay scheme.

Whenever possible, a sample should be at least 5 cm from the detector. True coincidence summing corrections should be applied to all peaks warranting such corrections. This is especially important when the source-to-detector distance is <5 cm (see 6.2.5). Large sample containers have a large mean source-to-detector distance even when they are in close proximity to the detector. This effect reduces the true coincidence summing effect.

5.5.3.4 Reentrant (Marinelli) beakers

The reentrant (or Marinelli) beaker (IEC 60973) is a beaker with a recessed well in its bottom that allows the beaker to fit over and around the detector housing. This geometry provides high sensitivity for the measurement of gamma-ray-emitting radionuclides. Depending on the size of the reentrant beaker and the detector efficiency, significant true coincidence summing corrections may be required and corresponding corrections should either be estimated or applied. The test described in 9.1 may be useful in demonstrating if true coincidence summing corrections can be avoided.

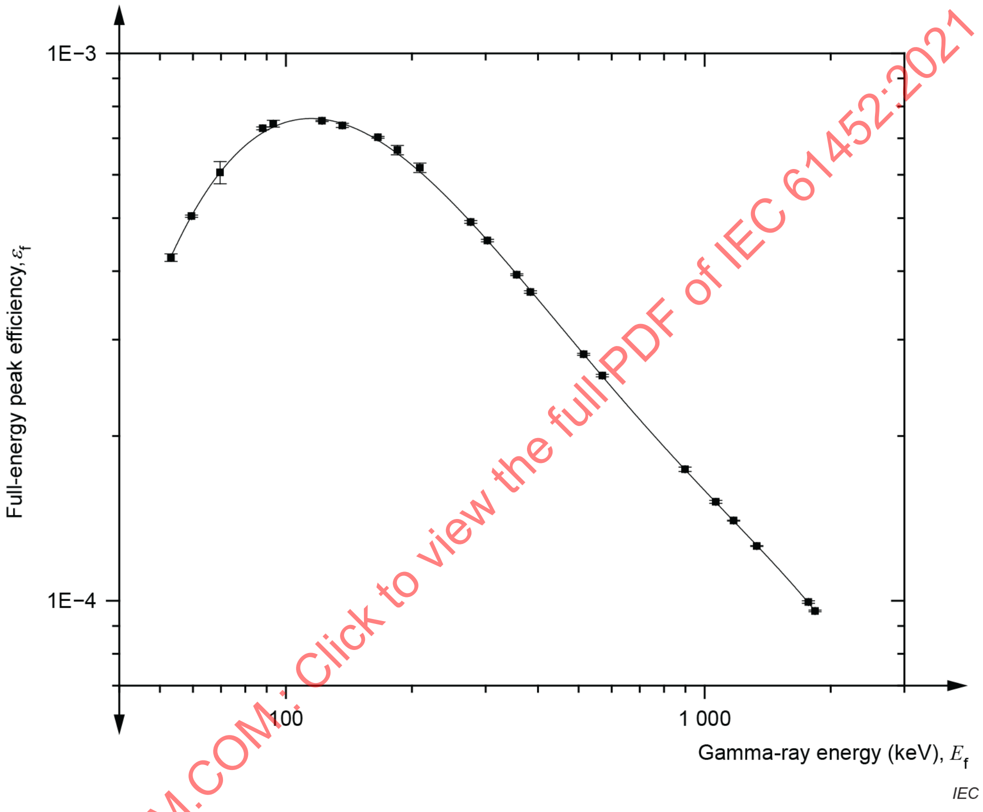
5.5.4 Efficiency function

The full-energy-peak efficiency values, $\varepsilon(E)$ for energy E, should be plotted either a) as $\varepsilon(E)$ on a log-log graph (see Figure 1) or b) as the function $Y = \varepsilon(E) \times E^a$ on a linear plot (a is a normalizing constant as shown in Figure 2); this latter function provides better resolution of the efficiency data when plotted.

There should be no large energy gaps between the efficiency values (especially below 300 keV) that would cause a large efficiency-interpolation uncertainty. Efficiency values for energies below the lowest measured energy efficiency point shall not be used to measure gamma-ray emission rates. Above approximately 300 keV the efficiency, as a function of energy, approximates a straight line to a few percent when plotted on a graph with a log-log scale. For many detectors, only a small deviation from this straight line exists between 500 keV and 1 000 keV. For 2 MeV < E < 4 MeV efficiency values may be estimated, with some loss in accuracy, by linear extrapolation beyond the highest measured efficiency value. However, it is best to avoid extrapolation beyond the highest measured efficiency.

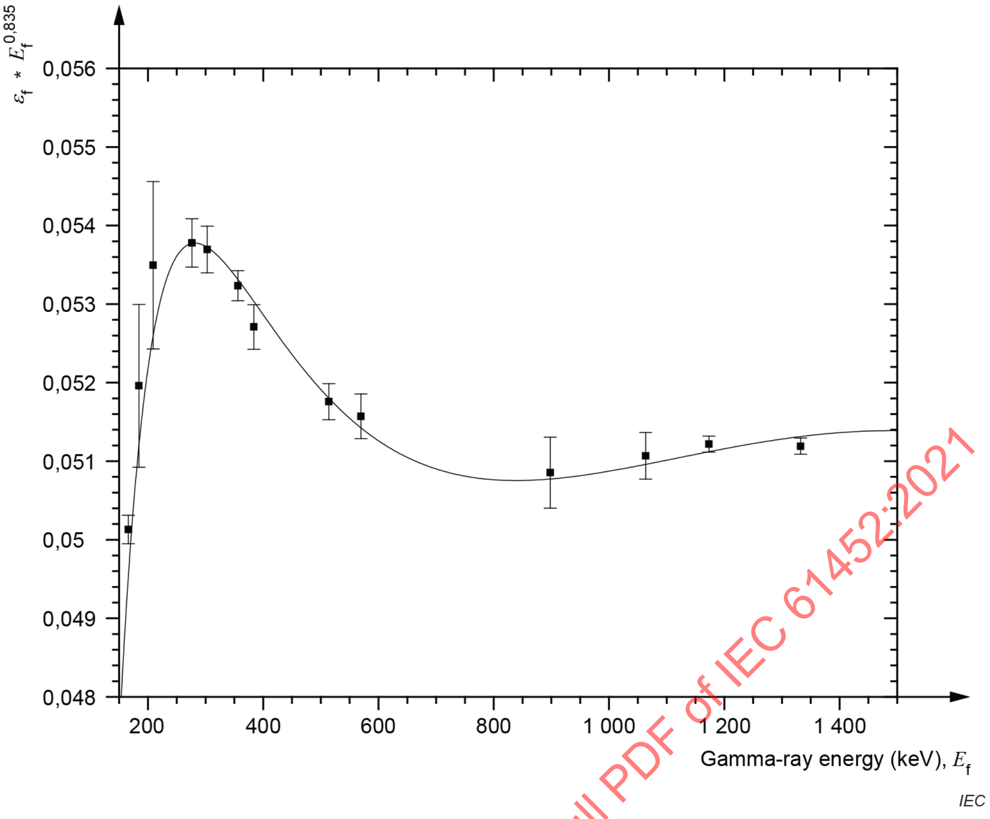
When fitting efficiency data with a function, the number of data points being fitted should exceed the number of functional parameters to be determined by about a factor of two and no large gaps should be present within the energy range being fitted if oscillations in the fitted function are to be avoided. Extrapolation of a fitted function beyond the measured efficiency data is subject to large biases.

Recommendation – Different functions may be used for the fit. The residuals of the fit and χ^2 divided by the degrees of freedom can provide information about the goodness of the fit. Comparing different fits can provide information that could allow for the selection of the best possible fit for the efficiency data. The user can define two (or more) energy regions to fit the efficiency data: the consistency between the set of fits should be checked around the cross points.



NOTE Efficiency as a function of energy on a log-log plot for a point source at a 45 cm source-to-detector distance for a coaxial HPGe spectrometer.

Figure 1 – Full-energy-peak efficiency as a function of gamma-ray energy



NOTE Part of the data in Figure 1 plotted as $\varepsilon_f E_f(\text{keV})^{0,835}$ versus gamma-ray energy $E_f(\text{keV})$, where ε_f is the full-energy peak efficiency at $E_f(\text{keV})$ and where $E_f(\text{keV})$ is the gamma-ray energy.

Figure 2 – $\varepsilon_f E_f(\text{keV})^{0,835}$ as a function of gamma-ray energy

6 Gamma-ray measurements with HPGe spectrometers

6.1 Measurement of gamma-ray energies

The gamma-ray energies are normally derived from a previous energy calibration (see 5.3 and 5.4). Sometimes it is desirable to derive the energies directly from the spectrum. In this case, calibration shall be performed using identified gamma-rays having accurately known energies.

6.2 Measurement of gamma-ray emission rates and radionuclide activities

6.2.1 General

The sample shall be counted in the same source-to-detector geometry as used for the establishment of the efficiency unless the differences can be accurately compensated for by calculation. The gamma-ray spectrum shall be accumulated at the same gain as used in the energy calibration and for a counting time sufficient to give an acceptable statistical uncertainty. The energies of the gamma-ray peaks present in the spectrum shall be identified by use of the energy calibration described above.

Warning – If the sample geometry (e.g., source-to-detector distance, source type) is not the same as for the source used to establish the efficiency, then additional corrections to account for differences in solid angles, source area/volume and/or matrices are needed.

Obtain the net area counting rate in each of the gamma-ray peaks of interest as described in 5.4. Obtain the full-energy-peak efficiency for each of the peaks of interest from the efficiency function as described in 5.5.4. Calculate the number of gamma-rays emitted per second, $R(E)$, for each of the full-energy peaks as follows:

$$R(E) = (n(E) - B(E))\frac{\prod C_\mathrm{i}}{\varepsilon(E)} \tag{2}$$

where

$n(E)$ is the net counting rate in the full-energy peak at energy E; $N(E)/T_\mathrm{L}$, where $N(E)$ is the net peak area and T_L is the counting live time;

$B(E)$ is the net counting rates of any interfering peaks in the background;

$\varepsilon(E)$ is the full-energy peak efficiency;

C_i are the multiplicative correction factors (where i = d, r, c and a, as defined below); and

Π is the operator that takes the product of the correction factors.

The correction factors, C_i include correction for decay during the counting period ($\mathrm{i = d1}$) and from the start time of counting period to a reference time ($\mathrm{i = d2}$), pulse pile-up ($\mathrm{i = r}$), true coincidence summing ($\mathrm{i = c}$), and differences in the attenuation of the sample counting geometry from that in the standard ($\mathrm{i = a}$). Each correction factor shall be applied whenever its magnitude is expected to exceed one third of the acceptable inaccuracy in the resulting gamma-ray emission rate.

If there is only one nuclide contributing to the full energy peak, the nuclear decay rate or activity (in units of becquerel, Bq) is calculated by dividing the gamma-ray emission rate (in units of s^{-1}) by the appropriate gamma-ray emission intensity, $P(E)$, see formula (3).

$$A = \frac{R(E)}{P(E)} \tag{3}$$

6.2.2 Subtraction of interference peaks in the background

Large samples may shield the detector from the ambient background radiation, causing a lower counting rate than if an instrumental background (no sample present) were obtained. In addition, all soils contain some natural ^{40}K, Th and U radioactivity. Thus, whenever large low-activity samples are counted, a background spectrum shall be obtained with a "blank" sample in the same geometry and of the same density. The background count shall be accumulated for at least the same counting time as used in counting the samples to be analysed.

The "blank" sample background spectrum, normalized for the same counting time, could be subtracted from the sample spectrum, channel-by-channel, or preferably, the background spectrum should be analysed to obtain the background-peak counting rates, $B(E)$, at energies E. If the latter method is used (it is preferred), the background-peak counting rates are then subtracted from the respective net peak area counting rates, $n(E)$, in the sample spectrum (see formula (2)). By subtracting the respective background peak counting rates from those of the sample, the statistics of each count are preserved. This method is also less sensitive to small changes in the energy calibration. If possible, the "blank" spectrum should be scanned to be sure that all background peaks are identified and that no false peaks are reported.

6.2.3 Radioactive decay

6.2.3.1 General

The correction factor that normalizes for radioactive decay is normally composed of two components:

a) decay during the counting interval, and

b) decay from a previous time, usually a sample-irradiation time (e.g., neutron-activation analysis) or a sample-collection time or reference time.

When sampling radioactivity or performing an irradiation over a time period comparable to the half-lives of the radionuclides of interest, a third correction factor shall be applied. These correction factors will be designated C_{d1}, C_{d2}, and C_{d3}, respectively. The combined correction factor for radioactive decay can be expressed as:

$$C_d = C_{d1} \times C_{d2} \times C_{d3} \tag{4}$$

Figure 3 illustrates the time to which each correction is made for the decay correction formulas given in 6.2.3.2, 6.2.3.3 and 6.2.3.4.

6.2.3.2 Radioactive decay during the counting period

If significant decay occurs during the counting time (>10 %) it will be necessary to correct the net peak area so that the gamma-ray emission rates or the radionuclide activities are determined at the start time of the count. If this correction is not applied when decay during the counting time is significant, the measured gamma-ray emission rates will apply to sometime after the start of the count. The following formula gives this multiplicative correction:

$$C_{\mathrm{d1}} = \frac{\lambda T_{\mathrm{r}}}{1 - e^{-\lambda T_{\mathrm{r}}}} \tag{5}$$

where

C_{d1} is the correction factor for decay during the count;

λ is the decay constant, $\lambda = \ln(2)/T_{1/2}$ in which $T_{1/2}$ is the radionuclide half-life;

T_r is the real time of the count; and

e = 2,71828..., base of the natural logarithm.

This correction assumes a constant dead time during the acquisition period. A bias will be introduced if the dead time varies significantly.

6.2.3.3 Radioactive decay from the start time of counting period to a reference time

The correction for radioactive decay to a reference time from the start time of a count is determined from the following formula:

$$C_{d2} = e^{\lambda T_{\mathrm{d}}} \tag{6}$$

where

C_{d2} is the correction factor for decay from the reference time to the start time of the count period;

T_d is the decay time from a reference time to the start of the counting period.

6.2.3.4 Correction during the sampling time or the irradiation period

Two options may be used to calculate the correction to the start of a sample activation at a constant rate during irradiation or a constant rate of activity collection (see Figure 3). If one is interested in the number of atoms in the unirradiated sample that are available for activation during an irradiation such as for neutron-activation analysis, then a saturated activity is useful (i.e., number of atoms which would be activated at saturation). In this option:

$$C_{\mathrm{d3}} = \frac{1}{1 - e^{-\lambda T_{\mathrm{i}}}} \tag{7}$$

where

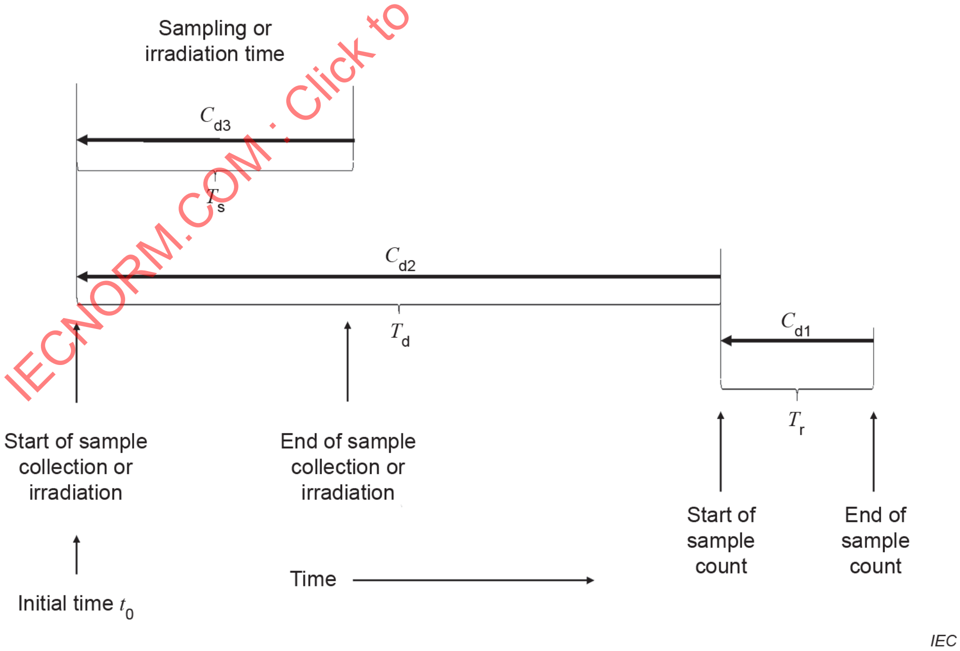
T_{i} is the irradiation duration.

If one is collecting at a uniform rate already activated nuclides, such as in the collection of radionuclides in reactor effluent studies, then a value representing the disintegration rate of the sampled stream at time T_{s} may be of interest (see Figure 3). In this case:

$$C_{\mathrm{d3}} = \frac{\lambda T_{\mathrm{s}}}{1 - e^{-\lambda T_{\mathrm{s}}}} \tag{8}$$

where

T_{s} is the sampling duration.



NOTE Specification of various events expressed in units of time, where t_0 is the initial time, T_{s} is the sampling or irradiation time, T_{d} is the decay time of the sample, and T_{r} is the real time. The arrows point to the times to which the respective decay corrections apply.

Figure 3 – Specification of times for decay corrections

6.2.4 Pulse pile-up (random summing)

The pulse pile-up correction shall be applied whenever the desired accuracy of the measurement requires it. The pulse pile-up correction, C_{r}, may be measured by one of several methods. Three popular methods are:

- method 1: use of a movable intense low-energy gamma-ray source and a fixed higher-energy source, or alternatively, use of a set of sources whose relative activities are precisely known with source counting rates ranging from about 300 s^{-1} up to about 40 000 s^{-1};
- method 2: use of live-time extension techniques whereby the live time is extended to compensate for displaced pile-up pulses; and
- method 3: the use of a precision pulser of known repetition rate (injected into the preamplifier test input).

Although each method gives a slightly different correction, the difference between methods is typically less than 20 % of the correction. The type of ADC and the type of peak-area measurement algorithm can affect the amount of energy dependency in the pulse pile-up correction.

A description of the source-set method is given in A.7.2. Descriptions of methods 2 and 3 for measuring the pulse pile-up correction can be found in A.7.3 and A.7.4, respectively, and the literature. The use of these or other types of electronic circuitry to correct automatically for pile-up is permitted if validated by passing the test in Pulse pile-up (random summing) described in 7.6.

Recommendation – Follow manufacturer provided recommendations when setting up the system. When analog electronics are used, the amplifier shaping-time constant should be selected to optimize the overall performance of the spectrometer when high counting rates are to be recorded (i.e., balance the energy resolution against counting rate performance). Larger shaping-time indices ($f_{1/2}$ > 5 µs) typically yield better energy resolution but poorer counting-rate performance. For amplifiers operating at an unipolar shaping-time constant of $f_{1/2}$ = 5 µs, the pulse width at the 1 % pulse-level is approximately 12 µs; at input counting rates <1 000 s^{-1}, the pulse pile-up correction is usually <1 % and may be neglected.

Warning – High counting rates can lead to dead-time inaccuracies in the ADC and to pile-up of pulses in the amplifier. Although the live timer of the multichannel analyser only records time while the pulse-processing circuitry is able to accept pulses, at very high rates (>10 000 s^{-1}), differences in the live timer can be significant (>5 %). For digital electronics, use the pile-up rejection provided by the manufacturer and verify that the peak area and resolution is not affected for the entire counting range needed.

Warning – Pile-up rejectors may be useful at high counting rates because they prevent the storing of piled-up pulses in the spectrum and thereby reduce the continuum between and underlying the spectral peaks. However, if the pile-up rejection process is amplitude dependent, unacceptably large errors in the correction of the net peak-area counting rates could result. Therefore, the use of pile-up rejection with or without live-timing or loss-free techniques is permitted only if the user demonstrates that it does not introduce an unacceptable error to the measurement, see 7.6. Available commercially are loss-free counting modules and programs that monitor the dead-time during data acquisition to compensate for a varying dead-time. The reader is referred to the manufacturer’s manuals, brochures and catalogues for further information. The performance of these systems shall be verified prior to use for constant counting rates with the performance test in Pulse pile-up (random summing) described in 7.6. The principles upon which some loss-free counting modules work may cause them to distort counting statistics.

6.2.5 True coincidence (cascade) summing

True coincidence summing corrections shall be applied whenever counting conditions result in significant coincident summing of gamma-rays associated with the radionuclides of interest. It should be assumed that these corrections are necessary for all counting geometries in which the mean sample-to-detector distance is <10 cm unless demonstrated otherwise.

Coincidence summing depends on the radionuclide decay scheme and on the detector efficiency. There are two kinds of effects: "summing-out" which results in the removal of counts from the full energy peak (FEP) and "summing-in" which results in adding counts in the FEP (e.g., the low-probability 2 734 keV crossover peak in the decay of ^{88}Y can contain counts from the summing of the high-probability 898 keV and 1 836 keV cascade gamma-rays). Programs developed for use in determining true coincidence summing corrections, C_c, for specific radionuclides and source-detector geometries are described in the literature ([2][1], [28], [29], [42], [43]). A brief description of true coincidence summing, and the equations needed to determine the loss or gain of counts to a gamma-ray peak is provided in Annex C.

Precaution – Characteristic X-rays produced from electron-capture and internal-conversion transitions also can sum with gamma-rays when thin-window detectors are used to measure both low-energy X-rays and gamma-rays. One alternative to reduce this effect is to place an absorber (e.g., 0,5 mm thick copper, 1 mm thick tin) between the source and the detector if this practice does not interfere with the basic intent of the measurement. Photon attenuation corrections shall be applied when using an absorber (including beta absorbers) if the efficiency curve was not obtained with the absorber in place. If software is used to perform the coincidence summing corrections, there is no need to add the absorbers.

