

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

National Committee on Radiation Instrumentation, N42

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Secretariat

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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 3 000 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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Foreword

(This Foreword is not a part of ANSI N42.14-1991, American National Standard 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 N42.2. Drafts were reviewed by members of Committee N42, Subcommittee N42.2, 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 September 28, 1990.

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

1. Introduction

The purpose of this standard is to provide a basis for the routine calibration and use of germanium (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 correct procedures for the calibration and use of such detectors. This standard is primarily intended 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), multi-channel 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 Appendix D 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. These pulses are 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 will display a spectrum of one or more peaks with an approximately normal (Gaussian) distribution corresponding to photons that interacted with the detector.

A source emission rate, $R(E)$, for a gamma ray of energy E is determined by dividing the

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

- (1) The loss of pulses due to pulse pileup (at high counting rates)
- (2) Cascade (coincidence) summing
- (3) The decay of the source during sampling (e.g., with air filters) and counting
- (4) The decay of the source from a previous time to the counting period
- (5) Attenuation of photons within and external to the source that is not accounted for by the full-energy peak efficiency calibration

2. Scope

This standard establishes methods for the calibration and use of 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. Minimum requirements for automated peak finding are stated. This standard establishes 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 cascade 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 establishment of data libraries for radionuclide identification, decay corrections, and the conversion of gamma-ray emission rates to decay rates.

The measurement of X-ray emission rates is not included because different functional fits are required for X-ray peaks, which have intrinsically different peak shapes than gamma-ray peaks. Further, X-ray peaks are complex multiplets (e.g., the K X rays of Tl are composed of eight components that form four partially resolved individual peaks). 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 beyond the scope of this standard. Refer to ANSI N13.30 [1]¹ or to NUREG-1156 [17] for such methods.

3. Definitions

The following terms are defined for use in this standard.

3.1 Special Word Usage. 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.

3.2 Glossary

activity (A). The expected number of spontaneous nuclear decays (transformations) in unit time from a specified energy state (excluding prompt decays from a lower nu-

clear 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, where 3.7×10^{10} Bq equals 1 Ci, exactly.

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

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.

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.

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 **baseline**.)

baseline. 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:* **continuum**.

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).

cascade 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:* **coincidence summing**.

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

continuum. See **baseline**.

¹The numbers in brackets correspond to those of the references in Section 13.

coincidence summing. See **cascade summing of X and gamma rays**.

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.

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}).

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

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

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 **full-energy peak efficiency** and **total efficiency**.

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.

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.

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**.

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

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].

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**.

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.

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).

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.

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.

multichannel pulse-height analyzer (MCA). An electronic device that records and stores pulses according to their height. It consists of three function segments:

- (1) An ADC to provide a means of measuring pulse amplitude
- (2) Memory registers (one for each channel of the spectrum) to tally the number of pulses having an amplitude within a given voltage increment
- (3) 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.

NIST. US National Institute of Standards and Technology (formerly US National Bureau of Standards, NBS).

NSLR. A country's National Standardizing Laboratory for radioactivity measurements.

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.

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 Fig A1 for an example of proper adjustment for pole-zero cancellation).

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.

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 diameter x 7.6 cm high, for a source-to-detector distance of 25 cm.

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

where

$n(1332)$ = The measured full-energy peak counting rate
 $R(1332)$ = 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.

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.

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

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

3.3 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 (cascade 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
$N(E)$	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_L	The live-timed counting period
T_r	The elapsed-time counting period
$\alpha_K(E)$	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
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 uninterruptable power supply. Assure that the ambient temperature is stable and below 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.

PRECAUTION: When filling the detector dewar with liquid nitrogen (LN₂), avoid spilling it in the vicinity of the cryostat or preamplifier, which could lead to cracks in the vacuum seal rings of the cryostat or damage to the preamplifier. It is also wise to turn a count off during the filling process to avoid distortion of the measured spectrum due to microphonics.

