

American National Standard for Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides

Sponsor

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Abstract: Methods for the calibration and use of germanium spectrometers for the measurement of gamma-ray energies and emission rates over the energy range from 59 keV to approximately 3000 keV, and for the calculation of source activities from these measurements, are established. Minimum requirements for automated peak finding are stated. Methods for measuring the full-energy peak efficiency with calibrated sources are given. Performance tests that ascertain the proper functioning of the Ge spectrometer and evaluate the limitations of the algorithms used for locating and fitting single and multiple peaks are described. Methods for the measurement of, and the correction for pulse pileup are suggested. Techniques are recommended for the inspection of spectral-analysis results for large errors resulting from summing of cascade gamma rays in the detector. Suggestions are provided for the establishment of data libraries for radionuclide identification, decay corrections, and the conversion of gamma-ray rates to decay rates.

Keywords: calibration, cascade summing, decay rates, gamma-ray emission rates, germanium spectrometers, radionuclides, spectrometers

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Introduction

(This introduction is not part of ANSI N42.14-1999, American National Standard for Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides.)

This standard covers the techniques for the calibration and use of germanium spectrometers for the measurement of gamma-ray emission rates of radionuclides to assure the reliability of such measurements.

This standard is the responsibility of the Accredited Standards Committee N42 on Radiation Instrumentation. Committee N42 delegated the development of the standard to its Subcommittee N42.RM. Drafts were reviewed by members of Committee N42, Subcommittee N42.RM, and other interested parties, and the comments received were utilized in producing the standard as finally approved and issued. The standard was approved by Committee N42 letter ballot of 17 December 1997.

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American National Standard for Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides

1. Overview

1.1 Scope

This standard establishes methods for the calibration and use of germanium (Ge) spectrometers for the measurement of gamma-ray energies and emission rates over the energy range from 59 keV to approximately 3000 keV, and the calculation of source activities from these measurements. This standard establishes minimum requirements for automated peak finding and methods for measuring the full-energy peak efficiency with calibrated sources.

Performance tests are described that ascertain the proper functioning of the Ge spectrometer and 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 pileup are suggested. A test to ascertain the approximate magnitude of coincidence summing is described. Techniques are recommended for the inspection of spectral-analysis results for large errors resulting from summing of cascade gamma rays in the detector. Suggestions are provided for the construction of data libraries for radionuclide identification, decay corrections, and the conversion of gamma-ray emission rates to decay rates.

This standard does not address the measurement of X-ray emission rates because X-ray peaks have intrinsically different peak shapes than gamma-ray peaks. Further, X-ray peaks are complex multiplets (e.g., the K X rays of Tl are composed of eight components that form four partially resolved individual peaks). Also, this standard 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. Discussion of acceptable methods for measuring the lower limits of detection as they relate to specific radionuclides is also beyond the scope of this standard. See ANSI N13.30-1996¹ or ANSI N42.23-1996. For additional guidance, see NUREG-1156-1986 [B33].²

¹Information on references can be found in Clause 2.

²The numbers in brackets correspond to those of the bibliography in Annex A.

1.2 Purpose

The purpose of this standard is to provide a basis for the routine calibration and use of Ge 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 Ge gamma-ray spectrometry and are responsible for the development of procedures for the calibration and use of such detectors. This standard is primarily intended for guidance on how to set up and use a system for routine analytical measurements.

A typical gamma-ray spectrometer consists of a Ge detector with its liquid-nitrogen or mechanically refrigerated cryostat and preamplifier, detector bias supply, linear amplifier, analog-to-digital converter (ADC), multichannel storage of the spectrum, and data-readout devices. A radiation shield often surrounds the detector to reduce the counting rate from room-background radiation (see Annex E for shield construction guidelines). Photons (X and gamma rays) interact with the Ge crystal to produce 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 Ge crystal. Each pulse is amplified, shaped, and sorted according to pulse height, using an ADC 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 displays a spectrum of one or more peaks with an approximately normal (Gaussian) distribution corresponding to photons that interacted with the detector.

A source 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 live time, T_L , and the full-energy peak efficiency, $\epsilon(E)$, of the detector for the counting geometry used. A curve, table, or functional representation of the full-energy peak efficiency permits interpolation between available calibration points. Corrections may be needed for

- a) The loss of pulses due to pulse pileup (at high counting rates);
- b) Coincidence (cascade) summing;
- c) The decay of the source during sampling (e.g., with air filters) and counting;
- d) The decay of the source from a previous time to the counting period;
- e) Attenuation of photons within and external to the source that is not accounted for by the full-energy peak efficiency calibration.

2. References

This standard shall be used in conjunction with the following publications. When the following standards are superseded by an approved revision, the revision shall apply.

ANSI N13.30-1996, American National Standard Performance Criteria for Radiobioassay.³

ANSI N42.23-1996, American National Standard Measurement and Associated Instrument Quality Assurance for Radioassay Laboratories.⁴

IEEE Std 100-1996, IEEE Standard Dictionary of Electrical and Electronics Terms.⁵

IEEE Std 325-1996, IEEE Standard Test Procedures for Germanium Gamma-Ray Detectors.

³ANSI publications are available from the Sales Department, American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036, USA (www.ansi.org/).

⁴This publication is available from the Institute of Electrical and Electronics Engineers, 445 Hoes Lane, P.O. Box 1331, Piscataway, NJ 08855-1331, USA (www.standards.ieee.org/).