Precaution – Beta particles and associated bremsstrahlung emitted in the decay of beta-emitting radionuclides can contribute significantly to the spectral continuum. Further, the gamma-ray transitions promptly follow the beta radiation (assuming no isometric transitions). The coincidence summing of beta particles and associated bremsstrahlung with gamma-rays is not included in most true coincidence summing correction programs. Under these circumstances, insertion of a low-atomic-number absorber between the source and the detector can significantly reduce the detection of beta particles and thereby reduce their summing with gamma-rays. The correction for gamma-ray attenuation in the beta-particle absorber shall be included in the efficiency or provided as a separate correction.

6.2.6 Efficiency transfer corrections

6.2.6.1 General

Whenever counting geometrical conditions are different from the calibration ones, efficiency transfer corrections shall be introduced, these corrections should consider:

- changes in the sample-to-detector distance,
- differences in shape, matrix and density of the sample,
- external absorbers.

Efficiency transfer methods (numerical or Monte Carlo) can be used to compute these corrections (see Clause 12). In simple cases, where there is no change in the geometry, the attenuation correction can be computed as the product of two components which take account of external absorbers, C_{a1}, and differences in the matrix or density of identically shaped samples, C_{a2}. These corrections are then combined as:

$$C_a = C_{a1} \times C_{a2} \quad (9)$$

where

C_a is the attenuation correction.

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[1] Numbers in square brackets refer to the Bibliography.

6.2.6.2 Attenuation external to the sample

When an absorber is inserted between the sample and the detector, the correction for attenuation in the absorber is given by:

$$C_{\mathrm{a1}} = e^{\mu\rho d} \qquad (10)$$

where

μ is the mass attenuation coefficient (depending on the energy and the absorber material);

ρ is the density of the absorber; and

d is the mean thickness of the absorber.

For small sample-to-detector distances this formula may need to be modified or the correction measured directly.

Recommendation – The mass attenuation coefficient for different elements, compounds and mixtures can be determined experimentally [51] and can also be found, for example, at: https://physics.nist.gov/PhysRefData/Xcom/html/xcom1.html

6.2.6.3 Attenuation within the sample (self-attenuation)

6.2.6.3.1 General

When it is not possible to measure the full-energy-peak efficiency with a standard of the same matrix and density as the sample, an attenuation correction beyond that included in the full-energy-peak efficiency may be required (physical configurations of the standard and sample shall be the same). When the error resulting from no correction is expected to be significant, a correction shall be made. This correction is most important for low-energy gamma-rays or X-rays and samples containing high-atomic-number elements.

6.2.6.3.2 Calculation for uniform plane sample

The correction for self-attenuation in a homogeneous, extended sample may be calculated by numerical integration (of the transmission through a material layer, according to Beer-Lambert law) over the sample thickness. For source-to-detector distances that are large relative to the dimensions of a uniform plane sample, the relative correction for self-attenuation (not included in the measured efficiency) may be estimated to be:

$$C_{\mathrm{a2}} = \frac{\mu_{\mathrm{u}}(E)\,\rho_{\mathrm{u}}\,d}{1 - e^{-\mu_{\mathrm{u}}(E)\,\rho_{\mathrm{u}}\,d}} \times \frac{1 - e^{-\mu_{\mathrm{s}}(E)\,\rho_{\mathrm{s}}\,d}}{\mu_{\mathrm{s}}(E)\,\rho_{\mathrm{s}}\,d} \qquad (11)$$

where

$\mu_{\mathrm{u}}(E)$ is the mass attenuation coefficient at energy E of sample;

$\mu_{\mathrm{s}}(E)$ is the mass attenuation coefficient at energy E of efficiency-measuring standard;

ρ_{u} is the density of sample;

ρ_{s} is the density of efficiency-measuring standard; and

d is the thickness of the standard/sample.

6.2.6.3.3 Measured correction

The attenuation correction (relative to that of the efficiency-measuring standard) for samples of the same shape but composed of a different matrix/density may be measured. A standard of the same shape but different matrix/density with gamma-rays covering the energy range of interest should be counted under the same conditions as used to determine the efficiency curve. The emission rates of the gamma-rays from the original efficiency-measuring standard and from the present attenuation-correction standard shall be known with sufficient accuracy to determine the attenuation correction with adequate precision.

The relative attenuation correction, C_{a2}, can be calculated from:

$$C_{\mathrm{a2}} = \frac{R(E)\,\varepsilon(E)}{(n(E) - B(E))\prod_{\mathrm{i}\neq\mathrm{a}} C_{\mathrm{i}}} \tag{12}$$

where

$R(E)$ is the emission rate of the gamma-ray at energy E in the attenuation-correction standard;

$\varepsilon(E)$ is the full-energy-peak efficiency at energy E;

$n(E)$ is the net peak-area counting rate for the attenuation-correction standard of a gamma-ray of energy E;

$B(E)$ is the net peak-area counting rate of any background peak at energy E;

Π is the operator that takes the product of the correction factors; and

C_i are the corrections for counting rate (i = r); true coincidence summing (i = c); and decay prior to and during the count (i = d) for the attenuation-correction standard.

An alternative and probably more accurate method to measure the relative attenuation correction is with two uncalibrated samples identically shaped but with different matrices and with each uniformly spiked with an accurately measured aliquant, M, of a gamma-ray emitting radionuclide (see Clause A.9 for the preparation of working standards). The matrix/density of one sample, s, is identical to that of the standard used to measure the detector efficiency and the other sample, u, is the same as the samples whose density is different from the efficiency-measuring standard. Assuming corrections for pulse pile-up can be neglected, for a given energy, E, the relative attenuation correction, C_{a2}, is given by:

$$C_{\mathrm{a2}}(E) = \frac{(n_{\mathrm{s}}(E) - B_{\mathrm{s}}(E))\,M_{\mathrm{u}}}{(n_{\mathrm{u}}(E) - B_{\mathrm{u}}(E))\,M_{\mathrm{s}}} \tag{13}$$

where

$n_{\mathrm{s}}(E)$ and $n_{\mathrm{u}}(E)$ are the net counting rates in the peak areas of the standard and sample, respectively, at energy E;

$B_{\mathrm{s}}(E)$and $B_{\mathrm{u}}(E)$ are the net counting rates in the peak areas of the associated background spectra;

M_{s} is the mass of aliquant used to prepare the working standard whose matrix is identical to that of the standard used to measure the detector efficiency;

M_{u} is the mass of aliquant used to prepare the sample whose matrix is identical to that of the unknown samples to be measured.

7 Performance tests of the spectrometry system

7.1 General

The spectrometer tests enumerated in 7.2 to 7.6 shall be performed on a regularly scheduled basis. The frequency for performing each test should depend on the long-term stability and maintenance record of the particular system. A range of typical frequencies for noncritical applications is recommended with each test. In addition, the performance tests shall be made after an event (e.g., change of the settings of an electronic module, repair to the detector or the pulse-processing circuitry) that might result in changes to the system. The results of these performance tests shall be maintained as plots, tables or both. When the measured parameter falls outside reasonable limits, either the cause of failing the test shall be found and corrected or the uncertainty associated with the tested parameter shall be increased sufficiently to avoid quoting unrealistically small uncertainties in the reported gamma-ray emission rates. Actions taken and corrections made should be appropriately documented.

Some hardware allows for direct readout of, for example: leakage current from the preamplifier or cooling power from an electrically cooled detector. These parameters can be logged and tracked for assessing the state-of-health of the detection system.

7.2 Multichannel-analyser and digital signal processing clocks

The real-time clock shall be checked at the time the acquisition electronics is installed and after any maintenance. Follow the instructions in the manufacturer's user manual (also see Clause A.4).

Recommendation – It is possible to synchronize the computer clock associated with the multichannel-analyser using an atomic clock synchronization tool.

7.3 DC offset and pole-zero settings

The DC offset and the amplifier pole-zero settings shall be checked periodically (preferably quarterly or yearly) and after any related maintenance performed on the spectrometer. Follow the instructions in the manufacturer's user manual (also see Clause A.2).

7.4 Energy calibration

The system energy calibration shall be checked periodically (typically daily to semi-weekly) by counting a source(s) emitting gamma-rays whose energies are precisely known and cover the low-energy and high-energy regions of interest (e.g., ^{57}Co and ^{60}Co). A spectrum shall be acquired, and the peaks centroids determined. Compare the gamma-ray energies of interest with the peak centroids, if the peak centroids are outside the desired accuracy (usually, ±0,5 keV) from the expected gamma-ray energies then perform the energy calibration procedure as described in 5.3.

NOTE Some software packages use a gamma line in the energy spectrum to stabilize the system to maintain the energy calibration by adjusting the gain.

7.5 Spectrometer efficiency and energy resolution

The reproducibility of the full-energy-peak efficiencies and energy resolution shall be checked periodically (daily to weekly checks are recommended) using a radioactive source with a long half-life emitting at least a low-energy (about 100 keV) and a high-energy (>1 300 keV) gamma-ray (e.g., mix of ^{125}Sb, ^{154}Eu and ^{155}Eu, or ^{57}Co and ^{60}Co, or ^{152}Eu sources). The same counting geometry shall be used each time for these checks with a reproducible source-to-detector distance (preferably >10 cm). A spectrum with at least 20 000 counts in the full-energy peaks of interest (approximately 0,7 % counting uncertainty in the net peak areas) shall be acquired. Deviations in the peak counting rates, corrected for decay, of reference peaks that lie more than two estimated standard deviations outside of the mean shall be investigated. An alternative test is to apply a chi-square (χ^2) test to the measured net area counting rates of the reference peaks over time (see [4] and [5]).

The energy resolution at both high and low energies should be measured and recorded during these tests and any unexpected increase in the peak width investigated. A test similar to that given in Clause A.6 is recommended. A loss in resolution of the full-energy peaks can be due to noise in the electronics, a degradation in the charge collection in the detector crystal, or a degradation in the detector-cryostat vacuum causing an increase in the electronic noise. Loss of energy resolution may result in an increase in the uncertainty in the measured gamma-ray peak areas.

7.6 Pulse pile-up (random summing)

Laboratories with heavy sample loads may optimize the sample throughput by increasing the counting rates with minimal loss in peak position and area measurement accuracy. Therefore, operation of HPGe spectrometers at counting rates up to 20 000 s^{-1} and even higher may be desirable if pulse pile-up corrections are applied (see Clause A.7 for a description of the various correction techniques, and their limitations). The following test shall be performed to verify that the correction at high counting rates is being performed accurately. Peak analyses which define the peak area to include a large fraction of the tails are less likely to have energy-dependent corrections. If counting rates are kept below 1 000 s^{-1} (for amplifier time constant or shaping-time constant less than 3 μs), this test may not be required, and no correction may need to be applied.

Method of test:

This test is designed to check the pulse pile-up correction at two energies, 662 keV (^{137}Cs) and 2 614 keV (^{208}Tl). The gain should be adjusted to be the same as used in counting samples. Similar tests may be designed to check the pulse pile-up correction at lower or higher energies.

a) Accumulate a ^{137}Cs spectrum with a source that yields a counting rate between 200 s^{-1} and 500 s^{-1}. Collect at least 20 000 and preferably 50 000 counts in the 662 keV full-energy peak (net area). Record the real time or live time (if the use of live time constitutes a part of the correction method). The ^{137}Cs source will be referred to as the fixed source.

b) Determine the net counting rate and its uncertainty for the 662 keV peak area using the same method normally used to evaluate the peak area, including methods employed to correct for pile-up and dead time.

c) Without moving the ^{137}Cs source, introduce a high-activity ^{57}Co or other essentially mono-energetic low-energy (100 keV < E < 200 keV) source of gamma-rays. Position the added source behind the fixed ^{137}Cs source so that the gross counting rate is about the maximum counting rate to be encountered in the radionuclide analysis of samples.

d) Erase the first spectrum and accumulate another spectrum for the same live time as in step a).

e) Evaluate and correct the net counting rate of the 662 keV peak as in step b) (including any method employed to correct for pile-up and dead time). Propagate the uncertainty in the net area of the 662 keV peak with that in the correction factors (see Clause 11).

f) (optional) Repeat steps a) through e) for a fixed source of ^{228}Th and a high activity ^{137}Cs source (^{232}U, which is the parent of ^{228}Th, has a half-life of about 70 years and may be a substitute for ^{228}Th). Any difference between the corrected peak counting rate of the stationary source and that measured in step a) lying outside twice the uncertainties should be investigated.

To pass this test at the desired maximum counting rate, the difference with respect to the measured peak counting rate shall not exceed one-third of the acceptable total inaccuracy of the measured gamma-ray emission rate for routinely counted samples. Failure in a repeat test is an indication of the error associated with this correction.

8 Performance tests of the analysis software

8.1 General

These tests are indications of performance of the software analysis algorithms and may assist the user in setting discretionary limits in their parameters of the analysis algorithms. The gamma-ray analysis program should be demonstrated to find the peaks in a spectrum above a minimum “observable" level and measure their net peak area with associated uncertainties lying within two estimated standard deviations of their "true” values (a singlet peak with no appreciable continuum). If commercial software is purchased, the tests in this section using that software may be performed by the vendor with the analysis parameters and results of each individual test documented to the user. Documentation of acceptable results by the software vendor shall show sufficient evidence of the capability of the software algorithms to justify not repeating the tests in this clause (vendor tests shall be conducted with the software version to be used in practice). However, it is recommended that the user perform these tests to assure that the analytical parameters are optimized for the users’ applications.

If the software used allows for extracting and importing spectrum simulations, the time-consuming measurements listed in Clause 8 can be replaced by Monte Carlo simulations.

In the following tests, ^{65}Zn and ^{137}Cs sources are required (^{22}Na or ^{60}Co may be substituted for ^{65}Zn, but if ^{60}Co is used, a correction may be needed for the presence of the single-escape peak of the 1 173 keV gamma-ray at 662 keV). The purity of the ^{65}Zn or alternatively the ^{22}Na or ^{60}Co source should be examined prior to the tests in 8.2 and 8.3 by counting the source at a moderate counting rate (between 500 s^{-1} and 2 000 s^{-1}) and at a reproducible source-to-detector distance (to be used in 8.2 and 8.3 tests) until 10 000 counts are in each of the channels surrounding the 662 keV energy region. The counting rate should be high enough to overwhelm the normal background. At the end of the counting period examine the 662 keV energy region to be assured that no peaks are within 5 keV of 662 keV, and that the continuum in the 662 keV region is flat. The peak-finding algorithm may be applied to the spectrum to confirm that the 662 keV energy region is free of any peaks. Any visually observed peaks within 5 keV of 662 keV should be avoided, if possible, since it will limit the sensitivity of the tests. The net peak-area counting rate of an unavoidable interference peak should be measured so that its contribution can be subtracted.

The continuum counting rate in counts per channel per second shall be recorded as determined from the counting time and average number of counts in each continuum channel for use in 8.2 and 8.3 tests.

Erase the previous spectrum and count the ^{137}Cs source at a moderate counting rate (between 500 s^{-1} and 2 000 s^{-1}) and at a reproducible source-to-detector distance (distance at which this source is to be used in the Clause 8 tests) for a 1 000 s counting time or until the 662 keV net peak area has 5 000 counts. Record the FWHM of the 662 keV peak in channels and the net peak-area counting rate for use in 8.2 and 8.3 tests.

8.2 Test of automatic peak-finding algorithm

The following test has been designed to determine how well singlet peaks on a flat continuum at or above an "observable" level can be found (i.e., detected) with the peak-finding algorithm. This test is not intended to demonstrate, nor should it be confused with the ability to measure the lower limits of detection at a given level of confidence. It shall be performed prior to sample analyses that depend on the automatic finding of the full-energy gamma-ray peaks in a spectrum. Because the sensitivity of peak-finding algorithms is usually adjustable, this test should be performed for every sensitivity chosen for routine analysis. For this test the gain should be the same as used when counting samples for radioanalysis.

This test shall be used to satisfy the criteria set forth in 5.1 regarding the ability of the peak-finding algorithm to automatically find singlet peaks.

a) With the ^{137}Cs source (positioned at the same source-to-detector distance as selected in Clause 8), accumulate a spectrum for a counting time that will result in the full-energy 662 keV peak having an expected net area of about 50 counts.

b) Execute the peak-finding algorithm routine. It shall find the peak every time in five measurements as described in step a). (This requirement was chosen to avoid one detection by chance in a single trial and the extensive time required to test more severe statistical criteria.)

Precaution – Some peak-finding algorithms require that the counts comprising the peak vary smoothly from channel to channel. In this case, peak areas with less than 50 counts in the net area may not be consistently found. If this is a limitation of the peak-finding algorithm, it should be so recorded.

c) Erase the spectrum, and with the same ^{137}Cs source at its designated source-to-detector distance, accumulate a new spectrum with 100 counts in the 662 keV peak.

d) Remove the ^{137}Cs source and insert a second source (e.g., ^{65}Zn, ^{22}Na or ^{60}Co). Continue the count (do not erase spectrum) for an additional counting time that will result in each channel of the continuum on each side of the ^{137}Cs peak having an expected number of counts equal to 720/(FWHM). The FWHM is the energy resolution expressed in channels of the 662 keV peak as measured above.

e) A flat continuum may be added by drawing normally distributed random numbers.

f) Execute the peak-finding algorithm routine. The peak at 662 keV should be found. Repetitive counts are not recommended due to the extensive time required; therefore, specific conclusions are limited in a statistical sense. If the peak is not found, repeat the test (steps c) through e)). If it is not found the second time, increase L_p (see note below) and rerun the test. Record the value of L_p for which the peak is found.

Hint – For the above and following procedures the effort and time required may be reduced by stopping the count and analyzing it at a continuum whose expected counts per channel correspond to L_p = 4,65 (see note below). If the peak is found, the ^{65}Zn count should be continued (do not erase the spectrum) for preset times corresponding to sequentially smaller values of L_p until either the peak cannot be found or L_p = 2,33.

NOTE The peak-finding algorithm is expected to find a peak in a spectrum whose area A is:

$$A = L_p\sqrt{2{,}55\ \mathrm{FWHM}\ y_i}$$

where:

- 2,55 is based on ±3σ for a Gaussian peak,
- FWHM is the full width in channels at half maximum of the peak,
- y_i are the average counts in each baseline channel, and
- L_p = 2,33 corresponds to the value of L_p initially suggested for this test. A multiplying factor 2,33 < L_p < 4,65 may be substituted if the peak-finding algorithm cannot find peaks for L_p = 2,33 or too many false peaks are found at this sensitivity (a judgment call).

g) Erase the spectrum, and with the ^{137}Cs source at its designated source-to-detector distance, accumulate a new spectrum with 1 000 counts in the 662 keV peak.

h) Remove the ^{137}Cs source and insert the second source (i.e., ^{65}Zn, ^{22}Na or ^{60}Co) and continue the count for a counting time that will result in an expected $(7{,}2 \times 10^4)$/(FWHM) number counts in each of the continuum channels (FWHM in channels). These many counts may take an entire day to accumulate even at a counting rate of 2 000 s^{-1}.

i) Execute the peak-finding algorithm routine. The 662 keV peak should be found when the peak-finding algorithm is applied. If it cannot find the peak, repeat the test (steps f) through h)). If it is not found the second time, increase L_p (see note in step f)) and rerun the test. Record the value of L_p for which the peak is found.

If the peak cannot be found for L_p = 4,65, the sensitivity parameter of the peak-finding algorithm should be adjusted, and the test rerun so that the identification criteria in note in step f) can be met. Consult the users' manual of the analytical program or the manufacturer for additional help in adjusting the sensitivity of the peak-finding algorithm parameters. Information pertaining to minimum detection can be found in the literature (see [6]).

If the software used allows for extracting and importing simulated spectra, then a simulation can be used as an alternative to the time-consuming steps d) and g). The spectrum that was counted in step c) (and/or step f)) should be exported out of the software. A random number should be drawn for each channel in the spectrum from the Poisson distribution with the average number of events set so that the average number of counts are the ones specified in step d) (and/or step g)). The simulated spectrum with a flat continuum should be added to the exported measured spectrum and the resulting spectrum should be imported into the software. Steps d) (and/or h)) can then be executed on the imported spectra. This would allow for repeated trials of the experiment without adding a lot of measurement time.

8.3 Test of independence of peak-area from the gross peak-height to continuum-height ratio

This test uses the same sources and source-to-detector distances as are used in 8.2 and requires the same high purity for the ^{65}Zn, ^{22}Na, or ^{60}Co spectrum in the energy region covered by the 662 keV peak.

a) With the ^{137}Cs source placed at its designated source-to-detector distance, accumulate a spectrum until approximately 5 000 counts are recorded in the gross area of the 662 keV peak.
b) Measure the net peak area and its uncertainty with the spectral analysis program used and to be tested.
c) Remove the ^{137}Cs source.
d) Without erasing the original spectrum, add counts to the continuum underlying the 662 keV peak by accumulating a spectrum with the ^{65}Zn source at its designated source-to-detector distance (^{22}Na or ^{60}Co may be substituted for ^{65}Zn; but if ^{60}Co is used, a correction for the presence of the single-escape peak of the 1 173 keV gamma-ray at 662 keV may need to be made).
e) Continue the counting until each channel in the continuum surrounding the 662 keV peak contains $(4,9 \times 10^4)/(\text{FWHM})$ counts. These many counts may take one or more days to accumulate. This step can be replaced by simulations as described in 8.2.
f) Determine the net area of the 662 keV peak and its uncertainty with the spectral analysis program being tested. If there is a 662 keV peak in the ^{65}Zn or background spectrum, its contribution shall be subtracted (see Clause 8).
g) The net peak area from step f) with its uncertainty at the 95 % confidence level (i.e., two estimated standard deviations) should encompass the net peak area counts measured in step b).

