



Designation: **E181 – 10 E181 – 17**

Standard Test Methods for Detector Calibration and Analysis of Radionuclides¹

This standard is issued under the fixed designation E181; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover general procedures for the calibration of radiation detectors and the analysis of radionuclides. For each individual radionuclide, one or more of these methods may apply.

1.2 These test methods are concerned only with specific radionuclide measurements. The chemical and physical properties of the radionuclides are not within the scope of this standard.

1.3 The measurement standards appear in the following order:

	Sections
Spectroscopy Methods:	
Calibration and Usage of Germanium Detectors	3 – 12
Calibration and Usage of Scintillation Detector Systems:	13 – 20
Calibration and Usage of Scintillation Detectors for Simple Spectra	16
Calibration and Usage of Scintillation Detectors for Complex Spectra	17
Counting Methods:	
Beta Particle Counting	25-26
Aluminum Absorption Curve	27 – 31
Alpha Particle Counting	32 – 39
Liquid Scintillation Counting	40 – 48

1.4 Additional information on the set-up, calibration and quality control for radiometric detectors and measurements is given in Guide C1402 and Practice D7282.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Document

2.1 *ASTM Standards:*²

[C1402 Guide for High-Resolution Gamma-Ray Spectrometry of Soil Samples](#)

[D7282 Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements](#)

[D7283 Test Method for Alpha and Beta Activity in Water By Liquid Scintillation Counting](#)

[E170 Terminology Relating to Radiation Measurements and Dosimetry](#)

¹ These test methods are under the jurisdiction of ASTM Committee E10 on Nuclear Technology and Applications.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

SPECTROSCOPY METHODS

3. Terminology

3.1 Definitions:

3.1.1 *certified radioactivity standard source*—a calibrated radioactive source, with stated accuracy, whose calibration is certified by the source supplier as traceable to the National Radioactivity Measurements System (1).³

3.1.2 *check source*—a radioactivity source, not necessarily calibrated, that is used to confirm the continuing satisfactory operation of an instrument.

3.1.3 *FWHM*—(full width at half maximum) the full width of a gamma-ray peak distribution measured at half the maximum ordinate above the continuum.

3.1.4 *national radioactivity standard source*—a calibrated radioactive source prepared and distributed as a standard reference material by the U.S. National Institute of Standards and Technology.

3.1.5 *resolution, gamma ray*—the measured FWHM, after background subtraction, of a gamma-ray peak distribution, expressed in units of energy.

3.2 Abbreviations:

3.2.1 *MCA*—Multichannel Analyzer.

3.2.2 *SCA*—Single Channel Analyzer.

3.2.3 *ROI*—Region-Of-Interest.

3.3 For other relevant terms, see Terminology E170.

3.4 *correlated photon summing*—the simultaneous detection of two or more photons originating from a single nuclear disintegration.

3.5 *dead time*—the time after a triggering pulse during which the system is unable to retrigger.

NOTE 1—The terms “standard source” and “radioactivity standard” are general terms used to refer to the sources and standards of National Radioactivity Standard Source and Certified Radioactivity Standard Source.

CALIBRATION AND USAGE OF GERMANIUM DETECTORS

4. Scope

4.1 This standard establishes methods for calibration, usage, and performance testing of germanium detectors for the measurement of gamma-ray emission rates of radionuclides. It covers the energy and full-energy peak efficiency calibration as well as the determination of gamma-ray energies in the 0.06 to 2-MeV energy region and is designed to yield gamma-ray emission rates with an uncertainty of $\pm 3\%$ (see Note 2). This method applies primarily to measurements that do not involve overlapping peaks, and in which peak-to-continuum considerations are not important.

NOTE 2—Uncertainty U is given at the 68 % confidence level; that is, $U = \sqrt{\sum \sigma_r^2 + 1/3 \sum \delta_i^2}$ where δ_i are the estimated maximum systematic uncertainties, and σ_r are the random uncertainties at the 68 % confidence level (2). Other methods of error analysis are in use (3, 4).

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5. Apparatus

5.1 A typical gamma-ray spectrometry system consists of a germanium detector (with its liquid nitrogen cryostat, preamplifier, and possibly a high-voltage filter) in conjunction with a detector bias supply, linear amplifier, multichannel analyzer, and data readout device, for example, a printer, plotter, oscilloscope, or computer. Gamma rays interact with the detector to produce pulses which are analyzed and counted by the supportive electronics system.