⁵IEEE publications are available from the Institute of Electrical and Electronics Engineers, 445 Hoes Lane, P.O. Box 1331, Piscataway, NJ 08855-1331, USA (www.standards.ieee.org/).

3. Definitions

The word “shall” denotes a requirement, the word “should” denotes a recommendation, and the word “may” denotes permission. To conform to this standard, Ge spectrometry shall be performed in accordance with the requirements of this standard, but not necessarily with its recommendations; however, justification should be documented for deviations from a recommendation.

For the purposes of this standard, the following terms and definitions apply. IEEE Std 100-1996 should be referenced for terms not defined in this clause.

3.1 abundance: *See:* **emission probability per decay [$P(E)$ or $P_\gamma(E)$].**

3.2 activity (A): The expected number of spontaneous nuclear decays (transformations) in unit time from a specified energy state (excluding prompt decays from a lower nuclear level) for a given amount of a radionuclide. Its standard unit (SI) is the becquerel (Bq), where one Bq equals one decay per second. Activity has often been expressed in curies (Ci), where 3.7×10^{10} Bq equals 1 Ci, exactly.

3.3 ADC conversion gain: The number of channels over which the full amplitude span can be spread; usually 2048–8192 channels are used for Ge gamma-ray spectrometry.

3.4 analog-to-digital converter (ADC): An electronic device used to convert the amplitude of a voltage pulse from analog to digital format.

3.5 attenuation: The 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 detector crystal.

3.6 background: Spectral data including peaks not caused by the source but rather resulting from radioactive decay occurring in the surrounding environment or resulting from cosmic-ray interactions in or adjacent to the detector. *See also:* **continuum.**

3.7 baseline: *See:* **continuum.**

3.8 calibration: The 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).

3.9 cascade summing: *See:* **coincidence summing of x and gamma rays.**

3.10 cascade transitions: Gamma rays in the radioactive decay of a single atom that are emitted sequentially and within the resolving time of the spectrometer.

3.11 coincidence summing of x and gamma rays: The simultaneous detection of two or more photons originating from a single nuclear disintegration that results in only one observed (summed) pulse. *Syn:* **cascade summing; true coincidence summing.**

3.12 combined uncertainty: The uncertainty resulting from combining category A and category B uncertainties, as defined by the Bureau International des Poids et Mesures (BIPM), using standard statistical methods. Category A uncertainties are evaluated by applying statistical methods to a series of repeated measurements and are characterized by the estimated standard deviation, s_A ; category B uncertainties are assigned to quantities whose variation is not explicitly observed. Category B uncertainties are determined by estimating from other information an approximation to a corresponding “standard deviation,” s_B , whose existence is assumed. They are combined as if they are all standard deviations.

3.13 continuum: The 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. *Syn:* **baseline.**

3.14 counting rate: The rate at which detector pulses are being registered in a selected voltage interval. The unit is reciprocal seconds (i.e., s^{-1}).

3.15 crossover transition: A gamma ray occurring between two nonadjacent nuclear levels.

3.16 dead time: The time during which the ADC is unable to process input pulses because it is processing a previous pulse.

3.17 detection efficiency: The ratio between the number of selected pulses recorded per unit time to the number of photons emitted by the source per unit time. *See also:* **full-energy peak efficiency; total efficiency.**

3.18 direct current (dc) level: The input or output voltage level on a dc-coupled instrument when there are no pulses present. For Ge spectrometer systems, the dc level of an output signal from a linear amplifier should be matched to the input requirements for dc levels of an ADC if the gain conversion scale is to include the origin.

3.19 elapsed time (T_r): Counting time uncompensated for periods in which an instrument might be unable to respond. Elapsed time of a count equals live time plus dead time.

3.20 emission probability per decay [$P(E)$ or $P_\gamma(E)$]: The probability that a radioactive decay will be followed by the emission of the specified radiation. Gamma-ray emission probabilities are often expressed per 100 decays. *Syn:* **yield; abundance.**

3.21 energy resolution, full width at half maximum (FWHM): The width of a peak at half of the maximum peak height with the baseline removed.

3.22 energy resolution, full width at tenth maximum (FWTM): The width of a peak at one-tenth of the maximum peak height with the baseline removed. [For a normal (Gaussian) distribution, FWTM is 1.823 times its FWHM.]

3.23 full-energy peak: A peak in the spectrum resulting from the complete (total) absorption of a photon of a given energy in the active volume of the Ge crystal and the collection of all of the resulting charge. *Syn:* **photopeak.**

3.24 full-energy peak efficiency [$\epsilon(E)$]: The 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 source-to-detector distance.

3.25 gamma-ray branching ratio [$f(E)$]: For a given excited state, the ratio of the emission rate of a particular gamma ray to the total transition rate from the level (not to be confused with emission probability per decay).

3.26 gamma-ray emission rate [$R(E)$]: The rate at which a gamma ray of a given energy from the decay of a particular radionuclide is emitted from a given source. The gamma-ray emission rate is the activity times the gamma-ray emission probability.

3.27 live time (T_L): The time interval of a count during which a counting system is capable of processing input pulses. Elapsed (or real) time equals live time plus dead time.

3.28 Monte Carlo: A modeling technique in which the interaction of a gamma-ray of energy E with matter is predicted by simulation.