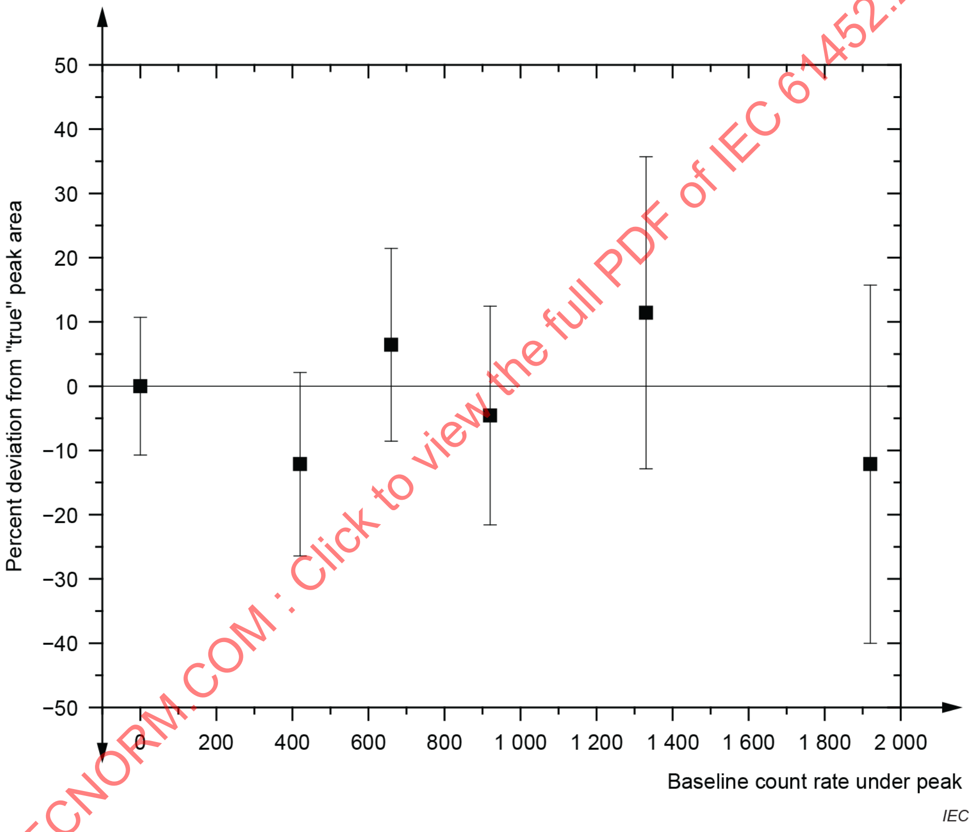
If these criteria are not met, repeat this test. If these criteria cannot be met, there may be a systematic bias in the peak-area-measurement algorithm. Figure 4 and Table 1 illustrate examples of the deviation of the net peak area from the "true" area (singlet peak area with no added continuum) as a function of the continuum height for a peak area of 110 counts and 9 844 counts, respectively.

Table 1 – Net-peak areas as a function of continuum height

Continuum[a] Y_i	Measured net peak area	Uncertainty[b] %	Deviation from first count %
1	9 844	1,0	0
3 500	9 799	2,2	-0,5
7 500	9 968	2,9	+1,3
20 500	10 262	4,6	+4,2
39 000	9 746	5,9	-1,0

[a] Average counts in each continuum channel surrounding peak.

[b] Uncertainty in the net peak area represents one estimated standard deviation.



NOTE The uncertainty in the deviation is shown as vertical lines through the data points. The "true' peak area is defined as the net area with no added continuum from a second source. The horizontal axis represents the integral continuum counts under the peak.

Figure 4 – Deviation in measured net peak area as a function of continuum height

8.4 Test of the doublet-peak finding and fitting algorithms

This test consists of two parts. The first part is designed to evaluate spectral-analysis programs with the capability of automatically finding doublet peaks (i.e., with doublet peaks, at least one side of the peak does not return to the continuum due to the presence of another peak). The second part of the test is designed to evaluate programs with the capability of fitting a doublet peak to determine the net area of each of the component peaks.

These tests can be replaced by Monte Carlo simulations (see 8.2) or other mathematical methods.

If measurements are performed, the gain of the spectrometer should be about the same as used to accumulate routine spectra, and the amplifier should have a gain control that can be adjusted to move a peak position by only a few channels to produce a doublet peak. Alternately, an adjustable offset on the ADC by which a peak can be shifted several channels (e.g., ADC zero level) can be used to produce doublet peaks. Before beginning the tests, the amplifier fine gain or digital offset should be calibrated so that the ^{60}Co 1 332 keV peak channel position can be raised or lowered in increments of approximately one half of one FWHM.

The test shall be carried out as follows:

a) Accumulate a spectrum with a ^{60}Co source (other long half-life radionuclides emitting a gamma-ray with a high-emission intensity and free of interferences may also be used). The source should have a low counting rate (approximately 500 s^{-1}) and be located at least 5 cm from the detector. The count should be accumulated for a pre-set live time so that about 20 000 and preferably 50 000 counts are in the net peak area.

b) Use the analysis program to find the 1 332 keV peak and measure its net peak-area counting rate. This value will be referred to as the “true" net peak-area counting rate and should be so recorded. Store the spectrum so that it can be recalled.

c) Adjust the gain or ADC offset so that the 1 332 keV peak is raised the equivalent of one FWHM. To the spectrum accumulated in step b) add the data accumulated for an equal live time using the new amplifier gain (or ADC offset).

d) Use the spectral analysis program to find the original and shifted 1 332 keV peaks and measure the net peak-area counting rates and associated uncertainties of each component. Each component should be found and each of their net peak-area counting rates should agree with that measured in step b) to within one-third of the acceptable inaccuracy (all uncertainties combined, see Clause 11) of the measured gamma-ray emission rate.

e) If the net peak-area counting rates are not determined to within one-third of the acceptable inaccuracy, repeat the test. If the test cannot be passed on the second attempt, rerun the test (steps a) through d)) with a peak separation of 1,5 FWHM or greater until these criteria are met. Record the separation at which these criteria are met.

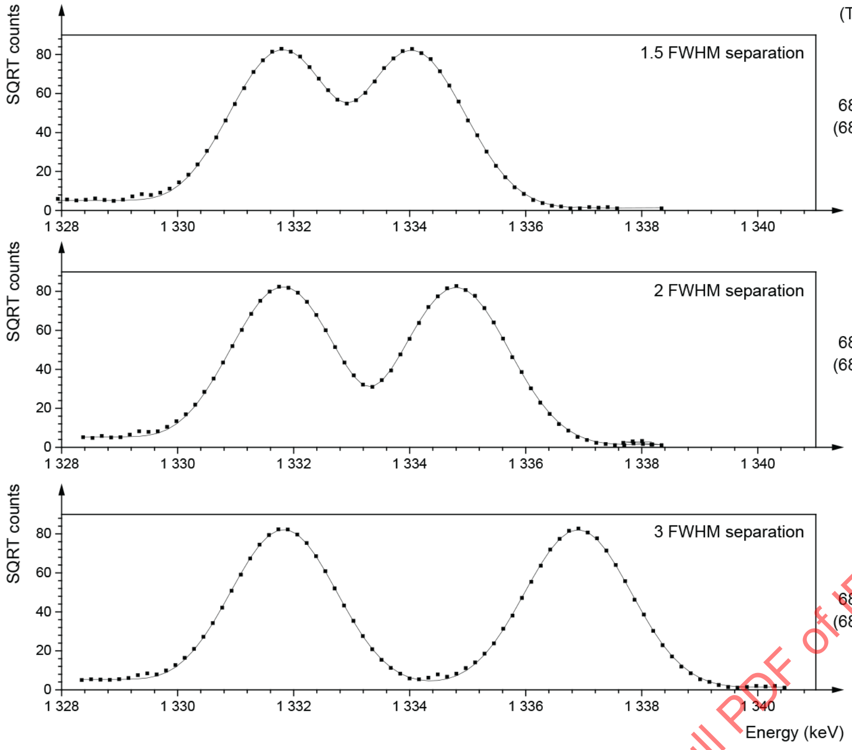
f) Repeat steps a) through e) but change the gain or ADC offset so that the 1 332 keV peak is raised the equivalent of two FWHM and reduce the second acquisition time by a factor of ten. Each of the component peaks should be found and each of the net peak-area counting rates should agree (within one-third of the acceptable inaccuracy) with the net peak-area counting rate measured in step b).

g) If the net peak-area counting rates are not determined to within one-third of the acceptable inaccuracy, repeat the test (step f)). If the test criteria cannot be met on the second attempt, rerun the test with the second acquisition time reduced by a factor of five or less until the test criteria can be met. Record the net peak area ratio for which the test criteria are met.

h) Repeat step f) but change the gain or ADC offset to lower the peak by two FWHM.

i) The criteria to be met in this test are the same as specified in step f).

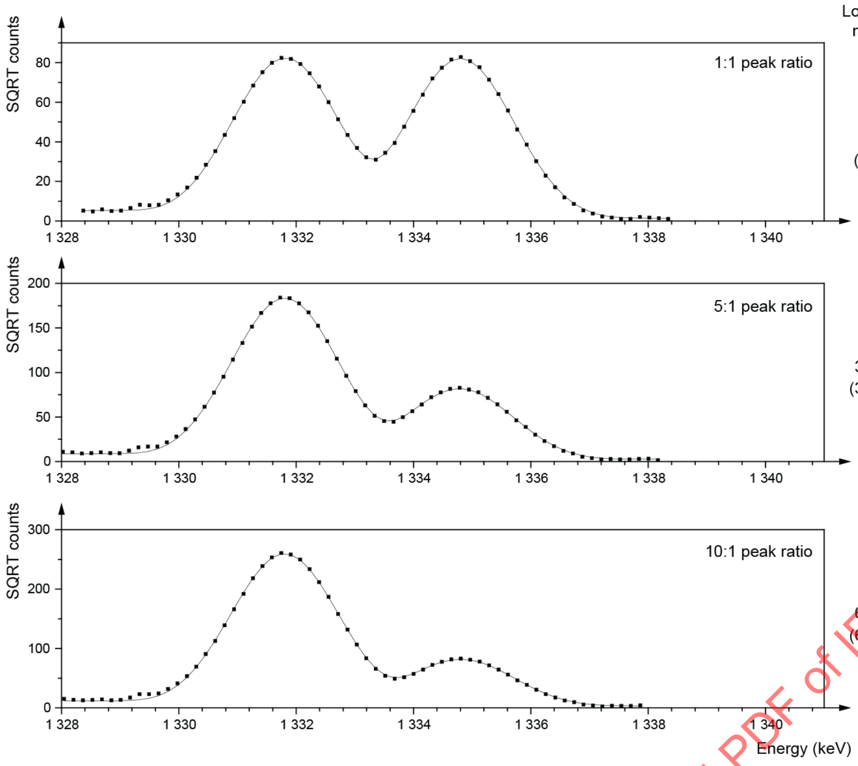
Precaution – If the peak-finding algorithm has difficulty finding both components of the doublet, it may be necessary to adjust the peak-finding parameters (see 8.1). Additional tests for other separations and for other net peak area ratios are recommended to better define the capabilities and limitations of the peak-finding and area-measuring algorithms. Figure 5 indicates the deviation in the net areas resulting from various separations of equal-sized doublets and Figure 6 indicates the deviation in the net areas resulting from the weaker (less active) of two unequal-sized doublets for the peak separations given.



Peak area			
Low energy measure (True)	% Deviation	High energy measure (True)	% Deviation
68 192 (68 146)	0,07	67 877 (68 146)	−0,39
68 164 (68 146)	0,03	67 863 (68 146)	−0,42
68 081 (68 146)	−0,10	67 963 (68 146)	−0,24

NOTE Deviation in doublet net peak area as a function of peak separation in units of FWHM for two equally sized components. The net peak-area counts are quoted here as described in the text.

Figure 5 – Deviation in equally sized doublet peak areas for different separations



Peak area			
Low energy measure (True)	% Deviation	High energy measure (True)	% Deviation
68 164 (68 146)	−0,07	67 863 (68 146)	−0,42
340 226 (340 730)	−0,15	67 727 (68 146)	−0,61
680 281 (681 460)	−0,17	67 601 (68 146)	−0,80

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NOTE Deviation from true peak area for weaker of two unequally sized doublet components. The net peak-area counts are quoted here as described in the text.

Figure 6 – Deviation in unequally sized doublet peak areas for different pulse-height ratios

9 Verification of the entire analysis process

9.1 Assessment of the magnitude of true coincidence summing

Although true coincidence summing is radionuclide, geometry and detector specific, operators need a quick and simple method of assessing the relative magnitude of this effect for various detection geometries and different size detectors in order to determine if additional (and substantially more complicated) corrections are required. It is important to note that the following test does not provide a correction factor that can be applied to measurements other than the test measurement, but rather allows an assessment of the relative magnitude of true coincidence summing at a given geometry. This test, best carried out before measuring the detector efficiency, can be conducted with any two sources or a mixture of these sources. One of the radionuclides emits a gamma-ray free of true coincidence summing and the other emits a gamma-ray close in energy, but well resolved from the first gamma-ray, but is affected by true coincidence summing. Several pairs of radionuclides can meet these conditions including: ^{60}Co(1 173)/^{65}Zn(1 115), ^{56}Co(846)/^{54}Mn(835), ^{88}Y(898)/^{54}Mn(835), and ^{154}Eu(591)/^{125}Sb(600), where the first radionuclide listed is affected by true coincidence summing and the second is (almost) free of true coincidence summing. The associated gamma-ray energies in keV are in parentheses. It is best if the source is composed of a mixture of both radionuclides; however, if the sources are not mixed to produce one source, their orientation shall be noted so that they are always counted in the same orientation.

The following test is one method designed to measure the change in the area ratio of the summing gamma-ray, $N(s)$, to that of the peak free of summing, $N(n)$, relative to the measured ratio at a distant reference position where the summing effect is negligible. This test will use a mixed radionuclide source containing ^{125}Sb, and ^{154}Eu (a spectrum of a long-lived mixed radionuclide standard is shown in Figure 8), but one of the other radionuclide pairs may be substituted. As the source-to-detector distance increases, this ratio will approach a constant as shown in Figure 7 (the normalized ratio approaches one). With this source, if the detector does not observe the Te K X-rays (27 keV to 32 keV) resulting from internal conversion in the decay of ^{125}Sb, the 600 keV full-energy peak does not lose counts due to true coincidence summing even when the sample is counted close to the detector and only a very few counts are added (172 keV gamma-rays may sum with 428 keV gamma-rays to add counts to the 600 keV peak). (See ^{125}Sb decay scheme.)

This change can be expressed in terms of ratios of net peak areas, $N(E)_\mathrm{i}$, for the energies E = 591 keV and 600 keV at each distance, i, that a spectrum was accumulated:

$$r_\mathrm{i} = \frac{N(591)_\mathrm{i}}{N(600)_\mathrm{i}} = \frac{N(s)_\mathrm{i}}{N(n)_\mathrm{i}} \tag{14}$$

By setting the ratio at one of the farther distances (e.g., 10 cm to 15 cm source-to-detector distance where the cascade summing is negligible) equal to r_r (i.e., reference distance), the following ratio, R_i, as a function of distance (at distance i = r the ratio will be one by definition) can be measured:

$$R_\mathrm{i} = \frac{r_\mathrm{i}}{r_\mathrm{r}} = \frac{N(591)_\mathrm{i}/N(600)_\mathrm{i}}{N(591)_\mathrm{r}/N(600)_\mathrm{r}} = \frac{N(s)_\mathrm{i}/N(n)_\mathrm{i}}{N(s)_\mathrm{r}/N(n)_\mathrm{r}} \tag{15}$$

The cascade-coincidence-summing correction (multiplicative) factor for the 591 keV gamma-ray peak area is C_c = 1/(1 – R_i). The value of R_i for gamma-rays in a cascade, that are emitted from other radionuclides, are typically within a factor of two of the R_i measured for the 591 keV gamma-ray for the same detector, sample size, and counting geometry.

Precaution – Crossover gamma-rays for which counts are summed into the peak due to two or more gamma-rays, should not be used in spectral analyses at close source-to-detector distances, unless the correction factor is calculated and applied.

A cascade-coincidence-summing correction may not be required at a given distance i if extended samples with a mean distance greater than 5 cm are to be counted and an uncertainty of 10 % is acceptable or if the error due to not making the correction is insignificant. A 10 cm source-to-detector distance is used in the test below for r_r, but a 15 cm source-to-detector distance is recommended for HPGe detectors having relative efficiency greater than 20 %.

Method of test:

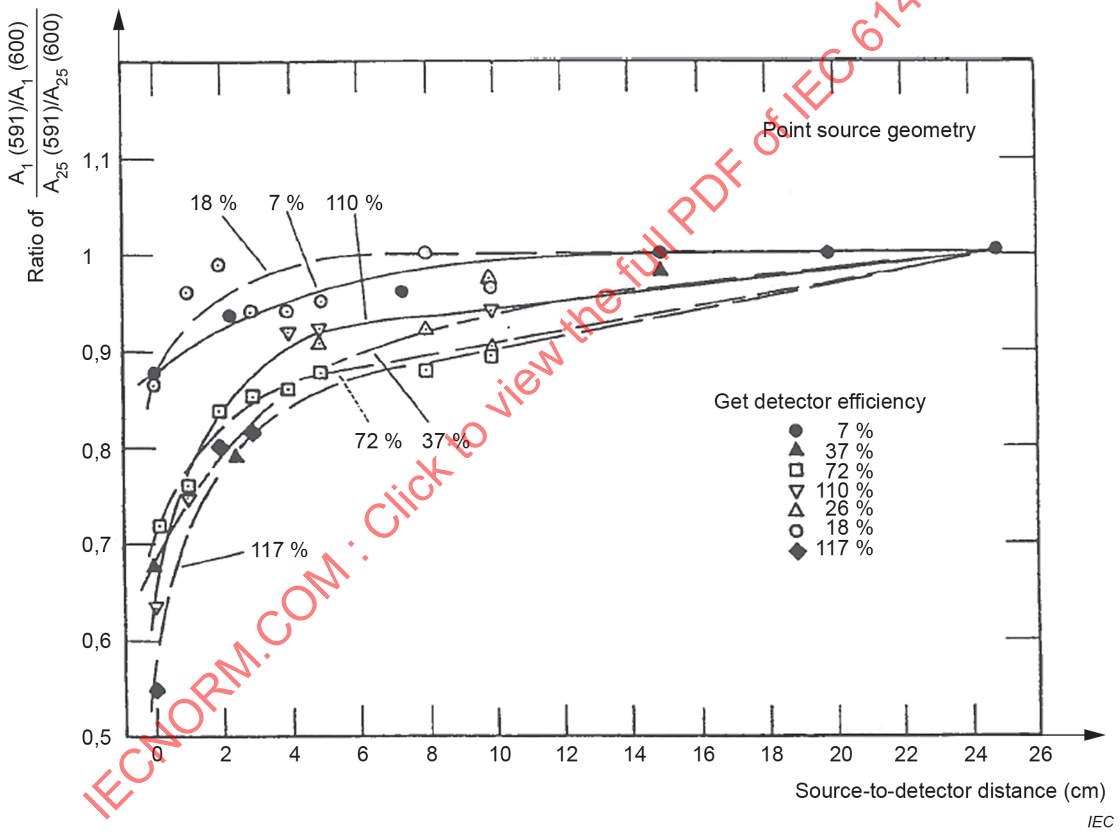
a) With a standard mix of ^{125}Sb and ^{154}Eu in solution form, prepare (or purchase) a sample of the geometry and matrix to be tested (see Clause A.8 on the preparation of working standards). The counting rate of the sample should not exceed 1 000 s^{-1} when positioned at the source-to-detector distance in question.

b) Count the sample at the smaller source-to-detector distance in question until at least 20 000 and preferably 50 000 counts are in the net areas of the 591 keV and 600 keV gamma-ray peaks. Determine the ratio, r_i, from the net peak areas.

c) Count the sample at a 10 cm mean source-to-detector distance until at least 20 000 and preferably 50 000 counts are in the net peak areas of the 591 keV and 600 keV gamma-rays of ^{154}Eu and ^{125}Sb, respectively. Compute the ratio, r_r, from the net areas indicated above.

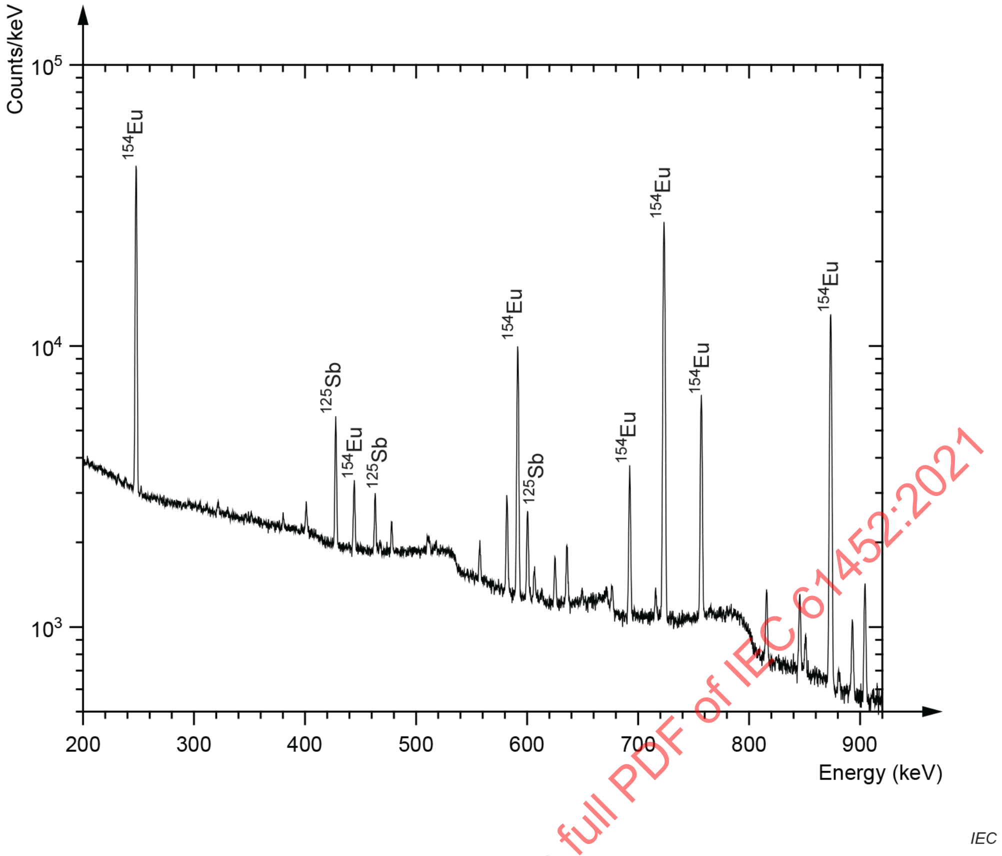
d) Compute the ratio, $R_\mathrm{i} = r_\mathrm{i}/r_\mathrm{r}$ from the ratios r_i and r_r measured in steps b) and c), respectively.