Follow the instructions of the manufacturer for installation and preliminary testing of the individual components (i.e., detector with cryostat and preamplifier, linear spectrometry amplifier, high-voltage bias supply, ADC, and multichannel storage). Adjust the linear amplifier gain so that the highest energy gamma ray of interest has a voltage pulse just below the maximum voltage pulse acceptable to the ADC input (see specifications of the manufacturer). Check the accuracy of the 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 Appendix A. Measure the energy resolution (i.e., FWHM and FWTM) at 122 keV (⁵⁷Co) and at 1332 keV (⁶⁰Co), and the peak-to-Compton ratio at 1332 keV as described by the user manual of the detector manufacturer (IEEE Std 325-1986 [2] and Appendix A may be useful in performing the above adjustments and measurements). 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 conditions 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 assure 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, the user should adjust the various spectrometer settings, including the amplifier time constant, baseline restorer,

pole-zero cancellation, etc., for optimal performance under routine counting conditions before conducting the performance tests in this standard. Timing of any blanking signals for reset preamplifiers should be checked, for example.

5. Calibration Procedures

5.1 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-area criteria for a singlet, are approximately the same intensity, and are separated by 1 FWHM. (see Section 8 for the test procedures). Optimization of the peak-search parameters of the peak-finding algorithm is left to the user or software vendor. These should be adjusted so that statistically significant peaks are found with a minimum number of false peaks being reported (see performance tests in Section 8).

5.2 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., > 3 FWHM separation) are recommended over those with overlapping interferences (i.e., < 3 FWHM separation) whenever there is a choice.

WARNING: The peak-fitting algorithm may influence the energy dependence of pileup corrections (see 7.5 and A5.1).

Recommendation. It is very helpful to be able to evaluate visually 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.

5.3 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 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 A2.2.

The calibration source should be positioned at the same distance as it is for the samples and counted for a duration that gives sufficient counts in the peaks to permit measurement of the peak positions with a precision of < 0.2 keV. The peak positions at the calibration energies should be used to specify the coefficients of the fitting function (linear or quadratic), which are stored for subsequent evaluation of the energies of the measured peaks in the sample spectra. If the Ge spectrometer has long-term pulse-height stability, the energy scale should not need to be measured 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. Reference [9] describes one method to measure the pulse-height nonlinearity employing sources. (In this method, ^{110m}Ag may be substituted for ^{82}Br and ^{88}Y or ^{144}Ce - ^{144}Pr may be substituted for ^{56}Co .)

5.4 Efficiency Calibration Measurement. Two separate approaches are considered for the calibration of the detector efficiency:

- (1) Measurement of a standardization coefficient for a specific gamma ray and radionuclide by direct comparison with a standard source of known activity
- (2) Measurement of the full-energy peak efficiency as a function of energy

For approach (2), two cases are considered:

- Source-to-detector distances that are greater than approximately 10 cm
- Small source-to-detector distances that are required for sensitivity (low-activity samples) but may compromise the accuracy of the source configuration geometry and add the requirement for cascade-summing corrections.

Section A6 contains efficiency-measuring procedures for each of these experimental conditions.

If calculations are used in place of measurements to deduce efficiencies or to infer efficiencies for one counting geometry from those measured for another, satisfactory agreement shall be demonstrated by direct measurement for at least one typical geometry.

5.4.1 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 cascade 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 located inside the well of a Ge well detector (also see first Precaution in 6.2.4). Therefore,

the standardization coefficient method is the only efficiency calibration technique recommended for these or similar experimental conditions.

5.4.2 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 cascade 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.

5.4.2.1 Large Source-to-Detector Distances. At large sample-to-detector distances (typically, a source-to-detector distance that is ≥ 10 cm), the error in the replication of the sample-detector geometry, and in the correction for cascade summing of gamma-ray pulses in the detector, can usually be neglected for routine measurements. For these reasons, measurements at large sample-to-detector distances are preferred over measurements close to the detector, provided the source activity is high enough to achieve an acceptable accuracy within the allotted counting time.

5.4.2.2 Small Source-to-Detector Distances. The analysis of low-activity-level samples is performed at many different types of nuclear facilities. Effluent and environmental samples typically require small sample-to-detector distances and long counting times. These experimental conditions can result in large uncertainties and/or errors due to proportionally large background contributions, nonreproducibility of the sample-to-detector distance, and large cascade-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 cascade summing of gamma rays at a 5 cm source-to-detector distance for a point source of some radionuclides.