3.29 multichannel analyzer: See: **multichannel pulse-height analyzer.**

3.30 multichannel pulse-height analyzer (MCA): An electronic device that records and stores pulses according to their height. 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; an input/output section that permits transfer of the spectral information to other devices, such as a computer, oscilloscope display, or permanent storage media (disk or magnetic tape storage). Syn: **multichannel analyzer.**

3.31 peak-to-Compton ratio for the 1332 keV ^{60}Co peak: The ratio of the full-energy peak height, for ^{60}Co measured at 1332 keV, to the average height of the corresponding Compton plateau between 1040 keV and 1096 keV.

3.32 Peak-to-total ratio: The net area of a full-energy mono-energetic peak divided by the counts in the full spectrum detected, including scattering contributions making up the spectrum (e.g., backscattering) from said gamma ray.

3.33 photopeak: See: **full-energy peak.**

3.34 pole-zero cancellation: The pole-zero adjustment on the shaping amplifier adjusts the zero location of the pole-zero network to cancel exactly the preamplifier output pole and thus provide single-pole (i.e., no under or overshoot) response of the signal pulse at the amplifier output. This operation converts the long-tailed preamplifier pulse to a short-tailed pulse suitable for signal optimization and subsequent pulse-height analysis. Proper pole-zero cancellation is an absolute necessity to prevent spectral degradation at moderate (2000 s^{-1}) rates. (See Figure B.1 for an example of proper adjustment for pole-zero cancellation.)

3.35 pulse pileup: 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. 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. Pulse pileup is a function of the square of the counting rate and of the amplifier pulse width. Syn: **random summing.**

3.36 random summing: See: **pulse pileup.**

3.37 relative full-energy peak efficiency for detector specification (ϵ_r): The ratio of full-energy peak detection efficiency for a point source of ^{60}Co (1332 keV photons) to that of a NaI(Tl) crystal, 7.6 cm in diameter $\times$ 7.6 cm in height, for a source-to-detector distance of 25 cm.

$$\epsilon_r = 0.833 \times 10^3 \times \frac{n(1332)}{R(1332)} \quad (1)$$

where

$n(1332)$ is the measured full-energy peak counting rate,

$R(1332)$ is the 1332 keV gamma-ray emission rate from the ^{60}Co source.

The constant is the reciprocal of the full-energy peak efficiency of the NaI(Tl) detector at that energy and distance.

3.38 standardization coefficient [$S_c(i, E)$]: A factor used for the direct conversion of a net area counting rate of a gamma-ray peak of a given energy, E , and from a specific radionuclide, i , to the activity of that radionuclide.

3.39 total efficiency [$\epsilon_t(E)$]: The ratio of the number of pulses in the entire energy spectrum due to an X or gamma ray of a given energy, E , to the number of that X- or gamma-ray photons emitted by the source for a specified source-to-detector distance. The total efficiency can be affected by the shield design due to photon scattering.

3.40 true coincidence summing: *See: coincidence summing of x and gamma rays.*

3.41 uncertainty: The likely inaccuracy of a reported value, expressed in terms of estimated standard deviations. *See also: combined uncertainty.*

3.42 yield: *See: emission probability per decay [$P(E)$ or $P_\gamma(E)$].*

3.43 Symbols

A	the activity, or decays per second, of a sample
$B(E)$	net counting rate of an interfering background peak at energy E
C_i	multiplicative correction factor, where i is: a (attenuation), c (coincidence summing), d (decay), or r (pileup)
e	2.71828...; base of natural or Napierian logarithms, $\ln$
$f(E)$	the fraction of transitions from a level that produces gamma rays with energy E
M_s	mass of aliquant used to prepare working standard whose matrix is identical to that of the standard used to measure the detector efficiency
M_u	mass of aliquant used to prepare sample whose matrix is identical to that of the unknown samples to be measured
$N(E)$	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
$P(E)$	the probability per decay for a gamma ray with energy E
$R(E)$	the emission rate of gamma rays with energy E by nuclei in a sample
$S_c(i, E)$	the fraction of decays of radionuclide, i , in a sample that leads to counts in a full-energy peak corresponding to a gamma ray of energy E
T_d	the decay time from an initial time to start of a count
T_i	the irradiation time
T_L	the live-timed counting period
T_r	the elapsed-time counting period

T_s	the sampling time
x	channel position of a gamma-ray peak
$\alpha_K(E)$	the ratio of transitions of energy E that occur by K internal conversion to those that emit gamma rays
$\alpha_i(E)$	the ratio of transitions of energy E that occur by internal conversion to those that emit gamma rays
$\epsilon(E)$	the full-energy peak efficiency for gamma rays of energy E
$\epsilon_t(E)$	the 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
Π	an operation that involves taking products (analogous to Σ designating an operation that involves taking sums)
ω_K	the fraction of atomic K vacancies that lead to K X rays

4. Installation of instrumentation

Locate the Ge spectrometer where stable, clean power is available or provide it via a power conditioner or uninterruptible power supply. Ensure that the ambient temperature is stable and preferably between 16 °C (60 °F) and 27 °C (80 °F), or install it in an air-conditioned equipment rack. Shield the spectrometer from interfering background radiation, including natural radiation emitted from the walls, floor, and ceiling, or resulting from cosmic rays.