Cascade summing corrections may not be required if $R_\mathrm{i} = r_\mathrm{i}/r_\mathrm{r} > 0{,}97$. The acceptable inaccuracy of the routine gamma-ray measurements at this geometry and for this sample matrix will determine the amount of cascade summing that can be tolerated without applying a correction. If corrections are to be avoided and $R_\mathrm{i} < 0{,}97$, then either the mean source-to-detector distance shall be increased until the above test satisfies the accuracy requirements of the routine measurements or the uncertainty in the measured gamma-ray emission rates shall be increased to account for the difference associated with not correcting for cascade summing. Figure 7 shows plots of R_i as a function of source-to-detector distance for a point source counted with small and large HPGe detectors. A crude estimate of this difference, expressed in percent, is $100(1 - r_\mathrm{i}/r_\mathrm{r})$.



NOTE Cascade summing of the ^{154}Eu 591 keV peak as a function of the source-to-detector distance, i, for point source geometry for different sizes and shapes of HPGe detectors. The percentages associated with each curve are the detection efficiencies relative to a 76,2 mm × 76,2 mm (3 in × 3 in) NaI(Tl) detector (a point source emitting 1 332 keV gamma-rays from ^{60}Co at a source-to-detector distance of 25 cm). The data points are from experimental results with the curves normalized to a 25 cm source-to-detector distance. (Courtesy Sanford Wagner and Richard Seymour, EG&G ORTEC Inc., Oak Ridge, TN, USA.)

Figure 7 – Cascade-summing corrections for a ^{154}Eu 591 keV gamma-ray



NOTE HPGe gamma-ray spectrum for the NIST long-lived radionuclide mix (i.e.,^{125}Sb and ^{154}Eu).

Figure 8 – Partial HPGe gamma-ray spectrum of a long-lived mix

9.2 Deviations in the relative full-energy-peak efficiency

The deviations in the relative full-energy-peak efficiency over the energy range of the efficiency curve shall be determined. This should be done by counting at least one radionuclide which emits gamma-rays that span the energy region of interest and whose relative abundances are precisely known. Some radionuclide sources that have relatively long half-lives (exceeding one week) with spectra containing strong (large peak-height to continuum ratio) singlet gamma-rays include a mix of ^{125}Sb and ^{154}Eu; ^{106}Ru/^{106}Rh, ^{125}Sb, ^{140}Ba/^{140}La, ^{152}Eu and ^{232}U with its daughter activities in equilibrium.

Method of test:

a) Count a sample, in the geometry to be tested, that contains a radionuclide whose relative gamma-ray emission intensities are accurately known, until 20 000 to 100 000 counts are accumulated in the net areas of the peaks of interest.

b) Analyse the spectrum following normal procedures and apply all applicable corrections. Deduce the measured relative gamma-ray intensities by using formula (2) and then normalize the strongest gamma-ray associated with each radionuclide to that given in the decay-data library or in the literature.

c) Compare the measured relative gamma-ray intensities to those in the decay-data library or in the literature.

d) The percentage deviation between the measured and known values for gamma-rays, with peak-height to continuum ratios greater than two, shall be within two estimated standard deviations of the propagated uncertainty and preferably not greater than one-third of the desired combined uncertainty (see Clause 11) in the gamma-ray emission rate or radionuclide activity.

Precaution – Although it is tempting to use the agreement in the relative intensities of the gamma-rays emitted from a radionuclide as an indication of the lack of true coincidence summing of cascade gamma-rays in the detector, caution shall be exercised. The magnitude of the cascade summing correction in many radionuclides, although specific for each gamma-ray, may be almost the same. Therefore, lack of agreement in the relative gamma-ray intensities of a radionuclide may indicate the need for corrections for cascade summing. However, agreement in the relative gamma-ray intensities does not assure that cascade summing corrections are small or zero.

Recommendation – Decay data used for these measurements should be obtained from the Evaluated Nuclear Structure Data File (ENSDF) (https://www.nndc.bnl.gov/)[25] or from the Decay Data Evaluation Project (DDEP) (http://www.lnhb.fr/nuclear-data/)[26].

9.3 Accuracy of the full-energy-peak efficiency

The accuracy of the full-energy-peak efficiency shall be verified, when activity is to be quantitatively measured (gamma-ray emission rates or disintegration rates measured), by performing the test in 9.2 with standards of radionuclides whose gamma-ray emission rates are accurately known and are from or are traceable to a country's national metrology institute (NMI) for radioactivity measurements. In this case, the gamma-ray-emission rates are deduced in step b) from formula (2) and the gamma-ray-emission rates are compared to the standard values in step c) in 9.2. Lack of agreement indicates neglected or erroneous corrections.

10 Radionuclide identification

10.1 General

Identification and quantification of the radionuclides associated with the measured gamma-ray energies and emission rates are performed by many gamma-ray spectral-analysis programs. This requires a decay-data library which should contain a carefully selected set of the radionuclides anticipated likely to be present in the sample spectra and background spectra. The spectral-analysis program should permit the editing of this library only by a qualified individual at the laboratory and changes should be documented. The information that should be contained in a decay-data library includes the radionuclides and their half-lives, associated gamma-ray energies, and their gamma-ray emission intensities per decay. The uncertainties on each of these values should also be included in the decay-data library. These parameters, which are normally available from recent decay-data evaluations (see [26] and [27]), should have their reference documented.

Since radionuclide identifications are primarily based on the measured energies of associated gamma-rays, an energy window should be established that is broad enough to permit some energy mismatch but not so broad as to include many misidentifications.

An energy-search window, W_s, that is fixed (W_s = 0,5 keV) or increasing with energy in the range of 0,3 keV < W_s < 2,0 keV is near optimum.

Precaution – With analysis programs that include radionuclide identification, it may seem reasonable to place all the gamma-rays from all the radionuclides that could possibly be observed into the decay-data library. However, limitations of radionuclide identification software process suggest that judicious selection be made, and a reduced list of radionuclides and associated key gamma-rays be established. Some analysis programs only use for identification one intense gamma-ray that is expected to be free of interference from gamma-rays of other radionuclides. Because circumstances will arise where the radionuclide associated with certain gamma-ray peaks present in the spectrum will not be automatically identified, the identification process is not without its deficiencies. Samples to be analysed from different origins should have separate decay-data libraries that are selected for efficient yet complete radionuclide identification.

10.2 Identification through multipeak analysis and correction for interference from other radionuclides

With some analysis programs, the activity of a radionuclide is computed from the most intense gamma-ray peak. When peak interferences arise from another gamma-ray, this method will give a result that is too high. In an attempt to correct for these types of interferences some analysis programs use a multipeak analysis technique in which the activity is derived from a weighted (usually by the inverse of the uncertainty, see formula (18)) average of the activities measured using several gamma-ray peaks. Some programs even allow for the discarding of outlier values from this calculated average. Some programs can even separate a peak into its individual radionuclide contributions when the possible interference gamma-rays can be identified. For most spectra, these techniques give good results, but with complex spectra containing many peaks (e.g., fresh reactor coolant water), even these techniques can lead to large uncertainties.

Precaution – Whichever technique is used to identify the radionuclides and their activities, an experienced gamma-ray spectrometrist should oversee the entire analysis process and be actively involved in the quality assurance of the data and results.

10.3 Detection limits

When identifying and quantifying the radionuclides that are impurities in a sample, detection limits should be provided over the entire energy range of the measurement. This will provide information about the minimum detectable activity (MDA) of the different radionuclides present in the source. See ISO 11929 and [14, 34],

11 Uncertainties and uncertainty propagation

Uncertainties shall be evaluated using the Bureau International des Poids et Mesures (BIPM), JCGM 100. There are several ways to report the numerical results and the respective uncertainty, see JCGM 100:2008, 7.2.2. The number of digits used to report the result need to be consistent with the uncertainty and the coverage factor used to report the uncertainty. For example, three possible ways to report an activity are listed below:

- A = 25 (5) Bq
- or A= (25 ± 5) Bq
- or A = 25 Bq with 20 % relative standard uncertainty

If an expanded uncertainty needs to be reported, then the coverage factor needs to be specified.

All uncertainties, including those not derived from a complete statistical analysis, should be stated as approximations to standard deviations (of the mean, if the mean is used). This convention follows that proposed by the BIPM. It allows all of the uncertainty components to be propagated by statistical rules and results in a combined uncertainty.

Table 2 lists the formulas for propagating uncertainties for several common algebraic expressions (see [9] and [4]). The principal components of the combined standard uncertainty in a measured gamma-ray emission rate are those in the efficiency calibration standards, the measurement of the peak areas, the reproducibility of the sample-detector geometry, the pulse pile-up, the cascade-summing corrections, the decay corrections during and prior to counting, the attenuation corrections when the sample matrix differs from that of the standard, the detector full-energy-peak efficiency interpolation, and the subtraction of the gamma-ray peaks in the "blank" background that are at the same energies as those in the sample [31]. The uncertainties in the gamma-ray emission intensities should be propagated with the other uncertainty contributions when the activity is being calculated. The tests of performance described in Clauses 7, 8 and 9 should provide a good initial estimate of the uncertainty components for a given HPGe spectrometer and analysis program(s). These estimates should be refined as experience is gained. Typical ranges for these uncertainty components are listed in Table 3.

Whenever a set of values with equal-size uncertainties are to be averaged, a simple mean is computed, along with its standard deviation, as follows:

$$\bar{x} = \sum_{i=1}^{n} \frac{x_i}{n} \tag{16}$$

$$s_x^2 = \frac{1}{n-1}\left[\sum_{i=1}^{n}(x_i - \bar{x})^2\right] \tag{17}$$

where

x_i are the values to be averaged;

n is the number of values to be averaged;

$\bar{x}$ is the average value; and

s_x^2 is the experimental variance (the experimental standard deviation is the square root of the variance).

Whenever a set of data with associated uncertainties is to be averaged, a weighted average should be taken:

$$\bar{x} = \left[\sum_{i=1}^{n} w_i x_i\right] \Big/ \left[\sum_{i=1}^{n} w_i\right] \tag{18}$$

where

$w_i = 1/s_i^2$

The uncertainty in the weighted average is dependent upon whether the data are internally consistent as determined by the chi-squared (χ^2) test. If $\chi^2/(n-1) \gg 1$, where $n - 1$ is the number of degrees of freedom, the data are inconsistent or the uncertainties in the individual measurements are underestimated. The following two formulas represent the internal variance (19) and the external variance (20) of weighted means.

$$s^2(\bar{x}, 1) = 1 \Big/ \left[\sum_{i=1}^{n} w_i\right] \tag{19}$$

$$s^2(\bar{x}, 2) = \left[\sum_{i=1}^{n} w_i (x_i - \bar{x})^2\right] \Big/ \left[(n-1)\sum_{i=1}^{n} w_i\right] \tag{20}$$

$$\chi^2(x) = \sum_{i=1}^{n} (x_i - \bar{x})^2 \; w_i \tag{21}$$

where the larger value of $s^2(\bar{x}, 1)$ and $s^2(\bar{x}, 2)$ is chosen for the uncertainty in the weighted average.

Whenever it is necessary to multiply the combined standard uncertainty by a factor to obtain the expanded uncertainty (a more conservative overall uncertainty), the multiplying factor (coverage factor) shall be stated (e.g., reporting uncertainties at about a 95 % confidence level, corresponds to a coverage factor k = 2).

Whenever environmental and other very low-level activity samples are analysed, the gamma-ray emission rates or activity should be reported with two uncertainties: the uncertainty in the net peak area (or counting rate), and the combined uncertainty. The uncertainty in the net peak area will indicate whether or not the peak is truly a positive peak while the combined uncertainty will indicate the accuracy with which the reported value is known.

For accurate estimates of combined standard uncertainty, it may be necessary to consider correlations between the component uncertainties. An efficiency curve derived in part from several gamma-rays emitted from one radionuclide will have correlated uncertainties for those points. See [1] and [9]. Fitting a function to the efficiency calibration data will introduce correlations between the uncertainties of efficiencies evaluated by the fitted function. Gamma emission intensities could also have a correlated component, and it is often tabulated in the nuclear data as a “normalisation uncertainty”, this uncertainty can be large, and should be included when present.

In the case of a component which cannot be described by differentiable functions, the estimation of associated uncertainty may require the use of a Monte Carlo method, see [25].

Table 2 – Uncertainty propagation for simple functions

Function[1]	Manipulation	Uncertainties[2]
$y = a_1x_1 + a_2x_2$	Addition	$s_y = \sqrt{a_1^2 s_{x_1}^2 + a_2^2 s_{x_2}^2}$
$y = a_1x_1 - a_2x_2$	Subtraction	$s_y = \sqrt{a_1^2 s_{x_1}^2 + a_2^2 s_{x_2}^2}$
$y = ax_1x_2$	Multiplication	$\frac{s_y}{y} = a\sqrt{\left[\frac{s_{x_1}^2}{x_1}\right]^2 + \left[\frac{s_{x_2}^2}{x_2}\right]^2}$
$y = ax_1/x_2$	Division	$\frac{s_y}{y} = a\sqrt{\left[\frac{s_{x_1}^2}{x_1}\right]^2 + \left[\frac{s_{x_2}^2}{x_2}\right]^2}$
$y = x^m$	Power of x	$\frac{s_y}{y} = \frac{m\,s_x}{x}$
$y = e^{-ax}$	Power of e	$\frac{s_y}{y} = a\,s_x$
$y = f(x_1, x_2)$	Function of two variables	$s_y = \sqrt{\left[\frac{\delta f}{\delta x_1}\right]^2 s_{x_1}^2 + \left[\frac{\delta f}{\delta x_2}\right]^2 s_{x_2}^2}$

[1] a, a_1, a_2 and m are constants. x, x_1 and x_2 are independent variables. y is the dependent variable.

[2] s_i is the estimated standard deviation in variable i.

Table 3 – Uncertainty contributions

Uncertainty contribution	Typical magnitude %[1]
Activity of standard source	0,1 to 2
Source aliquots	0,1 to 3
Peak-area measurement	0,1 to 30
Background peak-area variations	0,0 to 100
Variations in sample attenuation	0,0 to 50
Source-to-detector geometry	0,1 to 10
ADC live timer	0,0 to 0,5
Pulse pile-up (high counting rate)	0,0 to 30
Cascade summing	0,0 to 100
Decay of radionuclide	0,0 to 5
Gamma-ray emission intensity	0,1 to 20
Full-energy-peak efficiency	0,5 to 15

[1] Uncertainties are one estimated standard deviation expressed as a percentage.

12 Mathematical efficiency and correction factors modelling

12.1 General

Mathematical approaches can be used to calculate the efficiency of detectors and correction factors. The two main ways are deterministic or stochastic. While the first one uses an analytical approach, which leads to numerical solutions, the last one is based on random sampling (Monte Carlo simulation). Different software based on pure analytical approach have been developed and some codes have been developed with a mixed approach using both analytical calculation and Monte Carlo simulation.

Monte Carlo simulation is a practical tool to examine photon interactions in an experimental setup. Monte Carlo software packages can be divided in two different types:

- "generalist" sofware (e.g., GEANT4 [52], PENELOPE [53], MCNP [54], EGS [55], PHITS [59]) which are multipurpose codes (the efficiency or correction factor calculations are not directly implemented) and
- "dedicated" software (e.g., DETEFF [62], GESPECOR [28], ISOCS/LABSOCS [61], SCAL [60]) which have been conceived with dedicated practical calculations for gamma-ray spectrometry.

There are some considerations to be made when choosing a Monte Carlo code as Monte Carlo codes may use different databases for cross-sections values and use different approaches for modelling the electron transport. It shall be noted that two important parameters can influence the Monte Carlo simulation results that are:

- the cut-off energy of the different tracked particles;
- the number of channels and the definition of the FEP depending on the channel bin width.

In order to perform these simulations, details about the detector dimensions and construction are needed that shall be provided by the detector manufacturer. Validation measurements shall be performed to assess the accuracy of the simulations.

In addition, Monte Carlo may be used to determine the uncertainty contributions listed in Table 3 (see e.g., https://uncertainty.nist.gov/).

12.2 Mathematical full energy peak efficiency calculations

12.2.1 General

These codes can also be broadly divided into two categories, one that calculates the efficiency directly (e.g., GEANT4, PENELOPE, MCNP, GESPECOR, ISOCS/LabSOCS) (see [32], [35], [36], [37], [38], [39], [40], [41]) or one that calculates the ratio between a geometry with known efficiency and a geometry with unknown efficiency (e.g., ANGLE [64], EFFTRAN [63], ETNA [29]). Other codes not listed here may also be available.

12.2.2 Construction of the detector model

The use of mathematical simulations to determine detection efficiency requires the geometrical parameters of the experimental conditions, and particularly, the internal dimensions of the detector. For codes that calculate the efficiency directly, a more precise knowledge of the internal dimensions than can be known from the detector manufacturing process is often required. For efficiency transfer calculation this is not as important as these calculations are based on relative calculations from experimental values and small deviations in the internal dimensions will cancel out. Validation measurements are needed to determine that the manufacturing dimensions used in the code give satisfactory agreement, or to optimize the detector geometry. It is therefore required to optimize the most important detector parameters with measurements using sources with known activities. It is recommended to use sources that are traceable to a national metrology institute and with a geometry that is well known. The most important detector parameters to optimize are the crystal radius and height, the thickness of the inactive crystal layers or dead layer, window thickness and the distance from the end cap to the front of the crystal. The measurements used to validate the detector models should be sensitive to these parameters. The minimum series of measurements that allows for determining these parameters are:

- A source emitting multiple low- and high-energy gamma-rays (e.g., ^{152}Eu, ^{166m}Ho) positioned at large source-to-detector distance (i.e., larger than 20 cm). A large source-to-detector distance minimizes the impact of any inaccuracy of the source position and source dimensions. In addition, it minimizes true coincidence summing effects and sources emitting multiple gamma-rays can be used. The highest energy should be high enough that there will be interactions in the entire crystal volume.
- A source close to the detector end cap with a low and medium or high energy. The higher energy should be high enough that the interaction in the inactive layers are insignificant. The close distance between the source and detector makes true coincidence summing effects large and the source should preferably not have a radionuclide that exhibits true coincidence summing.
- If samples are used that have activity on the side of the detector, for example Marinelli beakers, it is also important to measure a source located on the side of the detector. A large source-to-detector distance is preferable to minimize source positioning errors and true coincidence summing.

Recommendation – Taking an X-ray or computer tomography (CT) image of the detector will allow better determination of the internal dimensions of the detector [44]. Scanning using a collimated point source may help to determine active volume dimensions.

The measurements should be performed to reach a minimum net peak area of 20 000 for the peaks used to calculate the full energy peak efficiencies and the count rates should be low enough that pile-up correction can be neglected. The Monte Carlo simulations should be performed with high enough statistics on the simulations (or convergence) so that the uncertainty on the simulation is small compared to other uncertainty components. When the full energy peak efficiency has been calculated for each measurement condition, the parameters of the detector model can be optimized to reach agreement between the measured and simulated full energy peak efficiencies. The validated detector model or the efficiency response of the model should be used when calculating the full energy peak efficiencies.

Once the detector model has been validated it will remain valid as long as the detector response is shown to be unchanged. This can be achieved by repeated measurement from the front and the side of the detector with a source emitting a low-energy and a high-energy gamma-ray.

Extrapolating to higher energies than the highest energy used for the measurements is permitted as long as the energy used was high enough that there were interactions in the entire crystal and that all the relevant physical phenomena are taken into account when the full energy peak efficiencies are calculated.

The state-of-the-art efficiency calibration, achieved by a very careful experimental calibration combined with Monte Carlo simulations [35, 36] corresponds to reaching an uncertainty of 0,2 % in the energy range from 50 keV to 1 400 keV for a point source measurement.

Consequently, it is absolutely necessary to validate the results of Monte Carlo simulations by comparison of the calculated values with experimental efficiencies, if possible, for several energies and different geometrical conditions, to optimize more accurately the input parameters of the detector model (see [37], [38], [39], [40]).

Another way to determine key parameters of the detector is based on scanning using a strongly collimated source to determine the dimensions of the active volume and the crystal-to-end-cap distance [45].

12.2.3 Creation of sample geometries

The sample geometries should be created as closely as possible to the physical sample that is modelled. Use of manufacturer data for the container and physical measurements using calibrated callipers are recommended. The most important parameters of the sample geometries are:

- Container wall thickness
- Container shape. The parts of the sample that are closest to the detector are the most important to model correctly. Many beakers have curved bottoms and when used with a small detector to end cap distance modelling the curvature correctly will increase the agreement between the modelled response and the true response of the detector.
- Sample material
- Sample density
- Sample homogeneity

12.2.4 Validation of the detector and sample container

It is good practice to validate at least one container per detector and every sample container on any detector according to the procedure described in 9.3. A lack of agreement indicates that either some corrections are incorrect or that the detector model or sample geometry was not modelled correctly.

12.2.5 Estimation of uncertainties for Monte Carlo codes for full energy peak efficiencies

The estimation of uncertainties for the Monte Carlo calculation of the geometry should take into account:

- The uncertainty in the full energy peak efficiency calculated to validate the detector model, including the uncertainty in the source activity, the uncertainty in peak areas, and source positioning
- The agreement between the measurement and the detector model described in 12.2.2
- The agreement with the validation of the sample container as described in 12.2.4

- The uncertainty in the container position
- The convergence or estimated uncertainty of the calculation.

Caution – Many Monte Carlo codes calculate a convergence, or an estimated uncertainty based on statistics on the simulations. This value is only one component of the full energy peak efficiency uncertainty and should not be used as the uncertainty of the geometry of interest.

