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(Revision of
ANSI N42.14-1991)

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

Sponsor

Accredited Standards Committee on Radiation Instrumentation, N42

Accredited by the

American National Standards Institute

Secretariat

The Institute of Electrical and Electronics Engineers, Inc.

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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 N13.30-1996[1] or ANSI N42.23-1996. For additional guidance, see NUREG-1156-1986 [B33].[2]

[1]Information on references can be found in Clause 2.

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

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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, $\varepsilon(E)$, 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.

[3]ANSI 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/).

[4]This 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/).

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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.

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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 [$\varepsilon(E)$]: 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.

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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 × 7.6 cm in height, for a source-to-detector distance of 25 cm.

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

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.

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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 [$\varepsilon_t(E)$]: 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

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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

$\alpha_t(E)$ the ratio of transitions of energy E that occur by internal conversion to those that emit gamma rays

$\varepsilon(E)$ the full-energy peak efficiency for gamma rays of energy E

$\varepsilon_t(E)$ 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 (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. 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 (^{57}Co) and at 1332 keV (^{60}Co), 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-

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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.

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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, $\varepsilon(E)$ for energy E, should be plotted either as $\varepsilon(E)$ on a log-log graph (see Figure 1) or as the function

$$Y = \varepsilon(E) \times E^{a} \tag{2}$$

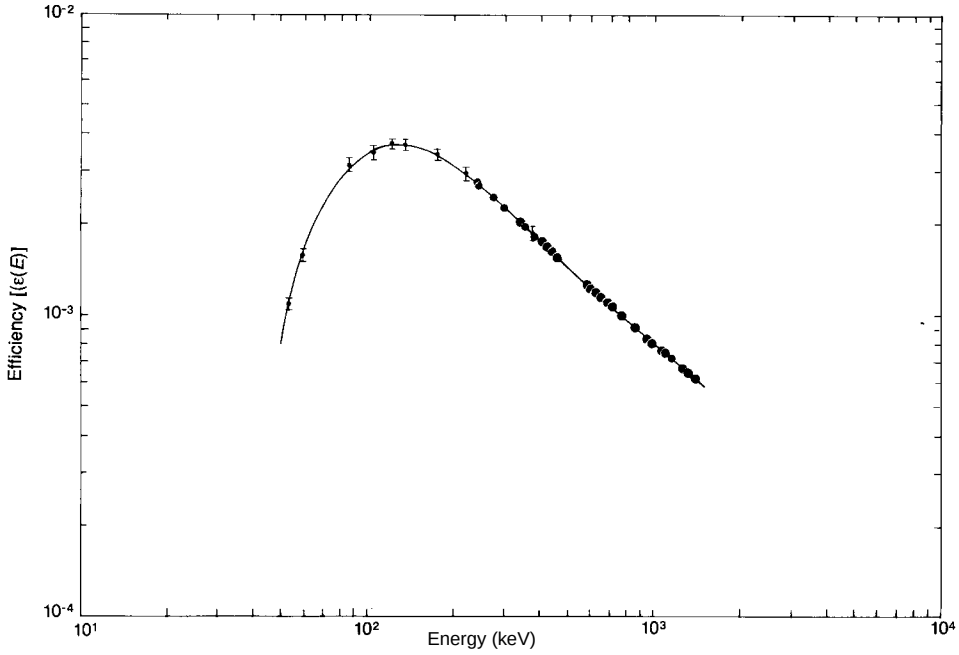
on a linear plot (a is a normalizing constant that can be determined for each efficiency curve separately), as shown in Figure 2. This function provides better resolution of the efficiency data when plotted. There should be no large gaps in energy between the measured or calculated efficiency values (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.

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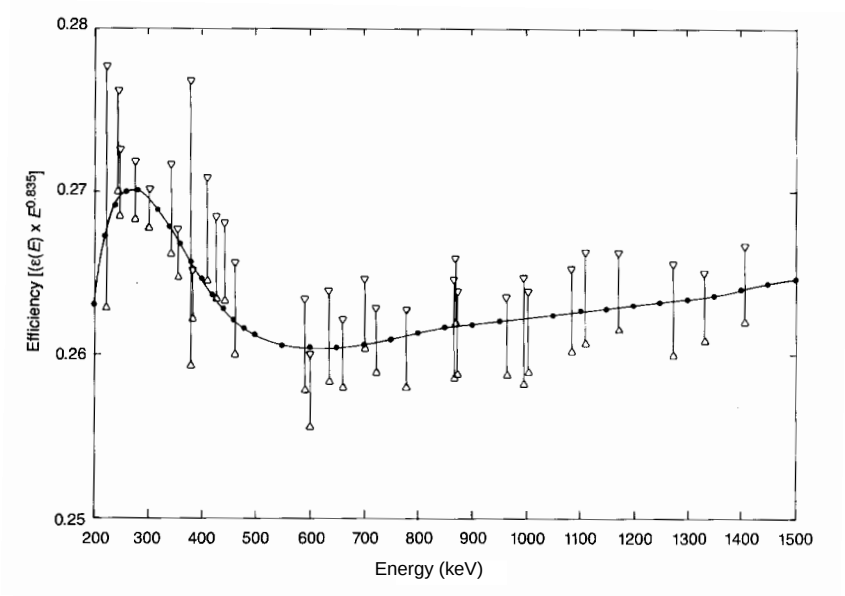
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NOTE—Efficiency as a function of energy on a log-log plot for a point source at a 15 cm source-to-detector distance for a coaxial Ge spectrometer.

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Figure 1—Full-energy peak efficiency



NOTE—The same data as in Figure 1, plotted as $\varepsilon \times E$ (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—$\varepsilon \times E$ (keV) as a function of gamma-ray energy

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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-

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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:

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

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, 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).

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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

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$$C_{a_1} = e^{\mu\rho d} \tag{5}$$

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

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

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

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

where

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

$\varepsilon(E)$ 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,

$C_{i \neq a}$ 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, $P(E)$.

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

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6.6.1 Radioactive decay

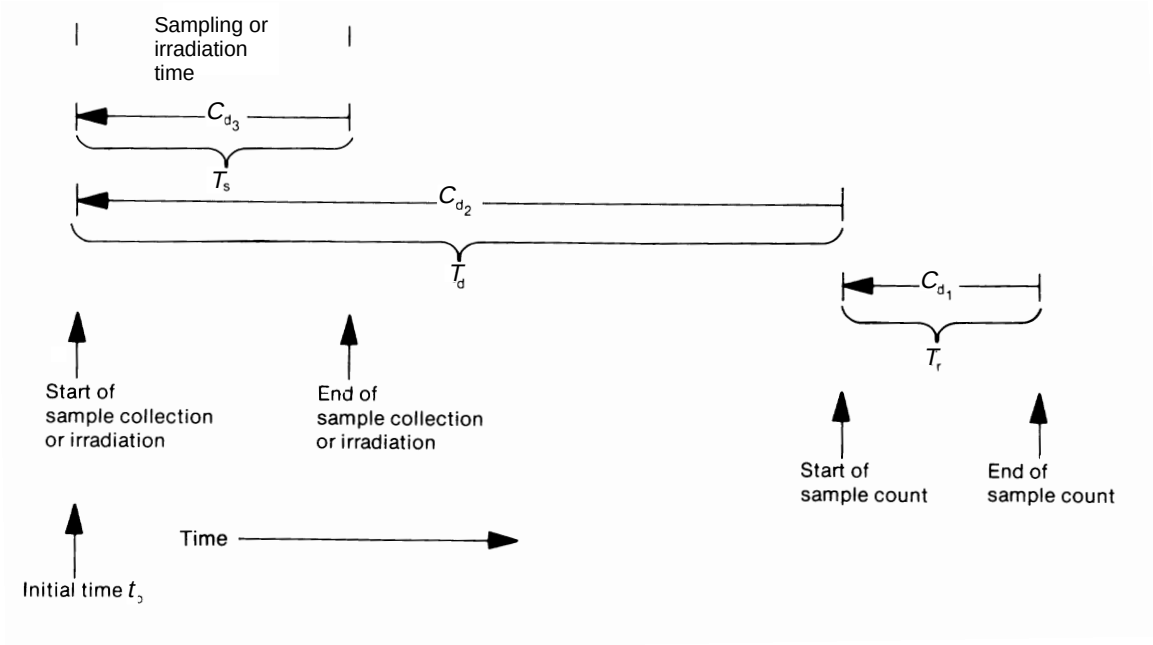
The correction factor that normalizes the nuclide activity for radioactive decay is normally composed of the following components:

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.

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

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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:

$$C_{d_1} = \frac{\lambda T_r}{1 - e^{-\lambda T_r}} \tag{11}$$

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:

$$C_{d_2} = e^{\lambda T_d} \tag{12}$$

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.

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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

$$C_{d_3} = \frac{1}{1 - e^{-\lambda T_i}} \tag{13}$$

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

$$C_{d_3} = \frac{\lambda T_s}{1 - e^{-\lambda T_s}} \tag{14}$$

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.

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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.

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7.4 System efficiency

The reproducibility of the full-energy peak efficiencies 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 (greater than 1300 keV) gamma ray (e.g., the National Institute of Standards and Technology (NIST) mix of ^{125}Sb, ^{154}Eu, and ^{155}Eu, or the equivalent). The same counting geometry shall be used each time for these checks with a reproducible source-detector distance (preferably greater than 10 cm). Acquire sufficient counts in the spectrum so as to obtain at least 20 000 counts (approximately 0.7% counting uncertainty) in the net peak areas of the peaks used. Deviations in the peak counting rates, corrected for decay, of either the low- or high-energy peaks that lie more than two estimated standard deviations outside of the mean shall be investigated. An alternate test is to apply a chi-square test to the measured net-area counting rates of the low- and high-energy peaks over time. A good description of the application of the chi-square test to radiation measurements is given on page 55 of Tsoulfanidis [B40].

7.5 System resolution

The energy resolution at both high and low energies should be measured and recorded periodically (e.g., during efficiency calibration tests in 7.4) and any unexpected increase in the peak width investigated. A test similar to that given in B.4 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 of measured gamma-ray peak areas.

8. Verification of the analysis process

Verification of the analysis process is carried out through a set of performance tests of the analysis software, and through an evaluation of the accuracy of the parameters used to convert a net peak area counting rate to a gamma-ray emission rate or to an activity. The gamma-ray analysis program should be demonstrated to find peaks in a spectrum and to measure their net area above a minimum “observable” level. It shall measure the net peak-area counting rates within two estimated standard deviations of their “true” values (for a singlet peak with no appreciable baseline), and the accuracy shall be determined with which the net peak-area counting rates of equal-size doublet peaks for a given separation in FWHM increments can be measured.

If commercial software is purchased, the tests in this clause 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 be 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, we recommend that the user perform these tests to assure that analytical parameters are optimized for the applications of the users.

The accuracy of the pulse-pileup correction and the detector efficiency values, which are needed to convert a net peak-area counting rate to a gamma-ray emission rate, is also verified through test procedures. In the case of coincidence summing, only an estimate of the magnitude of the correction is determined by test, leaving the user with the decision to either apply a correction or to modify the counting geometry to reduce the size of the correction to a negligible amount. Other factors, e.g., radioactive decay, attenuation, or gamma-ray emission probability, are reported in the literature and a reliable source for these values that can be referenced should be used.

Quality results from the tests in the following subclauses can only be achieved if the quality of the acquired spectra is high, with no interfering peaks present from the background or contaminants that cause interferences with the peaks of interest. Therefore, a background spectrum shall be acquired for a count time not less than the count time of the longest acquisition time used in the performance tests, and the spectral purity

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of each radioactive source used shall be verified by acquiring a count of sufficient time to disclose any potential interference. Interferences shall be avoided whenever possible, or a correction applied when necessary.

8.1 Test of automatic peak-finding algorithm

The following test has been designed to determine how well singlet peaks on a flat baseline that are at or above an "observable" level can be detected with the peak-finding algorithm.[6] 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 is used when counting samples for radioanalysis.

This test shall be used to satisfy the criteria set forth in 6.1 regarding the ability of the peak-finding algorithm to find singlet full-energy gamma-ray peaks automatically.

a) With the ^{137}Cs source [positioned at the same source-to-detector distance as selected to measure its net peak-area counting rate and FWHM (see Clause 8)], accumulate a spectrum for a counting time that results in the full-energy 661 keV peak having an expected net area of about 50 counts.

b) Execute the peak-finding algorithm routine. The peak-finding algorithm should find the peak every time in five trials. (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.)

CAUTION

Some peak-finding algorithms require that the counts comprising the peak be smoothly varying 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 another spectrum for a counting time that results in an expected net area in the 661 keV peak of 100 counts.

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 results in each channel of the baseline on each side of the ^{137}Cs peak having an expected number of counts equal to 720/(FWHM). FWHM is the energy resolution expressed in channels of the 661 keV peak at 1 FWHM, as measured in step a).

e) Execute the peak-finding algorithm routine. The peak at 661 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 and rerun the test.[7] Record the value of L_p for which the peak is found.

NOTE—The effort and time required may be reduced by stopping the count and analyzing it at a baseline whose expected counts per channel correspond to $L_p = 4.65$.[7] If the peak is found, the count (only ^{65}Zn source now being counted) 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 $L_p = 2.33$.

[6]The peak-finding algorithm is expected to find a peak in a spectrum whose area, 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 $L_p = 2.33$, which corresponds to the value of L_p 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 if too many false peaks are found at this sensitivity (a judgment call).

[7]See Footnote 6.

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f) Erase and then accumulate a new spectrum with the ^{137}Cs source at its designated source-to-detector distance for a counting time that results in an expected net area in the 661 keV peak of 1000 counts.

g) 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 (7.2×10^4)/(FWHM) counts in each of the baseline channels (FWHM in channels). This many counts may take an entire day to accumulate, even at a counting rate of 2000 s^{-1}.

h) Execute the peak-finding algorithm routine. The 661 keV peak should be found when the peak-finding algorithm is applied. If the peak-finding algorithm cannot find the peak, repeat the test [steps (f) through (h)]. If it is not found the second time, increase L_p and rerun the test.[7] 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 Footnote 6 can be met. Consult the user 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 Head [B20] and McNelles and Campbell [B30].

8.2 Test of independence of peak-area from the gross peak-height-to-baseline-height ratio

This test uses the same sources and source-to-detector distances as are used in 8.1 and requires the same high purity for the ^{65}Zn, ^{22}Na, or ^{60}Co spectrum in the energy region covered by the 661 keV peak.

a) With the ^{137}Cs source placed at its designated source-to-detector distance, accumulate a spectrum until approximately 5000 counts are recorded in the gross area of the 661 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 baseline underlying the 661 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 1173 keV gamma ray at 661 keV may need to be made).

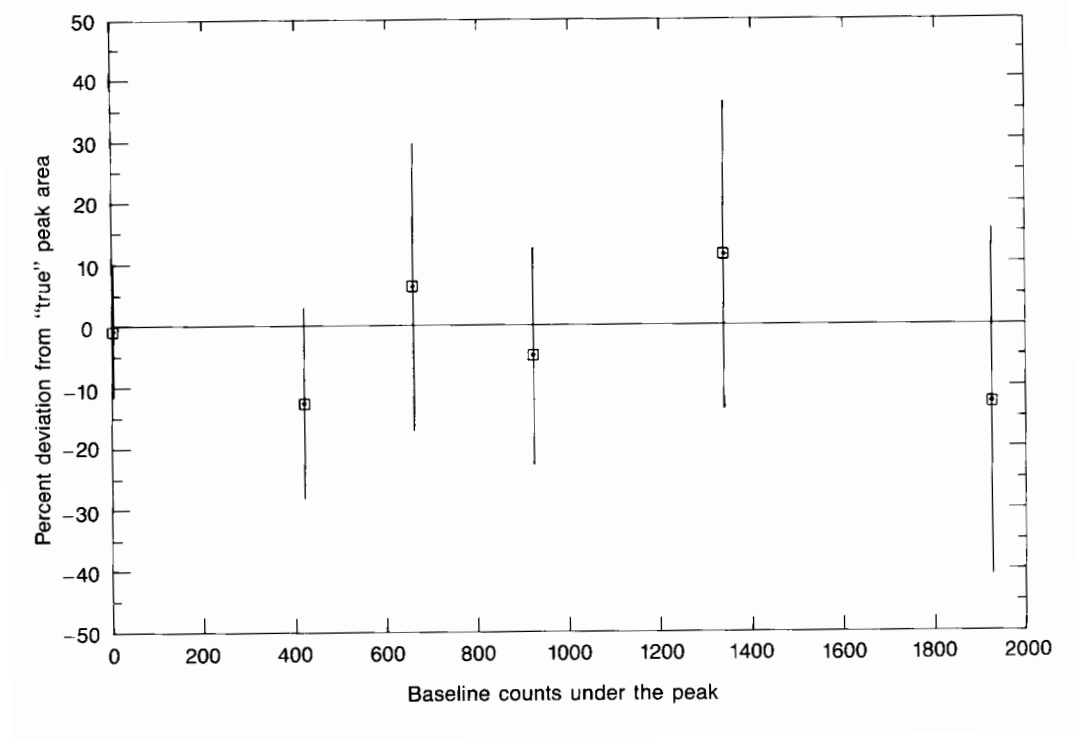
e) Continue the counting until each channel in the baseline surrounding the 661 keV peak contains (4.9×10^4)/(FWHM) counts. This many counts may take one or more days to accumulate.

f) Measure the net area of the 661 keV peak and its uncertainty with the spectral analysis program being tested. If there is a 661 keV peak in the ^{65}Zn or background spectrum, its contribution shall be subtracted (see Clause 8).

g) The measured 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 error 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 baseline) as a function of the baseline height for a peak area of 110 counts and 9844 counts, respectively.