6. Summary of Methods

6.1 The purpose of these methods is to provide a standardized basis for the calibration and usage of germanium detectors for measurement of gamma-ray emission rates of radionuclides. The method is intended for use by knowledgeable persons who are responsible for the development of correct procedures for the calibration and usage of germanium detectors.

³ The boldface numbers in parentheses refer to the list of references at the end of these methods.

6.2 A source emission rate for a gamma ray of a selected energy is determined from the counting rate in a full-energy peak of a spectrum, together with the measured efficiency of the spectrometry system for that energy and source location. It is usually not possible to measure the efficiency directly with emission-rate standards at all desired energies. Therefore a curve or function is constructed to permit interpolation between available calibration points.

7. Preparation of Apparatus

7.1 Follow the manufacturer's instructions for setting up and preliminary testing of the equipment. Observe all of the manufacturer's limitations and cautions. All tests described in Section 12 should be performed before starting the calibrations, and all corrections shall be made when required. A check source should be used to check the stability of the system at least before and after the calibration.

8. Calibration Procedure

8.1 *Energy Calibration*—Determine the energy calibration (channel number versus gamma-ray energy) of the detector system at a fixed gain by determining the channel numbers corresponding to full energy peak centroids from gamma rays emitted over the full energy range of interest from multip peaked or multinuclide radioactivity sources, or both. Determine nonlinearity correction factors as necessary (5).

8.1.1 Using suitable gamma-ray compilations (6-14), plot or fit to an appropriate mathematical function the values for peak centroid (in channels) versus gamma energy.

8.2 Efficiency Calibration:

8.2.1 Accumulate an energy spectrum using calibrated radioactivity standards at a desired and reproducible source-to-detector distance. At least 20 000 net counts should be accumulated in each full-energy gamma-ray peak of interest using National or Certified Radioactivity Standard Sources, or both (see 12.1, 12.5, and 12.6).

8.2.2 For each standard source, obtain the net count rate (total count rate of region of interest minus the Compton continuum count rate and, if applicable, the ambient background count rate within the same region) in the full-energy gamma-ray peak, or peaks, using a tested method that provides consistent results (see 12.2, 12.3, and 12.4).

8.2.3 Correct the standard source emission rate for decay to the count time of 8.2.2.

8.2.4 Calculate the full-energy peak efficiency, E_f , as follows:

$$E_f = \frac{N_p}{N_\gamma} \quad (1)$$

where:

E_f = full-energy peak efficiency (counts per gamma ray emitted),

N_p = net gamma-ray count in the full-energy peak (counts per second live time) (Note 3) (see 8.2.2), and

N_γ = gamma-ray emission rate (gamma rays per second).

NOTE 3—Any other unit of time is acceptable provided it is used consistently throughout.

8.2.5 There are many ways of calculating the net gamma-ray count. The method presented here is a valid, common method when there are no interferences from photopeaks adjacent to the peak of interest, and when the continuum varies linearly from one side of the peak to the other.

8.2.5.1 Other net peak area calculation methods can also be used for single peaks, and must be used when there is interference from adjacent peaks, or when the continuum does not behave linearly. Other methods are acceptable, if they are used in a consistent manner and have been verified to provide accurate results.

8.2.5.2 Using a simple model, the net peak area for a single peak can be calculated as follows:

$$N_A = G_s - B - I \quad (2)$$

where:

G_s = gross count in the peak region-of-interest (ROI) in the sample spectrum,

B = continuum, and

I = number of counts in the background peak (if there is no background peak, or if a background subtraction is not performed, $I = 0$).

8.2.5.3 The net gamma-ray count, N_p is related to the net peak area as follows:

$$N_p = \frac{N_A}{T_s} \quad (3)$$

where T_s = spectrum live time.

8.2.5.4 The continuum, B , is calculated from the sample spectrum using the following equation (see Fig. 1):

$$B = \frac{N}{2n} (B_{1s} + B_{2s}) \quad (4)$$

where:

- N = number of channels in the peak ROI,
- n = number of continuum channels on each side,⁴
- B_{1s} = sum of counts in the low-energy continuum region in the sample spectrum, and
- B_{2s} = sum of counts in the high-energy continuum region in the sample spectrum.