Recommendation – If the geometry that is being measured has a shape that cannot be made into a source and the geometry has to be approximated to be able to model in the software, the uncertainties can be increased to account for these approximations. Counting the sample at a larger source-to-detector distance will reduce the uncertainty due to approximations of the sample geometry.

12.3 Estimation of uncertainties from geometry variations

Full energy peak efficiencies rely on the sample being measured having the same geometry as the calibration source or on the model used in the Monte Carlo code. In practice this can be hard to achieve, for example the density of a soil sample may differ from the density of the calibration source or the density of the soil in the matrix.

Monte Carlo codes also allow to assess the uncertainty in the full energy peak efficiency due to changes in a specific parameter in the geometry between the sample and the calibration source (or reference geometry).

a) The assumption is that a parameter in the geometry has a nominal value of “a” but can vary between the values “b” and “c”. The parameter can for example be the source-to-detector distance, the matrix density, fill height or the bottom thickness of a beaker.

b) Calculate the full energy peak efficiency for the energy region of interest with the parameter set to the nominal value “a”.

c) Calculate the full energy peak efficiency for the energy region of interest for the parameter set to the extreme values “b” and “c”.

d) Compare the full energy peak efficiencies calculated in step b) with the two calculated in step c). If the difference is small compared to other uncertainties from the measurement setup then the uncertainty from the variation of this parameter are negligible.

e) Repeat the process for every parameter that can vary from the samples to the source geometry or the reference geometry in the Monte Carlo simulation.

If one or more parameters are determined to be non-negligible then the uncertainties should be increased.

If more than one parameter is determined to have non-negligible contribution to the uncertainty, the uncertainty from the combination of parameters can be estimated as follows:

f) Determine the nominal values, the extreme values and the distribution function, for example normal or uniform, for each parameter that was determined to influence the full energy peak efficiency.

g) Determine values of all parameters according to their limits and probability distribution.

h) Calculate the full energy peak efficiencies for energies in the energy interval of interest.

i) Calculate the mean and standard deviation of the full energy peak efficiencies for all models calculated

j) Repeat step g) to i) until convergence on the mean and standard deviation has been achieved.

The standard deviation calculated in step j) can be used as an estimate of the uncertainty from the sample variations.

With Monte Carlo codes it is possible to adjust all parameters so that they match the sample and to re-run the computation of the full energy peak efficiency and avoid the necessity of increasing the uncertainty due to sample variations, but it may be necessary to perform a specific calculation for each sample.

Recommendation – This procedure for determining uncertainty for sample variations can be done for source-based efficiency calibrations as well as Monte Carlo simulations. Uncertainties from sample variations are not limited to Monte Carlo based calibrations.

12.4 Efficiency transfer

Without caution, Monte Carlo computation of full-energy peak efficiency can deviate by up to 10 % or more from the experimental values, see [46]. However, it was shown [47] that the efficiency transfer method can provide satisfactory results to compute the FEP efficiency for experimental conditions different from the calibration ones (sample position, size, composition, etc.). For such computation, it is necessary to run the Monte Carlo simulation twice, to calculate the ratio of the efficiencies for the sample of interest and for the calibration source and to multiply it with the FEP efficiency in the calibration condition. The advantage of the method is that in the calculated ratio, many inaccuracies in the detector model cancel out to a large degree, making it possible to work directly with non-optimized detector parameters.

Dedicated codes, based on the Moens principle [65], also take advantage of an experimental reference calibration; correction factors to the actual sample configuration are expressed as ratios between efficiencies in the two configurations considered. Compared to the pure Monte Carlo approach, since they are specifically designed, these codes are generally user-friendly and provide faster results.

Recommendation – Care should be taken to avoid computing geometry transfer factors for very different geometrical conditions. On the other hand, the effect of slight geometrical variations, such as filling height of the sample, can be examined by computing this factor, which can be useful to assess associated uncertainties.

12.5 True coincidence summing corrections

There are two main ways of calculating true coincidence summing correction factors. One way is to calculate the efficiency response when all particles are emitted in the same history (with coincidence) and when every particle is emitted in a separate history (without coincidence). This requires knowledge of the radionuclide decay scheme. For generalist codes, specific modules may be used [48]. The correction factor is then the ratio of the two efficiency responses. Figure 9 shows examples of Monte Carlo simulation (using PENELOPE) results performed for ^{134}Cs in some typical geometrical conditions: HPGe P-type and N-type detectors (6 cm diameter and 6 cm thickness) with point source at different source-detector distances, water sample (9 cm diameter and 4 cm thickness) and filter (cellulose, 8 cm diameter and 3 mm thickness) both at 1 mm from the detector window. The plotted results are the ratios between efficiencies computed without and with coincidence for some gamma emissions, i.e. the coincidence summing correction factors to be applied to the relevant experimental peak area. These are mainly due either to summing-out (795 keV and 802 keV) or summing-in (1 365 keV) effects.

The second way is to calculate the total efficiency (the probability that any energy is deposited in the detector) for the energies involved in the decay of nuclide and then use formulas similar to the formulas in Annex C to calculate the correction factors.

One of these codes can be used to correct for true coincidence summing correction when performing source-based efficiency calibration and when measuring samples with sample-to-detector distances less than 5 cm as described in 6.2.5. If the code is able to correct for gamma – X-ray coincidences, adding an absorber to filter x-rays out are no longer needed.

NOTE Full energy peak efficiencies calculated from Monte Carlo codes are inherently free from coincidence summing corrections and no correction on the full energy peak calibration is necessary.

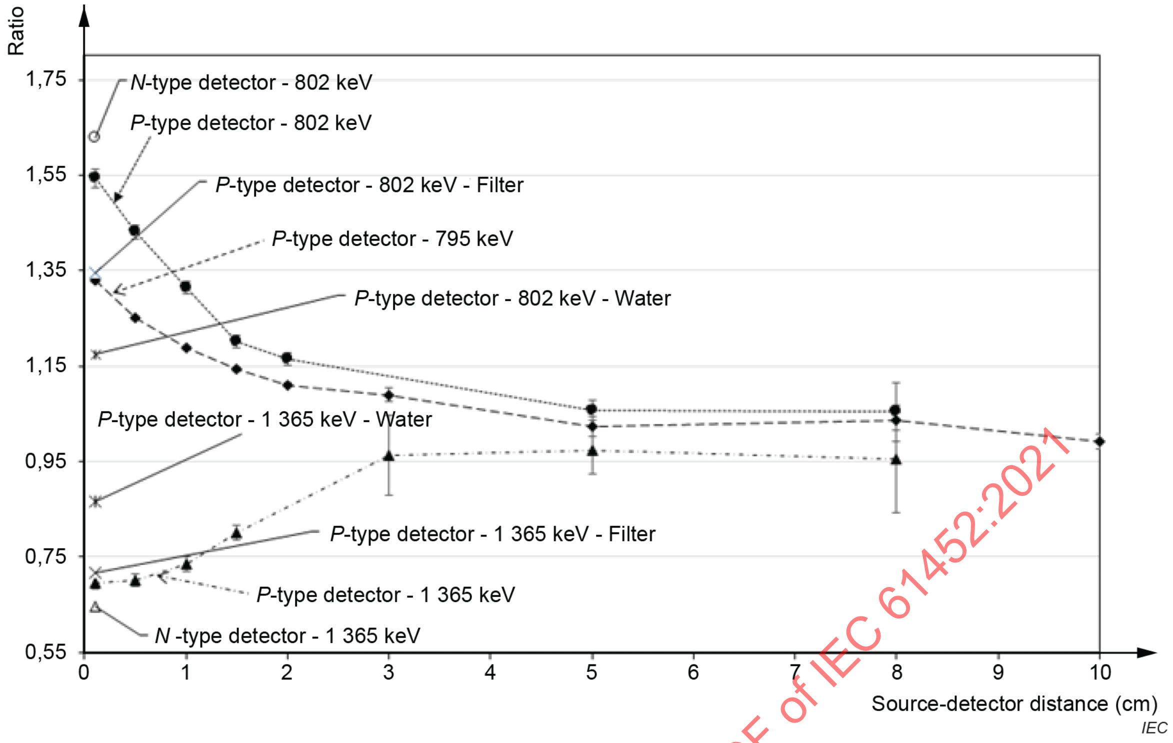


Figure 9 – Results of Monte Carlo simulation to compute true coincidence summing correction factors: example of ^{134}Cs in different geometrical conditions (point or volume) (filter or water) source at different distances from the HP-Ge detector window

Annex A
(informative)

Procedures for characterization of a HPGe gamma-ray spectrometer

A.1 General

The following procedures have been tested and found to be helpful in the set-up and calibration of HPGe spectrometers. They have been gathered from several sources, some unpublished, and are provided in this annex for the convenience of the reader.

A.2 Adjustment of the pole-zero cancellation and direct current level

A.2.1 Rationale for systems using analog electronics

To ensure that the pole-zero cancellation and direct current (DC) level of the amplifier output has been adjusted to provide proper processing of a signal pulse by an MCA system. The following procedure may be used to satisfy the test in 7.3 of the standard for resistive feedback preamplifiers. For transistor reset preamplifiers, the amplifier pole-zero adjustment should be taken out (i.e., no resistance).

A.2.2 Adjustment of the pole-zero cancellation

a) Place a ^{137}Cs or ^{60}Co source at a distance from the detector that establishes a counting rate of about 500 counts per second in the full-energy peak (approximately 2 000 counts per second in the spectrum).

b) Set the oscilloscope for a deflection of 100 mV/cm and a sweep speed of 10 µs/cm.

c) Connect the unipolar output of the amplifier to the input of the oscilloscope. If the amplifier has a baseline restorer, it should be turned off.

d) Adjust the pole-zero potentiometer of the amplifier to bring the long tail component of the trailing edge of the pulses to a flat pulse baseline (no long negative (undershoot) or positive tail (overshoot)). Figure A.1a shows pulses properly adjusted for pole-zero and Figure A.1b and Figure A.1c are examples of pulses unadjusted for pole-zero.

e) Document completion of this procedure in the instrument logbook.

A.2.3 Adjustment of the direct current (DC) level

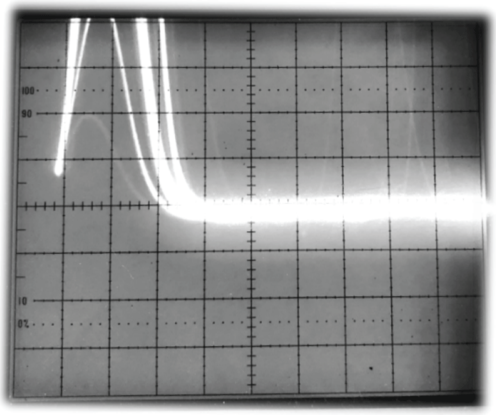
a) Check that the appropriate amplifier shaping-time constant, baseline restorer, input polarity, and approximate gain have been selected. Check the ADC conversion gain, lower-level discriminator and zero settings, and confirm that the ADC is in the singles mode.

b) Remove all radioactive sources from the shield and disconnect the ADC input from the amplifier output.

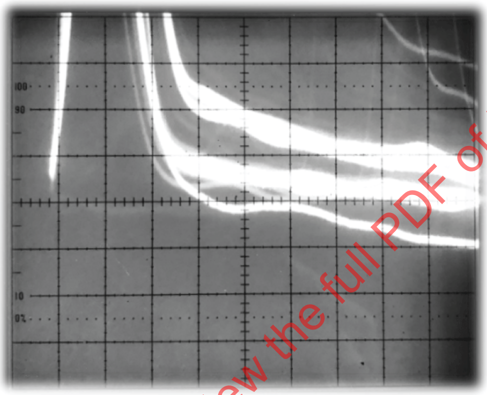
c) Set the oscilloscope for a deflection of 20 mV/cm and a sweep speed of 10 µs/cm and connect the unipolar output of the amplifier to the input of the oscilloscope. The baseline restorer should be at the position to be used in routine counting.

d) Adjust the DC level with the DC trim potentiometer on the amplifier so that the horizontal trace on the oscilloscope does not shift vertically when the AC/DC switch on the oscilloscope is toggled or, alternately, when the signal cable to the oscilloscope is connected and disconnected.

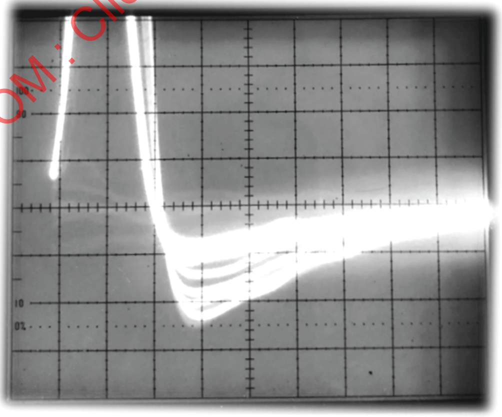
e) Document completion of this procedure in an instrument logbook assigned to this spectrometer. Reconnect the ADC input from the amplifier output if it was disconnected to perform this test.



a) Amplifier output of pulses properly adjusted for pole-zero cancelation



b) Amplifier output of pulses not properly adjusted for pole-zero cancelation (positive tail or overshoot)



c) Amplifier output of pulses not properly adjusted for pole-zero cancelation (negative tail or undershoot)

Figure A.1 – Amplifier output pulses showing correct and incorrect pole-zero cancellation

A.3 Adjustment of the lower-level discriminator (LLD), ADC zero and initial energy scale

A.3.1 Rationale

To ensure that noise or the lower-level discriminator does not block the processing of valid pulses from low-energy gamma-rays, and to permit direct comparisons of spectra taken on different detectors by setting the same gain and zero. The initial energy scale may be set at the same time that the ADC zero and the lower-level discriminator are adjusted. The following procedure may be used to satisfy 5.3.

A.3.2 Adjustment of the lower-level discriminator

a) Place an ^{241}Am source or other low-energy source (use a source whose photon energy is at or below that of the lowest photon to be routinely analysed) in the detector shield and establish a counting rate of about 500 counts per second.

b) Set the lower-level discriminator of the ADC above the noise so that noise is not processed by the ADC, but below the energy of the lowest gamma-ray to be analysed.

Precaution – The lower-level discriminator should not be set below 0,5 % of the full scale.

c) Document completion of this procedure in the instrument logbook and record the LLD setting (if possible).

A.3.3 Adjustment of the ADC zero and initial energy scale

This procedure is designed for adjustment of the zero and energy scale for the following gains (keV/channel):

a) a gain of 0,375 keV/channel (8 192 channel spectrum) using ^{232}U + daughters;

b) a gain of 0,500 keV/channel (4 096 channel spectrum) using ^{88}Y and ^{139}Ce or ^{182}Eu; and

c) a gain of 0,750 keV/channel (4 096 channel spectrum) using ^{232}U in equilibrium with its daughters. A source of ^{228}Th in equilibrium with its daughters is a suitable alternative for ^{232}U.

This procedure can be used with other gain scales by choosing to place a low-energy and a high-energy gamma-ray in preselected channels of the spectrum such that the intercept of the energy equation is approximately zero.

d) Select the energy scale and conversion gain applicable to the experimental setup. Use Table A.1 to determine the radionuclide(s) to be used, the low and high gamma-ray energies, and the associated channels in which the gamma-rays are to be located.

Table A.1 – Adjustment of energy channels to yield energy equation with zero intercept

Gain keV/channel	Channel	Lower-energy peak			Upper-energy peak		
		Isotope	Energy[1]	Channel	Isotope	Energy[1]	Channel
0,250	8 192	^{139}Ce	165,86	663	^{88}Y	1836,07	7344
0,250	8 192	^{152}Eu	121,78	487	^{152}Eu	1408,01	5632
0,375	8 192	^{212}Pb[2]	238,63	636	^{208}Tl [2]	2614,51	6972
0,500	4 096	^{139}Ce	165,86	332	^{88}Y	1836,07	3672
0,500	4 096	^{152}Eu	121,78	244	^{152}Eu	1408,01	2816
0,750	4 096	^{212}Pb [2]	238,63	318	^{208}Tl [2]	2614,51	3486

[1] Energies are those given in [27]. Energies are in keV.

[2] From ^{232}Th decay chain.

e) With the sources inside the detector shield, establish the source-to-detector distance at which the total or gross counting rate is about 500 counts per second. If two sources are required in this procedure, adjust them so that the gamma-ray peaks from each source have good peak-to-baseline ratios.

f) Adjust the fine gain so that the higher-energy gamma-ray peak is located at the channel location (within one channel) specified in Table A.1.

g) Adjust the zero of the ADC so that the lower energy gamma-ray peak is located at the channel location (within one channel) specified in Table A.1.

h) Repeat steps c) and d) until the higher and lower gamma-ray peaks are both located (within one channel) at the positions specified in Table A.1.

i) From the peak energies and the channel positions calculate the energy scale with the following formulas:

$$G = \frac{E_{(\mathrm{high})} - E_{(\mathrm{low})}}{CH_{(\mathrm{high})} - CH_{(\mathrm{low})}} \qquad \text{(A.1)}$$

$$Z_i = E_{(\mathrm{high})} - G \times CH_{(\mathrm{low})} \qquad \text{(A.2)}$$

where

$E_{(\mathrm{high})}$ and $E_{(\mathrm{low})}$	are the energies of the high and low gamma-ray peaks in the spectrum,
$CH_{(\mathrm{high})}$ and $CH_{(\mathrm{low})}$	are the channel positions of the high and low gamma-ray peaks in the spectrum,
G	is the gain in keV/channel, and
Z_i	is the zero intercept in keV.

j) Document completion of this procedure in the instrument logbook including the ADC conversion gain, amplifier gain (keV/channel), the two gamma-ray energies selected and the respective channels at which the gamma-ray peaks were positioned.

A.4 Check of the multichannel analyser (MCA) real-time clock

A.4.1 Rationale

To ensure that accurate measurement of the counting time is achieved, the elapsed-time clock of the ADC is checked. The following procedure may be used to satisfy the test in 7.2. A pulse-height-stable pulser with a precise repetition rate is required. A calibrated oscilloscope may be useful to adjust the pulse-height of the pulser pulses.

A.4.2 Instructions

a) Disconnect all inputs to the amplifier.

b) Check that the appropriate amplifier shaping-time constant, baseline restorer, input polarity, and gain have been selected. Check the ADC conversion gain, lower-level discriminator and zero settings, and confirm that the ADC is in the singles pulse-height analysis mode.

c) Select a pulser repetition rate between 50 and 1 000 pulses per second. The shape (i.e., rise and fall times) of the pulser pulses feeding the amplifier does not need to closely approximate the size and shape of preamplifier output gamma-ray pulses for this purpose.

d) Connect the output of the pulser to the input of the amplifier, and collect a spectrum for a preset counting time of 100 s to 1 000 s.

e) Sum the counts stored in the MCA and compare with the number of pulses that were emitted from the pulser during the counting time.

f) If the measured number of pulser pulses deviates from the number emitted from the pulser by more than 0,3 % the reason for the difference shall be identified and corrected.

g) Document the results in a logbook assigned for this spectrometer.

A.5 Digital electronics

Digital electronics combine the amplifier, ADC and high voltage power supply into a single unit. In a digital system, the signal is sampled as soon as possible by an ADC, these samples are then digitally processed (i.e., filtering, dead-time processing, amplification, pile-up rejection) [49]. Digital electronics have a large choice of parameters, the instrument manual and manufacturer specifications should be used to correctly setup this type of system.

These systems have improved methods to account for counting losses at high counting rates by extending the acquisition time, are stable against variations in temperature and provide a better energy resolution compared to analog electronics.

Digital pulse processing (DPP) or digital signal processing (DSP) is now replacing the traditional analog pulse processing. It generally includes the amplifier and ADC (including also high-voltage and preamplifier (PA) low voltage supply) in a single module. Compared to the traditional analog approach, in which the conversion to digital signal is performed only at the end of the chain, the digitization is performed as close as possible to the source in a DPP system. The PA output is sampled and digitized at a high frequency, producing a string of discrete digital values. Then, as provided by the conventional amplifier plus ADC setup, this signal is filtered and shaped using numerical algorithms to reduce the noise and determine the voltage amplitude. It shall be mentioned that this mathematical processing allows a large choice of peak shapes (e.g., triangle, trapezoid, etc.) and associated parameters. In spectrometry-dedicated systems, the MCA is also included.

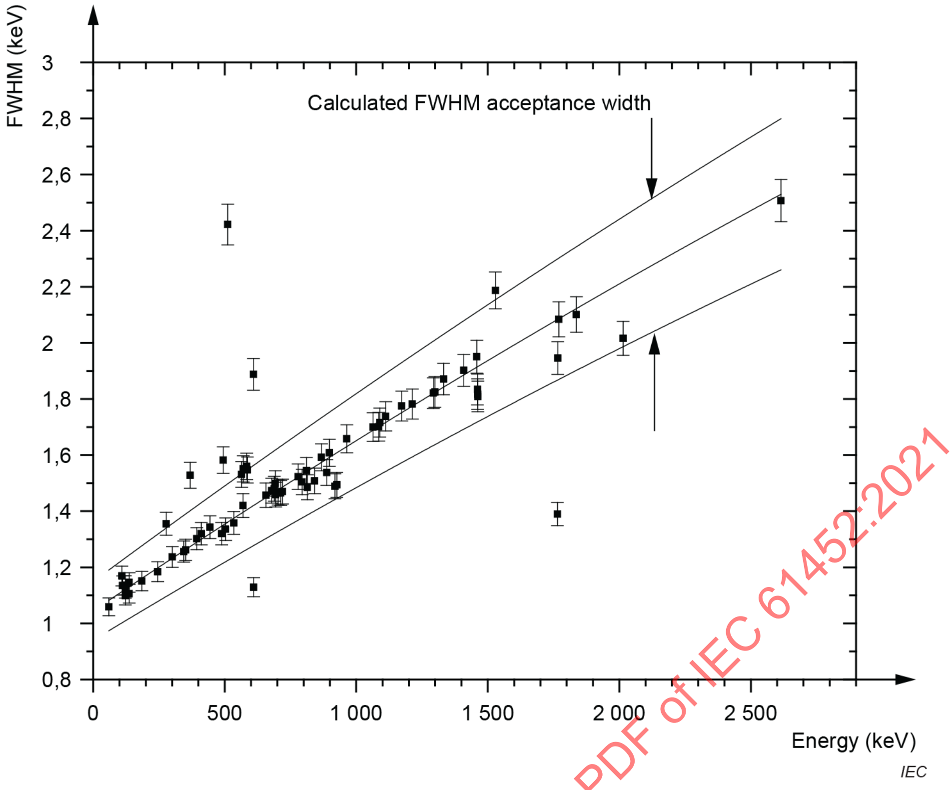
Due to the large number of possible choices, the flexibility of DPP can nevertheless represent a drawback for the user in determining optimum settings. It can also be more difficult to check the results of changing the settings, but the use of the oscilloscope often included in commercial DPP modules should help with the optimization of the latter. As with the analog systems, the DPP setting adjustment influences the spectrum quality in terms of energy resolution and peak shape directly and, ultimately, the uncertainties associated with peak area. The result of an automatic adjustment shall be checked and the use of options such as ‘permanent automatic adjustment depending on the counting conditions’ should be avoided.