Whenever possible, a sample should be at least 5 cm from the detector, and only those peaks whose corrections for cascade 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 6.2.4). Large sample containers have a large mean source-to-detector distance even when they are in close proximity to the detector. This effect reduces the cascade-summing effect.

5.4.2.3 Reentrant (Marinelli) Beakers.

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. Depending on the size of the Marinelli beaker and the detector efficiency, significant cascade-summing corrections may be required. If Marinelli beakers are to be used

- (1) Corrections for cascade summing should either be estimated or applied
- (2) Large volume (e.g., approximately 4 L) Marinelli beakers should be used to increase the mean sample-to-detector distance (the uncertainties in the gamma-ray emission rates may need to be increased)

The test in Section 9 may be useful to demonstrate if cascade-summing corrections can be avoided.

5.4.3 Efficiency Function or Lookup Table.

The full-energy peak efficiency values, $\epsilon(E)$ for energy E , should be plotted either as $\epsilon(E)$ on a log-log graph (see Fig 1) or as the function $Y = \epsilon(E) \times E^a$ on a linear plot (a is a normalizing constant), as shown in Fig 2. This latter function provides better resolution of the efficiency data when plotted. There should be no large energy gaps between the efficiency values (especially below 300 keV) that would cause a large efficiency-interpolation 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 megaelectronvolts), 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

- (1) 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 [4] and [10]) or by
- (2) Fitting the efficiency data with a suitable function (e.g., see [12], [15], [21], and [24]).

6. Gamma-Ray Measurements With Ge Spectrometers

6.1 Measurement of Gamma-Ray Energies.

The gamma-ray energies are normally derived from a previous energy calibration (see 5.3). 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 gamma rays present in the spectrum whose 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. Under these conditions the width (FWHM) and mean (peak position) of the normal (Gaussian) distribution function are fixed, and a linear fit is made to measure the peak height. In these types of analyses the width, as a function of the peak energy, must be known. Also a more accurate energy calibration may be required.