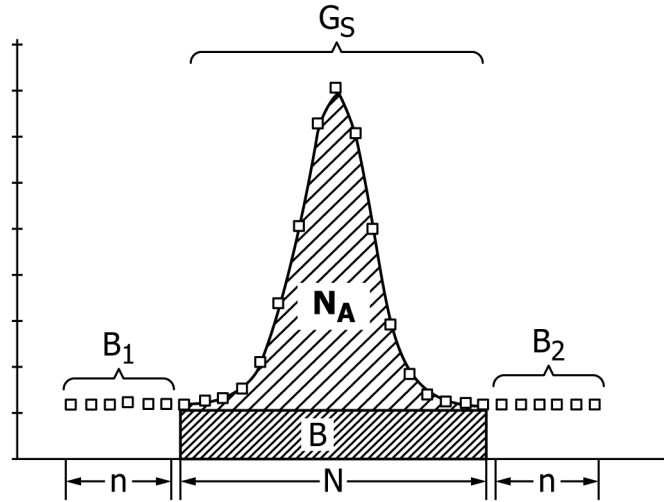


FIG. 1 Typical Spectral Peak With Parameters Used in the Peak Area Determination Indicated

NOTE 4—These equations assume that the channels that are used to calculate the continuum do not overlap with the peak ROI, and are adjacent to it, or have the same size gap between the two regions on both sides. A different equation must be used, if the gaps are of a different size.

The peaked background, I , is calculated from a separate background measurement using the following equation:

$$I = \frac{T_s}{T_b} I_b \quad (5)$$

where:

- T_s = live time of the sample spectrum,
- T_b = live time of the background spectrum, and
- I_b = net background peak area in the background spectrum.

If a separate background measurement exists, the net background peak area is calculated from the following equation:

$$I_b = G_b - B_b \quad (6)$$

where:

- G_b = sum of gross counts in the background peak region (of the background spectrum), and
- B_b = continuum counts in the background peak region (of the background spectrum).

The continuum counts in the background spectrum are calculated from the following equation:

$$B_b = \frac{N}{2n} (B_{1b} + B_{2b}) \quad (7)$$

where:

- N = number of channels in the background peak ROI,
- n = number of continuum channels on each side (assumed to be the same on both sides),
- B_{1b} = sum of counts in the low-energy continuum region in the background spectrum, and
- B_{2b} = sum of counts in the high-energy continuum region in the background spectrum.

8.2.5.5 If the standard source is calibrated in units of Becquerels, the gamma-ray emission rate is given by:

$$N_\gamma = AP_\gamma \quad (8)$$

⁴ For simplicity of these calculations, n is assumed to be the same on both sides of the peak. If the continuum is calculated using a different number of channels on the left of the peak than on the right of the peak, different equations must be used.

where:

A = number of nuclear decays per second, and

P_γ = probability per nuclear decay for the gamma ray (7-14).

8.2.6 Plot, or fit to an appropriate mathematical function, the values for full-energy peak efficiency (determined in 8.2.4) versus gamma-ray energy (see 12.5) (15-23).

9. Measurement of Gamma-Ray Emission Rate of the Sample

9.1 Place the sample to be measured at the source-to-detector distance used for efficiency calibration (see 12.6).

9.1.1 Accumulate the gamma-ray spectrum, recording the count duration.

9.1.2 Determine the energy of the gamma rays present by use of the energy calibration obtained under, and at the same gain as 8.1.

9.1.3 Obtain the net count rate in each full-energy gamma-ray peak of interest as described in 8.2.2.

9.1.4 Determine the full-energy peak efficiency for each energy of interest from the curve or function obtained in 8.2.5.

9.1.5 Calculate the number of gamma rays emitted per unit live time for each full-energy peak as follows:

$$N_\gamma = \frac{N_p}{E_f} \quad (9)$$

When calculating a nuclear transmutation rate from a gamma-ray emission rate determined for a specific radionuclide, a knowledge of the gamma-ray probability per decay is required (7-14), that is,

$$A = \frac{N_\gamma}{P_\gamma} \quad (10)$$

9.1.6 Calculate the net peak area uncertainty as follows:

$$S_{N_A} = \sqrt{G_s + \left(\frac{N}{2n}\right)^2 (B_{1s} + B_{2s}) + \left(\frac{T_s}{T_b}\right)^2 (S_{Ib})^2} \quad (11)$$

where:

$$S_{Ib} = \sqrt{G_b + \left(\frac{N}{2n}\right)^2 (B_{1b} + B_{2b})} \quad (12)$$

and

S_{N_A} = net peak area uncertainty (at 1 σ confidence level),

G_s = gross counts in the peak ROI of the sample spectrum,

G_b = gross counts in the peak ROI of the background spectrum,

N = number of channels in the peak ROI,

n = number of continuum channels on each side (assumed to be the same on both sides for these equations to be valid),

B_{1s} = continuum counts left of the peak ROI in the sample spectrum,

B_{2s} = continuum counts right of the peak ROI in the sample spectrum,

B_{1b} = continuum counts left of the peak ROI in the background spectrum,

B_{2b} = continuum counts right of the peak ROI in the background spectrum,

T_s = live time of the sample spectrum, and

T_b = live time of the background spectrum.