CAUTION

When filling the detector dewar with liquid nitrogen (LN₂), 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. It is also prudent to turn a count off during the filling process and restart it after a suitable delay. This practice should be done to avoid distortion of the measured spectrum due to microphonics or shifts due to cooling of various electronic components external to the cryostat.

Follow the instructions of the manufacturer for installation and preliminary testing of the individual components (i.e., detector with cryostat and preamplifier, spectrometry amplifier, high-voltage bias supply, ADC, and multichannel analyzer). Check the accuracy of the multichannel pulse-height analyzer (MCA) clocks and adjust the dc offset and the pole-zero setting on the amplifier, following the procedures given in the user manual of the manufacturer or those in Annex B. Measure the energy resolution [i.e., full width at half maximum (FWHM) and full width at tenth maximum (FWTM)] at 122 keV (⁵⁷Co) and at 1332 keV (⁶⁰Co), and the peak-to-compton ratio at 1332 keV as described by the user manual of the detector manufacturer, or in IEEE Std 325-1996, or in Annex B. The ADC lower-level discriminator should be set above the noise, but it shall be below the lowest energy gamma-ray peak of interest.

Recommendation. Often the instrumentation associated with a Ge 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 share the same ground. This can result in ground loops that adversely affect the detector resolution. To avoid such loops, the instrument grounds should be isolated and connected to local ground at only one point.

If the measured values do not meet the specifications of the manufacturer within reasonable tolerances, the calibration should not proceed until the problem has been corrected. All settings and experimental condi-

tions shall be documented in a logbook for each specific Ge spectrometer, and any changes or maintenance shall be so indicated at the time they occur.

The experimental conditions recommended by the manufacturer for initial installation of the instrumentation are usually intended to ensure that the Ge spectrometer meets the various specifications (e.g., energy resolution) claimed by the manufacturer. These conditions often are not optimum for routine analyses. Therefore, after the initial installation checks, the user should adjust the various spectrometer settings, including the amplifier time constant, baseline restorer, and pole-zero cancellation, for optimal performance under normal counting conditions before conducting the performance tests described in this standard.

5. Calibration procedures

5.1 Energy calibration

The energy calibration (gamma-ray energy as a function of channel number) of the Ge spectrometer system shall be established by measuring the positions of selected, single full-energy gamma-ray peaks with large peak-height-to-background ratios, and whose energies are known precisely. The gamma rays should cover the energy range of interest. Laboratories with more than one Ge spectrometer may find it 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. A procedure to set the ADC zero and gain is described in B.2.1.

The calibration source should be counted for a duration that gives sufficient counts in the peaks to permit measurement of the peak positions with a precision of less than 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. If the Ge spectrometer has long-term pulse-height stability, the energy scale should not need to be checked more than once each day using an energy calibration source. Uncertainties on 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. Gehrke et al. [B14] describes one method for measuring the pulse-height nonlinearity employing sources (other sources may also be used as long as their peaks adequately cover the energy range of interest).

5.2 Efficiency calibration

The following approaches may be 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;
- c) Calculation of the peak efficiency as a function of energy with the use of Monte Carlo or other calculation techniques.

If calculations are used to determine efficiencies or to infer efficiencies for a counting geometry different from the measured efficiency (usually a point source geometry), direct measurement of a known working standard shall be demonstrated for the different counting geometry.

5.2.1 Standardization 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. This method measures a standardization coefficient for a selected gamma ray emitted from each specific radionuclide. The standardization coefficient converts the net peak-area counting rate to the activity of the radionuclide being measured. The need to interpolate between measured efficiency values, to correct for coincidence summing, and to account for the uncertainty in the gamma-ray emission probability is eliminated with this method. If the counting rates of the samples are low or closely approximate those of the standard, pulse-pileup corrections can also be neglected.

Severe 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 are located inside the well of a Ge well detector (see Caution 1 in 6.6.2). Therefore, the standardization coefficient method is the only efficiency calibration technique recommended for these or similar experimental conditions.

5.2.2 Detector efficiency as a function of energy

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 pileup and coincidence summing of gamma rays. The corresponding radionuclide activities can be calculated from the gamma-ray emission rates when the gamma-ray emission probabilities are known.

5.2.3 Efficiency function or lookup table

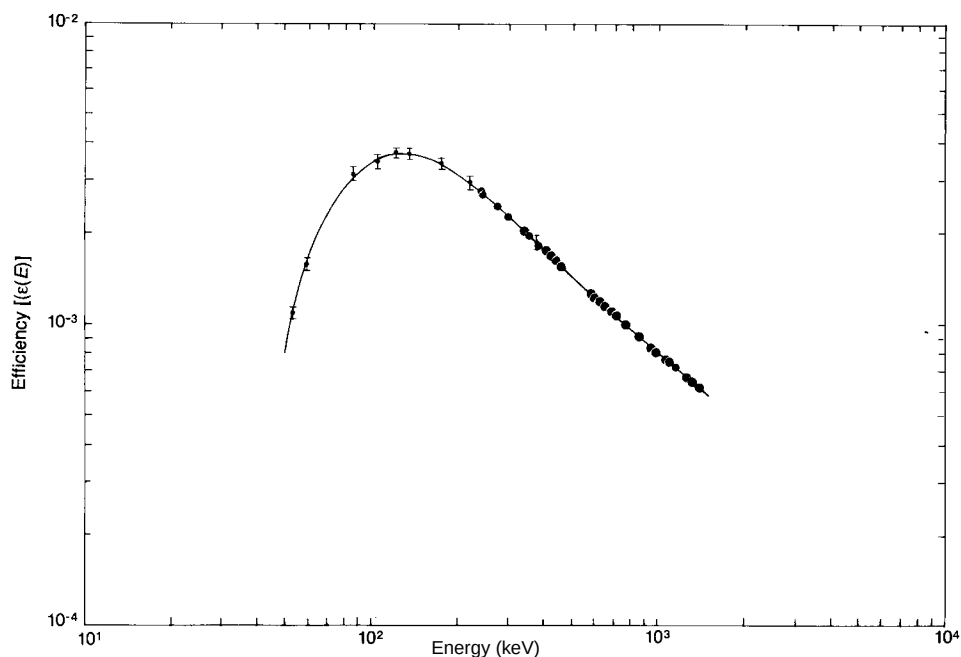
The full-energy peak efficiency values, $\epsilon(E)$ for energy E , should be plotted either as $\epsilon(E)$ on a log-log graph (see Figure 1) or as the function