6.2 Measurement of Gamma-Ray Emission Rates and Radionuclide Activities.

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

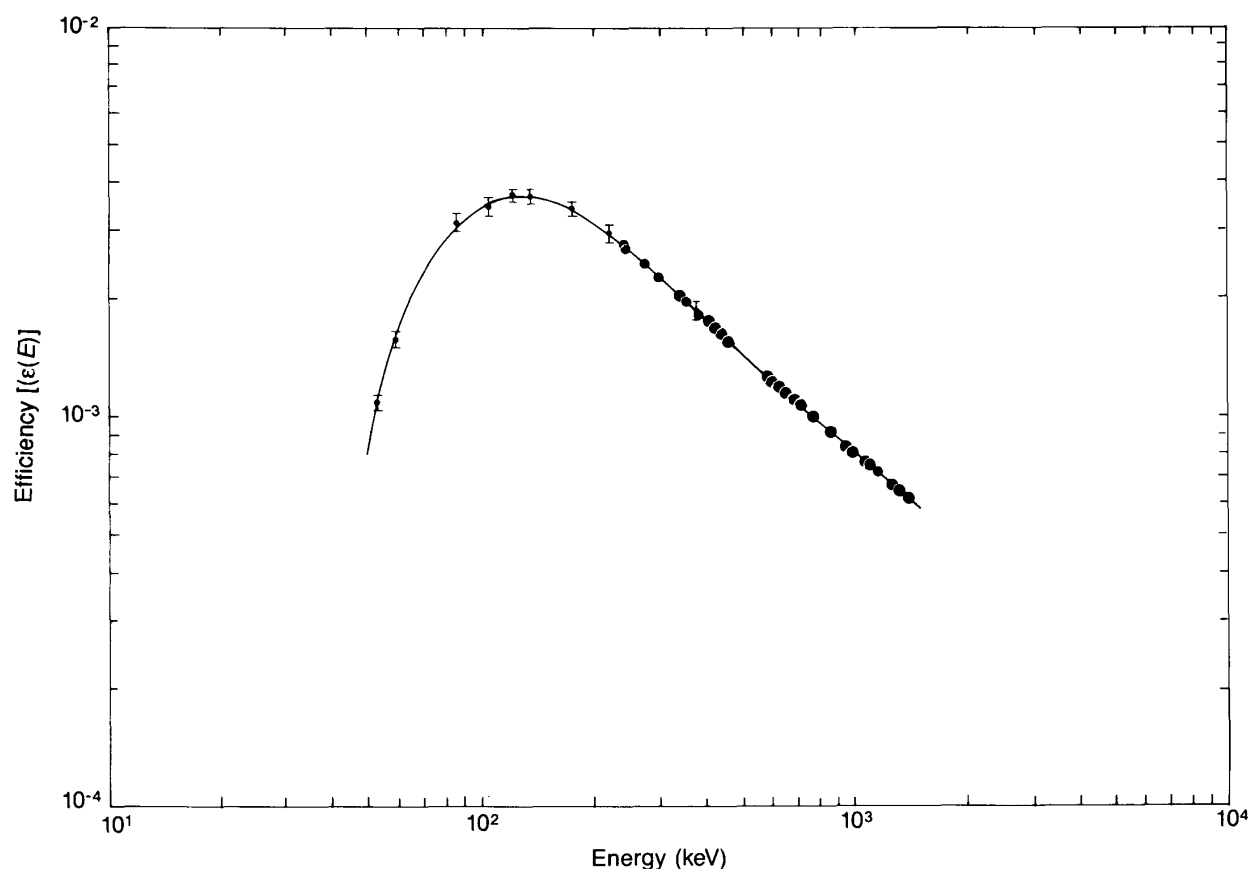


Fig 1
Full-Energy Peak Efficiency

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 (from R. G. Helmer, EG & G Idaho, Inc., P. O. Box 1625, Idaho Falls, ID 83415, USA).

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 above. Obtain the net-area counting rate in each of the gamma-ray peaks of interest, as described in 5.2. 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 5.4.3. Calculate the number of gamma rays emitted per second, $R(E)$, for each of the full-energy peaks as follows:

$$R(E) = [n(E) - B(E)] \times \frac{\pi C_i}{\varepsilon(E)} \quad (\text{Eq 2})$$

where

$n(E)$ = 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

$B(E)$ = Net counting rates of any interfering peaks in the background

$\varepsilon(E)$ = Full-energy peak efficiency (counts in the net peak per gamma-ray emitted)

C_i = Correction factors (where $i = d, r, c$ and a , as defined below)

π = An operation of taking products (analogous to Σ designating an operation of taking sums)