A.6 Measurement of energy resolution and peak-to-Compton ratio

A.6.1 Rationale

To verify the resolution specifications of the detector and to ensure that the ability of the detector to discriminate full-energy peaks has not degraded with time. Through the measurement of the FWHM of several full-energy peaks ranging from <100 keV to >1 000 keV, an approximately linear function can be determined as shown in Figure A.2. For a given detector, a rigorous expression relating the peak width (in energy units) to the energy of the full-energy peak is $(\text{FWHM})^2 = mE + b$ where E is the energy and m and b are constants. This function may be useful in peak fitting or in judging the quality of a fit. Often when the vacuum of the detector cryostat begins to degrade, or an electronic component degrades or fails, it can be first recognized from the degradation of the energy resolution.

The following procedure may be used to satisfy the test in 7.5. Sources of ^{57}Co and ^{60}Co approximating point source geometry (with activities of about 40 kBq to 200 kBq) are needed in this procedure.



NOTE Resolution of the full-energy-peak as a function of gamma-ray energy. Peaks whose FWHM fall outside the region contained by the outer two lines are questionable and may be either poor fits or spectral artefacts that are not real peaks. This is one method by which false peaks can be identified.

Figure A.2 – Distribution of FWHM of spectral peaks as a function of energy

A.6.2 Measurement of the energy resolution at 122 keV and 1 332 keV

A.6.2.1 Resolution at 122 keV

a) Establish the settings of the HPGe spectrometer system so that the gain is approximately 0,20 keV/channel.

b) Center a ^{57}Co source on the detector axis at a distance from the detector that establishes a counting rate of about 500 counts per second.

c) Acquire a spectrum with about 3 000 counts in the peak centre channel for the gamma-ray at 122,06 keV. If possible, adjust the fine gain so that there is an integer channel located within ±0,1 channel of the peak position and reacquire a spectrum. (Having an integer channel located at the mean position of the peak will simplify the measurement of the energy resolution.)

d) Perform an energy calibration using the 122,06 keV and 136,47 keV gamma-rays (by inclusion of a ^{60}Co source, the gain may be measured more accurately as in step d) of A.6.2.2). If the ADC zero was previously set, the intercept term should be close to zero even at a different gain.

e) The FWHM is determined with the peak analysis program.

f) Document the FWHM energy resolution measured at 122,06 keV.

A.6.2.2 Resolution at 1 332 keV

a) Center the source on the detector axis at a distance from the detector to establish a counting rate of 500 counts per second. Locate the ^{60}Co source in the position it occupied in the above energy resolution measurement procedure.

b) Acquire a spectrum with about 3 000 counts in the peak channel for the gamma-ray at 1 332,49 keV.

c) Perform an energy calibration using the 122,06 keV and 1 332,49 keV gamma-rays.

d) Determine the FWHM of the 1 332,49 keV peak with the procedure described above.

e) Document the FWHM energy resolution measured at 1 332,49 keV.

NOTE It is possible to perform both measurements in one step using simultaneously ^{57}Co and ^{60}Co source(s).

A.6.3 Measurement of the peak-to-Compton ratio for ^{60}Co

a) With the ^{60}Co spectrum accumulated in A.6.2.2, sum the counts in the spectral region from 1 040 keV to 1 096 keV, and divide the sum by the number of channels over which the sum was taken. This result is the average number of counts in the channels of the Compton region below the 1 332,49 keV peak.

   Precaution – When measuring the peak-to-Compton ratio for the purpose of verifying the manufacturer's specification, the region surrounding and in between the source and the detector should be at least 10 cm from any mass that may scatter ^{60}Co gamma-rays into the detector so as to cause an increase in the counts in the Compton continuum region of the spectrum and a reduction in the measured peak-to-Compton ratio.

b) Calculate the peak-to-Compton ratio by dividing the 1332,49 keV peak height (counts in the mean channel position of the peak) by the average Compton counts calculated in step a).

c) Document completion of this procedure in the instrument logbook with the peak-to-Compton value and the energy (1 332,49 keV) at which it was measured.

A.7 Correction for losses due to counting rate

A.7.1 Rationale

To correct for all counting-rate effects that can result in the loss of counts from the full-energy peaks and to inform the user of the various methods in use to carry out this correction, as well as their limitations.

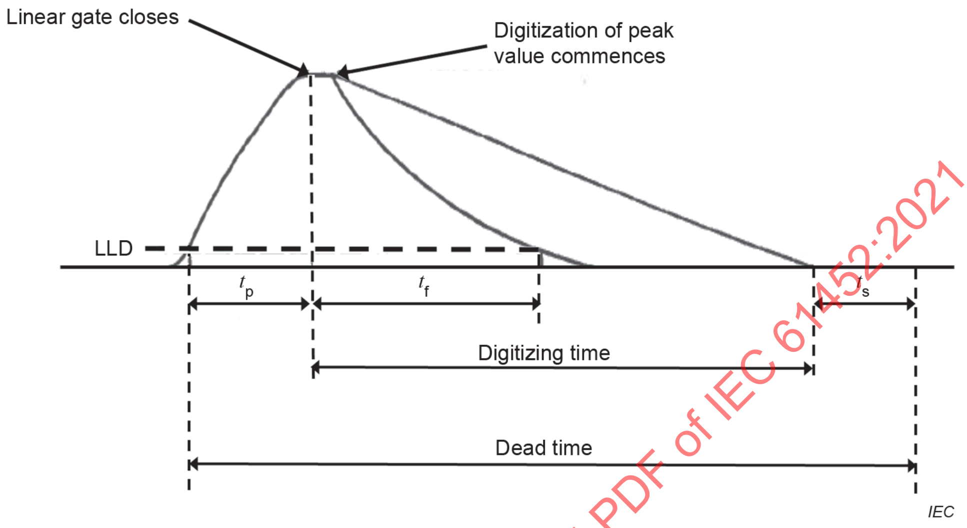
System dead time is the amount of time during a counting period that the pulse-height analysis system cannot correctly process another pulse. As shown in Figure A.3 the dead time as defined here includes the amplifier output pulse width (from the time it crosses the lower-level discriminator) plus any extra time required for digitalization (may be variable) plus the digital storage time, t_S. By turning off the live-time clock during these dead times and calculating peak-area counting rate (net peak area divided by counting live time), a correction for counting losses to the spectral peaks due to dead time may be accomplished. Most modern analog-to-digital converters (ADC) provide a close approximation for this correction. The live-time clock is turned off whenever the lower-level discriminator is exceeded by incoming pulses, and it remains off until the voltage level returns below the lower-level discriminator setting and the digital value of the previous pulse is stored. The live-time clock does not, however, correct for counts lost from the peak due to pile-up losses (see 3.1).

Methods by which pulse pile-up corrections can be made are:

a) empirical or source;
b) live-time extension;
c) pulser; and
d) virtual pulser and add "N" counts method.

(The method used in 7.6 is a variation of the empirical method and can be adopted to measure the first-order correction for pulse pile-up by measuring the net peak-area counting rate of the fixed source as a function of the integral counting rate resulting from the movable source). Each of these techniques is discussed in A.7.2 through A.7.5.

In any pile-up loss correcting technique, it is absolutely necessary to characterize the limitations of the equipment and technique used, and to operate within the limits of the system. This characterization involves the technique employing a fixed and a movable source or, alternatively, could involve the technique employing a set of sources of accurately known intensity ratios (see A.7.2). By using these techniques to demonstrate system performance, some operation outside the expected limits of a spectrometer system may be justified if its operation is well characterized and corrections can be empirically applied.



NOTE Unipolar amplifier output pulse showing the LLD setting, the time t_p, to reach peak value, the time t_f, from peak value till the pulse falls below the LLD, the digitizing time, the dead time, and the time t_s, required to store an analysed pulse.

Figure A.3 – Specification of times for pulse processing by an ADC

A.7.2 Empirical or source method

This method (see [17]) empirically measures the amount of pulse pile-up as function of gross counting rate and of peak energy with a set of gamma-ray-emitting radioactive sources of a given radionuclide whose relative emission rates are known precisely. The relative emission rates are expressed as dilution factors, f_i, relative to the weakest source. (Sources are actually prepared as mass aliquots relative to the strongest source.) A set of seven or eight ^{152}Eu "point" sources ranging from a few hundred counts per second to approximately 20 000 s^{-1} or more should either be prepared by taking mass aliquots of a solution of the radionuclide or be procured from a reputable radionuclide supplier. The precision of the relative emission rates of these sources should be known to <0,5 %. The integral counting rate of the HPGe spectrometer should be measured (shield door closed) from the output of the amplifier with a scaler whose discriminator is set just above the noise. However, for counting rates below 20 000 s^{-1} and for an amplifier shaping-time constant $t_{1/2} \leq 5\ \mu\text{s}$, an acceptable method is to add all of the spectral counts (assuming that only a small fraction of pulses are truncated by the ADC upper level discriminator). This latter method of measuring the integral counting rate can be performed by a subroutine of the analysis program. With the source method of counting-rate correction, the losses due to pulse pile-up and ADC dead time are made separately.

Procedure:

a) Verify that all the spectrometer settings, including the gain, are the same as will be used when counting routine samples. Changes in the detector bias, pole-zero cancellation, DC off-set and amplifier shaping-time constant can cause significant changes in the pulse pile-up correction.

   Recommendation – When operating a HPGe spectrometer at high counting rates (i.e., >20 000 s^{-1}), it is best to choose an amplifier shaping-time constant that reduces the pulse pile-up without seriously degrading the energy resolution.

b) Acquire a background spectrum at the same gain and for a counting time equal to or exceeding that used for the weakest ^{152}Eu source to be counted. Measure the net areas of all the peaks present and divide each area by the live time of the count. Tabulate the net area counting rates and associated peak energies.

c) Count each of the sources comprising the set of sources, starting with the weakest, for a known counting time and at a chosen and reproducible source-to-detector distance so that comparable statistics are obtained (i.e., at least 20 000 and preferably 50 000 counts in the net peak areas of the gamma-rays of interest). The counting rate of the weakest source should be at or below 500 s^{-1} so that the correction for pile-up with this source is negligible. The "true" net peak counting rates for the other sources will be derived from this measurement and the known ratio of source aliquant masses[2]:

$$n_\mathrm{r}(E) = M_1(E) f_i \tag{A.3}$$

where, at energy E

$n_\mathrm{r}(E)$ is the "true" net peak counting rate for source i with a gross counting rate, r

$M_1(E)$ is the measured net peak counting rate of the weakest source (i = 1); and

f_i is the ratio of the mass aliquants of source i to source 1.

Select at least one low-energy gamma-ray (approximately 100 keV), one medium-energy gamma-ray (approximately 600 keV), and one higher-energy gamma-ray (>1 000 keV) to be analysed.

Precaution – Follow accepted procedures when handling high-activity radioactive sources to prevent unnecessary radiation exposure of personnel or accidental detection by other active counting systems in the laboratory. It is prudent to leak check all sources before use (and periodically thereafter), and to label all radioactive sources with their date, radiation field at contact, and activity. To avoid unwanted interferences, keep radioactive sources in a well shielded storage location when not in use.

d) Analyze each spectrum with the same analysis program as is used in the analysis of routine samples. Measure the net areas of the peaks of interest. If there is a corresponding peak in the background count, multiply the counting rate of the peak in the background count by the counting time for which the ^{152}Eu source was counted and subtract it from the net area. (The net area will already be corrected for the dead time attributed to the pulse processing occurring in the ADC.)

e) For a gross counting rate (or alternatively, the spectral counting rate), r, the correction is:

$$C_r(E) = \frac{n_r(E)}{M_r(E)} \tag{A.4}$$

f) Plot C_r as a function of r for each peak energy E of interest. These functions should closely approximate a family of straight lines converging at the intercept of zero integral counting rate as shown in Figure A.4. These corrections may be either incorporated into the analysis software or made manually. When the correction is energy dependent, the correction of peaks whose energies fall between those measured can be made either by an interpolation process or approximated by choosing the correction factor closest in energy to the peak to be corrected.

g) Document completion of this procedure in the instrument logbook.

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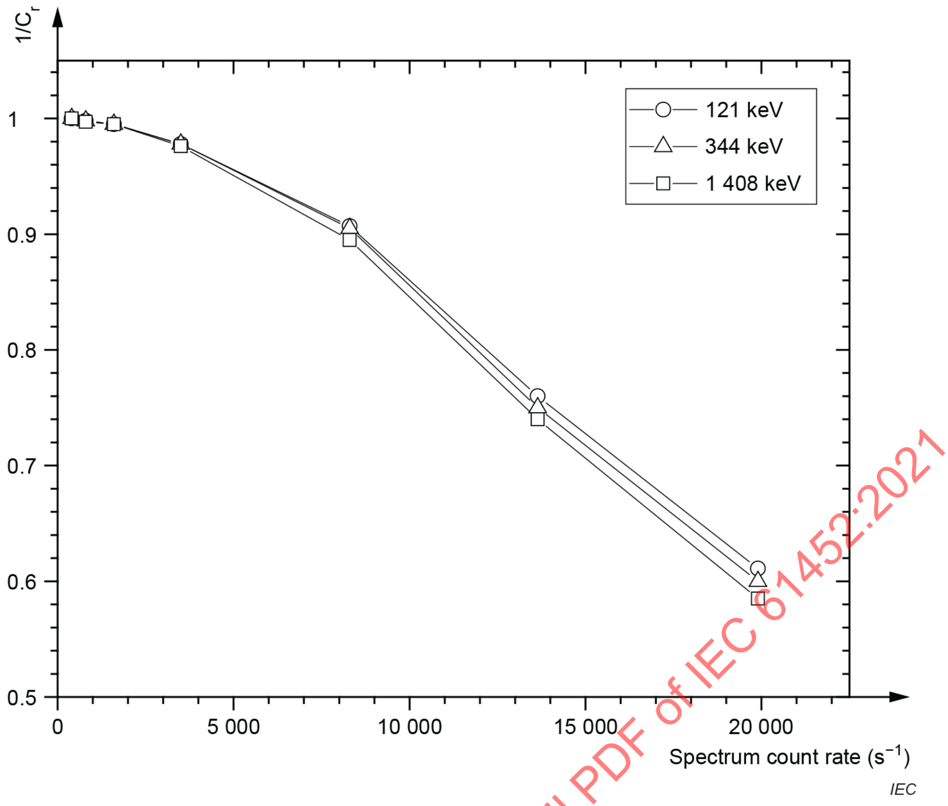
[2] If a set of ^{152}Eu sources whose relative emission rates are precisely known has been obtained from a radionuclide supplier, the values of f_i should be provided by the supplier.



NOTE Reciprocals of the multiplicative correction at three gamma-ray energies due to pulse pile-up as a function of the integral counting rate, *r*, for a 4 µs amplifier shaping-time constant and a fixed digitalized time.

Figure A.4 – Pulse pile-up correction as a function of integral counting rate

A.7.3 Live-time extension method (see [18])

In all modern ADCs an internal linear gate shuts the input off at peak detect time, thus preventing pile-up interference after peak detect time, t_p, (see Figure A.3). However, during time t_p pulse pile-up may occur, which will alter the pulse height and cause two pulses to be stored as one pulse within the spectrum. If one or both pulses would have fallen in a spectrum full-energy peak, it has now been removed, and the live-time correction will not restore it. The resultant pulse stored in the spectrum is an artifact, and in some systems may be removed by pulse pile-up rejection circuitry which inspects for multiple input pulses during time t_p. This is usually accomplished by a "fast" channel and discriminator whose pulse width is much shorter than t_p. Multiple pulses during t_p generate an inhibit signal to the ADC which causes it not to store the artifact pulse-height value. Three important issues about pile-up rejectors are to be realized:

a) The fast channel also has a finite dead time during which it cannot detect a pulse pile-up. Therefore, not all piled up pulses are removed from the spectrum.

b) The pile-up rejecter itself does not correct the peak area for the pulse lost.

c) Care shall be taken in the pile-up rejecter design to ensure that the pile-up rejecter is not energy dependent, i.e., more effective in one energy range than another.

Pile-up losses during time t_p have been compensated in various ways by different investigators, usually by extending the counting time by gating the live timer off. In most cases, the method involves counting all the fast channel pulses during a fixed real time and then extending the counting time long enough to compensate for the lost pulses. All such systems suffer from the fast channel having a finite dead time of its own, so that not all pile-up losses are compensated. The ADC peak-detector circuitry itself inserts an energy-dependent pile-up loss term because t_p may be longer for very small pulses than for large pulses. The absolute width of a gamma-ray peak increases as a function of energy for HPGe detectors, thus contributing to the energy dependence of pile-up losses. For example, an addition of approximately 5 mV to the pulse height, due to pile-up, on a pulse destined for a spectrum peak whose width is 4 mV will probably result in that pulse being removed from the spectrum peak, whereas, the same 5 mV addition to the pulse height on a pulse destined for a spectrum peak whose width is 15 mV may not result in that pulse being removed from the peak. Thus, a straightforward extension of counting time to compensate for pile-up losses is, at best, a first order correction though it does allow for more accurate spectrometry at higher rates than simple live-time correction. This method can be expected to correct the peak area to within ±2 % for counting rates up to 15 000 s^{-1} for an amplifier shaping-time constant <2 μs.

At counting rates high enough to be affected significantly by the fast-channel dead time, some have used an additional counting time extension which interpolates the error in the fast channel and forces the analyser to count even longer. These systems can raise the allowable input rates somewhat higher than for straightforward counting time extension but may not be able to provide for energy-dependent counting loss correction. Unless demonstrated otherwise, this method should not be assumed to be accurate above approximately 20 000 s^{-1}.

Warning – All of the live-time extension systems discussed in this section depend upon the relative spectral energy content not changing during the counting time. The corrections all assume a uniform correction for all peaks in the spectrum. The virtual pulse generator (see A.7.4) can be operated in the live-time-extension mode without many of the limitations stated above.

A.7.4 Pulser method (see [10], [14] and [17] to [22])

A.7.4.1 General

Another method of counting loss correction involves the use of a precision pulser. One or more fixed-amplitude pulses are injected into the preamplifier test input at a known rate, or at least a known number of pulses are injected into the preamplifier during the counting time. Then, in theory, any pulses that do not appear in the resulting spectral pulser peak were lost due to multichannel analyser dead time or pulse pile-up. Correction for losses from the spectral gamma-ray peaks may be made from the analysis of the pulser peak data. If carefully implemented, this technique can be very powerful and can correct even for energy-dependent counting losses. It cannot, however, do "real time" correction if the spectral shape changes during a given counting time.

There are several potential problems, but easily solvable, involved with the pulser method. The first of these deals with the charge injection itself. Applying a voltage pulse to the charge sensitive preamplifier test capacitor (see Figure A.5) injects an amount of charge equal to $V \times C_{\text{test}}$, as this voltage pulse returns to zero value, it removes this same amount of charge from the preamplifier input. If the pulse has a long exponential decay (commonly known as a "tail pulse"), a long tailed undershoot will occur at the shaping amplifier output which is a pulse baseline disturbance and will interfere with subsequent gamma-ray pulses to varying degrees depending upon the characteristics of the associated baseline restorer. The pulser pulses have a two-pole response whereas the gamma-ray pulses have a single-pole response at the charge amplifier output. The pole-zero cancellation in the shaping amplifier may be set to assume a stable pulse baseline following the gamma-ray pulses but not following the pulser pulses. As shown in Figure A.5, the only shaped pulser pulse which does not upset the shaping amplifier baseline long term is a square flat-top pulse. However, a square flat-top pulse produces both a positive and a negative pulse at the shaping amplifier output. The negative pulse shall be blocked from the restorer and ADC and correction made for the dead time it introduces. However, this time can be kept to less than 0,5 % in most cases.

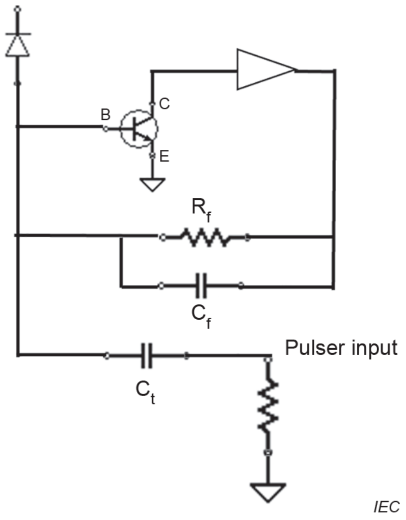
There are several pulser methods which can be used to measure the "system" dead time (i.e., due to pulse pile-up and analyser pulse processing time):

a) a pulse height variable and periodic pulse generator set at a fixed pulse height;

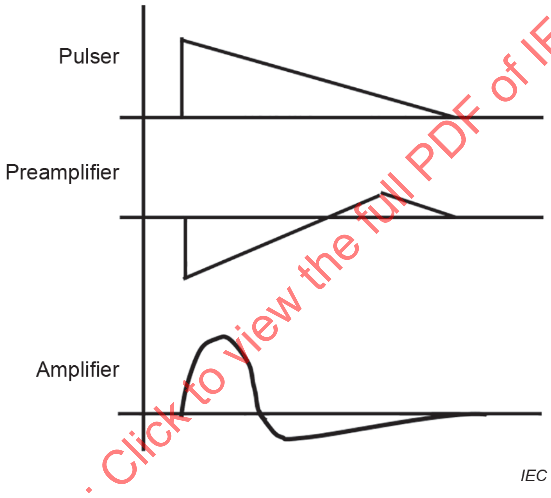
b) a pulse height variable pulse generator set at a fixed pulse height that injects pulses on demand (sometimes inappropriately referred to as a random pulse generator); and

c) a single or dual pulse generator of type 1 or 2 that is tagged and routed to a separate memory location as it is processed by the ADC.