$$Y = \epsilon(E) \times E^a \quad (2)$$

on a linear plot (a is a normalizing constant that can be determined for each efficiency curve separately), as shown in Figure 2. This function provides better resolution of the efficiency data when plotted. There should be no large gaps in energy between the measured or calculated efficiency values (especially below 300 keV) that would cause a large efficiency-interpolation error. 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 1000 keV. For $2 < E < 4$ (E is in 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. The efficiency data may be treated by either

- a) Forming a table of efficiency values as a function of energy, as obtained from a smooth curve drawn through the plotted data (e.g., see Cline et al. [B6] and Gehrke et al. [B15]); or by
- b) Fitting the efficiency data with a suitable function (see Gray and Ahmad [B17], McNelles and Campbell [B30], Singh [B37], and Yoshizawa et al. [B44]).

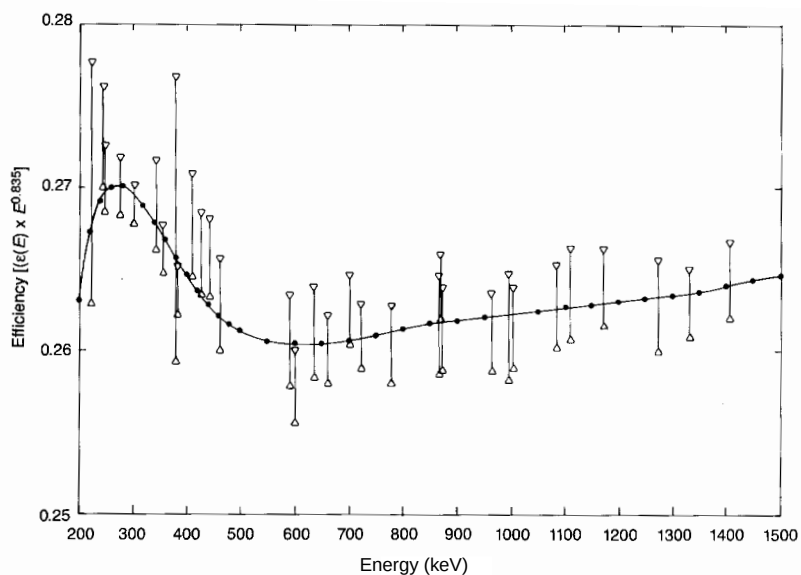
NOTE—Regardless of the efficiency calibration method selected, the peak areas for the method should be calculated in the same way as the peak areas are calculated during the sample analyses.



NOTE—Efficiency as a function of energy on a log-log plot for a point source at a 15 cm source-to-detector distance for a coaxial Ge spectrometer.

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Figure 1—Full-energy peak efficiency



NOTE—The same data as in Figure 1, plotted as $\epsilon \times E (\text{keV})^{0.835}$ versus Energy (keV) on a linear plot, where ϵ is the full-energy peak efficiency at E (keV), and E (keV) is the energy of the full-energy peak. The solid circles represent selected values taken from the plot and used to prepare the efficiency lookup table.

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Figure 2— $\epsilon \times E$ (keV) as a function of gamma-ray energy

6. Gamma-ray measurements with Ge spectrometers

6.1 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 background). The peak-fitting routine should be able to find multiplet peaks that meet the peak-to-baseline criteria for a singlet, are approximately the same intensity, and are separated by at least 1 FWHM (see performance tests in Clause 8). Optimization of the peak-search parameters of the peak-finding algorithm is left to the user or software vendor. The parameters 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).

6.2 Peak position and area measurement

The simplest method that consistently produces satisfactory results for the measuring conditions and analysis requirements is the best, and is the recommended method for measuring 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., greater than 3 FWHM separation) are recommended over those with overlapping interference (i.e., less than 3 FWHM separation) whenever there is a choice.

WARNING

The peak-fitting algorithm may influence the energy dependence of pileup corrections (see 8.4 and B.5.1).

Recommendation. It is very helpful to be able to visually evaluate the quality of any peak fit through a display of the spectral counts in the peak region with the fitted function and background 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.

6.3 Measurement of gamma-ray energies

The gamma-ray energies are normally derived from a previous energy calibration (see 5.1). Sometimes it is desirable to derive the gamma-ray energies directly from the sample spectrum. In this case, an energy calibration is performed on the sample spectrum using identified peaks present in the spectrum whose gamma-ray energies are accurately known.