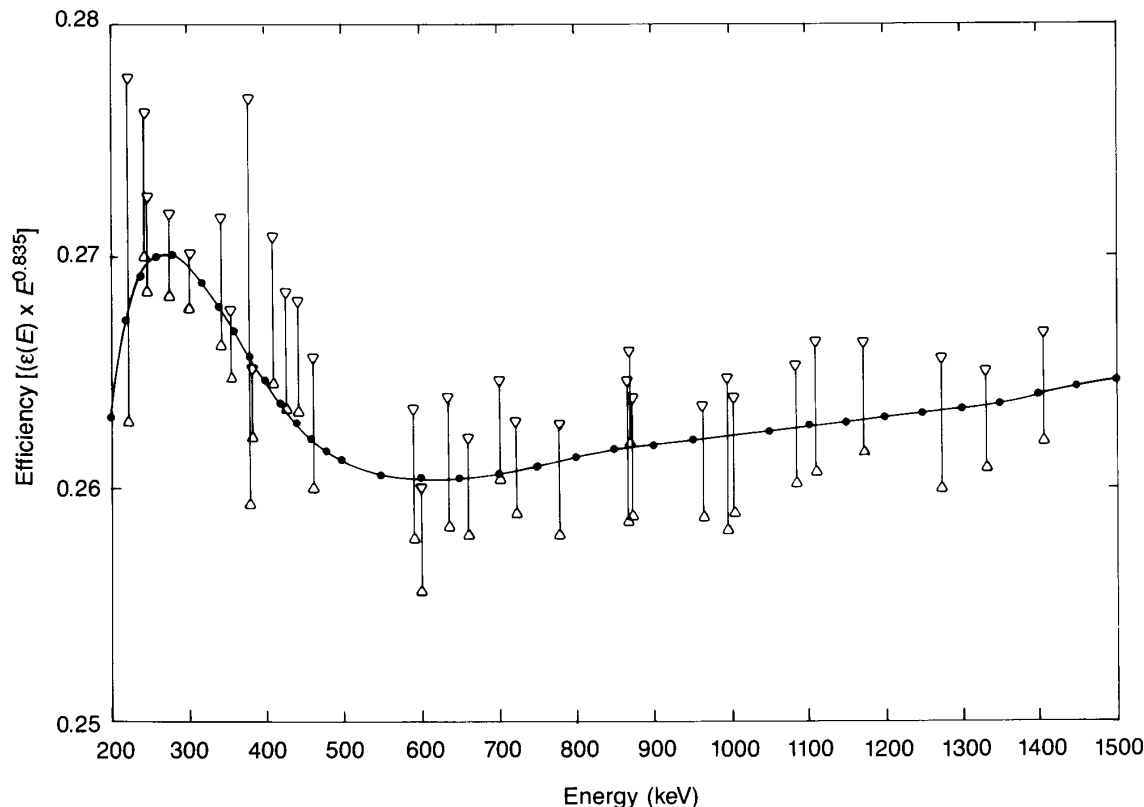


Fig 2
 $\epsilon \cdot E(\text{keV})$ as a Function of Gamma-Ray Energy

NOTE: The same data as in Fig 1, plotted as $\epsilon E(\text{keV})^{0.835}$ versus $E(\text{keV})$ on a linear plot where ϵ is the full-energy peak efficiency at $E(\text{keV})$ and $E(\text{keV})$ is the energy of the full-energy peak (from R. G. Helmer, EG & G Idaho, Inc., P.O. Box 1625, Idaho Falls, ID 83415, USA). The solid circles represent selected values taken from the plot and used to prepare the efficiency lookup table.

The correction factors include correction for decay during and prior to a count ($i = d$), pulse pileup ($i = r$), cascade summing ($i = c$), and differences in the attenuation of the sample counting geometry from that in the standard ($i = a$). 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.

The nuclear decay rate or activity is calculated by dividing the gamma-ray emission rate by the appropriate gamma-ray emission probability, $P(E)$.

$$A = \frac{R(E)}{P(E)} \quad (\text{Eq 3})$$

6.2.1 Subtraction of Interference Peaks in the Background. Large samples may shield the detector from the ambient background radiation, causing a lower counting rate than if an instrumental background (no sample present) were obtained. Thus, whenever large, low-activity samples are counted, a background spectrum shall be obtained with a "blank" sample in the same geometry and of the same density. The background count shall be accumulated for at least the same counting time as used in counting the samples to be analyzed. (All soils contain some natural ^{40}K , Th, and U radioactivity. If a "blank" containing no radioactivity is required for an analysis, it will be necessary to use a simulated "blank"

or calculate the attenuation in the background measured without the sample.) The "blank" sample background spectrum, normalized for the same counting time, could be subtracted from the sample spectrum, channel-by-channel, or, preferably, the background spectrum should be analyzed to obtain the background-peak counting rates, $B(E)$, at energies E . If the latter method is used (it is preferred), the background-peak counting rates are then subtracted from the respective net-area peak counting rates, $n(E)$, in the sample spectrum (see Eq 2). By subtracting the respective background-peak counting rates from those of the sample, the statistics of each count are preserved. This method is also less sensitive to small changes in the energy calibration. If possible, the "blank" spectrum should be scanned to be sure that all background peaks are identified and that no false peaks are reported.