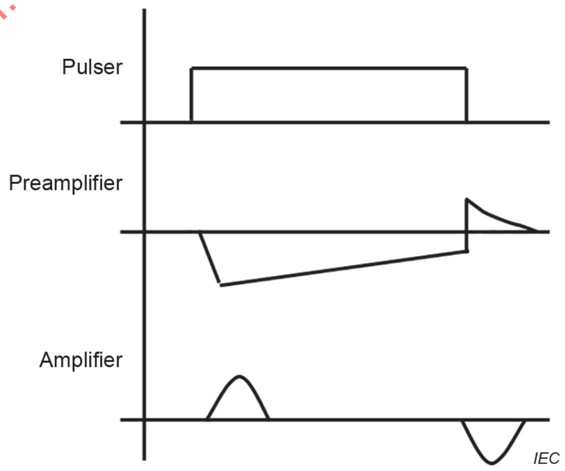
In all cases a square flat-top pulse is recommended to be injected into the input of the preamplifier through a charge injection capacitor internal to the detector cryostat. Methods using pulser peak areas to correct for counting losses out of varying width spectral peaks have been developed.



a) The portion of the preamplifier circuit showing where the pulse is injected



b) Pulse shapes as measured at the preamplifier and amplifier outputs resulting from the injection of a pulse with steep-rise time and a long-fall time



c) Pulse shapes as measured at the preamplifier and amplifier outputs resulting from the injection of a square, flat-injection of a square, flat-top pulse with a sharp rise and fall time

Figure A.5 – Preamplifier and amplifier pulse shapes resulting from different pulser shapes

A.7.4.2 Pulser method 1

This is the easiest pulser method to set up. It uses a variable amplitude and periodic pulser whose repetition rate is precisely known. It assumes that the source counting rates are constant during the count. Large variations (i.e., a factor of two or more) in counting rate or rapidly decaying sources may not be handled correctly. In addition, to avoid large systematic errors, the dead times produced by the pulser counting rate should be a factor of ten less than the dead time produced by the source being measured (i.e., pulser counting rates should be kept as low as adequate statistics will allow). Further, the shape of the pulser peak in the spectrum is different from a gamma-ray peak and allowances should be made for this difference. This can be done by:

a) transformation of the pulser peak to a Gaussian shaped peak with the same net pulser peak area but with the resolution of a gamma-ray of equivalent energy; or

b) minimization of the peak shape differences by measuring the peak areas by summation techniques.

This method can be expected to correct the peak area to within ±2 % for counting rates up to 50 000 s^{-1} for an amplifier shaping time constant <2 μs but it makes no provision for energy dependent counting losses.

A.7.4.3 Pulser method 2

This method can be used to accurately correct for pulse pile-up when counting rapidly decaying sources with a relative spectral shape that is constant with time (significant decay of similar half-life radionuclides during the counting period) or when counting sources with widely varying counting rates. It is based on the use of a pulse generator that can inject a pulse train at the preamplifier after a preset number of preamplifier output pulses have been counted (accurate counting of input pulses at high counting rates may be difficult). Some authors refer to this method as the random pulse generator technique. At the end of the counting period, the total number of injected pulses is compared with the number of these pulses that are recorded. Again, the differences in shape of the pulser and gamma-ray pulses shall be handled as described in method 1 above. The counting rate limitations of this method are similar to those of pulser method 1 (A.7.4.2).

A.7.4.4 Pulser method 3

This method involves the injection of a low- and a high-voltage pulse from a dual pulse generator with subsequent removal of these pulses so that they are not stored in the gamma-ray spectrum but rather in another memory location. By calibrating their pulse height relative to gamma-rays of known energy, the pulses can be made energy equivalent and used as an ongoing energy calibration of the spectrum. By storing the pulser information separate from the spectrum, the lower-energy pulses are not masked by the gamma-ray spectral counts at high counting rates; this method also avoids interference of the pulses with the gamma-ray peaks in the spectrum. This technique permits not only the correction for pulse pile-up as a function of energy but also the monitoring of the stability of the gain and zero. With this method peak areas may be corrected to an accuracy of ±2 % for counting rates as high as 250 000 s^{-1} for an amplifier shaping-time constant of <2 μs for the entire range of gamma-ray energies being detected.

A.7.5 Virtual pulser and add "N" counts method

A system which does a "real-time" correction is the method in which the MCA does an "add N" to the contents of a pulse-height channel. In the add "N" counts (see [23]), "N" is determined by counting the fast-channel pulses missed during pulse processing, adding a correction for fast-channel dead time, and adding "N" counts to the pulse-height channel being processed. In the virtual pulse generator method, (see [22] and [24]), "N" is determined by inspecting the ADC dead time and shaping amplifier dead time to determine the ratio of pulses that would have been processed or lost if pulses had been injected. The method does not actually inject pulses, so the "virtual" pulse rate may be very high and the "add N" can be adjusted in a short time frame with good accuracy to allow true "real time" correction. The virtual pulse generator can also be used in a live-time extension mode to correct for pulse pile-up. These methods make no energy-dependent correction.

However, this method does permit the counting rate to vary during a count. This method can be expected to accurately correct for pulse pile-up for peaks throughout the entire spectrum at integral counting rates up to 250 000 s^{-1}. Since "N" pulses have been added to a channel upon the processing of a pulse, determination of the uncertainty in the peak areas is no longer straightforward.

A.8 Measurement of the full-energy peak efficiency curve

A.8.1 Rationale

The full-energy-peak efficiency is used to convert the net counting rates in the full-energy peaks to gamma-ray emission rates. If the detector is calibrated for the analysis of specific radionuclides, the efficiency values (or standardization coefficients) need only be measured at the specific energies represented by the key gamma-ray(s) associated with each radionuclide. The following three sections provide procedures for three experimental conditions:

a) calibration for specific radionuclides;
b) measurement of the full-energy-peak efficiency as a function of the energy for large source-to-detector distances; and
c) measurement of the full-energy-peak efficiency as a function of energy for small source-to-detector distances.

These procedures may be used to satisfy the requirements of 5.5.

For these procedures, the radioactivity standards shall have the same geometrical shape and should preferably be composed of the same matrix material as the samples to be analysed. See 6.2.6 for the measurement or computation of attenuation corrections when the sample matrix has a different density than the calibration standard. If the attenuation correction is not made, an estimate of the magnitude of the error in the resulting gamma-ray emission rate shall be folded into its uncertainty. The standards shall be from a country's National Metrology Institute (NMI) or from a qualified supplier that has demonstrated its traceability to the NMI. Precise measurements of gamma-ray relative intensities and gamma-ray emission intensities are given in the literature (see [8], [50]).

A.8.2 Measurement of standardization coefficients for specific radionuclides

a) Accumulate energy spectra for a known live time using a radioactivity standard for each radionuclide of interest at the desired source-to-detector geometry. At least 20 000 and preferably 50 000 net counts shall be accumulated in the full-energy peaks of interest. The gross counting rate of each standard should simulate that of the samples. If possible, the counting rate should be kept below 1 000 s^{-1} so that pulse-pile-up corrections are not required. If the counting rate of the standard or samples are >1 000 s^{-1} and differ, corrections for pulse pile-up shall be applied (see Clause A.7).

b) Calculate the standardization coefficient, $S_c(i, E)$, using a gamma-ray of energy E with the following formula:

$$S_c(i,E) = \frac{[n(i,E) - B(E)] \times C_r \times C_d}{A(i)} \tag{A.5}$$

where

$n(i, E)$ is the net area counting rate (expressed in s^{-1}) in a full-energy peak of energy E associated with radionuclide i (i.e., the continuum as defined in the text is removed);

$B(E)$ is the net area counting rate (expressed in s^{-1}) in the energy region E, associated with a background peak;

C_r is the correction for random summing (see 6.2.4);

C_d is the correction due to decay during and prior to the count (see 6.2.3); and

$A(i)$ is the activity in becquerels (Bq), at the measurement time, of the radionuclide i in the calibration source.

c) When an unknown source activity of radionuclide i is measured:

$$A(i) = \frac{[n(i,E) - B(E)] \times C_r \times C_d}{S_c(i,E)} \tag{A.6}$$

d) Prepare a table listing the standardization coefficients for each of the radionuclides for which an analysis will be performed. Note that there can be a standardization coefficient for more than one gamma-ray for each radionuclide.

A.8.3 Measurement of the detector efficiency versus energy for large sample-to-detector distances

Standards prepared from a mix of all or a subset of the following radionuclides: ^{241}Am, ^{109}Cd, ^{57}Co, ^{139}Ce, ^{203}Hg, ^{51}Cr, ^{113}Sn, ^{85}Sr, ^{137}Cs, ^{88}Y, ^{65}Zn and ^{60}Co, from the long half-life mix of ^{125}Sb and ^{154}Eu, or from ^{152}Eu or ^{166m}Ho are the most popular standards in use. A spectrum of the mixed radionuclide standard mentioned here is given in Figure A.6. Other standardized radionuclides may also be used.

Precaution – Annihilation radiation (at 511 keV) is often present in spectra due to beta plus decay or pair production from the radionuclide sources or from the background. That peak may interfere with the measurement of the 514 keV gamma-ray of ^{85}Sr. Also, the single-escape peak of the 1 836 keV gamma-ray emitted in the decay of ^{88}Y lies at 1 325 keV and may interfere with the 1 332 keV gamma-ray emitted in the decay of ^{60}Co and the single-escape peak of the 1 173 keV gamma-ray emitted in the decay of ^{60}Co may interfere with the 662 keV gamma-ray emitted in the decay of ^{137}Cs.

a) Accumulate for a known live time a spectrum using one or more standards so that at least 20 000 and preferably 50 000 counts have been accumulated in the full-energy peaks of interest (only peaks whose peak to continuum ratio is at least 10 should be used to measure the efficiency). The standardized samples shall have the same geometrical shape and should be of the same matrix (material and density) as the routine samples to be analysed.

b) Determine the net counting rate of the full-energy peaks of interest using the same method for measuring the peak area as will be used for the routine analysis of samples.

c) Calculate the full-energy-peak efficiency $\varepsilon(E)$ using the following formula:

$$\varepsilon(E) = \frac{[n(E) - B(E)] \times \prod C_\mathrm{i}(E)}{R(E)} \qquad \text{(A.7)}$$

where

$\varepsilon(E)$	is the full-energy peak efficiency at energy E;
$n(E)$	is the net area counting rate in the full-energy peak of energy E;
$B(E)$	is the net area counting rate in a corresponding peak at energy E associated with the background spectrum;
$R(E)$	is the emission rate for gamma-ray with energy E (source activity time emission intensity of gamma-ray with energy E);
$C_i(E)$	are the correction factors for a radionuclide with the associated energy E (where i = d, r, c, and a as defined below); and
Π	is the operator that takes the product of the correction factors.

The correction factors shall be applied, as the experimental conditions and the accuracy require, for decay during and prior to a count ($i = d$), pulse pile-up ($i = r$), cascade summing ($i = c$), and variation in the attenuation of the sample relative to the standard ($i = a$).

d) After the efficiencies below 2 MeV have been measured, a plot of the efficiency as a function of energy shall be made (see 5.5.4). Efficiency values above 2 MeV may be measured with non-quantitative sources of the radionuclides ^{24}Na(1 369 keV, 2 754 keV), ^{56}Co(847 keV, 2 598 keV, 3 010 keV), ^{144}Ce-^{144}Pr(697 keV, 2186 keV) and/or ^{208}Tl(583 keV, 2 615 keV) (from a ^{228}Th source) by following steps e) through h). (For relative gamma-ray emission intensities for these radionuclides, see the footnote to Table A.1.) If efficiency values above 2 MeV are not required, go to step i).

e) Each sample containing a radionuclide that emits gamma-rays with energies greater than 2 MeV should be of the same geometrical shape and matrix as used in step a). Spectra shall be accumulated for a known counting time so that at least 20 000 and preferably 50 000 counts are accumulated in the gamma-ray peaks of interest.

f) The emission rate of the lower-energy gamma-ray shall be determined from the partial efficiency curve measured from steps a) to c).

g) The emission rate of the higher-energy gamma-ray is calculated from the emission rate of the lower-energy gamma-ray and their relative gamma-ray intensities shown in the following formula:

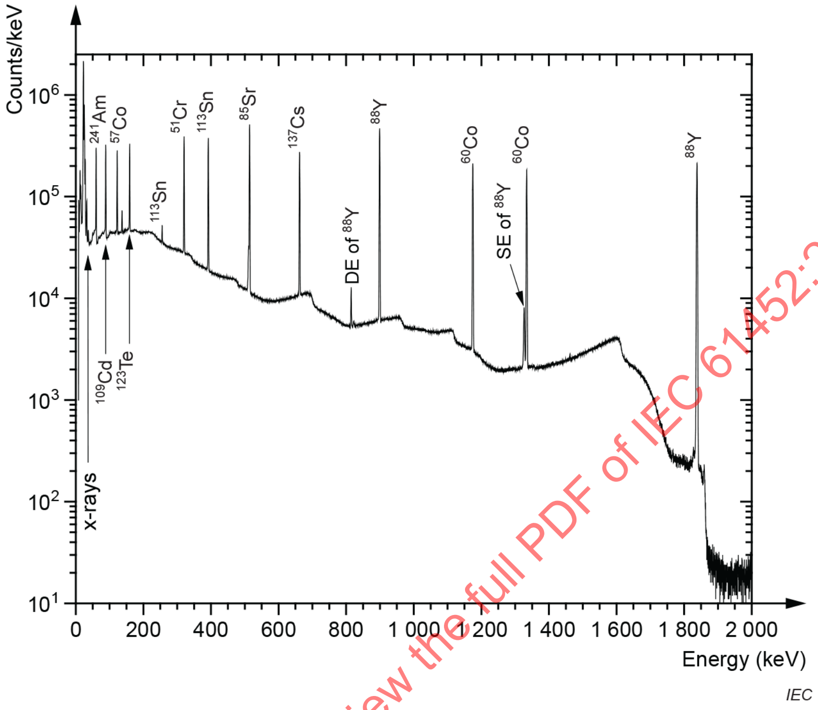
$$R(E_\mathrm{H}) = \frac{R(E_\mathrm{L}) \times P(E_\mathrm{H})}{P(E_\mathrm{L})} \qquad \text{(A.8)}$$

where

$R(E_\mathrm{L})$	is the emission rate of the lower-energy gamma-ray;
$R(E_\mathrm{H})$	is the emission rate of the higher-energy gamma-ray;
$P(E_\mathrm{L})$	is the emission intensity of the lower-energy gamma-ray; and
$P(E_\mathrm{H})$	is the emission intensity of the higher-energy gamma-ray.

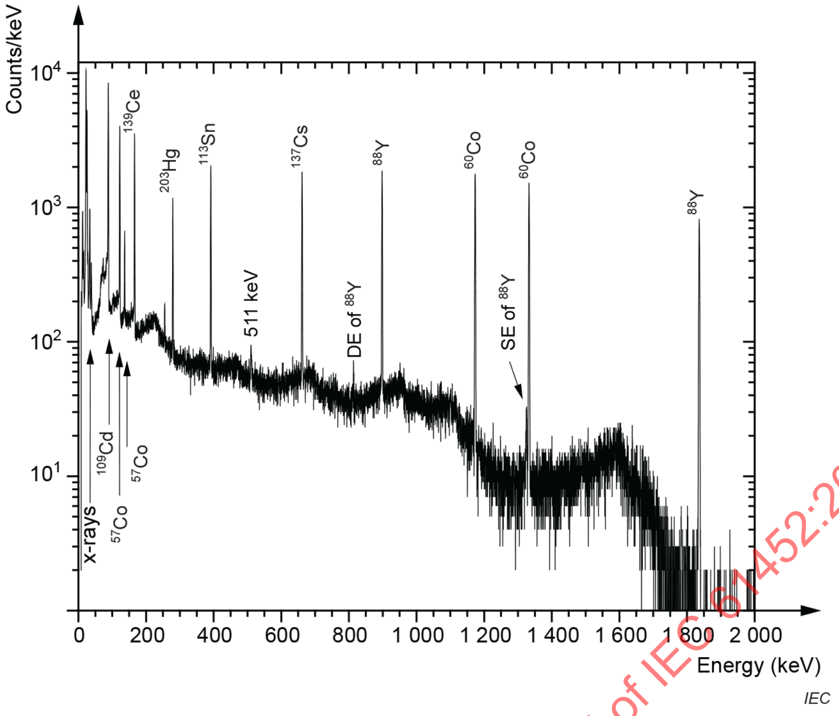
h) Use the gamma-ray emission rate determined in step g) to calculate the full-energy-peak efficiency of the higher energy gamma-ray as described in step c). If the sample containing a radionuclide that emits gamma-rays with energies greater than 2 MeV has been standardized, the efficiency can be measured directly from its known gamma-ray emission rate.

i) Complete the plot of the full-energy-peak efficiency as a function of energy using the method described in 5.5.4. From a set of reasonably spaced efficiency values taken from this plot determine a formula or formulas that best represent(s) the efficiency curve as a function of gamma-ray energy. When high accuracy or precision in the measured gamma-ray emission rates or relative intensities is required (i.e., <5 %), it is recommended that supplementary measurements be made to better define the efficiency curve. There is limit of how much the uncertainty can be reduced due to correlations between the input data.



NOTE SE and DE are the abbreviations for single and double escape peaks, respectively. These peaks are consecutive to pair production plus annihilation occurring from high-energy gamma-rays in the active volume of the detector where the annihilation photons escape from the active volume.

Figure A.6a – Spectrum of the mixed radionuclide standard containing ^{241}Am, ^{109}Cd, ^{57}Co, ^{123m}Te, ^{51}Cr, ^{113}Sn, ^{88}Sr, ^{137}Cs, ^{88}Y and ^{60}Co



NOTE SE and DE are the abbreviations for single and double escape peaks, respectively. These peaks are consecutive to pair production plus annihilation occurring from high-energy gamma-rays in the active volume of the detector where the annihilation photons escape from the active volume.

Figure A.6b – Spectrum of the mixed radionuclide standard containing ^{109}Cd, ^{57}Co, ^{139}Ce, ^{203}Hg, ^{113}Sn, ^{137}Cs, ^{88}Y and ^{60}Co

Figure A.6 – Gamma-ray spectrum of a mixed radionuclide standard

A.8.4 Measurement of the detector efficiency versus energy for small sample-to-detector distances

Coincidence summing of gamma-rays in cascade may cause large errors in the measured gamma-ray emission rates at small sample-detector distances if cascade summing corrections are not applied. Radionuclides whose associated gamma-rays are essentially free of cascade summing can be used to measure the full-energy-peak efficiency. For example, standards prepared from a mix of the following radionuclides; ^{241}Am, ^{109}Cd, ^{57}Co, ^{139}Ce, ^{203}Hg, ^{113}Sn, ^{85}Sr, ^{137}Cs, ^{54}Mn and ^{65}Zn are essentially free of detectable gamma-rays in cascade coincidence assuming that energies at or below the energies of the K X-rays of ^{139}Ce are essentially not detected. The radionuclides ^{54}Mn and ^{65}Zn should be used to replace ^{88}Y and ^{60}Co, respectively, because the latter two emit gamma-rays that are in coincidence. To measure the full-energy-peak efficiency above 2 MeV the radionuclide ^{144}Ce-^{144}Pr (697 keV, 2186 keV) may be used as described in A.8.3, steps d) through h) (see radionuclides used in A.8.3).

Follow the procedure described in A.8.3 using only the radionuclides listed above or other radionuclides that have been demonstrated to be free from cascade summing.

Warning – Some of the radionuclides recommended above for the measurement of the full-energy-peak efficiency at small sample detector distances emit X-rays or low-energy gamma-rays that are in coincidence with the gamma-ray of interest. Verify that these low energy photons are attenuated by the detector dead layer, the surrounding cryostat, an inserted (external) absorber or the sample itself so that these low-energy photons are not detected. When this is done, and the absorber is left in place for the counting of samples, no separate attenuation correction is required for the presence of this absorber since it is included in the full-energy-peak efficiency.

Warning – Although the resulting peak efficiencies will be free of cascade summing effects, in general a correction for cascade summing will be required when counting and analysing samples containing other radionuclides that emit cascade gamma-rays. For this reason, establishing an energy-efficiency curve without the capability to apply cascade-summing corrections is of limited value for tight geometries, unless the radionuclides to be analysed are known to be relatively free of cascade summing.

A.9 Preparation of reference sources from standard solutions

A.9.1 Rationale

It is important to use reliable and tested procedures to prepare working standards. Sometimes standards are not available as an off-the-shelf product in the geometry or matrix required. Under these conditions it is necessary to either purchase custom prepared sources, or to prepare them at the laboratory from solution standards that are documented to be traceable to an NMI. The preparation of aliquots by gravimetric techniques (a five-place balance is recommended) is usually preferred to volumetric methods because it is easier to learn and is less prone to error. Whenever possible, at least three standard samples should be prepared and cross-checked.

Precaution – The preparation of custom sources is an art that is best performed by technical staff who are experienced in the preparation of radioactivity standards and who are meticulous in carrying out laboratory procedures. All subsequent quantitative radioactivity measurements are directly affected by the accuracy of the standards used in the calibration of a spectrometer. Suitable precautions to avoid evaporation or uncontrolled release of radioactive contamination shall be taken when handling all radioactive materials once their containers have been opened. Resealing of unused liquid radioactivity standards is best accomplished by flame sealing in glass ampoules so that the ampoules are at least half-full.