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NOTE—Deviation of the measured net peak area from a "true" peak area of 110 counts as a function of the baseline height. 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 baseline from a second source. The horizontal axis represents the integral baseline counts under the peak.

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Figure 4—Deviation in peak area as a function of baseline height

Table 1—Measured net-peak areas as a function of the baseline height

Baseline (Y_i)[a]	Measured net peak area	Uncertainty[b]	Deviation from first count (%)
1	9 844	1.0	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 baseline channel surrounding peak.
[b]Uncertainty in the net peak area represents one estimated standard deviation.

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8.3 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 gamma-ray peaks (i.e., with doublet peaks, at least one side of the peak does not return to the baseline 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 gamma-ray peak to determine the peak areas of each of the component peaks. 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 1332 keV peak channel position can be raised or lowered in increments of approximately one-half of 1 FWHM.

a) With a ^{60}Co source (other long half-life radionuclides emitting a gamma ray with a high-emission probability and free of interferences may also be used), accumulate a spectrum. 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 preset live time so that about 20 000 counts, and preferably 50 000 counts, are in the peak area.

b) Use the analysis program to find the 1332 keV peak and measure its net peak-area counting rate. This value is 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 1332 keV peak is raised the equivalent of 1 FWHM. The spectrum accumulated in step (b) should be restored to the analyzer memory and additional 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 1332 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 accuracy (all uncertainties combined; see Clause 9) 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 accuracy, 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 1-1/2 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 1332 keV peak is raised the equivalent of 2 FWHM and reduce the second acquisition time by a factor of 10. 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 accuracy) 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 accuracy, 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 2 FWHM.

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

CAUTION

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).

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Additional tests for other separations and for other net peak-area ratios are recommended to define the capabilities and limitations of the peak-finding and area-measuring algorithms better. Figure 5 indicates the error in the net areas resulting from various separations of equal-sized doublets, and Figure 6 indicates the error in the net areas resulting from the weaker (less active) of two unequally sized doublets for the peak separations given.

8.4 Pulse pileup (random summing)

Laboratories with heavy sample loads may optimize the sample throughput by increasing the counting rates of the samples with minimal loss in peak position and area-measurement accuracy. Therefore, operation of Ge spectrometers at counting rates above 1000 s^{-1} may be used if pulse-pileup corrections are applied (see B.5 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 that 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 1000 s^{-1} (for amplifier-time constant less than 3 μs), this test may not be required, and no correction may need to be applied.

8.4.1 Test

This test is designed to check the pulse-pileup correction at two energies, 661 keV (^{137}Cs) and 2614 keV (^{208}Tl, which is a daughter of ^{228}Th). Other suitable sources may be used as well. The gain should be adjusted to be the same as used in counting samples. Similar tests may be designed to check the pulse-pileup 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 counts, and preferably 50 000 counts, in the 661 keV full-energy peak (net area). Record the elapsed time or live time (if the use of live time constitutes a part of the correction method). The ^{137}Cs source is referred to as the fixed source.

b) Measure the net counting rate and its uncertainty for the 661 keV peak area using the same method normally used to evaluate the peak area, including methods employed to correct for pileup and dead time.

c) Without moving the ^{137}Cs source, introduce a high-activity ^{57}Co or other essentially monoenergetic low-energy source of gamma rays (100 keV < E < 200 keV). Position the added source behind the fixed ^{137}Cs source so that the gross counting rate is approximately 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 time as in step (a).

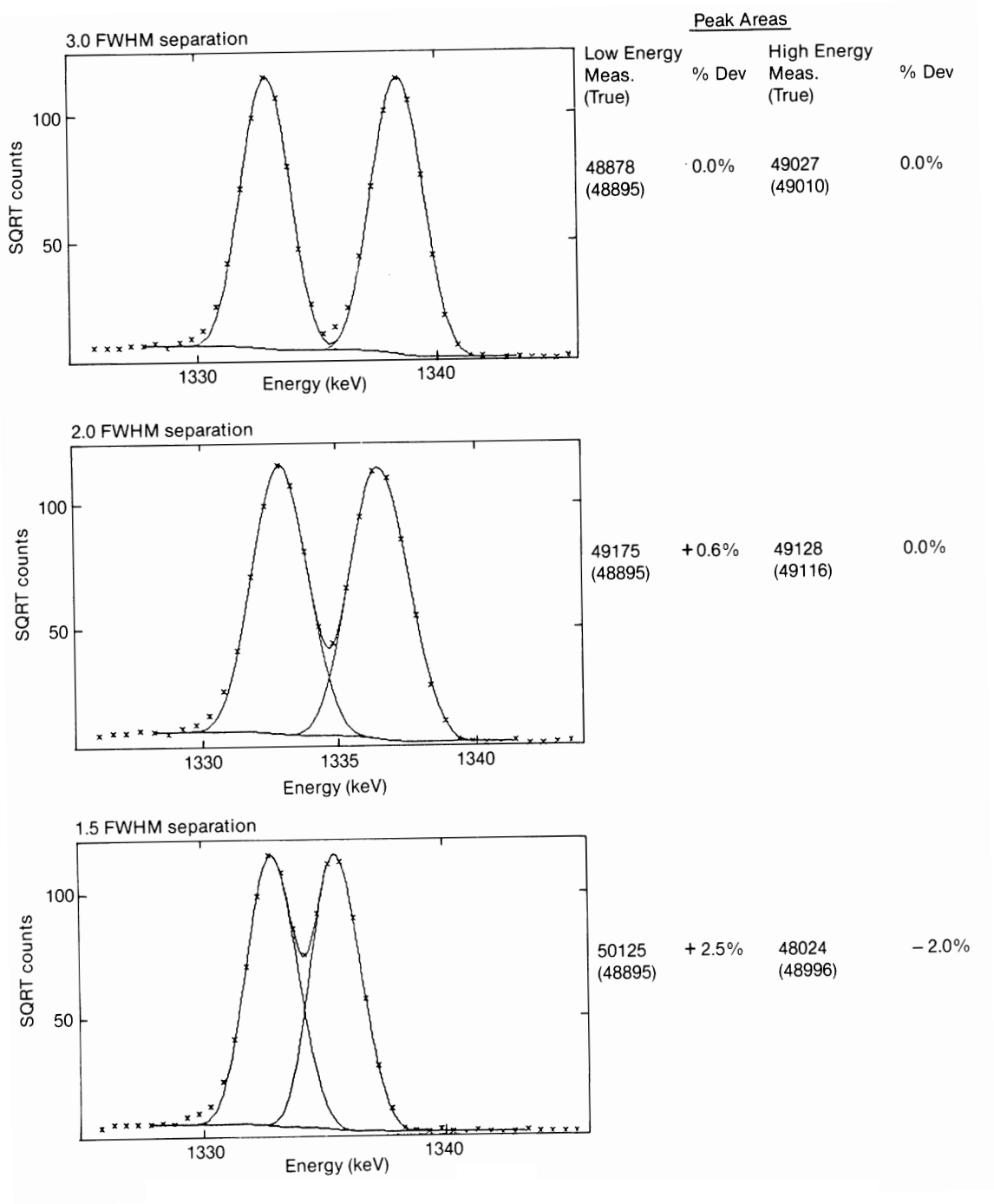
e) Evaluate and correct the net counting rate of the 661 keV peak as in step (b) (including any method employed to correct for pileup and dead time). Propagate the uncertainty in the net area of the 661 keV peak with that in the correction factors (see Clause 9).

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 y and is issued as a standard reference material by the National Institute of Standards and Technology (NIST), formerly known as the U.S. National Bureau of Standards]. Any difference between the corrected peak counting rate of the stationary source and that measured in step (a) that lies 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 accuracy 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.

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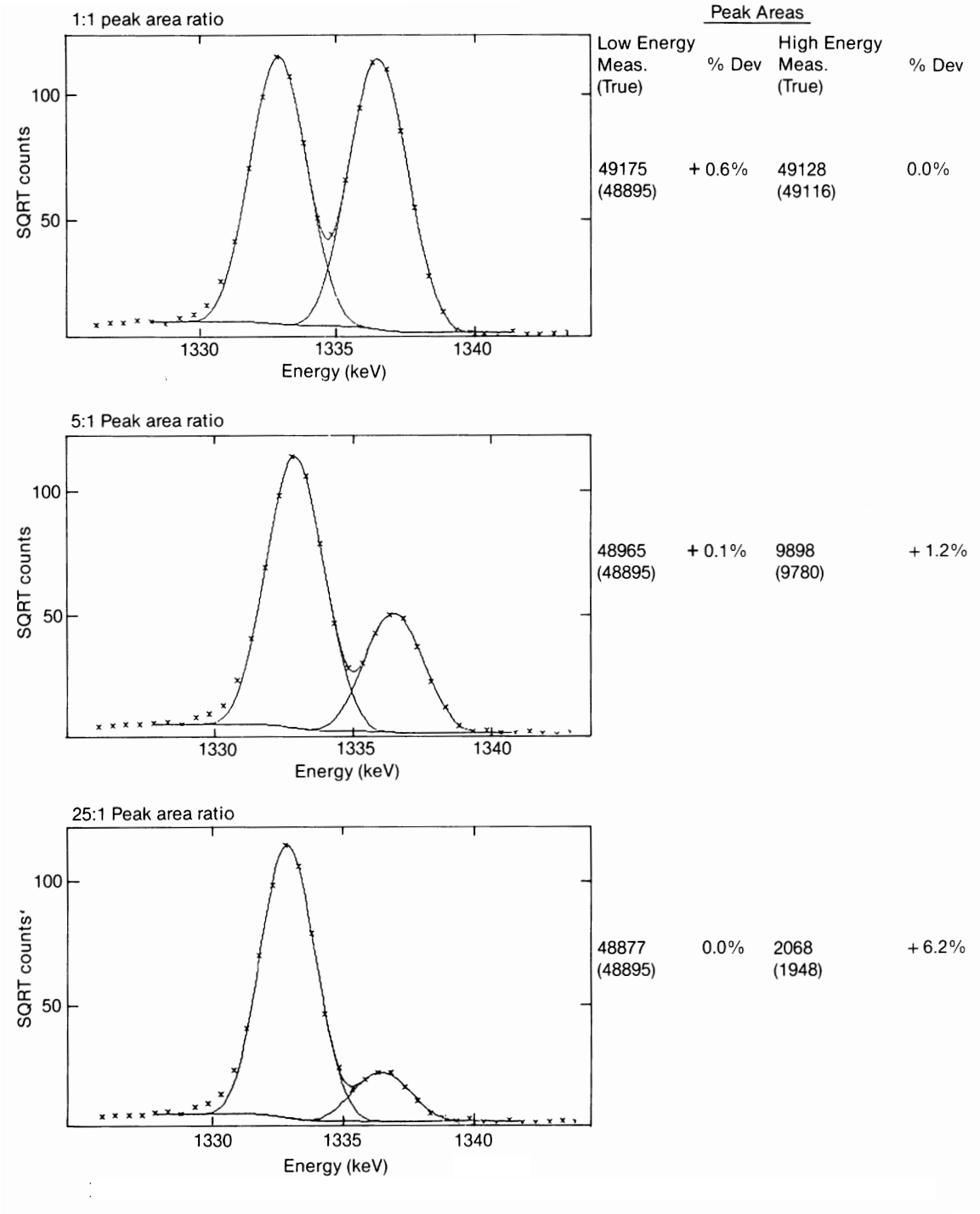


NOTE–Deviation in doublet peak area as a function of peak separation in FWHM for two equally sized components. Areas are quoted here rather than the net peak-area counting rate, as described in 8.3.

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

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NOTE–Deviation from true peak area for weaker of two unequally sized doublets. Areas are quoted here rather than the net peak-area counting rate, as described in 8.3.

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

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8.5 Assessment of the magnitude of coincidence summing

The magnitude of coincidence summing should be evaluated anytime the solid angle of a "point size" sample with the detector is greater than 0.02 steradians. When counting samples in close proximity to the detector (mean sample distance is less than or equal to 5 cm), the presence of coincidence summing shall be evaluated according to the procedure described in 8.5.1, or according to a method of equal sensitivity.

Although 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-sized detectors in order to determine if additional (and substantially more complicated) corrections are required. It is important to note that the test in 8.5.1 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 coincidence summing at a given geometry. This test, best carried out before measuring the detector efficiency, is conducted with a mixed radionuclide source that is composed of ^{125}Sb, ^{154}Eu, and ^{155}Eu, or the equivalent. If the source used in the test is a point source, the resultant curve of peak-area losses versus distance should be a worst-case result for summing losses out of the 591 keV peak from the decay of ^{154}Eu.

The test in 8.5.1 is designed to measure the change in the gamma-ray peak area ratio of the 591–600 keV gamma-ray peaks from the NIST long-lived mixed radionuclide standard, or the equivalent, relative to a distant reference position. As the source-detector distance increases, this ratio will approach a constant, as shown in Figure 7. Due to coincidence summing of the 591 keV gamma ray with other ^{154}Eu gamma rays (a spectrum of the NIST long-lived mixed radionuclide standard is shown in Figure 8), the 591 keV full-energy peak loses counts when the sample is counted close to the detector (see D.1.2 for the calculation of the coincidence-summing correction factor for the 591 keV gamma ray). If the detector does not observe the Te K X rays (27–32 keV) resulting from internal conversion in the decay of ^{125}Sb (due largely to the 35 keV transition), then the 600 keV full-energy peak does not lose counts due to 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 the ^{125}Sb decay scheme in Browne and Firestone [B5] and Firestone [B13].)