6.2.2 Radioactive Decay. The correction

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

- (1) Decay during the counting interval
- (2) 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 must be applied. These correction factors will be designated C_{d1} , C_{d2} , and C_{d3} , respectively. The combined correction factor for radioactive decay can be expressed as

$$C_d = C_{d1} \times C_{d2} \times C_{d3} \quad (\text{Eq 4})$$

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

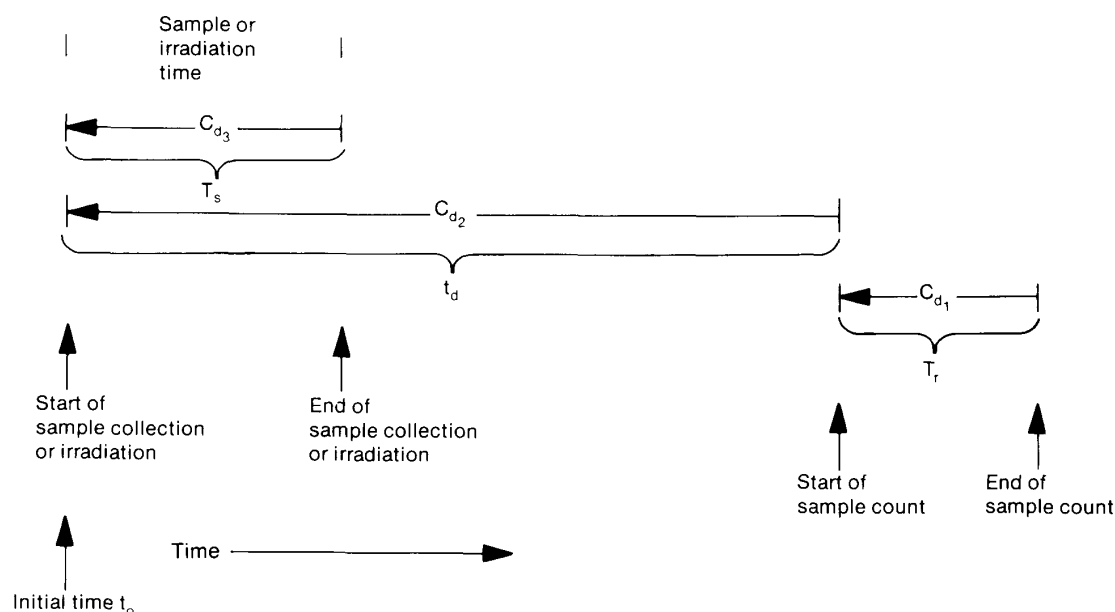


Fig 3
Specification of Times for Decay Corrections

NOTE: Specification of various events expressed in units of time, where t_0 is the initial time, T_s is the sample or 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.

6.2.2.1 Radioactive Decay During the Counting Period. If significant decay occurs during the counting time ($> 10\%$), 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:

$$C_{d1} = \frac{\lambda T_r}{1 - e^{-\lambda T_r}} \quad (\text{Eq 5})$$

where

- C_{d1} = The correction factor for decay during the count
 λ = $\ln 2/T_{1/2}$ ($T_{1/2}$ is the half-life)
 T_r = Real (elapsed) time of the count
 e = Base of the natural logarithm

This correction assumes a constant dead time during the acquisition period. An error will 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.

6.2.2.2 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:

$$C_{d2} = \exp(\lambda T_d) \quad (\text{Eq 6})$$

where

- C_{d2} = Correction factor for decay prior to the count
 T_d = Decay time from a prior time to the start of the count
 $\exp$ = The operation of raising the bracketed quantity to the power of e .

6.2.2.3 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 Fig 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,

$$C_{d3} = \frac{1}{[1 - \exp(-\lambda T_i)]} \quad (\text{Eq 7})$$

where

T_i = 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 Fig 3). In this case,

$$C_{d3} = \frac{\lambda T_s}{[1 - \exp(-\lambda T_s)]} \quad (\text{Eq 8})$$

where

T_s = Sampling duration

6.2.3 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_r , may be measured by one of several methods. Three popular methods are

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

Although each method gives a slightly different correction, the difference between meth-

ods is typically less than 20% of the correction. The type of ADC and the type of peak-area measurement algorithm can affect the amount of energy dependency in the pulse-pileup correction.