At other times, peak-area estimates may be required in specified energy regions of the spectrum to determine the possible presence or upper limit of specific gamma-ray peaks and associated radionuclides of particular interest, even when they were not found by the peak-finding algorithm. Often these spectral regions contain no visible peaks. In these types of analyses the width, as a function of the peak energy, shall be known. Also a more accurate energy calibration may be required.

6.4 Subtraction of background peaks

Large samples may shield the detector from the ambient background radiation, causing a lower counting rate than if an instrumental background (no sample present) was obtained. Thus, whenever large, low-activity samples are counted, a background spectrum should be obtained with a “blank” sample in the same geome-

try and of the same density. This “blank” shall be free of any radionuclides of interest. The background count shall be accumulated for at least the same counting time as used in counting the samples to be analyzed. (All soils contain some natural ^{40}K , Th, and U radioactivity.) The “blank” sample background spectrum should be analyzed to obtain the background-peak counting rates, $B(E)$, at energies E . The background-peak counting rates are then subtracted from the respective net-area peak counting rates, $n(E)$, in the sample spectrum [see Equation (3)]. By subtracting the respective background-peak counting rates from those of the sample, the statistics of each count are preserved. If possible, the “blank” spectrum should be scanned to be sure that all background peaks are identified and that no false peaks are reported.

6.5 Measurement of gamma-ray emission rates

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. Accumulate the gamma-ray spectrum at the same gain as used in the energy calibration and for a counting time sufficient to give an acceptable statistical uncertainty. Identify the energies of the gamma-ray peaks present in the spectrum by use of the energy calibration described in 5.1. Obtain the net-area counting rate in each of the gamma-ray peaks of interest, as described in 6.2. Obtain the full-energy peak efficiency for each of the peaks of interest from the efficiency look-up table or efficiency function, as described in 5.2.3. Calculate the number of gamma rays emitted per second, $R(E)$, for each of the full-energy peaks as follows:

$$R(E) = [n(E) - B(E)] \times \frac{\Pi C_i}{\varepsilon(E)} \quad (3)$$

where

- $n(E)$ is the net counting rate, $N(E)/T_L$ [where $N(E)$ is the net peak area and T_L is the counting (or live) time], in the full-energy peak at energy E ,
- $B(E)$ is the net counting rates of any interfering peaks in the background,
- $\varepsilon(E)$ is the full-energy peak efficiency (counts in the net peak per gamma ray emitted),
- C_i are the correction factors (where $i = a, c, d, \text{ and } r$; attenuation correction, coincidence summing correction, decay correction, and pileup correction, respectively),
- Π is an operation that involves taking products (analogous to Σ designating an operation that involves taking sums).

Each correction factor shall be applied whenever its magnitude is expected to exceed one-third of the desired accuracy in the resulting gamma-ray emission rate.

6.5.1 Pulse pileup (random summing)

The pulse-pileup correction shall be applied whenever the desired accuracy of the measurement requires it. The pulse-pileup correction, C_p , may be measured by one of several methods. Some popular methods are

- a) 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 the maximum expected count rate of samples analyzed.
- b) Use of live-time extension techniques, whereby the live time is extended to compensate for displaced pile-up pulses.
- c) 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 should be less than approximately 10% 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-pileup correction, but this dependency should be small.

A description of the method using a set of sources whose relative activities are precisely known is given in B.5.1. Descriptions of the methods in items b) and c) for measuring the pulse-pileup correction can be found in Knoll [B25] and Wiernik [B43]. The use of these or other types of electronic circuitry to correct automatically for pileup is permitted if validated by passing the test in 8.4.

Recommendation. The amplifier 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 time constants (greater than 2 μs) typically yield higher energy resolution but poorer counting-rate performance. For amplifier unipolar output pulse widths of about 12 μs (a time constant of about 2 μs), preamplifiers with resistive feedback, and input counting rates less than 1000 s^{-1} , the pulse-pileup correction is normally less than or equal to 1% and may be neglected.

WARNING

1—High counting rates can lead to dead-time inaccuracies in the ADC and to pileup of pulses in the amplifier. Although the live timer of the MCA only records time while the pulse-processing circuitry is able to accept pulses, at very high rates (greater than 10 000 s^{-1}), errors in the live timer can be significant (greater than 5%).

2—Pileup rejecters may be useful at high counting rates because they prevent the storage of piled-up pulses in the spectrum and thereby reduce the continuum between and underlying the spectral peaks. However, if the pileup rejection process is amplitude dependent, unacceptably large errors in the correction of the net peak-area counting rates could result. Therefore, the use of pileup 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. 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 brochures and catalogues of the manufacturer for further information. The performance of these systems shall be verified prior to use for constant counting rates with the performance test in 8.4.

3—The principles upon which loss-free counting modules work may cause them to distort counting statistics.

6.5.2 Attenuation corrections

Two components are considered for attenuation correction: external absorbers, C_{a_1} , and differences in the matrix or density of identically shaped samples, C_{a_2} . These corrections are then combined as

$$C_a = C_{a_1} \times C_{a_2} \quad (4)$$

where

C_a is the attenuation correction.