This change can be expressed in terms of ratios, r, of net peak areas, $N(E)_i$, for the energies E = 591 keV and 600 keV, at each distance, i, that a spectrum was accumulated.

$$r_i = \frac{N(591)_i}{N(600)_i} \tag{15}$$

By setting the ratio at one of the farther distances (e.g., 10–15 cm source-to-detector distance where the coincidence 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:

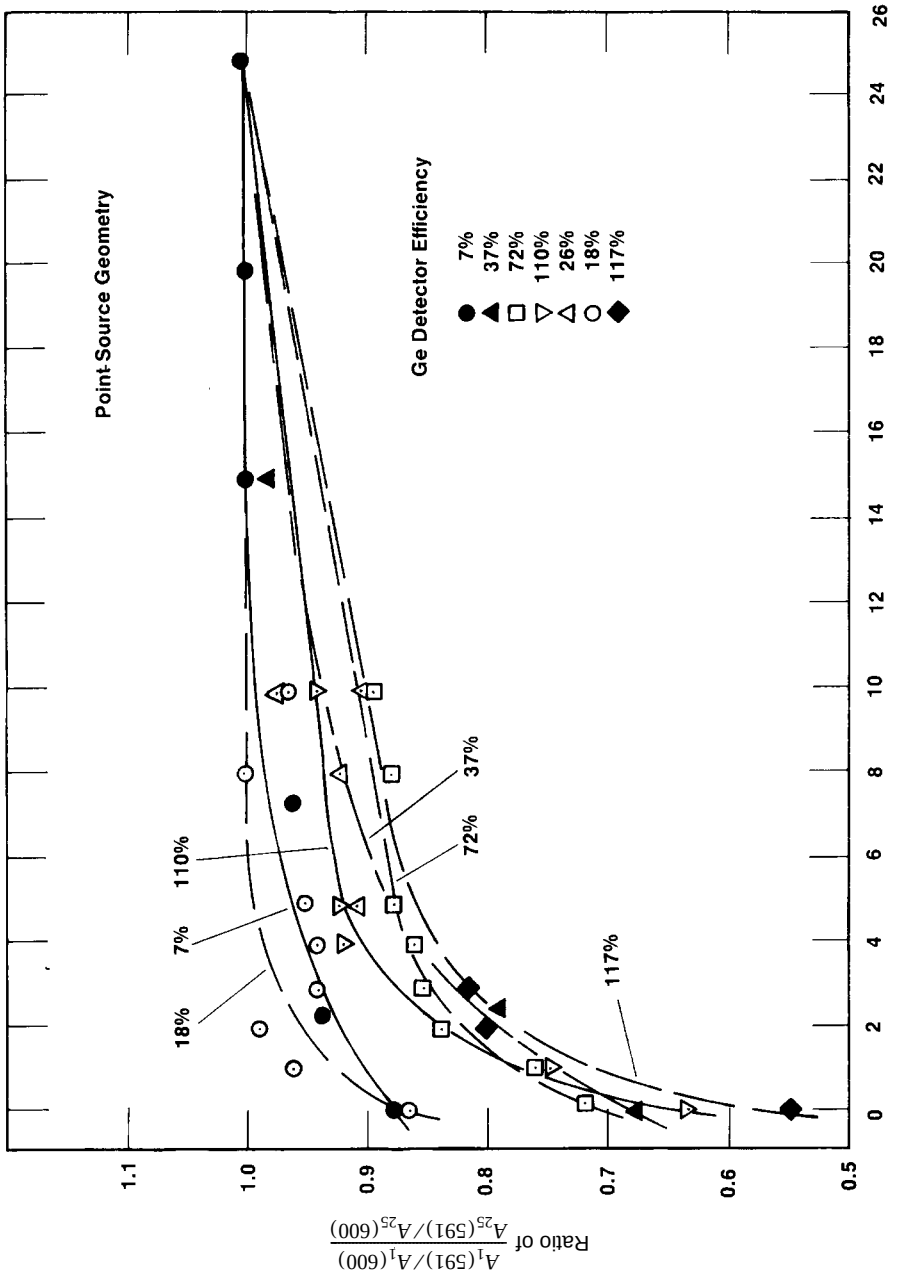
$$R_i = \frac{[N(591)_i]/[N(600)_i]}{[N(591)_r]/[N(600)_r]} \tag{16}$$

The coincidence-summing correction (multiplicative) factor for the 591 keV gamma ray is $C_c = 1/R_i$. The value of R_i, for gamma rays in a cascade that are emitted from other radionuclides, is 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.

CAUTION

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

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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 Ge detectors. The curves are normalized at a 25 cm source-to-detector distance. 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 1332 keV gamma rays from ^{60}Co at a source-to-detector distance of 25 cm).

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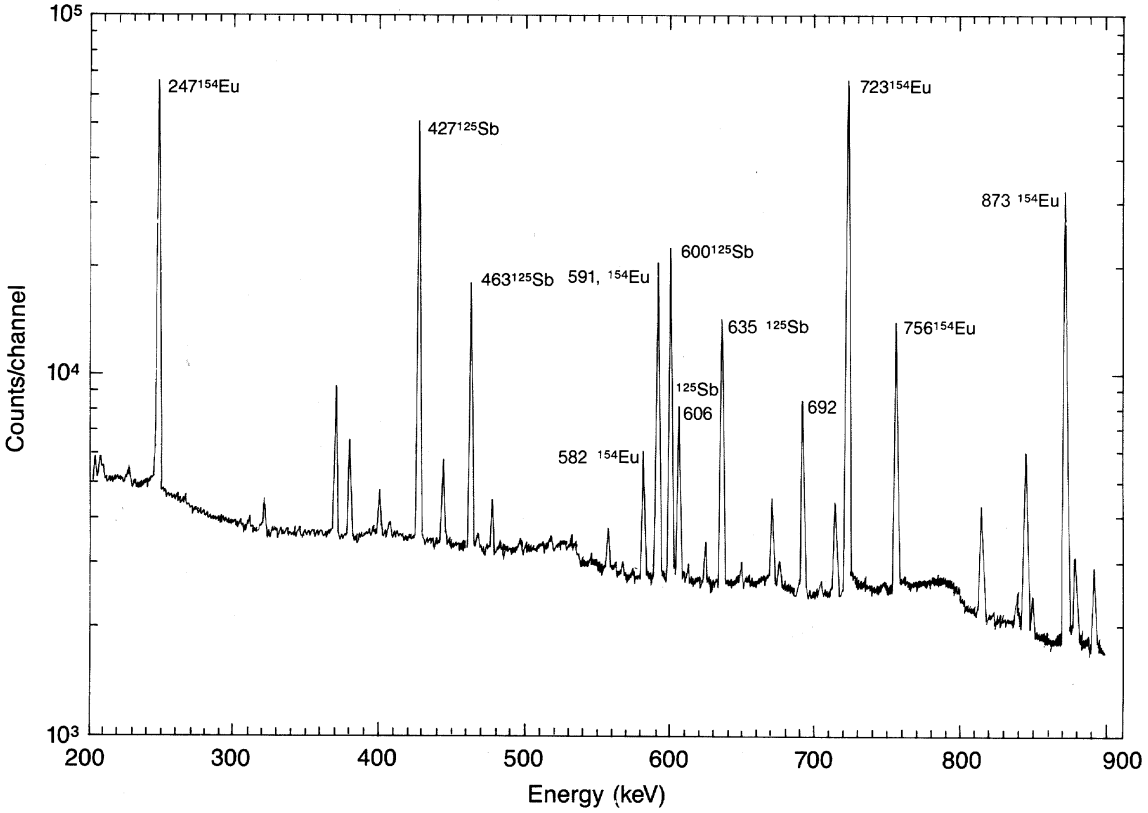
Figure 7—Cascade-summing corrections for a ^{152}Eu 591 keV gamma ray

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A coincidence-summing correction may not be required at a given sample-to-detector 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 for r_r in 8.5.1, but a 15 cm, or even 20 cm, source-to-detector distance is recommended for larger (greater than 20%) Ge detectors.



NOTE—Ge gamma-ray spectrum from the NIST long-lived radionuclide mix (i.e., ^{125}Sb, ^{154}Eu, and ^{155}Eu).

Figure 8—Partial Ge gamma-ray spectrum of NIST long-lived mix

8.5.1 Test

a) With a mix of ^{125}Sb, ^{154}Eu, and ^{155}Eu in solution form, prepare (or purchase) a sample to match the geometry and matrix to be tested (see B.7 on the preparation of working standards).[8] The counting rate of the sample should not exceed 1000 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 counts, and preferably 50 000 counts, are in the net areas of the 591 keV and 600 keV gamma rays. 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 counts, 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 peak areas.

[8]Calibrated point sources and solutions can be purchased directly from NIST, Gaithersburg, MD 20899-0001 (www.nist.gov/).

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d) Compute the ratio, $R_i = r_i/r_r$, from the ratios r_i and r_r measured in steps (b) and (c), respectively. Coincidence-summing corrections may not be required if $(1 - R_i)$ is less than or equal to one-third of the total uncertainty desired. (The acceptable accuracy of the routine gamma-ray measurements, at this geometry and for this sample matrix, determines the amount of coincidence summing that can be tolerated without applying a correction.) If corrections are to be avoided and $(1 - R_i)$ greater than one-third of the total uncertainty is desired, then either the mean source-to-detector distance shall be increased until the test in 8.5.1 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 error associated with not correcting for coincidence 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 Ge detectors.

8.6 Bias in the relative full-energy peak efficiency

The bias in the relative full-energy peak efficiency over the energy range of the efficiency curve shall be measured. This should be done by counting at least one radionuclide that emits gamma rays that span the energy region of interest and whose relative abundances (intensities) are precisely known. Some radionuclide sources that have relatively long half-lives (greater than 1 wk) with spectra containing strong (large peak height-to-baseline ratio) singlet gamma rays include the NIST mix (containing ^{125}Sb, ^{154}Eu, and ^{155}Eu), ^{106}Ru-^{106}Rh, ^{125}Sb, ^{140}Ba-^{140}La, ^{152}Eu, and ^{232}U with its daughter activities in equilibrium. Precise values for the relative intensities of these radionuclides can be found in Firestone [B13] and Gehrke [B15].

8.6.1 Test

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

b) Analyze the spectrum following normal procedures, and apply all applicable corrections. Deduce the measured relative gamma-ray intensities by using Equation (3), 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-baseline 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 9) in the gamma-ray emission rate or radionuclide activity.

CAUTION

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 coincidence summing of coincidence gamma rays in the detector, caution must be exercised. The magnitude of the coincidence-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 coincidence summing. However, agreement in the relative gamma-ray intensities does not assure that coincidence-summing corrections are small or zero.

8.7 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 with standards of radionuclides whose gamma-ray emission rates are accurately known and are from, or are traceable to NIST.

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In this case, the gamma-ray emission rates are deduced in step (b) of 8.6.1 using Equation (3) from 6.5, and the gamma-ray emission rates are compared to the standard values. Lack of agreement indicates neglected or erroneous corrections.

8.8 Radionuclide identification

Identification and quantification of the radionuclides associated with the measured gamma-ray energies and intensities are performed by many gamma-ray spectral-analysis programs. This requires a decay-data library that should contain a carefully selected set of the radionuclides anticipated 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 probabilities per decay. The uncertainties on each of these values may also be useful information to have in the decay-data library. These parameters, which are normally available from recent decay-data evaluations, should have their references documented. Some evaluated decay data are in Browne and Firestone [B5], ENSDF [B39], Kocher [B26], and Kocher [B27].

Since radionuclide identifications are based primarily 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.

CAUTION

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 computer memory or disk space, and especially computing time during the library-search process, suggests 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 may arise where the radionuclide associated with certain gamma-ray peaks present in the spectrum are not automatically identified, the identification process is not without its deficiencies. Samples to be analyzed from different origins should have separate decay-data libraries that are selected for efficient yet complete radionuclide identification.

8.9 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 can 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 intensity) 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 gamma-ray peaks (e.g., fresh reactor-coolant water), even these techniques can lead to large errors.

CAUTION

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.

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9. Uncertainties and propagation of uncertainty

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 Bureau International des Poids et Mesures (BIPM). It allows all of the uncertainty components to be propagated by statistical rules and results in a combined uncertainty (category A and B as defined in 3.12; see Taylor and Kuyatt [B38]). Table 2 lists the equations for propagating uncertainties for several common algebraic expressions. (See Chapter 2 of Tsoulfanidis [B40] for a more detailed discussion of uncertainties and their propagation.) The principal components of the combined uncertainty in a measured gamma-ray emission rate are those in the

a) Efficiency calibration standards;
b) Measurement of the peak areas;
c) Reproducibility of the sample-detector geometry;
d) Pulse pileup;
e) Coincidence-summing corrections;
f) Decay corrections during and prior to counting;
g) Absorption corrections when the sample matrix differs from that of the standard;
h) Detector full-energy peak efficiency interpolation;
i) Subtraction of the gamma-ray peaks in the “blank” background that are at the same energies as those in the sample.

Table 2—Uncertainties

Function[a]	Manipulation	Uncertainty[b]
$y = a_1x_1 - a_2x_2$	Addition/subtraction	$s_y = \sqrt{a_1^2 s_{x_1}^2 + a_2^2 s_{x_2}^2}$
$y = a_1x_1 + a_2x_2$	Addition/subtraction	$s_y = \sqrt{a_1^2 s_{x_1}^2 + a_2^2 s_{x_2}^2}$
$y = a_1x_1x_2$	Multiplication/division	$\frac{s_y}{y} = \sqrt{\left(\frac{s_{x_1}}{x_1}\right)^2 + \left(\frac{s_{x_2}}{x_2}\right)^2}$
$y = a_1\left(\frac{x_1}{x_2}\right)$	Multiplication/division	$\frac{s_y}{y} = \sqrt{\left(\frac{s_{x_1}}{x_1}\right)^2 + \left(\frac{s_{x_2}}{x_2}\right)^2}$
$y = x^m$	Power of x	$\frac{s_y}{y} = \frac{ms_x}{x}$
$y = e^{-ax}$	Power of e	$\frac{s_y}{y} = as_x$
$y = f(x_1, x_2)$	Function of two variables	$s_y = \sqrt{\left(\frac{\partial f}{\partial x_1}\right)^2 s_{x_1}^2 + \left(\frac{\partial f}{\partial x_2}\right)^2 s_{x_2}^2}$

[a] a, a_1, a_2, and m represent constants. x, x_1, and x_2 are independent variables. y is the dependent variable.
[b] s_i is the estimated standard deviation in variable i.

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The uncertainties in the gamma-ray emission probabilities should be propagated with the other uncertainty contributions when the activity is being calculated. The tests of performance described in this standard in Clause 7, Clause 8, and Clause 9 should provide a good initial estimate of the uncertainty components for any given Ge spectrometer and analysis program. These estimates should be refined as experience is gained. Typical ranges for these uncertainty components are listed in Table 3.

Table 3—Uncertainty contributions

Uncertainty contribution	Typical magnitude (%)[a]
Activity of standard source	0.1—2.0
Source aliquots	0.1—3.0
Peak-area measurement	0.1—30.0
Background peak-area variations	0.0—100.0
Variations in sample attenuation	0.0—50.0
Source-detector geometry	0.1—10.0
ADC live timer	0.0—0.5
Pulse pileup (high counting rate)	0.0—30.0
Coincidence summing	0.0—100.0
Decay of radionuclide	0.0—5.0
Gamma-ray emission probability	0.1—20.0
Full-energy peak efficiency	0.5—15.0

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

Whenever a set of values with equally sized uncertainties are to be averaged, a sample mean is computed along with its standard deviation as follows:

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i \tag{17}$$

$$s_x^2 = \frac{1}{(n-1)}\left[\sum_{i=1}^{n} (x_i - \bar{x})^2\right] \tag{18}$$

where

x_i are the values to be averaged,
n is the number to be averaged,
$\bar{x}$ is the average value,
s_x^2 is the sample variance (the standard deviation of the sample is the square root of the variance).

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

$$\mu_x = \frac{\sum_{i=1}^{n} w_i x_i}{\sum_{i=1}^{n} w_i} \tag{19}$$

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 $[\chi^2/(n-1)] >> 1$, 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 equations represent the internal variance and the external variance of weighted means.

Internal variance:

$$s^2(\mu_x, 2) = \frac{1}{\sum_{i=1}^{n} w_i} \tag{20}$$

External variance:

$$s^2(\mu_x, 1) = \frac{\sum_{i=1}^{n} w_i (x_i - \bar{x})^2}{(n-1)\sum_{i=1}^{n} w_i} \tag{21}$$

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

Whenever it is necessary to multiply the combined uncertainty by a factor to obtain a more conservative overall uncertainty, the multiplying factor shall be stated (e.g., 2, reporting uncertainties at about a 95% confidence level). Whenever a set of counts has been taken for a constant number of counts (i.e., the counting times vary), these counts shall be averaged using the harmonic mean (see Colle [B7]).

Whenever environmental and other very low-level activity samples are analyzed, 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 should be reported. The uncertainty in the net peak area indicates whether or not the peak is truly a positive peak, while the combined uncertainty indicates the accuracy with which the reported value is known.

For accurate estimates of combined 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 ANSI N13.30-1996.)

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Annex A

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[10]PTB-Ra-12e is a reprint of the report AAEC-LIB/TRANS-773, an English translation of the original (in German) report by G. C. Lowenthal, Lucas Heights Research Laboratories, Australian Atomic Energy Commission Research Establishment.

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[13]NUREG publications are available from the Superintendent of Documents, US Government Printing Office, P.O. Box 37082, Washington, DC 20013-7082, USA.

[14]NIST publications are available from the National Institute of Standards and Technology Library, Gaithersburg, MD 20899, USA (www.nist.org/).

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Annex B

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Procedures for characterization of a Ge gamma-ray spectrometer

The following procedures have been tested and found to be helpful in the set-up and calibration of Ge spectrometers. They have been gathered from several sources, some unpublished, and are provided in this annex for the convenience of the reader. The references given for each of the procedures provide additional information. Use of these procedures is optional, but they do meet the requirements of this standard.

B.1 Adjustment of the pole/zero cancellation and dc level

Rationale. Ensures that the pole/zero cancellation and the dc level of the amplifier output have been adjusted to provide proper processing of a signal pulse by an MCA system. This is required for each detector, preamplifier, amplifier combination, and whenever the shaping time is changed. Changes of the gain should not affect the pole/zero cancellation. The following procedure may be used to satisfy the test in 7.2.