A description of the source-set method is given in Section A7. Descriptions of methods (2) and (3) for measuring the pulse-pileup correction can be found in [14] and [23]. The use of these or other types of electronic circuitry to correct automatically for pileup is permitted if validated by passing the test in 7.5.

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 ($> 2 \mu\text{s}$) typically yield higher energy resolution but poorer counting-rate performance. For amplifier unipolar output pulse widths of about $12 \mu\text{s}$ (a time constant of about $2 \mu\text{s}$), preamplifiers with resistive feedback, and input counting rates $< 1000 \text{ s}^{-1}$, the pulse-pileup correction is normally $< 1\%$ and may be neglected.

WARNING: 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 ($> 10\,000 \text{ s}^{-1}$), errors in the live timer can be significant ($> 5\%$).

WARNING: 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 7.5.

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

6.2.4 Cascade (Coincidence) Summing.

Cascade-summing corrections shall be applied whenever counting conditions result in significant coincident summing of gamma rays associated with the radionuclides of interest. It should be assumed that these corrections are necessary for all counting

geometries in which the mean sample-to-detector distance is $< 5 \text{ cm}$ unless demonstrated otherwise.

Because the total efficiency (partial and total photon energy deposition in the detector) of 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 cascade 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 cascade-summing corrections, C_c , for specific radionuclides and source-detector geometries are described in [6] and [20]. A brief description of cascade summing and the equations needed to determine the loss or gain of counts to a gamma-ray peak is provided in Appendix C.

PRECAUTION: 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. 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.

PRECAUTION: Beta particles and associated bremsstrahlung emitted in the decay of beta-emitting radionuclides can contribute significantly to the spectral continuum. Further, the gamma-ray transitions promptly follow the beta radiation (assuming no isomeric transitions). The coincidence summing of beta particles and associated bremsstrahlung with gamma rays is not included in most cascade-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.

6.2.5 Attenuation Corrections. Two components are considered for attenuation correction: external absorbers, C_{a1} , and differences in the matrix or density of identically shaped samples, C_{a2} . These corrections are then combined as

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

where

C_a = Attenuation correction

6.2.5.1 Absorption External to the Sample.

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

$$C_{a1} = \exp(\mu \cdot \rho \cdot d) \quad (\text{Eq 10})$$

where

μ = Mass attenuation coefficient
 ρ = Density of the absorber
 d = Mean thickness through absorber

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

6.2.5.2 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 must be the same). When the error resulting from not correcting 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 [19]).

6.2.5.2.1 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 [19]). 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

$$C_{a2} = \frac{\mu_u(E) \cdot \rho_u \cdot d}{1 - \exp[-\mu_u(E) \cdot \rho_u \cdot d]} \times \frac{1 - \exp[-\mu_s(E) \cdot \rho_s \cdot d]}{\mu_s(E) \cdot \rho_s} \quad (\text{Eq 11})$$

where

$\mu_u(E)$ = Mass absorption coefficient at energy E of sample
 $\mu_s(E)$ = Mass absorption coefficient at energy E of efficiency-measuring standard
 ρ_u = Density of sample
 ρ_s = Density of efficiency-measuring standard
 d = Thickness of the standard/sample

6.2.5.2.2 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 must be known to a sufficient accuracy to determine the attenuation correction with adequate precision. The relative attenuation correction, C_{a2} , is calculated to be

$$C_{a2} = \frac{R(E) \cdot \epsilon(E)}{[n(E) - B(E)] [\tau C_{i \neq a}]} \quad (\text{Eq 12})$$

where

$R(E)$ = Emission rate of the gamma ray at energy E in the attenuation-correction standard
 $\epsilon(E)$ = Full-energy-peak efficiency at energy E
 $n(E)$ = Net peak-area counting rate for the attenuation-correction standard of a gamma ray of energy E
 $B(E)$ = Net peak-area counting rate of any background peak at energy E
 $C_{i \neq a}$ = Corrections for counting rate ($i = r$); cascade summing ($i = c$); and decay prior to and during the count ($i = d$) for the attenuation-correction standard

An alternative and probably more accurate method to measure the relative attenuation