6.5.2.1 Absorption 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

$$C_{a_1} = e^{\mu \rho d} \quad (5)$$

where

- μ is the mass attenuation coefficient,
- ρ is the density of the absorber,
- d is the mean thickness through absorber.

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

6.5.2.2 Absorption within the sample

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 the sample shall be the same). When the error resulting from not applying the correction is not insignificant, a correction shall be made. This correction is most important for low-energy gamma rays and sources containing high atomic-number elements (see Parker [B34]).

6.5.2.2.1 Calculated for uniform plane source

The correction for self-attenuation in a homogeneous, extended sample may be calculated by numerical integration over the sample volume (see Parker [B34]). 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 for uncollimated geometry to be

$$C_{a_2} = \frac{\mu_u(E)\rho_u d}{1 - \exp[-\mu_u(E)\rho_u d]} \times \frac{1 - \exp[-\mu_s(E)\rho_s d]}{\mu_s(E)\rho_s d} \quad (6)$$

where

- $\mu_u(E)$ is the mass absorption coefficient at energy E of sample,
- $\mu_s(E)$ is the mass absorption coefficient at energy E of efficiency measuring standard,
- ρ_u is the density of the sample,
- ρ_s is the density of the efficiency-measuring standard,
- d is the thickness of the standard/sample.

6.5.2.2.2 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, is counted under the same conditions as used to measure 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 to a sufficient accuracy to determine the attenuation correction with adequate precision. The relative attenuation correction, C_{a_2} , is calculated to be

$$C_{a_2} = \frac{R(E)\epsilon(E)}{[n(E) - B(E)][\prod C_{i \neq a}]} \quad (7)$$

where

- $R(E)$ is the emission rate of the gamma ray at energy E in the attenuation-correction standard,
- $\epsilon(E)$ 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 ,
- $C_{i \neq a}$ are the corrections for pileup ($i = r$),
($i = c$), coincidence summing,
($i = d$), decay prior to and during the count,
- $C_{i = a}$ is 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 each uniformly spiked with an accurately measured aliquant, M , of a gamma-ray emitting radionuclide (see B.7 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 than the efficiency-measuring standard. Assuming corrections for pulse pileup can be neglected, for a given energy, E , the relative attenuation correction, C_{a_2} , is given by

$$C_{a_2}(E) = \frac{[n_s(E) - B_s(E)]M_u}{[n_u(E) - B_u(E)]M_s} \quad (8)$$

where

- $n_s(E)$ is the net counting rates in the peak areas of the standard at energy E ,
- $n_u(E)$ is the net counting rates in the peak areas of the sample at energy E ,
- $B_s(E)$ is the net counting rates in the peak areas of the background spectra associated with the standard,
- $B_u(E)$ is the net counting rates in the peak areas of the background spectra associated with the sample,
- 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.

6.6 Calculation of nuclide activities

The nuclides are identified by comparing the list of observed peaks with the energies in a gamma library. The nuclear decay rate or activity, A , is calculated by dividing the gamma-ray emission rate, $R(E)$, by the appropriate gamma-ray emission probability, $P(E)$.

$$A = \frac{R(E)}{P(E)} \quad (9)$$

6.6.1 Radioactive decay

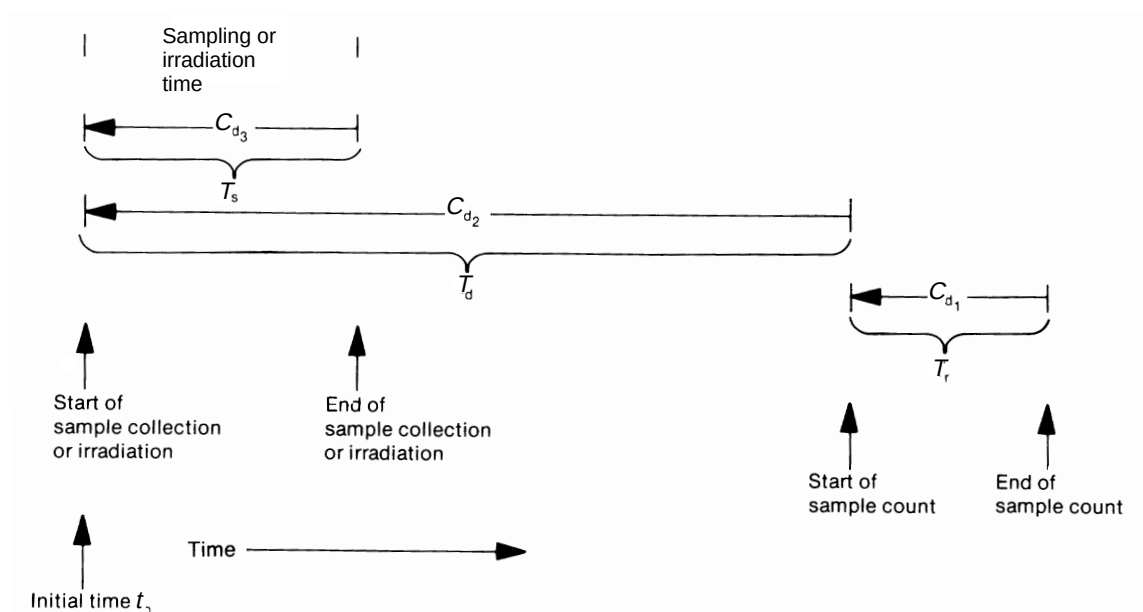
The correction factor that normalizes the nuclide activity for radioactive decay is normally composed of the following components:

- Decay during the counting interval; and
- Decay from a previous time, usually a sample-irradiation time (e.g., neutron-activation analysis) or a sample-collection 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 are designated C_{d_1} , C_{d_2} , and C_{d_3} , respectively. The combined correction factor for radioactive decay can be expressed as

$$C_d = C_{d_1} \times C_{d_2} \times C_{d_3} \quad (10)$$

Figure 3 illustrates the time to which each correction is made for the decay correction equations given in this subclause.