B.1.1 Manual adjustment

a) Place a ^{137}Cs source at a distance from the detector that establishes a counting rate of about 500 s^{-1} in the full-energy peak (approximately 2000 s^{-1} 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 baseline (no long negative or positive tail). Part (a) of Figure B.1 shows pulses properly adjusted for pole/zero. Parts (b) and (c) of Figure B.1 are examples of pulses unadjusted for pole/zero. Reset the position of the baseline restorer.
e) Document completion of this procedure in the instrument logbook.

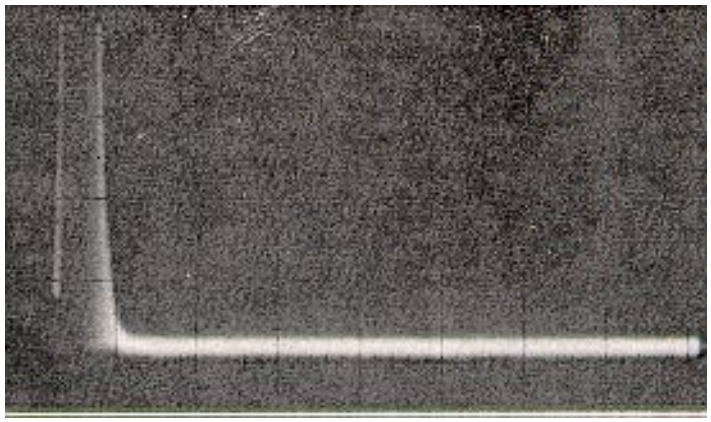
B.1.2 Automatic adjustment

Follow the manufacturer's instructions, or complete the following steps:

a) Place a ^{137}Cs source at a distance from the detector that establishes a counting rate of about 500–1000 s^{-1}.
b) Initiate the automatic setting of the pole/zero.
c) If possible, verify the pole/zero setting with an oscilloscope as described in B.1.1.
d) Document completion of this procedure in the instrument logbook.

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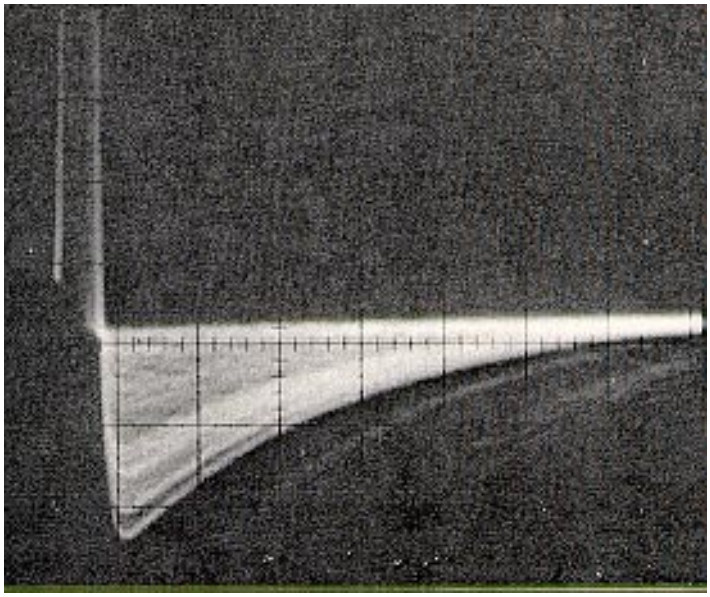
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(a) Amplifier output of pulses properly adjusted for pole-zero cancellation



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



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

Figure B.1—Amplifier output pulses showing correct and incorrect pole-zero cancellation

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B.1.3 Adjustment of the dc level

a) Check that the appropriate amplifier time constant, baseline restorer, input polarity, and approximate gain have been selected. Check the ADC conversion gain, lower-level discriminator (LLD), and zero settings. 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 pot 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, alternatively, 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.

B.1.4 Reference

See NCRP Report 58-1985 [B31].

B.2 Adjustment of the LLD, ADC zero, and initial energy scale

Rationale. Ensures that noise or the LLD does not block the processing of valid pulses from low-energy gamma rays, and permits 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 the ADC zero and the LLD are adjusted. The following procedures may be used to satisfy 5.3.

B.2.1 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 (in kiloelectron volts/channel):

a) a gain of 0.375 keV/channel (8192 channel spectrum) using ^{232}U in equilibrium with its daughters;

b) a gain of 0.250 keV/channel (8192 channel spectrum) using ^{88}Y or ^{152}Eu;

c) a gain of 0.500 keV/channel (4096 channel spectrum) using ^{88}Y and ^{139}Ce or ^{152}Eu;

d) a gain of 0.750 keV/channel (4096 channel spectrum) using ^{232}U in equilibrium with its daughters.[16]

B.2.1.1 Procedure for adjusting the ADC zero and initial energy scale

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.

a) Select the energy scale and conversion gain applicable to the experimental setup. Use Table B.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.

[16] A source of ^{228}Th is a suitable alternative to ^{232}U.

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Table B.1—Adjustment of energy scale to yield energy equation with zero intercept

Gain (keV/ channel)	Spectral channels	Lower peak			Upper peak		
		Energy[a]	Nuclide	Channel	Nuclide	Energy[a]	Channel
0.250	8192	165.86	^{139}Ce	663.4	^{88}Y	1836.06	7344.2
0.250	8192	121.78	^{152}Eu	487.2	^{152}Eu	1408.02	5632.2
0.375	8192	238.63	^{232}U daug	636.3	^{232}U daug	2614.53	6972.0
0.500	4096	165.86	^{139}Ce	331.7	^{88}Y	1836.06	3672.1
0.500	4096	121.78	^{152}Eu	243.6	^{152}Eu	1408.02	2816.1
0.750	4096	238.63	^{232}U daug	318.2	^{232}U daug	2614.53	3486.1

[a]Energies are those given on pages 351–367 of Debertin and Helmer [B10].

b) With the sources inside the detector shield, establish the source-to-detector distance at which the counting rate is about 500 s^{-1}. 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.

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

d) 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 B.1.

e) 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 B.1.

f) From the peak energies and the channel positions, calculate the energy scale using the following equations:

$$G = \frac{(E_u - E_L)}{(X_u - X_L)} \quad \text{(B.1)}$$

$$Z = E_L - G \times X_L \quad \text{(B.2)}$$

where

E_u is the energy of the upper gamma-ray peak in the spectrum,

E_L is the energy of the lower gamma-ray peak in the spectrum,

X_u is the channel position of the upper gamma-ray peak in the spectrum,

X_L is the channel position of the lower gamma-ray peak in the spectrum,

G is the gain (in keV/channel),

Z is the zero intercept (in keV).

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

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B.2.2 Adjustment of the LLD

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 analyzed) in the detector shield, and establish a counting rate of about 500 s^{-1}.

b) Set the LLD 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 analyzed.

CAUTION

The LLD should not be set below approximately 0.5% of the full scale.

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

B.3 Check of the MCA elapsed-time and live-time clocks

Rationale. Ensures that accurate measurement of the counting time is achieved by checking the elapsed-time clock of the ADC. The following procedure may be used to satisfy the test in 7.1. A pulse-height-stable pulser with a precise repetition rate is required [one based on a 60 cycle repetition rate (line frequency) is recommended for this purpose]. A calibrated oscilloscope may be useful to adjust the pulse-height of the pulser pulses.

B.3.1 MCA elapsed-time clock

a) Disconnect all inputs to the amplifier.

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

c) Select a pulser repetition rate between 50 pulses/s and 1000 pulses/s (a pulser based on the 60 cycle line frequency is recommended). The shape (i.e., rise and fall times) of the pulser pulses feeding the amplifier does not need to approximate closely 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 100–1000 s.

e) Sum the counts stored in the MCA, and compare with the number of pulses that was 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 (electronic or hardbound) assigned for this spectrometer.

B.3.2 Reference

See Helmer [B22].

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B.3.3 MCA live-time clock

A simple method for testing the live-time clock is proposed in Bartosek and Masek [B2]. This method requires construction of a simple circuit to generate a dead-time of exactly 50%, and therefore this procedure is only referenced.

B.3.4 Reference

See Bartosek and Masek [B2].

B.4 Measurement of the energy resolution and the peak-to-compton ratio

Rationale. Verifies the resolution specifications of the detector and ensures that the ability of the detector to energy discriminate full-energy peaks has not degraded with time. Through the measurement of the FWHM of several full-energy peaks ranging from less than 100 keV to greater than 1000 keV, an approximately linear function can be determined, as shown in Figure B.2. 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 7.4. Sources of ^{57}Co and ^{60}Co approximating point-source geometry (with activities of about 1–5 μCi) are needed in this procedure.

B.4.1 Measurement of the energy resolution at 122 keV and 1332 keV

B.4.1.1 Resolution at 122.1 keV

a) Establish the settings of the Ge 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 s^{-1}.

c) Acquire a spectrum with about 3000 counts in the peak channel for the gamma ray at 122.1 keV. If possible, adjust the fine gain so that there is an integer channel located within ± 0.1 channels of the peak position, and reacquire a spectrum. (Having an integer channel located at the mean channel of the peak simplifies the measurement of the energy resolution.)

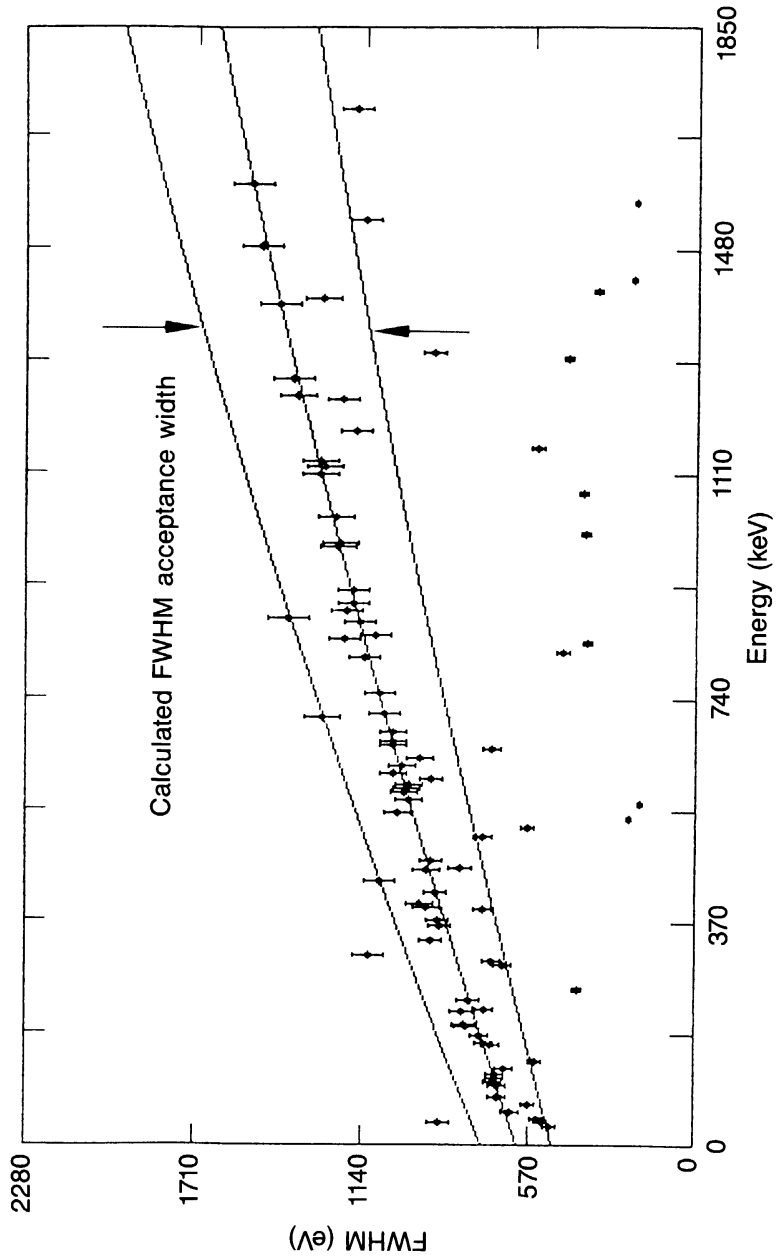
d) Perform an energy calibration using the 122.061 keV and 136.474 keV gamma rays (by inclusion of a ^{60}Co source, the gain may be measured more accurately, as in step (d) of B.4.1.2). If the ADC zero was previously set, the intercept term should be close to zero, even at a different gain.

e) If the FWHM is determined with the peak analysis program, go to step (i).

f) Subtract the baseline (see definition of baseline in Clause 3) from the 122.1 keV peak and determine the net peak area, *N*, by integration (also see alternate method in step (h) to measure the FWHM).

g) Measure the peak height, *Y,* at the centroid. If there is an integer channel at the estimated centroid, this simply consists of reading the counts in this channel from the MCA display, or from a listing of this spectral region, and subtracting the background counts estimated to be in this channel.

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NOTE—Resolution of the full-energy peak in FWHM units 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 artifacts that are not real peaks. This is one method by which false peaks can be identified.

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Figure B.2—Distribution of FWHM of spectral peaks as a function of energy

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h) Calculate the energy resolution with the following equation:

$$FWHM = 0.94 \times (N/Y) \quad \text{(B.3)}$$

where

N is the net peak area,

Y is the peak height.

An alternate method commonly used to measure the FWHM is to subtract the baseline channel-by-channel from the gross counts in the peak, determine the number of channels (to within ± 0.2 channels) above the half-height of the peak, and convert the number of channels to energy.

i) Document completion of this procedure in the instrument logbook with the FWHM energy resolution measured at 122.1 keV.

B.4.1.2 Resolution at 1332 keV

a) Establish the settings of the Ge spectrometer system so that the gain is approximately 0.35 keV/channel. This gain corresponds to placing the 1332 keV full-energy peak of ^{60}Co in channel 3800 (4096 channel analyzer).

b) Center the source on the detector axis at a distance from the detector to establish a counting rate of 500 s^{-1}. Locate the ^{60}Co source in the position used in the energy-resolution-measurement procedure for ^{57}Co.

c) Acquire a spectrum with about 3000 counts in the peak channel for the gamma ray at 1332.50 keV. If possible, adjust the fine gain so that there is an integer channel located within ± 0.1 channels of the mean channel position of the peak, and reacquire a spectrum.

d) Perform an energy calibration using the 1173.24 keV and 1332.50 keV gamma rays. If the ADC zero was previously set, the intercept term should be close to zero even at a different gain.

e) Determine the FWHM of the 1332.5 keV peak with one of the procedures described in B.4.1.1.

f) Document completion of this procedure in the instrument logbook with the FWHM energy resolution measured at 1332.5 keV.

B.4.2 Measurement of the peak-to-compton ratio for ^{60}Co

a) With the ^{60}Co spectrum accumulated in B.4.1.2, add the counts in the spectral region from 1040 keV to 1096 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 1332.5 keV peak.

b) Calculate the peak-to-compton ratio by dividing the 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 (1332.5 keV) at which it was measured.

B.4.3 Reference

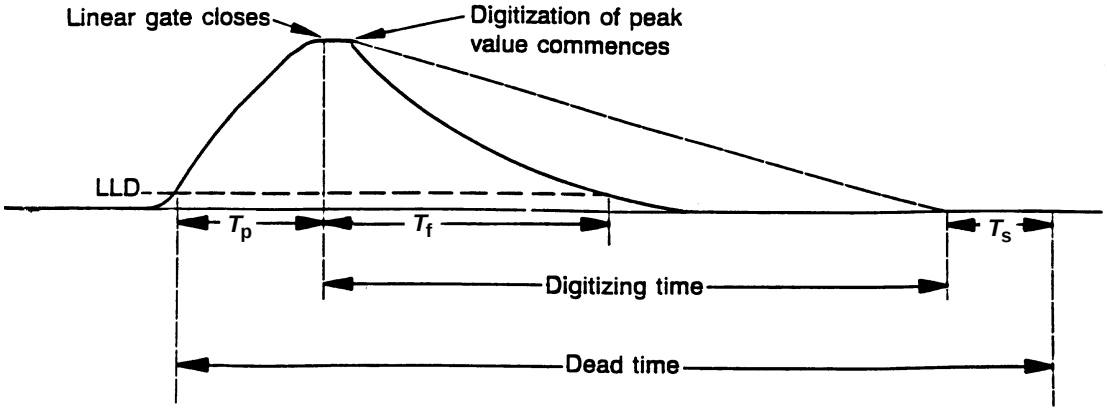
See Helmer [B22].