A.9.2 Preparation of standard sources

a) Obtain a standard solution of at least 5 ml volume whose radionuclide activity concentration is known. A solution containing stable carrier ions of the various radioelements should also be obtained.

b) Dilute the carrier solution, using pure filtered water, to as much as 5 l with an aqueous solution that has been pH adjusted with a suitable acid or base as recommended by the supplier of the standard solution.

c) From the activity concentration, calculate the amount of standard solution needed to prepare the custom standard (when using a four-place balance at least 0,5 g should be transferred to avoid unacceptably large errors). Although a five or six place balance is preferred since smaller aliquots can be accurately measured, they are probably not used routinely in many plant laboratories.

d) Obtain the tare mass of a small, flat-bottomed, plastic container from which small aliquots can be easily dispensed without loss due to splattering or to droplets sticking to the lip of the container. If there is a lid, it should be on during all mass measurements. A miniature plastic squeeze vial (i.e., Pasteur pipette) (5 ml to 20 ml) with the top heated and drawn out to a capillary or a volumetric pipette is ideal for removing radioactive solutions from a container and for dispensing small aliquots. A volumetric flask is best for larger sizes.

e) Transfer the standard solution to the small plastic container. If the activity concentration is given with the standard, it is not necessary to transfer all the solution. Obtain the mass of the container plus the standard solution. The mass from this step, minus that from step d) is the mass of the transferred solution.

f) Obtain the tare mass of the sample container that will hold the custom standard. Be sure that the container is identical in dimensions to the containers to be used for the samples to be analysed.

g) Transfer to the sample container enough carrier solution to half fill it.

h) Transfer to this container an amount of solution standard that will result in a sample that is neither too high nor too low in activity for the counting geometry to be used (a counting rate of <1 000 s^{-1} is recommended). Determine the mass of the transferred solution by the mass difference technique used in steps d) and e).

i) Fill this container to almost the desired volume with carrier solution while stirring vigorously to mix it thoroughly. Rinse the stirring rod with the remainder of the carrier solution over the container. Determine the full mass of the custom standard. Subtract from this mass the tare mass in step f).

j) Determine the total activity of each radionuclide present in the standard. Label the sample container with the activity concentration in becquerels per gram of solution (Bq/g) and associated date, volume and density of the sample, dimensions of the container, and other identifying features. Also record this information in a logbook.

Precaution – Residue or debris in a liquid sample can cause a partitioning of the radioactivity. Therefore, if such debris is present, it should be removed by filtration and the filtered material counted separately. The total activity of this type of sample is the sum of that in the solution and that on the filter.

A.9.3 Preparation of soil sources

The following procedure is for the preparation of soil standards for use in gamma-ray spectrometry (mass standards > 50 g). Soil samples of different densities may also be prepared from different types of soils. The soil should be counted to demonstrate that it does not contain any radionuclides that might interfere with the gamma-rays emitted from the radioactive standard.

a) Measure out into a plastic polyethylene bag with a zip locked closure the amount of soil (previously milled or sieved to a selected particle size) needed to fill the sample container to the proper volume.

b) Transfer to this bag a mass of solution standard that will result in a sample that is neither too high or low in activity for the counting geometry to be used. At least 5 g of solution should be transferred directly to the soil to minimize the possibility of an inhomogeneous standard. A resulting consistency of thick mud is ideal. If this would result in too high an activity, first dilute the standard solution using the procedure in A.9.2. Determine the activity concentration of the diluted standard from the masses of the transferred standard solution and of the transferred carrier solution. A counting rate of <1 000 s^{-1} is recommended.

c) Seal the bag and mix or knead the bag with its contents for 10 min to 15 min so that the solution becomes intimately mixed with all the soil. There should be enough soil to avoid the presence of any free liquid.

d) Let the sealed plastic bag sit overnight.

e) Knead the contents the following day for 10 min to 15 min. Be sure that there are no visible free-liquid droplets.

f) If the soil appears almost dry, carefully place the contents into the sample container. Be sure that it all fits into the sample container and fill it to the correct volume. If additional soil needs to be added, step c) should be repeated with added soil.

g) If the soil is wet or damp, carefully tear the plastic bag open over a plastic film and allow it to air dry. Be careful not to lose any of the soil.

h) If the soil has formed clods upon drying, place it into a clean plastic bag with a zip closure, seal it and break up the clods by kneading the soil through the plastic bag.

i) Carefully place the contents into the sample container. Be sure that it all fits into the sample container and fills it to the correct volume. If additional soil needs to be added, step c) should be repeated with the added soil.

j) Count the empty plastic bags overnight in close geometry with a HPGe spectrometer to be sure that no appreciable amount of radioactivity remained with the plastic bag. At least 99 % of the activity of each radionuclide shall be in the sample if that radionuclide is to be used as a standard.

k) Checks of homogeneity of the sample are best made by carefully dividing the mixed sample into several portions and counting each portion in the same sample geometry and sample-to-detector distance.

A.9.4 Preparation of filter sources

There are different methods to prepare filter source [56], [57], [58]. These methods rely on dispensing equal size drops of the solution regularly spaced on a filter or similar geometry. The solution drops can be dispensed on the filter using an automated or manual system.

It is important that the distribution of the radioactive material is homogeneous over the surface of the filter. Homogeneity can be demonstrated during construction.

Annex B
(informative)

Measurement of peak position, net area and their uncertainties

B.1 General

Both non-fitting and fitting methods can be employed to measure peak position and net peak area. Two methods will be outlined here, a simple-peak integration method that can be easily carried out with a calculator or small computer and a method in which a normal (Gaussian) function is fitted to the data after the continuum has been subtracted. Note that the peak area measurement method and the type of ADC used may influence the energy dependence observed in the measured pulse pile-up correction (see 7.6). Methods that use all the spectral peak, including low- and high-energy tails, will tend to have less energy dependence in the pile-up correction. (See [14].)

B.2 Non-fitting technique

A typical well-resolved spectral peak is illustrated in Figure B.1. The region comprising the peak extends from channel L to channel R and is equal to C channels that make up a known portion of the peak (e.g., >99,7 % of the area).

The net area of the peak is then obtained from:

$$N = G - C\frac{B_1 + B_2}{2c} \tag{B.1}$$

where

G is the gross counts (integral) under the peak;

C is the number of channels in the peak;

c is the number of background channels on each side of the peak;

B_1 is the sum over c channels of continuum below the peak;

B_2 is the sum over c channels of continuum above the peak.

The number of channels in B_1 and B_2 is determined by the shape of the spectrum adjacent to the peak and the channel-to-channel variation in the counts. When there are no close lying peaks, the number of channels in each B_1 and B_2 is typically between 1 and 1,5 times the FWHM of the peak where the FWHM is expressed in channels. The measured standard deviation associated with the net area A is given by:

$$s(N) = \left[G + \left[\frac{C}{2c}\right]^2 (B_1 + B_2)\right]^{0,5} \tag{B.2}$$

The background in each channel under the peak may be approximated by a straight line which is obtained from a linear fit to n channels of the continuum on each side of the peak. The peak position may then be determined from the first moment, x_0, given by:

$$x_0 = \frac{1}{N}\sum_{i=L}^{R} i\,N_i \tag{B.3}$$

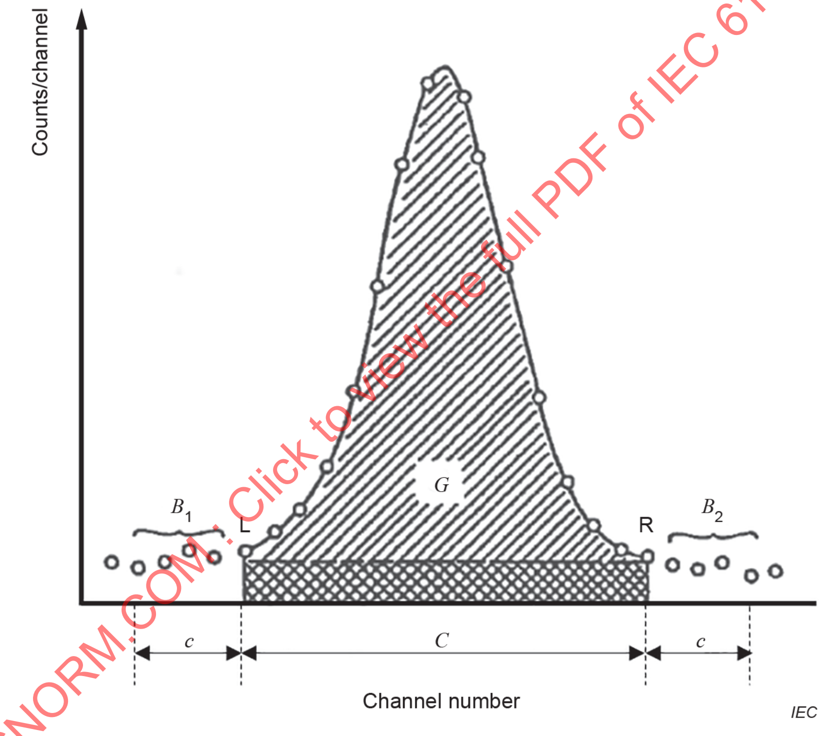
where

N is the net counts in the peak area;

N_i is the net counts in channel i after the background has been removed.

The square of the measured standard deviation associated with the first moment, if N and $\Sigma(i\,N_i)$ were independent, is given by:

$$s(x_0)^2 = \left[\frac{\Sigma(i\,N_i)}{N^2}\right]^2 s_N^2 + \frac{1}{N^2}\sum i^2\,s_{N_i}^2 \tag{B.4}$$



NOTE The gross counts, G, under the peak include all counts in channels C.

Figure B.1 – Well-resolved peak with continuum

B.3 Fitting techniques

The full-energy peak has a predominantly Gaussian shape with a tail on the low-energy side of the peak due to incomplete charge collection in the detector crystal. High-energy tails usually are an indication of high-counting-rate effects (Other minor components have also been identified by various gamma-ray spectrometrists). Because of its added complexity and counting-rate dependence, no attempt will be made to describe a function that will fit the tails of the full-energy peak. A Gaussian function is described by:

$$Y(x) = Y_0 e^{-(x-x_0)^2/(2\sigma^2)} \tag{B.5}$$

where

$Y(x)$	is the amplitude of the Gaussian function at channel; x
x	is the channel number;
Y_0	is the height of the Gaussian at the centroid channel;
x_0	is the centroid of the Gaussian;
σ	is the width of the Gaussian (FWHM = 2,355 σ).

This function is fitted to the data in the region of the peak – after the baseline has been subtracted – to determine Y_0, x_0 and f. Because x_0 and σ are in the exponent, a nonlinear fit is required. The area of a Gaussian is given by:

$$\frac{(Y_0)(\mathrm{FWHM})}{0{,}94} = \text{net area} \tag{B.6}$$

A least-squares fit is used to determine the values of the variable parameters p_j = Y_0, x_0 and σ respectively, that minimize the sum of the squares of the deviations of a set of experimental data points y_0 from the estimates $y_i(p_j)$ for the desired functional form, that is:

$$R^2 = \sum_{i=1}^{n} \{w_i[y_i - y_i(p_j)]\}^2 \tag{B.7}$$

is minimized, where the w_i are the weights associated with the y_i. The summation is over the n channels chosen to be used in the fit. The function $y_i(p_j)$ is expanded in a Taylor's series and truncated after the first derivatives of p_j. Given initial estimates p_j^0 of the parameters, $p_j = p_j^0 + \mathrm{d}p_j$ then:

$$y_i(p_j) \approx y_i(p_j^0) + \sum \left[\frac{\mathrm{d}y_i(p_j)}{\mathrm{d}p_j}\right]_0 (\mathrm{d}p_j) \tag{B.8}$$

For this truncated series, the conditions for R^2 to be a minimum, as a function of the $\mathrm{d}p_j$ are that the set of formulas:

$$\frac{\mathrm{d}R^2}{\mathrm{d}(\mathrm{d}p_j)} = 0 \tag{B.9}$$

all hold simultaneously. The solution of this set of equations gives the values $\mathrm{d}p_j$. Since the higher-order derivatives have been neglected, this process does not give the true minimum of formula (B.7). Therefore, formulas (B.8) and (B.9) are evaluated and solved again with the p_j^0 replaced by $p_j^0 + \mathrm{d}p_j$. This process is continued until all the $\mathrm{d}p_j$ approach zero (i.e., are less than some predetermined convergence criteria). When the parameters p_j are calculated, the variance and covariance terms of the matrix shall be taken into account if meaningful uncertainties are to be assigned to these parameters.

Annex C
(informative)

Formulas for the true coincidence summing correction of cascade gamma-rays

C.1 Formulas for true coincidence summing correction factors

C.1.1 General

True coincidence summing (see [2] and [3]) is due to the simultaneous detection of two or more gamma-rays, occurring in cascade from the decay of an excited nucleus in which the life-times of intermediate levels are short relative to the resolving time of the spectrometer (typically, a few µs). It results in the removal of counts from the channels at which the individual digitized peaks would occur ("summing-out") and places those counts in channels equivalent to the energy of the summed pulses. Since the full energy associated with both gamma-rays is often not detected, the sum spectrum consists primarily of a continuum. However, if the full energy from two full-energy peaks in cascade is deposited and all the charge is collected by the detector, the digitized count will be placed in a channel whose energy equivalent is equal to the sum of the individual cascade gamma-rays ("summing-in"). This is at the same energy as for a crossover transition and can result in an increase in the net area counting rate of this crossover peak.

The angular correlation between gamma-rays averaged over the solid angle of the detector at close source-to-detector distances is usually negligible and therefore has been neglected in this treatment.

Although the beta particles feeding the populated levels are also in true coincidence with the gamma-ray transitions, it is assumed that the beta rays are absorbed in the sample being counted or in the material surrounding the detector crystal. This is a good assumption for low-energy beta rays (i.e., <500 keV) but is a limiting feature of the true coincidence summing correction factors presented in this section.

These calculations can be performed using the software described in Clause 12. These calculations can also be performed using the equations shown below.

A limitation, which can be handled with the present method, is true coincidence summing between gamma-rays from one transition and X-rays emitted as a result of the internal conversion or electron capture process. Normally only K X-rays have enough energy to be observed by a HPGe spectrometer. This case is handled by determining the X-ray emission intensity in a manner similar to that used to determine the gamma-ray emission intensity. One method used to deal with many highly converted transitions is to overlay a level scheme in which the relative X-ray emission intensities are given for each transition.

$$p_{K,i} = \frac{(\omega_K)\,[\alpha_K(i)]}{1+\alpha_t(i)} \qquad \text{(C.1)}$$

where

$p_{K,i}$ is the K X-ray emission intensity of transition i;

ω_K is the K fluorescence yield for the daughter nuclide;

$\alpha_K(i)$ is the K conversion coefficient of transition i; and

$\alpha_t(i)$ is the total conversion coefficient of transition i.

For decay schemes with several transitions that decay by electron conversion in K shell, the total K X-ray emission intensity is equal to the sum of the individual K X-ray emission intensities of the transitions. The K X-ray emission intensity due to electron capture is $p_K \omega_K$ where p_K is the K-capture probability.

As an example, the correction factors in C.1.2 and C.1.3 are derived to demonstrate the complexity of these calculations and to aid those who cannot avoid the experimental conditions requiring true coincidence summing corrections.

C.1.2 True coincidence summing correction factors for a simple decay scheme

The decay scheme in Figure C.1 consists of three transitions, with “one" depopulating the second level and feeding the first, and "two” depopulating the first and feeding the ground state. Transition "three" depopulates the second excited level and feeds the ground state. All levels of the daughter nuclide are populated by beta decay of the parent radionuclide, and the transition probability for transition i is q_i. It is assumed that all the transitions depopulate both by gamma-ray emission and by internal conversion. The gamma-ray emission intensity, p_i, is related to the transition probability, q_i, by the following formula:

$$q_i = p_i[1 + \alpha_t(i)] \tag{C.2}$$

For simplicity, assume that the HPGe spectrometer is insensitive to the K X-rays emitted by the internal conversion process (i.e., $\varepsilon_K = 0$) so that counts are not lost from the net areas of full-energy gamma-ray peaks due to summing with K X-rays.

In the decay scheme shown in Figure C.1, there are three gamma transitions, designated gamma 1, gamma 2, and gamma 3. The probability P_1 of losing counts from the net peak area associated with gamma 1 because it is in true coincidence with gamma 2 is:

$$P_1 = (f_2)\varepsilon_t(2) \tag{C.3}$$

where

$\varepsilon_t(2)$ is the total efficiency for gamma-ray 2;

f_2 is the fraction of gamma-ray 2 that is in coincidence with gamma-ray 1.

Similarly:

$$P_2 = (f_1)\varepsilon_t(1) \tag{C.4}$$

where

$\varepsilon_t(1)$ is the total efficiency for gamma-ray 1

f_1 is the fraction of gamma-ray 1 that is in coincidence with gamma-ray 2

If none of transition 2 is internally converted (the total internal conversion coefficient is equal to zero), all the gamma-ray 1 transitions are in coincidence with gamma-ray 2 and f_2 is equal to one. If transition 2 is internally converted, the fraction f_2 is:

$$f_2 = \frac{p_2}{q_2} = \frac{1}{1 + \alpha_t(2)} \tag{C.5}$$

where $\alpha_t(2)$ is the total conversion coefficient of transition 2.

The probability of not observing a count in the gamma-ray 2 peak area due to a coincidence with gamma-ray 1 is somewhat different from that given above. Because of the beta feeding of the first excited state, only a fraction of the 2 transitions originate from the depopulation of level 2 above i.e., (i.e., ${}^{q_1}/_{q_2}$). Moreover, only a fraction f_1, of these transitions coming from level 2 above are gamma-rays. Therefore:

$$f_1 = \frac{1}{1+\alpha_t(1)}\left[\frac{q_1}{q_2}\right] \tag{C.6}$$

Simplifying:

$$f_1 = \left[\frac{p_1}{p_2}\right]\frac{1}{1+\alpha_t(2)} \tag{C.7}$$

The crossover transition 3 can only have counts added into the net peak area of the full-energy gamma-ray 3 peak, as none of the other transitions are in cascade with this transition. The probability that gamma-ray 1 and gamma-ray 2 both leave all their energy in the active volume of the detector is:

$$P_3 = (f_3)\,[\varepsilon(1)]\,[\varepsilon(2)] \tag{C.8}$$

where

f_3 is the fraction of transitions 1 and 2 that cascade with both emitting a gamma-ray;

$\varepsilon(1)$ is the full-energy peak efficiency of gamma-ray 1, and

$\varepsilon(2)$ is the full-energy peak efficiency of gamma-ray 2.

The fraction, f_3, is simply the combined probability of gamma-ray emission from each of the cascading transitions:

$$f_3 = \frac{(p_1)(p_2)}{(q_2)} \tag{C.9}$$

From the probabilities P_1 and P_2, the multiplicative true coincidence summing correction factors, $C_c(1)$ and $C_c(2)$ are calculated to be:

$$C_c(1) = \frac{1}{1-P_1} \tag{C.10}$$

and

$$C_c(2) = \frac{1}{1-P_2} \tag{C.11}$$

To determine the true coincidence summing correction factor for the crossover transition it is necessary to know the probability, $P_g(3)$, of detecting the crossover gamma-ray 3:

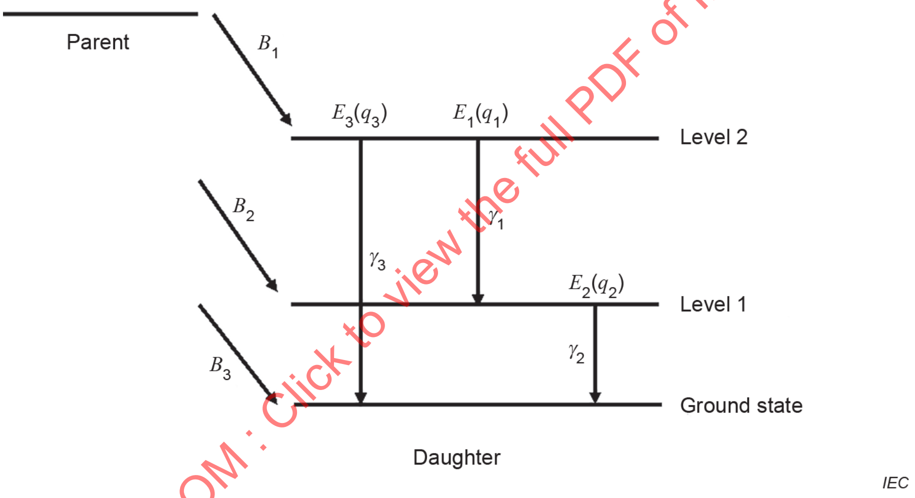
$$P_g(3) = (p_3)[\varepsilon(3)] \tag{C.12}$$

where $\varepsilon(3)$ is the full-energy-peak efficiency of gamma-ray 3.

Hence the true coincidence correction factor for the crossover gamma-ray 3 is:

$$C_c(3) = \frac{1}{1 + \left[{P_3}/{P_g(3)}\right]} \tag{C.13}$$

The plus ("+") sign in the denominator of formula (C.13) is due to the fact that a fraction of the observed counts is due to true coincidence summing into the peak and need to be removed.



NOTE A simple decay scheme with transitions one and two in cascade and transition three crossing over one and two.

Figure C.1 – A three-transition decay scheme

C.1.3 Correction factor for the 591 keV gamma-ray emitted in the decay of ^{154}Eu

C.1.3.1 General

As seen from Figure C.2 the 591 keV gamma-ray is not a crossover transition. Therefore, no counts will sum into the 591 keV full-energy peak. However, there are two cascade branches that can sum with the 591 keV gamma-ray and thereby remove counts from the 591 keV full-energy peak:

a) the 591 keV, 1 005 keV, 123 keV branch; and

b) the 591 keV, 756 keV, 248 keV, 123 keV branch.