NOTE—Specification of various events expressed in units of time, where t_0 is the initial time, T_s is the sample of irradiation time, T_d is the decay time of the sample, and T_r is the real (elapsed) time. The arrows point to the times to which the respective decay corrections apply.

Figure 3—Specification of times for decay corrections

6.6.1.1 Radioactive decay during the counting period

If significant decay occurs during the counting time (greater than 10%), it may be necessary to correct the net peak area so that the gamma-ray emission rates or the radionuclide activities are determined for 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 some time after the start of the count.) The following equation gives this multiplicative correction:

$$C_{d_1} = \frac{\lambda T_r}{1 - e^{-\lambda T_r}} \quad (11)$$

where

- C_{d_1} is the correction factor for decay during the count,
- T_r is the real (elapsed) time of the count,
- e is the base of the natural logarithm,
- λ is the radionuclide decay constant ($\ln 2/T_{1/2}$),
- $T_{1/2}$ is the radionuclide half-life.

This correction assumes a constant dead time during the acquisition period. An error may be introduced if the dead time varies significantly.

Corrections for decay during the counting time can often be avoided by measuring time from the middle of a count. When this is done, the error introduced is only 0.5% if the counting period is half of the half-life of the decaying radionuclide.

6.6.1.2 Radioactive decay prior to the counting period

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

$$C_{d_2} = e^{\lambda T_d} \quad (12)$$

where

- C_{d_2} is the correction factor for decay prior to the count,
- λ is the radionuclide decay constant,
- T_d is the decay time from a prior time to the start of the count,
- e is the base of the natural logarithm, whose value is approximately 2.7.

6.6.1.3 Correction during the sampling time or 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 that would be activated at saturation). In this option

$$C_{d_3} = \frac{1}{1 - e^{-\lambda T_i}} \quad (13)$$

where

C_{d_3} is the correction factor for decay during irradiation,
 e is the base of the natural logarithm,
 λ is the radionuclide decay constant,
 T_i is the irradiation duration.

If one is collecting already activated nuclides at a uniform rate, 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

$$C_{d_3} = \frac{\lambda T_s}{1 - e^{-\lambda T_s}} \quad (14)$$

where

C_{d_3} is the correction factor for decay during sample collection,
 T_s is the sampling duration.

6.6.2 Coincidence (cascade) summing

Coincidence summing corrections should 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 less than 5 cm, unless demonstrated otherwise.

Because the total efficiency of the detector (partial and total photon energy deposition in the detector) is greater than the full-energy peak efficiency, especially at higher energies, the summing of pulses that are in true coincidence normally results in the removal of counts from the full-energy peak. However, this may not be true for low-probability crossover gamma-ray transitions, where counts are added to the peak by the summing of the full-energy pulses from high-probability coincidence gamma rays (e.g., the low-probability 2734 keV crossover peak in the decay of ^{88}Y can contain counts from the summing of the high-probability 898 keV and 1836 keV cascade gamma rays). Programs developed for use in determining coincidence summing corrections, C_c , for specific radionuclides and source-detector geometries are described in Debertin and Schoetzig [B11] and Schima and Hoppes [B36]. A brief description of coincidence summing and the equations needed to determine the loss or gain of counts to a gamma-ray peak is provided in Annex D.

CAUTION

1—Characteristic X rays produced from electron-capture and internal-conversion transitions also can sum with coincident gamma rays when thin-window detectors that detect both low-energy X rays and gamma rays are used (e.g., ^{139}Ce or ^{125}Sb). One alternative, when using thin-window detectors, is to place a 1 mm thick tin absorber 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.

2—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 isomeric transitions). The coincidence summing of beta particles and associated bremsstrahlung with gamma rays is not included in most coincidence summing correction programs. Under these circumstances, insertion of a low-atomic-number beta-ray absorber between the source and the detector can significantly reduce the detection of beta particles, and thereby reduce their summing with gamma rays. Be sure that correction for gamma-ray attenuation in the beta-ray absorber is included in the efficiency or provided as a separate correction.

7. Performance tests of the spectrometry system

The spectrometer tests enumerated in 7.1 through 7.5 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., 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.

7.1 MCA clocks

The elapsed-time clock shall be checked at the time the MCA is installed and after any maintenance to it. Follow the instructions in the user manual of the manufacturer (see also B.3).

7.2 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 user manual of the manufacturer (see also B.1).

7.3 Energy calibration

The system energy calibration shall be checked periodically (typically daily to semiweekly) by counting the source(s) emitting gamma rays whose energies are precisely known and cover the region of interest. Initial gain and zero, and nonlinear terms may be determined first with the procedure described in B.2. Repeat the energy calibration if any of the measured energies lie outside the desired accuracy (usually ± 0.2 keV). Energy calibrations can be checked with spectral peaks of known energy.