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B.5 Correction for losses due to counting rate

Rationale. Corrects for all counting-rate effects that can result in the loss of counts from the full-energy peaks, and informs 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 count that the pulse-height analysis system cannot correctly process a pulse. As shown in Figure B.3, the dead time as defined in the previous sentence includes the amplifier output pulse width (from the time it crosses the LLD) 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 the 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 ADCs provide a close approximation for this correction. The live-time clock is turned off whenever the LLD is exceeded by incoming pulses, and it remains off until the voltage level returns below the LLD 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 pileup losses (see 3.27 and 3.35).



NOTE—Unipolar amplifier output pulse showing the LLD setting; the time, T_p, to reach peak value; the time, T_f, from peak value until the pulse falls below the LLD; the digitizing time; the dead time; and the time, T_s, required to store an analyzed pulse.

Figure B.3—Specification of times for pulse processing by an ADC

Methods by which pulse-pileup corrections can be made are

a) Empirical (see Debertin [B9] and Helmer [B22]);
b) Live-time extension (see Bartosek and Masek [B2]);
c) Pulser (see Bolotin et al. [B3], Debertin and Schoetzig [B12], Hartwell and Goodwin [B19], Johnson et al. [B24], and Wiernik [B43]);
d) Virtual pulser and add “N” counts method (see Harms [B18], Johnson et al. [B24], and Nix et al. [B32]).

B.5.1 through B.5.4 discuss each of these techniques. (The method used in 7.5 is a variation of the empirical method and can be adopted to measure the first-order correction for pulse pileup by measuring the net peak-area counting rate of the fixed source as a function of the integral counting rate resulting from the moveable source.)

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In any pileup-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 that employs a fixed and a moveable source or, alternatively, could involve the technique employing a set of sources of accurately known intensity ratios (see B.5.1). 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.

B.5.1 Source method

This method empirically measures the amount of pulse pileup as functions of the gross counting rate and of the 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 50 000 s^{-1} or more should be prepared either by taking mass aliquots of a solution of the radionuclide or by procuring them from a reputable radionuclide supplier. The precision of the relative emission rates of these sources should be known to less than 0.5%. The integral counting rate of the Ge 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 50 000 s^{-1} and for an amplifier time constant of less than or equal to 2 µs, 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.

B.5.1.1 Procedure

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

   Recommendation. When operating a Ge spectrometer at high counting rates (i.e., greater than 5000 s^{-1}), it is best to choose an amplifier time constant that reduces the pulse pileup 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-detector distance so that comparable statistics are obtained (i.e., at least 20 000 counts, 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 pileup with this source is small (less than 1%). The "true" net peak counting rates for the other sources will be derived from this measurement and the known ratio of source aliquant masses.[17]

$$n_r(E) = M_1(E) f_i \qquad \text{(B.4)}$$

where at energy E

$n_r(E)$ are the "true" net peak counting rates 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),

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

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

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Select at least one low-energy gamma ray (approximately 100 keV), one medium-energy gamma ray (300– 800 keV), and one higher-energy gamma ray (greater than 1000 keV) to be analyzed.

CAUTION

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 first before use (and periodically thereafter) and to label all radioactive sources with their date, radiation field at contact, activity, and custodian. 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 these net counts from the ^{152}Eu net area. (The net area shall 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

$$C_r(E) = \frac{n_r(E)}{M_r(E)} \tag{B.5}$$

where

$C_r(E)$ is the correction factor,

$n_r(E)$ are the “true” net peak counting rates for source *i* with a gross counting rate, *r,*

$M_r(E)$ is the measured net peak counting rate of the weakest source ($i = 1$).

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 B.4. These corrections may either be 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. With the source method of counting-rate correction, the losses due to pulse pileup and ADC dead time are made separately.

B.5.2 Live-time extension method

In all modern ADCs, an internal linear gate shuts the input off at peak detect time, thus preventing pileup interference after peak detect time, T_p (see Figure B.3). However, during time T_p, pulse pileup may occur, which alters the pulse height and cause two pulses to be stored as one pulse within the spectrum. If one or both of these pulses would have fallen in a spectrum full-energy peak, it has now been removed, and the live-time correction cannot restore it. The resultant pulse stored in the spectrum is an artifact, and in some systems may be removed by pulse-pileup 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. It is important to realize the following about pileup rejecters:

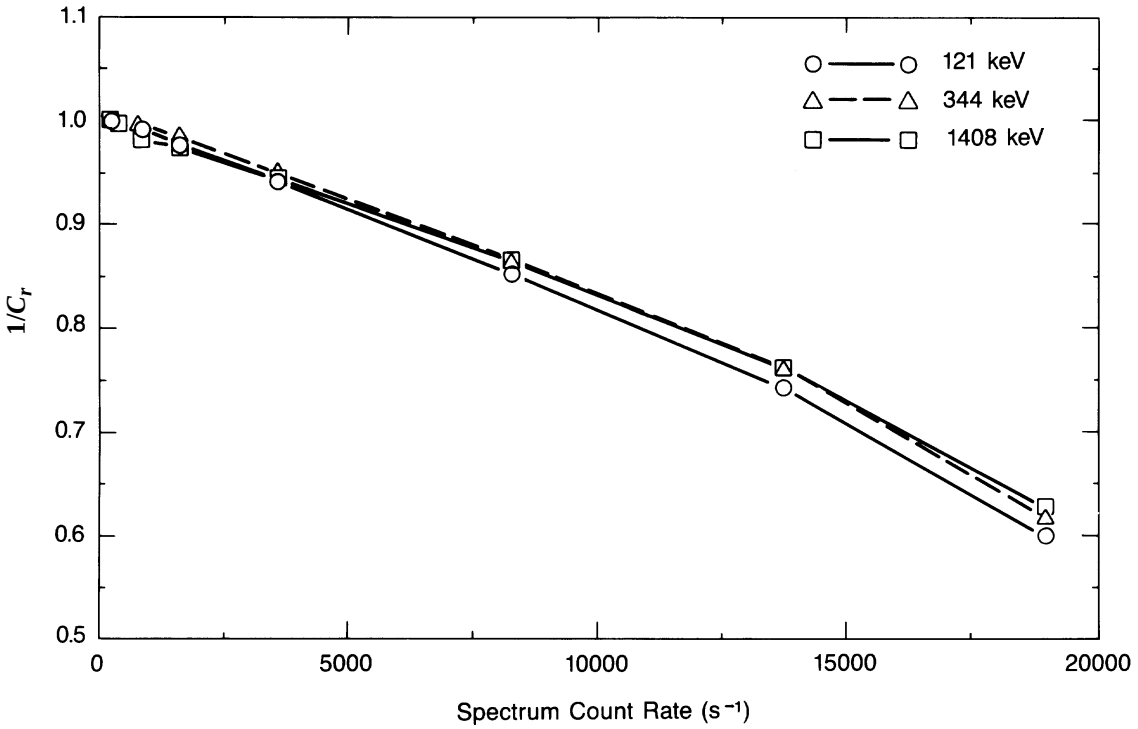
a) The fast channel also has a finite dead time during which it cannot detect a pulse pileup. Therefore, not all piled-up pulses are removed from the spectrum.

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b) The pileup rejecter itself does not correct the peak area for the pulse lost.

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



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

Figure B.4—Pulse-pileup correction as a function of integral counting rate

Andai and Jedlovsky [B1] describes a method for correcting for losses based on information from the pileup rejection circuitry. However, not all pileup pulses can be identified.

Pileup losses during time T_p have been compensated for in various ways by different investigators, usually by extending the counting time by gating off the live timer. 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 pileup losses are compensated. The ADC peak-detector circuitry itself inserts an energy-dependent pileup-loss term because T_p is longer for very small pulses than for large pulses. The intrinsic width of a gamma-ray peak increases as a function of energy for Ge detectors, thus contributing to the energy dependence of pileup losses. For example, an addition of approximately 1 mV to the pulse height, due to pileup, on a pulse destined for a spectrum peak whose width is 4 mV, probably results in that pulse being removed from the spectrum peak when the peak is fit by a Gaussian function to determine the net area, whereas the same 1 mV addition to the pulse height on a pulse destined for a spectrum peak whose width is 10 mV may not result in that pulse being removed from the net peak area. Thus, a straightforward extension of counting time to compensate for pileup losses is, at best, a first-order correction, though it does allow for accurate spectrometry at higher rates than a simple live-time correction. 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 time constant less than or equal to 2 μs. At counting rates high enough to be affected significantly by the fast-channel dead time, some have used an additional

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counting-time extension, which interpolates the error in the fast channel and forces the analyzer 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 50 000 s^{-1}.

WARNING

All of the live-time extension systems discussed in this clause depend upon no change occurring in the relative spectral energy content during the counting time. The corrections all assume a uniform correction for all peaks in the spectrum. The virtual pulse generator (see B.5.4) can be operated in the live-time extension mode without many of the limitations stated previously.

B.5.3 Pulser method

Another method of counting-loss correction involves the use of a precision pulser. One or more fixed-amplitude pulser 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 MCA dead time or pulse pileup. 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, but easily solvable, problems 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 B.5) injects an amount of charge equal to the applied voltage times the test capacitance. As this voltage pulse returns to zero value, it removes this same amount of charge from the preamplifier input. If the voltage pulse is a long-tailed pulse, a long-tailed undershoot occurs at the shaping amplifier output, which is a baseline disturbance and interferes 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 baseline following the gamma-ray pulses, but not following the pulser pulses. As shown in Figure B.5, the only shaped pulser pulse that does not upset the shaping amplifier baseline in the 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 is 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 that can be used to measure the “system” dead time (i.e., due to pulse pileup and analyzer pulse-processing time)

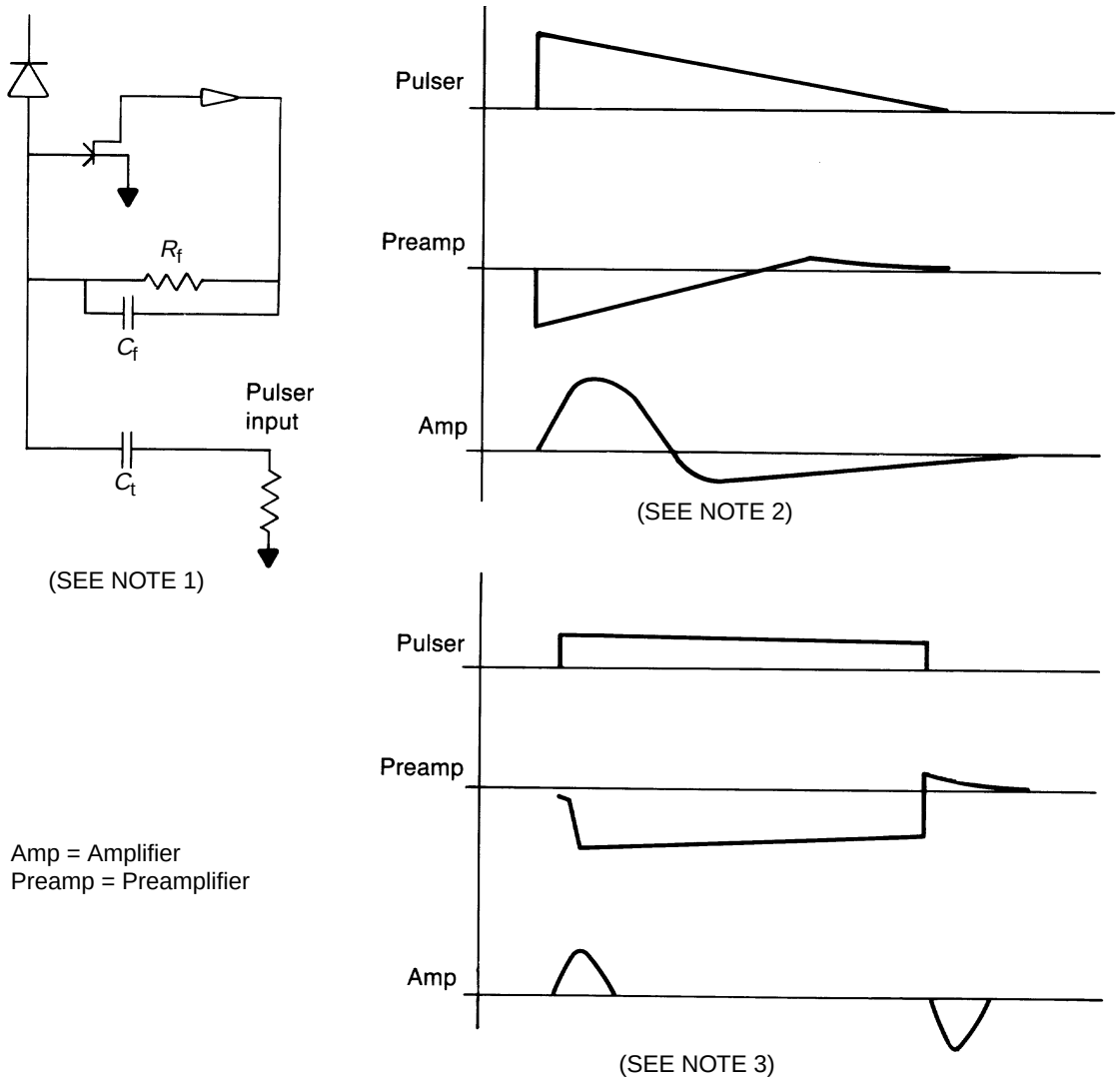
a) A pulse-height variable and periodic pulse generator set at a fixed pulse height;
b) A pulse-height variable pulse generator set at a fixed pulse height that injects pulses on demand (sometimes inappropriately referred to as a random pulse generator);
c) A single or dual pulse generator of type (a) or (b) that is tagged so that it can be routed to a separate memory location upon being 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 of using pulser peak areas to correct for counting losses out of varying-width spectral peaks have been developed. These methods allow for an energy-dependent correction for pileup losses, a special peak-fitting algorithm for the pulser peaks, identification of induced gain and/or zero shift due to large changes in the counting rate, and a redetermination of the gain parameters when a new steady-state gain condition has been established.

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NOTES

1—The portion of the preamplifier circuit showing where the pulser pulse is injected.

2—Pulse shapes as measured at the preamplifier and amplifier outputs resulting from the injection of a pulser pulse with a steep rise time and a long fall time.

3—Pulse shapes as measured at the preamplifier and amplifier outputs resulting from the injection of a pulser pulse with a square, flat-top pulse with sharp rise and fall times.

Figure B.5—Preamplifier and amplifier pulse shapes resulting from different pulser shapes

B.5.3.1 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 the 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 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

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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; and

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

Bolotin and Strauss [B3], Debertin and Schoetzig [B12], and Johnson et al. [B24] give details of this method. 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 time constant less than or equal to 2 μs, but it makes no provision for energy-dependent counting losses.

B.5.3.2 Pulser method 2

This method can be used to accurately correct for pulse pileup 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 pulser pulse into the 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 pulser pulses is compared to the number of these pulses that are recorded. Again, the differences in shape of the pulser and gamma-ray pulses are handled as described in B.5.3.1. Debertin and Schoetzig [B12] and Wiernik [B43] give details of this method. The counting rate limitations of this method are similar to those in B.5.3.1.

B.5.3.3 Pulser method 3

This method involves the injection of a low- and a high-voltage pulser pulse from a dual pulse generator, with subsequent removal of these pulser 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 pulser 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 pulser pulses are not masked by the gamma-ray spectral counts at high counting rates; this method also avoids interference of the pulser pulses with the gamma-ray peaks in the spectrum. This technique permits not only the correction for pulse pileup as a function of energy, but also the monitoring of the stability of the gain and zero. Hartwell and Goodwin [B19] and Johnson et al. [B24] give details of this method. 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 time constant of less than or equal to 2 μs, for the entire range of gamma-ray energies being detected.

B.5.4 Virtual pulser and add "*N*" counts method

A system that 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 Harms method (see Harms [B18]), *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 Westphal [B41] and Westphal [B42]), *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 pileup. 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 correct accurately for pulse pileup 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.

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B.6 Measurement of the full-energy peak efficiency curve

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. Subclauses B.6.1 through B.6.3 provide procedures for the following 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;
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 5.2.

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 analyzed. See 6.5.2 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 NIST or from a qualified supplier that has demonstrated its traceability by participation in a Measurements Assurance Program with NIST. Browne and Firestone [B5], ENSDF [B39], Gehrke et al. [B15], Kocher [B26], Kocher [B27], McNelles and Campbell [B30], Singh [B37], and Yoshizawa et al. [B44] report precise measurements of gamma-ray relative intensities and gamma-ray emission probabilities.

B.6.1 Measurement of standardization coefficients for specific radionuclides

a) Accumulate energy spectra for known live times using a radioactivity standard for each radionuclide of interest at the desired source-to-detector geometry. At least 20 000 net counts, 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, so that pulse-pileup corrections are not required. If the counting rate of either the standard or samples are greater than 1000 s^{-1}, and if the counting rates differ, correction for pulse pileup shall be applied (see B.5).
b) Calculate the standardization coefficient, $S_c(i, E)$, using a gamma ray of energy E with the following equation:

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

where

$n(i, E)$ is the net area counting rate (i.e., the baseline as defined in the text is removed) in a full-energy gamma-ray peak of energy E associated with radionuclide i,

$B(E)$ is the net area counting rate in the energy region E associated with a background peak,

C_r is the correction for pulse pileup (random summing) (see 6.5.1),

C_d is the correction due to decay during and prior to the count (see 6.6.1),

$A(i)$ is the activity (in Bq) of the calibration source radionuclide i.

c) Prepare a table listing the standardization coefficients for each of the radionuclides for which an analysis has been performed. Note that there can be a standardization coefficient for each gamma ray of each radionuclide.

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When an unknown source of radionuclide *i* is measured, its activity is calculated as

$$A(i) = [n(i, E) - B(E)] \times C_r \times C_d / S_c(i, E) \quad \text{(B.7)}$$

B.6.2 Measurement of the detector efficiency versus energy for large sample-to-detector distances

At large sample-to-detector distances (typically, a source-to-detector distance that is greater than or equal to 10 cm), the error in the replication of the sample-detector geometry, and in the correction for coincidence summing of gamma-ray pulses in the detector, can usually be neglected for most measurements. For these reasons, measurements at large sample-to-detector distances are preferred over measurements close to the detector (i.e., within 5 cm of the detector window), provided the source activity is high enough to achieve an acceptable statistical precision within the allotted counting time.

Standards prepared from a mix of ^{109}Cd, ^{57}Co, ^{139}Ce, ^{203}Hg, ^{113}Sn, ^{85}Sr, ^{137}Cs, ^{88}Y, and ^{60}Co (^{241}Am may be included in this mix); from the long half-life mix of ^{125}Sb, ^{154}Eu, and ^{155}Eu; or from ^{152}Eu are the most popular standards in use. A spectrum of the first mixed radionuclide standard mentioned in the previous sentence is given in Figure B.6. Other standardized radionuclides may also be used.

CAUTION

Annihilation radiation (at 511 keV) is often present in spectra due to positron decay, or pair production from the radionuclide sources or from the background, and may interfere with the measurement of the 514 keV gamma ray of ^{85}Sr.

a) Accumulate, for a known live time, a spectrum using one or more standards so that at least 20 000 counts, and preferably 50 000 counts, have been accumulated in the full-energy peaks of interest (only peaks whose peak-to-baseline 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 analyzed.

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

c) Calculate the full-energy peak efficiency, ε, using the following equation:

$$\varepsilon(E) = [n(E) - B(E)] \times \Pi C_i(E) / R(E) \quad \text{(B.8)}$$

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,

$B(E)$ is the net area counting rate in a corresponding peak associated with the background spectrum,

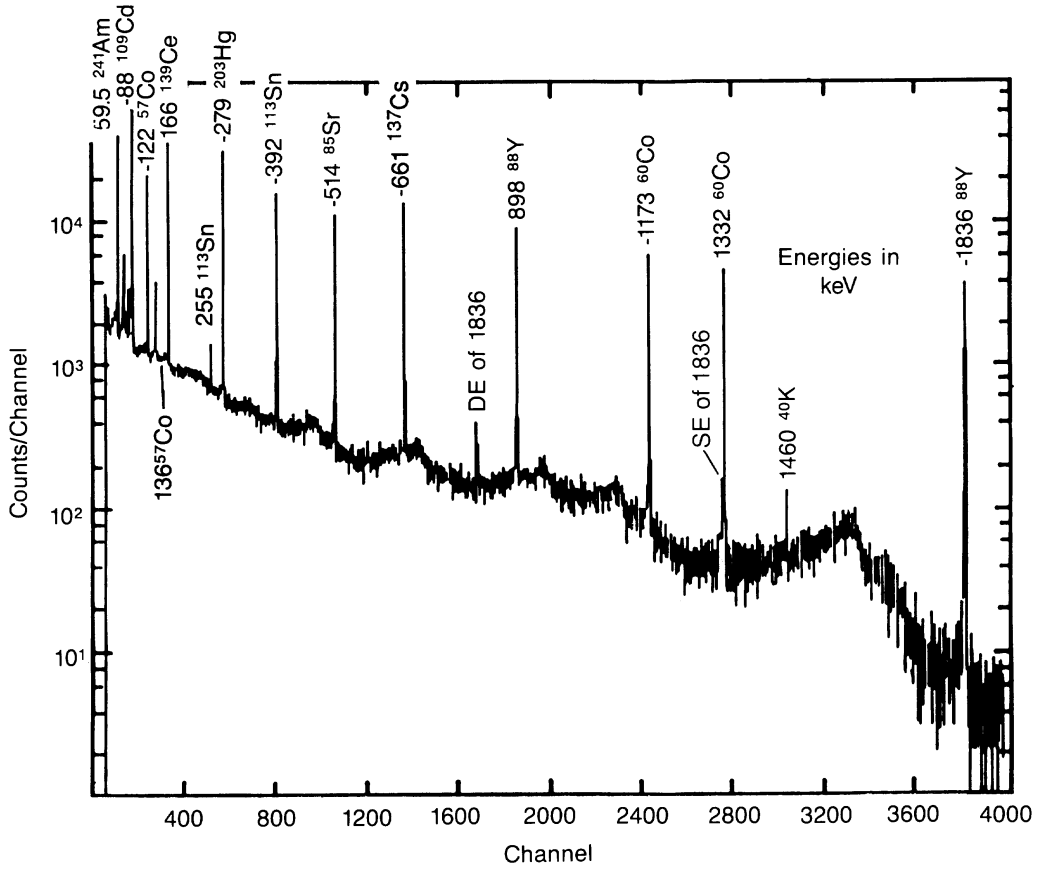
$R(E)$ is the emission rate for gamma ray with the associated energy E,

$C_i(E)$ are the correction factors for a radionuclide with the associated energy E (where i = a, c, d, and r, as defined in the following paragraph).

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

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NOTE–Spectrum of the mixed radionuclide standard containing ^{241}Am, ^{109}Cd, ^{57}Co, ^{139}Ce, ^{203}Hg, ^{113}Sn, ^{85}Sr, ^{137}Cs, ^{88}Y, and ^{60}Co. SE and DE are the abbreviations for single and double escape peaks, respectively. These peaks are caused by pair production occurring from high-energy gamma rays in the active volume of the detector.

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Figure B.6—Gamma-ray spectrum of a mixed radionuclide standard

d) After the efficiencies below 2 MeV have been measured, a plot of the efficiency as a function of energy shall be made (see 5.2.3). Efficiency values above 2 MeV may be measured with nonquantitative sources of the radionuclides ^{24}Na (1368, 2754), ^{144}Ce-^{144}Pr (696, 2185) and/or ^{228}Th (583, 2614) by following steps (e) through (h). Relative gamma-ray emission probabilities for these radionuclides can be found in Debertin and Helmer [B10]. 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 counts, 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) through (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 probabilities shown in the following equation:

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$$R(E_h) = R(E_L) \times P(E_h)/P(E_L) \quad \text{(B.9)}$$

where

$R(E_h)$ is the emission rate of the higher-energy gamma ray,

$R(E_L)$ is the emission rate of the lower-energy gamma ray,

$P(E_h)$ is the probability of the higher-energy gamma ray,

$P(E_L)$ is the probability of the lower-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 5.2.3. From a set of reasonably spaced efficiency values taken from this plot, either develop a full-energy peak efficiency look-up table as a function of energy, or determine an equation or equations 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., less than 5%), it is recommended that supplementary measurements be made to define the efficiency curve better.

B.6.2.1 Reference

See Debertin and Helmer [B10].

B.6.3 Measurement of the detector efficiency versus energy for small sample-to-detector distances

Effluent and environmental samples typically require small sample-to-detector distances and long counting times. These experimental conditions can result in large uncertainties and/or errors due to proportionally large background contributions, irreproducibility of the sample-to-detector distance, and large coincidence summing effects resulting from the detection of two or more coincident gamma rays from the same nucleus. For example, an uncertainty of ± 1.0 mm in the source-to-detector distance reproducibility at 5 cm results in an additional approximate 4% uncertainty in the net peak area for a point source and a 5 cm diameter Ge detector. A similar size or larger error can result from the coincidence summing of gamma rays at a 5 cm source-to-detector distance for a point source of some radionuclides.

Thus, whenever possible, a sample should be at least 5 cm from the detector, and only those peaks whose corrections for coincidence summing are small should be analyzed. However, when small source-to-detector distances are required, the magnitude of these uncertainties and/or errors should be assessed (see 6.6.5), and whenever possible, coincidence summing corrections applied. Large sample containers have a large mean source-to-detector distance even when they are in close proximity to the detector. Large samples reduce, but do not eliminate the coincidence summing effect. Coincidence summing of gamma rays in cascade may cause large errors in the measured gamma-ray emission rates at small sample-detector distances if coincidence-summing corrections are not applied. Radionuclides, whose associated gamma rays are essentially free of coincidence summing, can be used to measure the full-energy peak efficiency. For example, standards prepared from a mix of the 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 coincidence, assuming that energies at or below the K X rays of ^{139}Ce are not detected. The radionuclides ^{54}Mn and ^{65}Zn should be used to replace ^{88}Y and ^{60}Co, respectively (see radionuclides used in B.6.2), because the latter two radionuclides each emit gamma rays

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that are in coincidence. To measure the full-energy peak efficiency above 2 MeV, the radionuclide ^{144}Ce-^{144}Pr (696, 2185) may be used, as described in B.6.2, steps (d) through (h).

Follow the procedure described in B.6.2, using only the radionuclides listed in the previous paragraph or other radionuclides that have been demonstrated to be free from coincidence summing.

WARNINGS

1—Some of the radionuclides recommended 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 by the detector crystal. 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.

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

The reentrant or Marinelli beaker 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. Regardless of the size of the Marinelli beaker and the detector efficiency, coincidence-summing corrections are required for nuclides with cascading gamma lines.

B.6.3.1 Reference

See Debertin and Helmer [B10].

B.7 Preparation of working standards from standard solutions

Rationale. Prepares working standards using reliable and tested procedures. Sometimes standards are not available as an off-the-shelf product in the geometry or matrix required. Under these conditions, it is necessary either to purchase custom-prepared sources or to prepare them at the laboratory from solution standards that are documented to be traceable to NIST. 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 (see Mann and Rytz [B29] and NCRP Report 58-1985 [B31]). Whenever possible, at least three standard samples should be prepared and cross-checked.

CAUTION

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 quantitative subsequent 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 need to be taken when handling all radioactive materials once their containers have been opened. Resealing of unused liquid standards is best accomplished by flame sealing in glass ampoules so that the ampoules are at least

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B.7.1 Preparation of solution standards

a) Obtain a solution standard volume of at least 5 mL 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 decimal-place balance, at least 0.5 g should be transferred to avoid unacceptably large errors). A five or six decimal-place balance is preferred since smaller aliquots can be accurately measured.

d) Obtain the tare mass of a small, flat-bottom, plastic container from which small aliquots can be easily dispensed without loss due to splattering or 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 (5–20 mL), with the top heated and drawn out to a capillary, 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 of 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 holds the custom standard. (Be sure that the container is identical in dimension to the containers to be used for the samples to be analyzed.)

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

h) Transfer to this container an amount of solution standard that results in a sample that is neither too high or too low in activity for the counting geometry to be used (a counting rate of less than 1000 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 from step (f).

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

CAUTION

Residue or debris in a liquid sample can cause a partitioning of the radioactivity. Therefore, if such debris is present in the sample, 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.

B.7.2 Preparation of soil standards

The following procedure is for the preparation of soil standards for use in gamma-ray spectrometry (i.e., relatively high mass standards—greater than 10 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.

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a) Measure into a plastic polyethylene bag with a closure that is zip locked 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 results in a sample that is neither too high nor low in activity for the counting geometry to be used. Sufficient mass of solution should be transferred directly to the soil to minimize the possibility of a nonhomogeneous 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 B.7.1. Determine the activity concentration of the diluted standard from the masses of the transferred standard solution and the transferred carrier solution.

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

d) Let the sealed plastic bag sit overnight.

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

f) If the soil is essentially dry, 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 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 lock closure, seal it, and break up the clods in the soil by applying finger pressure through the plastic bag until the particle size is consistent.

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 Ge spectrometer to be sure that no appreciable amount of radioactivity remains 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.

B.7.3 Preparation of filter standards

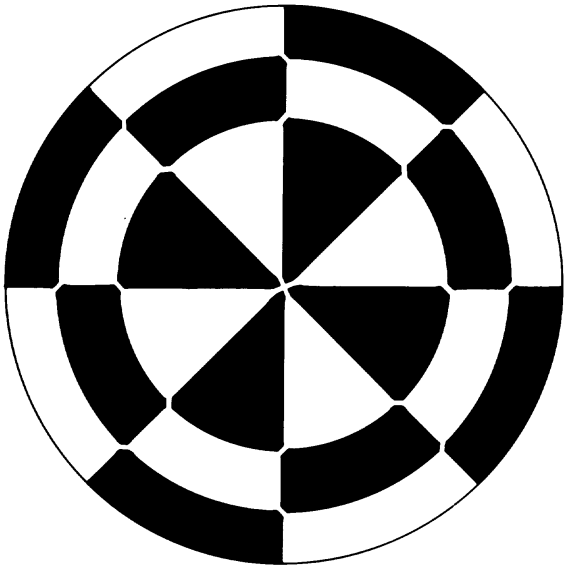
a) Prepare a pattern, using paper- or glass-fiber filter paper of the same diameter as the samples to be calibrated, by dividing the circular filter into four to eight pie-shaped sectors of equal area. Then divide it into three or more rings of equal area so that the filter is divided into 12 to 24 equally sized areas (see Figure B.7).

b) Draw the pattern on a translucent sheet of paper or plastic film, as used in mechanical drawings. The pattern should show through when a second sheet of adhesive-coated plastic film or wide polyester tape is placed adhesive side up over the pattern. A light box may be helpful to see the pattern through the second sheet of plastic.

c) Cut half of the filter containing the pattern into its individual equal area pieces. Carefully trim the corners of each piece so that neither the tips nor the sides of the individual pieces touch other pieces as a mosaic is constructed, with the filter material forming a full circle but covering only half of the pattern, with no sides or corners touching.

d) Place each individual piece onto the adhesive side of the tape or adhesive-coated plastic, as shown in Figure B.7.

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NOTE—The individual sections of filter shown in black are equal areas and do not touch each other. The standard is formed by placing the individual sections and sectors cut out of the filter paper onto the adhesive side of a translucent film through which the pattern can be seen so as to form a mosaic. An equal deposit of standard solution is placed on each piece of paper (shown in black) forming the mosaic. This allows the preparation of a pseudo-uniformly deposited source with no significant migration of the activity.

Figure B.7—Mosaic pattern of an air-filter standard

e) Place equally sized amounts of a standard solution onto each piece of the filter (shown in black in Figure B.7) making up a mosaic, using the mass aliquot technique described in B.7.1, so that the total mass of the deposited solution is accurately known.

f) Allow the deposited solution to air-dry overnight.

g) Cover the dried filter mosaic with a piece or pieces of clear polyester tape so that the radioactivity is totally contained. Trim off the excess backing, being careful not to remove any portion of the radioactivity from the filter standard.

h) Check the filter for leakage of radioactivity. Homogeneity in the filter-paper standard could be demonstrated during construction by not fastening the individual mosaic pieces of radioactive filter paper to the adhesive-backed plastic film until the activities in the individual pieces have dried and have been compared by counting them on a Ge spectrometer.

NOTE—Other less cumbersome methods have been used successfully by experienced laboratory chemists by making individual deposits on filters that do not wick. However, the method in this subclause is recommended for the less experienced radiochemistry laboratory worker.

B.7.4 Reference

See NCRP Report 58-1985 [B31].

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Annex C

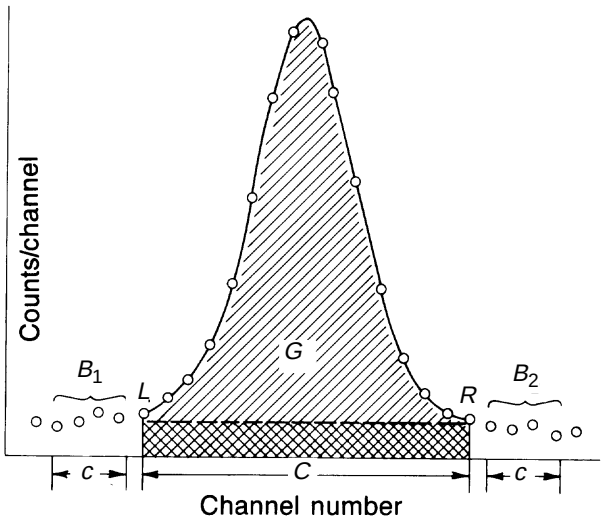
(informative)

Measurement of peak position, net area, and their uncertainties

Both nonfitting 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 baseline 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-pileup correction (see 8.4). Methods that use all of the spectral peak, including low- and high-energy tails, tends to have less energy dependence in the pileup correction.

C.1 Nonfitting technique

A typical well-resolved spectral peak is illustrated in Figure C.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., greater than or equal to 99.7% of the area).



NOTE—The gross count, *G*, under the peak includes all counts in channels *C*.

Figure C.1—Well-resolved peak with continuum baseline

The net area of the peak, *N*, is then obtained from

$$N = [G - C\,(B_1 + B_2)/2c] \quad \text{(C.1)}$$

where

- G are 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 baseline below the peak,
- B_2 is the sum over c channels of baseline above the peak.

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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 standard deviation associated with the net area N is given by

$$s(N) = \sqrt{G + \left(\frac{C}{2c}\right)^2 (B_1 + B_2)} \tag{C.2}$$

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 baseline on each side of the peak. The peak position may then be determined from the first moment, x_0, given by

$$x_0 = \frac{1}{N} \sum_{i=L}^{R} iN_i \tag{C.3}$$

where

L is the left boundary of the peak,
R is the right boundary of the peak,
N are the net counts in the peak area,
N_i are the net counts in channel i after the background has been removed.

The standard deviation associated with the first moment, if N and $\Sigma(iN_i)$ were independent, is given by

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

where

s_N is the standard deviation associated with N,
s_{N_i} is the standard deviation associated with N_i.

C.2 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 is made to describe a function that fits the tails of the full-energy peak. A Gaussian function is described by

$$Y(x) = Y_0 \exp\left[-(x - x_0)^2 / (2f^2)\right] \tag{C.5}$$

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,
f is the width of the Gaussian (FWHM = $2.355f$).

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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 f are in the exponent, a nonlinear fit is required. The net area of a Gaussian is given by

$$(Y_0)\left[\frac{FWHM}{0.94}\right] = \text{net area} \tag{C.6}$$

A least-squares fit is used to determine the values of the variable parameters, p_j, where $p_j = Y_0$, x_0, and f, respectively, that minimize the sum of the squares of the deviations, R^2, of a set of experimental data points, y_i, from the estimates $y_i(p_j)$ for the desired functional form; that is,

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

is minimized, where 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, $p_j = p_j^0 + dp_j$, then

$$y_i(p_j) \approx y_i(p_j^0) + \sum\left[\frac{dy_i(p_j)}{dp_j}\right]_0 (dp_j) \tag{C.8}$$

For this truncated series, the conditions for R^2 to be a minimum, as a function of the dp_j, are that the set of equations

$$\frac{dR^2}{d(dp_j)} = 0 \tag{C.9}$$

all hold simultaneously. The solution of this set of equations gives the values dp_j. Since the higher-order derivatives have been neglected, this process does not give the true minimum of Equation (C.7). Therefore, Equation (C.8) and Equation (C.9) are evaluated and solved again with the p_j^0 replaced by $(p_j^0 + dp_j)$. This process is continued until all the dp_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.

C.3 References

See Cline et al. [B6], Debertin and Helmer [B10], Helmer [B22], and Parker [B35].

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Annex D

(informative)

Equations for the correction of coincidence gamma-ray summing

D.1 Equations for coincidence-summing correction factors

Coincidence summing 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 lifetimes of intermediate levels are short relative to the integration time of the amplifier. It results in the removal of counts from the channels at which the individual digitized peaks would occur, and places these 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 is placed in a channel whose energy equivalent is equal to the sum of the individual cascade gamma rays. 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-detector distances is usually negligible, and therefore has been neglected in this treatment.

Although the beta rays 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., less than 500 keV), but is a limiting feature of the coincidence correction factors presented in this clause.

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

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

where

$p_{K,i}$ is the K X-ray emission probability 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*,

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

For decay schemes with several transitions that decay by K conversion, the total K X-ray emission probability is equal to the sum of the individual K X-ray emission probabilities of the transitions.

As an example, the correction factors in D.1.1 and D.1.2 are derived to demonstrate the complexity of these calculations and to aid those who cannot avoid the experimental conditions requiring coincidence-summing corrections.

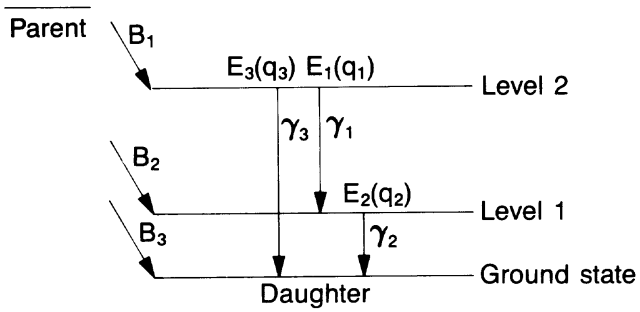
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D.1.1 Summing correction factors for a simple decay scheme

The decay scheme in Figure D.1 consists of transitions 1, 2, and 3. Transition 1 depopulates the second level and feeds the first, transition 2 depopulates the first level and feeds the ground state, and transition 3 depopulates the second 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 of the transitions depopulate by both gamma-ray emission and by internal conversion. The gamma-ray emission probability, p_i, is related to the transition probability by the following equation:

$$q_i = p_i[1 + \alpha_t(i)] \quad \text{(D.2)}$$



NOTE—A simple decay scheme with transitions 1 and 2 in coincidence, and transition 3 crossing over transitions 1 and 2.

Figure D.1—A three-transition decay scheme

For simplicity, assume that the Ge 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.

For the decay scheme given in Figure D.1, the probabilities, P_1 and P_2, of not observing a count in the net peak area associated with transitions 1 and 2, respectively, due to a true coincidence with the other gamma ray, is given by

$$P_1 = (f_2)\varepsilon_t(2) \quad \text{(D.3)}$$

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.

and

$$P_2 = (f_1)\varepsilon_t(1) \quad \text{(D.4)}$$

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.

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If none of transition 2 is internally converted (the total internal conversion coefficient is equal to zero), all of 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

$$f_2 = p_2/q_2 = 1/[1 + \alpha_t(2)]) \quad \text{(D.5)}$$

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 in Equation (D.4). Because of the beta feeding of the first excited state, only a fraction of the 2 transitions originate from the depopulation of level 2 (i.e., q_1/q_2). Moreover, only a fraction, f_1, of these transitions coming from level 2 are gamma rays. Therefore,

$$f_1 = 1/[1 + \alpha_t(1)]\ [(q_1/q_2)] \quad \text{(D.6)}$$

Simplifying,

$$f_1 = (p_1/p_2)[1/[1 + \alpha_t(2)]] \quad \text{(D.7)}$$

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 coincidence with this transition. The probability that gamma ray 1 and gamma ray 2 both leave all of their energy in the active volume of the detector is

$$P_3 = (f_3)[\varepsilon(1)][\varepsilon(2)] \quad \text{(D.8)}$$

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,

$\varepsilon(2)$ 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.

$$f_3 = (p_1)(p_2)/q_2 \quad \text{(D.9)}$$

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

$$C_c(1) = 1/(1 - P_1) \quad \text{(D.10)}$$

and

$$C_c(2) = 1/(1 - P_2) \quad \text{(D.11)}$$

To determine the 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.

$$P_g\,(3) = (p_3)[\varepsilon(3)] \quad \text{(D.12)}$$

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where

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

Hence, the coincidence-summing correction factor for the crossover gamma ray 3, $C_c(3)$, is

$$C_c(3) = \frac{1}{[P_g(3) + P_3]/[P_g(3)]} \quad \text{(D.13)}$$

The addition sign in the denominator of Equation (D.13) is due to the fact that a fraction of the observed counts are due to coincidence summing into the peak and need to be removed.

D.1.2 Correction factor for the 591 keV gamma ray emitted in the decay of ^{154}Eu

As seen from Figure D.2, the 591 keV gamma ray is not a crossover transition. Therefore, no counts can 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, 1005 keV, 123 keV branch;
b) The 591 keV, 756 keV, 248 keV, 123 keV branch.

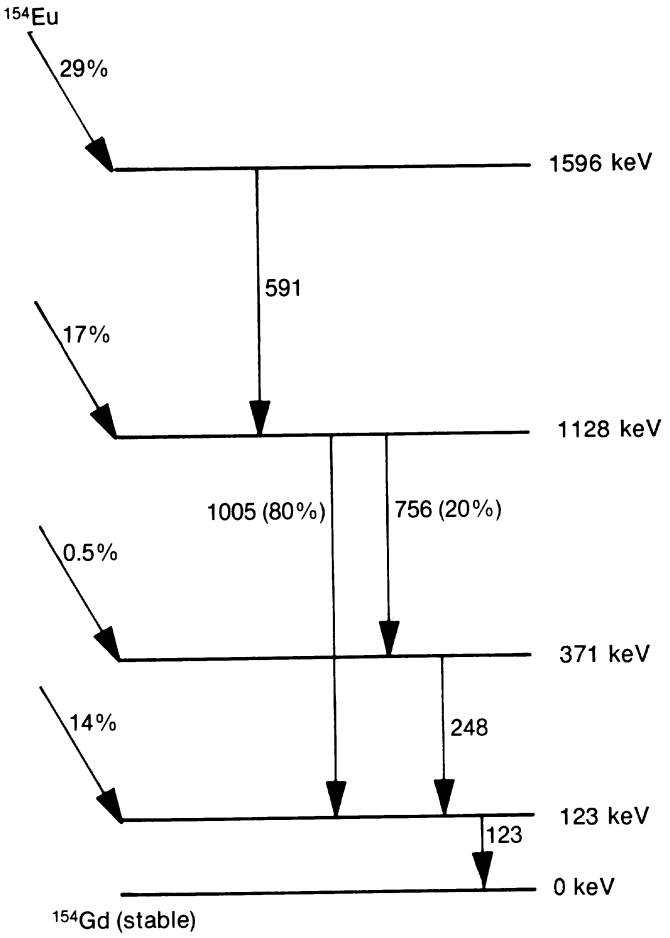


Figure D.2—Partial decay scheme of ^{154}Eu

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The coincidence-summing probability for each branch is calculated separately and combined for the total correction factor. Since the K X rays (approximately 45 keV) resulting from the internal conversion process are detected by many Ge spectrometers and are in true coincidence with the 591 keV gamma ray, these X rays are included in the correction for coincidence summing out of the 591 keV full-energy peak. In this example, only terms linear in ε_t are considered. This simplification is appropriate only if the total efficiency, ε_t, is less than 25%.

D.1.2.1 Correction factor for the 591 keV, 1005 keV, 123 keV branch

The probability of not observing a count in the 591 keV full-energy peak due to coincidence summing in this first branch, $P_1(591)$, is equal to the sum of the probabilities of losing a count from the 591 keV full-energy peak by it summing with a prompt count from one of the other gamma rays or a K X ray in this branch of the cascade (i.e., a count, in or below the full-energy peak, associated with a 1005 keV or a 123 keV gamma ray or with the K X rays from internal conversion of the 123 keV transition).

$$P_1(591) = (f_{1005})\varepsilon_t(1005) + (f_{1005})(f_{123})\varepsilon_t(123) + (f_{1005})[f_{(\text{K X ray})}]\varepsilon_t(\text{K X ray}) \quad \text{(D.14)}$$

where

f_{1005} is the fraction of 1005 keV gamma rays that depopulate the ^{154}Gd 1128 keV level,

f_{123} is the fraction of 123 keV gamma rays that depopulate the ^{154}Gd 123 keV level,

$f_{(\text{K X ray})}$ is the fraction of K X rays resulting from depopulation of the 123 keV level,

$\varepsilon_t(i)$ is the total efficiency of the ith gamma ray or the Gd K X ray.

For the 1128 keV level, 79.7% of the depopulating transitions are due to the emission of a 1005 keV gamma ray. Since the total internal conversion of this transition is very small, it can be neglected.

$$f_{1005} = 0.797 \quad \text{(D.15)}$$

For the 123 keV level, all of the depopulating transitions are due to the emission of a 123 keV transition, but not all 123 keV transitions are in the form of a gamma ray. Some of these transitions depopulate the 123 keV level by the internal electron conversion process, i.e., the excited nucleus gives up its energy to one of the orbiting electrons that escapes its orbit, leaving a vacancy. This vacancy is filled by an electron in one of the outer shells. When this occurs, the excess energy of the electron filling the vacated shell is released either by the emission of a characteristic X ray or by the emission of Auger electrons. Therefore,

$$f_{123} = 1/[1 + \alpha_t(123)]) \quad \text{(D.16)}$$

where

$\alpha_t(123)$ is the total conversion coefficient of the 123 keV transition.

The fraction of transitions that depopulate the 123 keV level by K internal conversion that result in the emission of a Gd K X ray is given by

$$f_{(\text{K X ray})} = (\omega_K)[\alpha_K(123)]/[1 + \alpha_t(123)] \quad \text{(D.17)}$$

where

ω_K is the K fluorescence yield for Gd,

$\alpha_K(123)$ is the K conversion coefficient of the 123 keV transition.

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Substituting the values of f from Equation (D.15), Equation (D.16), and Equation (D.17) into Equation (D.14) gives the probability of not observing a count in the 591 keV full-energy peak due to coincidence summing in this first branch.

$$P_1(591) = (0.797)\varepsilon_t(1005) + (0.797)\{1/[1 + \alpha_t(123)]\}\varepsilon_t(123) + (0.797)(\omega_K)[\alpha_K(123)]\{1/[1 + \alpha_t(123)]\}[\varepsilon_t(\text{K X ray})] \quad \text{(D.18)}$$

D.1.2.2 Correction factor for the 591 keV, 756 keV, 248 keV, 123 keV branch

The probability of not observing a count in the 591 keV full-energy peak due to coincidence summing in the second branch, $P_2(591)$, is equal to the sum of the probabilities of losing a count from the 591 keV full-energy peak by summing it with a prompt count from one of the other gamma rays or a K X ray in this branch of the cascade (i.e., a count, in or below the full-energy peak, associated with a 756 keV, 248 keV, or 123 keV gamma ray, or with the K X rays from internal conversion of the 248 keV or 123 keV transitions).

$$P_2(591) = (f_{756})\varepsilon_t(756) + (f_{756})(f_{248})\varepsilon_t(248) + (f_{756})(1.00)(f_{123})\varepsilon_t(123) + (f_{756})(f_{1\text{K X ray}})\,\varepsilon_t(\text{K X ray}) + (f_{756})(1.00)(f_{2\text{K X ray}})\varepsilon_t(\text{K X ray}) \quad \text{(D.19)}$$

where

f_{756} is the fraction of 756 keV gamma rays that depopulate the ^{154}Gd 1128 keV level,

f_{248} is the fraction of 248 keV gamma rays that depopulate the 371 keV level,

f_{123} is the fraction of 123 keV gamma rays that depopulate the ^{154}Gd 123 keV level,

$f_{1\text{K X ray}}$ is the fraction of K X rays that are emitted in the depopulation of the 371 keV level due to the K conversion process of the 248 keV transition,

$f_{2\text{K X ray}}$ is the fraction of K X rays that are emitted in the depopulation of the 123 keV level due to the K conversion process of the 123 keV transition,

$\varepsilon_t(i)$ is the total efficiency of the ith gamma ray or the Gd K X ray.

The use of (1.00) in the third and in the last terms of Equation (D.19) results from the fact that all depopulation of the 371 keV and 123 keV levels passes through the 248 keV and 123 keV transitions, respectively.

For the 1128 keV level, 20.2% of the depopulating transitions are due to the emission of a 756 keV gamma ray. This transition is only weakly converted (assume the internal conversion to be negligible). Hence,

$$f_{756} = 0.202 \quad \text{(D.20)}$$

For the 123 keV and 371 keV levels, all of the depopulating transitions are due to the emission of a 123 keV or 248 keV transition, respectively, but not all of the 248 keV transitions are in the form of a gamma ray. As with the 123 keV transition, some of these transitions depopulate their levels by the internal electron process.

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$$f_{248} = 1/[1 + \alpha_t(248)] \quad \text{(D.21)}$$

$$f_{123} = 1/[1 + \alpha_t(123)] \quad \text{(D.22)}$$

where

$\alpha_t(248)$ is the total conversion coefficient of the 248 keV transition,

$\alpha_t(123)$ is the total conversion coefficient of the 123 keV transition.

The fraction of transitions that depopulate the 123 keV and 248 keV levels by K internal conversion that result in the emission of a Gd K X ray are, respectively, given by

$$f_{2K\ X\ ray} = (\omega_K)[\alpha_K(123)]\{1/[1 + \alpha_t(123)]\} \quad \text{(D.23)}$$

and

$$f_{1K\ X\ ray} = (\omega_K)\{\alpha_K(248)\}\{1/[1 + \alpha_t(248)]\} \quad \text{(D.24)}$$

where

ω_K is the K fluorescence yield for Gd,

$\alpha_K(123)$ is the K conversion coefficient of the 123 keV transition,

$\alpha_K(248)$ is the K conversion coefficient of the 248 keV transition.

Substituting the values of *f* from Equations (D.20) through (D.24) into Equation (D.19) gives the probability of not observing a count in the 591 keV full-energy peak due to coincidence summing in this second branch.

$$\begin{aligned} P_2(591) = {} & (0.202)\varepsilon_t(756) + (0.202)\{1/[1 + \alpha_t(248)]\}\varepsilon_t(248) + \\ & (0.202)(1.00)\{1/[1 + \alpha_t(123)]\}\varepsilon_t(123) + \\ & (0.202)(\omega_K)[\alpha_K(248)]\{1/[1 + \alpha_t(248)]\}\varepsilon_t(\text{K X ray}) + \\ & (0.202)(1.00)(\omega_K)[\alpha_K(123)]\{1/[1 + \alpha_t(123)]\}\varepsilon_t(\text{K X ray}) \end{aligned} \quad \text{(D.25)}$$

The total probability, $P(591)$, of not observing a count in the 591 keV gamma-ray peak due to coincidence summing is given by

$$P(591) = P_1(591) + P_2(591) \quad \text{(D.26)}$$

From Equation (D.18) and Equation (D.25), the probability of not detecting a gamma ray in the 591 keV peak due to coincidence summing is

$$\begin{aligned} P(591) = {} & (0.797)\varepsilon_t(1005) + (0.797)\{1/[1 + \alpha_t(123)]\}\varepsilon_t(123) + \\ & (0.797)(\omega_K)[\alpha_K(123)]\{1/[1 + \alpha_t(123)]\}\varepsilon_t(\text{K X ray}) + (0.202)\varepsilon_t(756) + \\ & (0.202)\{1/[1 + \alpha_t(248)]\}\varepsilon_t(248) + (0.202)(1.00)\{1/[1 + \alpha_t(123)]\}\varepsilon_t(123) + \\ & (0.202)(\omega_K)[\alpha_K(248)]\{1/[1 + \alpha_t(248)]\}\varepsilon_t(\text{K X ray}) + \\ & (0.202)(1.00)(\omega_K)[\alpha_K(123)]\{1/[1 + \alpha_t(123)]\}\varepsilon_t(\text{K X ray}) \end{aligned} \quad \text{(D.27)}$$

Substituting numeric values into Equation (D.27) [$\omega_K = 0.934$, $\alpha_K(123) = 0.662$, $\alpha_K(248) = 0.0814$, $\alpha_t(123) = 1.20$, and $\alpha_t(248) = 0.11$] yields

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$$P(591) = (0.797)\varepsilon_t(1005) + (0.202)\varepsilon_t(756) + (0.454)\varepsilon_t(123) + (0.182)\varepsilon_t(248) + (0.294)\varepsilon_t(\text{K X ray}) \quad \text{(D.28)}$$

Finally, the multiplicative coincidence-summing correction factor, C_c, for the 591 keV gamma ray is given by

$$C_c = 1/[1 - P(591)] \quad \text{(D.29)}$$

The corrections for gamma rays in other cascades or emitted from other radionuclides are calculated in the same way. Obviously, it is necessary to know the decay scheme, including the relative depopulation of each of the excited states, the internal conversion coefficients, and the total and full-energy peak efficiencies of the detector as a function of energy if the correction factors are to be accurate.

D.1.3 General Case

For the general case with j gamma rays in cascade, the fractional loss to first order is given by

$$C_c = \frac{1}{\left[1 - \sum_{i=1}^{j} f_i \varepsilon_t(i)\right]} \quad \text{(D.30)}$$

where

f_i is the fraction of coincidence photons of energy i in coincidence with the gamma ray of interest,
$\varepsilon_t(i)$ is the total efficiency of the coincidence photon of energy i.

D.1.4 References

See Debertin and Schoetzig [B11] and Schima and Hoppes [B36].

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Annex E

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Construction of shields for Ge spectrometers

E.1 Construction materials

It is recommended that the following good practices be observed in shield construction regardless of the activity level of the samples being counted. The shielding for detectors used in a radioanalytical laboratory to count samples should be constructed from low-activity lead or steel that is free from the natural thorium and uranium decay chains as well as ^{40}K, ^{60}Co, and ^{137}Cs. Only the Ge crystal housing should be located inside the shield, with the preamplifier located outside the shield. This is most easily accomplished with horizontal cryostats, especially if the detector comes with an offset-port dewar. Structural materials used in the detector housing and the cold finger should be of high-purity steel, copper, magnesium, or specially fabricated, radiation-free aluminum. The preamplifier and molecular sieve should be made from materials low in radioactivity and either located outside the shield or well shielded from the detector crystal.

Use of carbon steel as a structural material inside the shield without verification that it is free of ^{60}Co should be avoided. Use of aluminum inside the shield without verification that it is free of uranium, thorium, or other radioactive contaminants should also be avoided. If a graded shield without a tin layer is required to remove Pb K X rays generated in the lead, it should be made from a 2 mm thick tin sheet. To attenuate the K X rays of tin, a 0.5 mm copper sheet may be used to cover the tin. Copper material that is 1 cm thick also may be used as a graded shield when the shield is made from lead.

E.2 Shield design

One of two possible shield designs should be considered based on the activity level of the samples to be counted and the variety of sample sizes and shapes to be counted.

E.2.1 Shield design for detectors counting a variety of low- or high-activity-level samples

The following guidelines are provided:

a) The floor, ceiling, walls, and door of the shield should be at least 10 cm thick lead or 15 cm thick steel. An extra 5 cm of lead along those sides facing a concrete floor or a cinderblock wall can further reduce the ^{40}K background. Thicker sides do not, in general, significantly improve the shielding, due to cosmic-neutron multiplicity.

b) The inside walls of the high Z material comprising the shield should be at least 10 cm from the detector housing to reduce the probability of backscattered radiation entering the detector.

c) To reduce the presence of radioactive gas (e.g., ^{226}Ra) inside the shield, the shield can be continuously purged with nitrogen or air free of radioactive gas or aerosol particles. Sometimes the nitrogen off-gas from the detector dewar is vented inside the shield interior for this reason.

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E.2.2 Shield design for detectors counting only environmental samples of the same size and shape

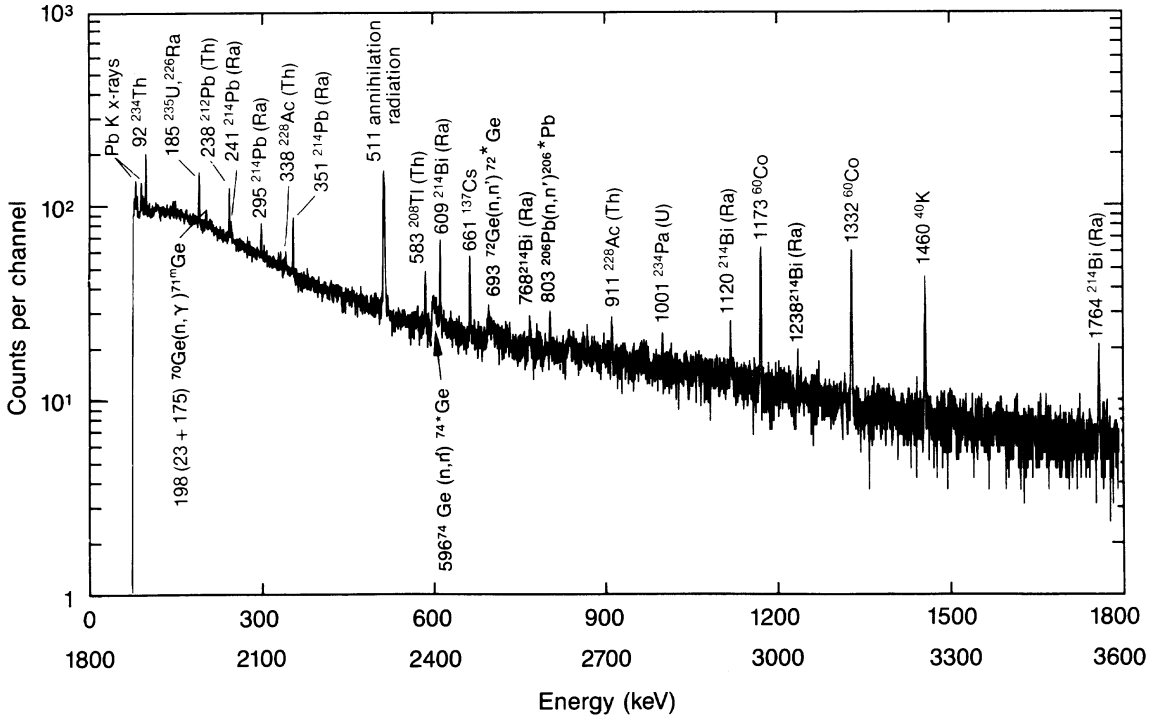
For samples at or below environmental radioactivity levels, it is recommended that only specially designed detectors or cryostats be purchased. An inner cylindrical sleeve of approximately 1 cm thick high-purity copper may be used to shield 46 keV gamma rays from ^{210}Pb and the lead-fluorescence K X rays from the detector. At very low counting rates, spectral peaks may be observed from prompt gamma-ray emission from the detector (Ge) and surrounding material (e.g., Al, Cu, Cd, and Pb) due to nuclear excitation by cosmic-ray-generated neutrons. If the excitation of surrounding material by cosmic-ray-generated neutrons causes problems, special shields designed to shield against these neutrons should be built (see Brodzinski et al. [B4]). The air gaps between the shield, copper sleeve, and detector should be tightly sealed to reduce the background from radon and progeny. If properly constructed, a very low background can be achieved, and gamma rays with energies below 50 keV from the sample can be observed above the background.

A background gamma-ray spectrum taken for a 64 h counting time on a 160 cm^3 Ge coaxial detector (the relative efficiency, ε_{rel}, is approximately 40%) with no sample present is shown in Figure E.1, and a background spectrum with a 4 L Marinelli beaker filled with deionized water (radon free) on the same detector is shown in Figure E.2. The shield is surrounded by 10 cm thick lead except where a cinderblock wall is adjacent to it. On the sides adjacent to the cinderblock wall, the lead is 15 cm thick. The spectra show not only the natural radioactive elements present in the environment surrounding the detector, but also many gamma rays resulting from reactions of degenerated cosmic-ray neutrons with the detector crystal and other surrounding material. The flux of degenerated cosmic-ray neutrons is particularly noticeable through the nuclear reactions with the detector and surrounding materials in laboratories at elevations greater than 1000 m above sea level.

E.3 References

See Brodzinski et al. [B4], Debertin and Helmer [B10], and Lindstrom et al. [B28].

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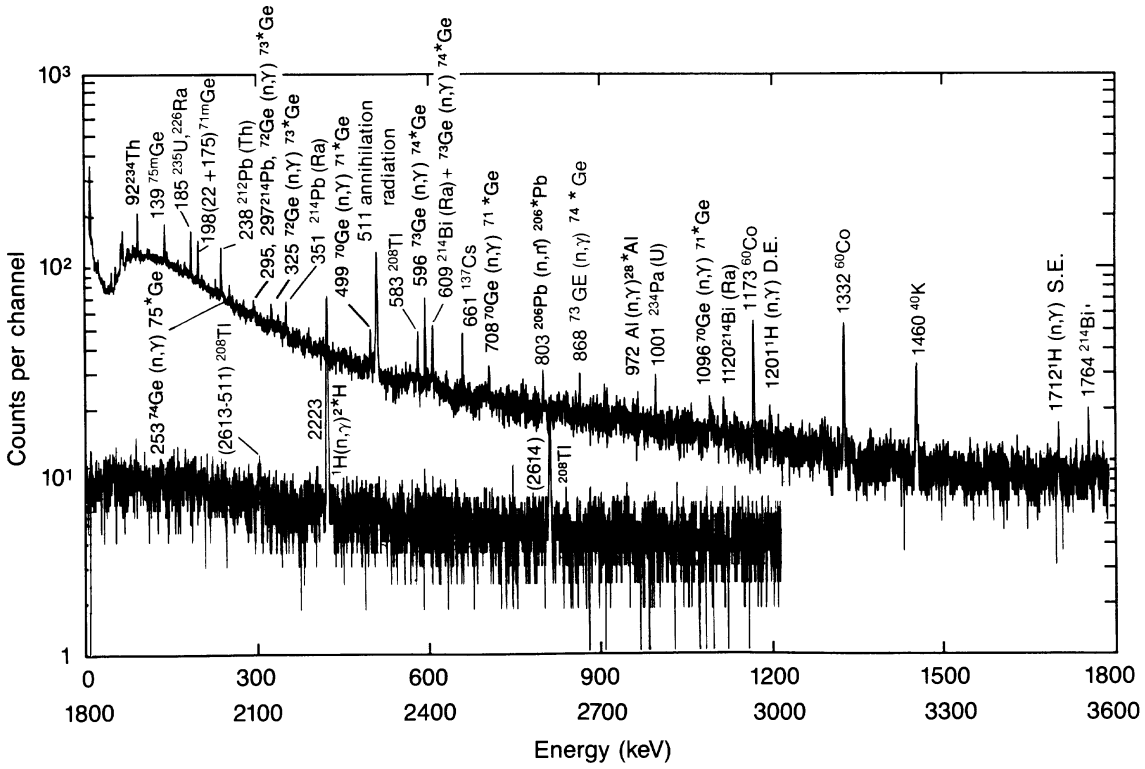


NOTE—The counting time was 64 h, and the count was taken with a 38% coaxial Ge detector completely surrounded by a 10–15 cm thick lead shield lined with 1 mm thick tin sheet. The support for the ceiling of the shield is a brass plate. The lead walls of the shield adjacent to the cinderblock walls of the room are 15 cm thick. The background peaks are caused by the man-made radionuclides in the ambient background (e.g., ^{137}Cs); members of the natural decay chains (e.g., ^{214}Bi); and peaks resulting from the interaction of neutrons from degenerated cosmic-ray particles upon various materials of the detector (e.g., ^{71m}Ge), lead shield [e.g., $^{206}Pb(n,g)^{206*}Pb$ where the asterisk represents an excited state], sample (see Figure E.2), or surrounding material [e.g., $^{27}Al(n,g)^{28*}Al$]. If the shield were lined with cadmium sheet instead of tin, a peak may be observed at 558 keV due to the $^{113}Cd(n,g)^{114*}Cd$ reaction. This spectrum was taken at a laboratory that is at an elevation of approximately 1500 m.

Figure E.1—Background gamma-ray spectrum taken with no sample

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NOTE—Background gamma-ray spectrum taken on the same spectrometer and for the same counting time as used in Figure E.1, but with a water sample in a 4 L Marinelli beaker. Observe that the contribution from the man-made and natural radionuclides is less since the water sample is acting as a shield. Note that the Doppler-broadened peaks at 596 keV and 693 keV from Figure E.1 are not observable. These peaks in Figure E.1 were caused by energetic neutrons. in this spectrum, the neutrons have been thermalized by the water in the Marinelli beaker and are more likely to interact with the detector itself without causing a Doppler broadening of the observed peaks. Therefore, a larger number of peaks from $^{A}Ge(n,g)^{(A+1)}*Ge$ (the asterisk represents an excited state) reactions are observed along with the reaction $^{1}H(n,g)^{2}H$ with a 2223 keV gamma ray (deuteron binding energy) from the water itself. This spectrum was taken at a laboratory that is at an elevation of approximately 1500 m.

Figure E.2—Background gamma-ray spectrum taken with a Marinelli beaker sample of water

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