If there is no separate background measurement, or if no background subtract is performed, $S_{Ib} = 0$.

9.1.7 For other sources of error, see Section 11.

10. Performance Testing

10.1 The following system tests should be performed on a regularly scheduled basis (or, if infrequently used, preceding the use of the system). The frequency for performing each test will depend on the stability of the particular system as well as on the accuracy and reliability of the required results. Where health or safety is involved, much more frequent checking may be appropriate. A range of typical frequencies for noncritical applications is given below for each test.

10.1.1 Check the system energy calibration (typically daily to semiweekly), using two or more gamma rays whose energies span at least 50 % of the calibration range of interest. Correct the energy calibration, if necessary. Sample counting must be halted or redone if the system energy calibration is found to be inadequate.

10.1.2 Check the system count rate reproducibility (typically daily to weekly) using at least one long-lived radionuclide. Correct for radioactive decay if significant decay (>1 %) has occurred between checks.

10.1.3 Check the system resolution (typically weekly to monthly) using at least one gamma-ray emitting radionuclide (24).

10.1.4 Check the efficiency calibration (typically monthly to yearly) using a National or Certified Radioactivity Standard (or Standards) emitting gamma rays of widely differing energies.

10.2 The results of all performance checks shall be recorded in such a way that deviations from the norm will be readily observable. Appropriate action, which could include confirmation, repair, and recalibration as required, shall be taken when the measured values fall outside the predetermined limits.

10.2.1 In addition, the above performance checks (see 10.1) should be made after an event (such as power failures or repairs) which might lead to potential changes in the system.

11. Sources of Uncertainty

11.1 Other than Poisson-distribution uncertainties, the principal sources of uncertainty (and typical magnitudes) in this method are:

11.1.1 The calibration of the standard source, including uncertainties introduced in using a standard radioactivity solution, or aliquot thereof, to prepare another (working) standard for counting (typically $\pm 3\%$).

11.1.2 The reproducibility in the determination of net full-energy peak counts (typically $\pm 2\%$).

11.1.3 The reproducibility of the positioning of the source relative to the detector and the source geometry (typically $\pm 3\%$).

11.1.4 The accuracy with which the full-energy peak efficiency at a given energy can be determined from the calibration curve or function (typically $\pm 3\%$).

11.1.5 The accuracy of the live-time determinations and pile-up corrections (typically $\pm 2\%$).

12. Precautions and Tests

12.1 *Random Summing and Dead Time:*

12.1.1 *Precaution*—The shape and length of pulses used can cause a reduction in peak areas due to random summing of pulses at rates of over a few hundred per second (25, 26). Sample count rates should be low enough to reduce the effect of random summing of gamma rays to a level where it may be neglected, or one should use pile-up rejectors and live-time circuits, or reference pulser techniques of verified accuracy at the required rates (27-33).

NOTE 5—Use of percent dead time to indicate whether random summing can be neglected may not be appropriate.

12.1.2 *Test:*

12.1.2.1 If the maximum total count rate (above the amplifier noise level) ever used is less than 1000 s^{-1} and the amplifier time constant is less than $5\text{ }\mu\text{s}$, this test need not be performed. Otherwise, accumulate a ^{60}Co spectrum with a total count rate of less than 1000 counts per second until at least 25 000 counts are collected in the 1.332 and 1.173 MeV full-energy peaks. A mixed isotopic point source may be used. Record the counting live time. The source may be placed at any convenient distance from the detector.

12.1.2.2 Evaluate the activity of ^{60}Co utilizing first the full photon peak area at 1.332 MeV and then the area at 1.173 MeV, including any methods employed to correct for pile-up and dead time losses.

12.1.2.3 Without moving the ^{60}Co source, introduce a ^{57}Co source, or any other source with no gamma rays emitted with an energy greater than 0.662 MeV. Position the added source so that the highest count rate used for gamma-ray emission rate determinations has been achieved.

12.1.2.4 Erase the first spectrum and accumulate another spectrum for the same length of time as in 12.1.2.1. The same live time may be used, if the use of live time constitutes at least a part of the correction method.

12.1.2.5 Evaluate the activity of ^{60}Co utilizing first the full photon peak area at 1.332 MeV and then the area at 1.173 MeV, including any methods employed to correct for pile-up and dead time losses. For the correction method to be acceptable, the resolution must not have increased beyond the range of the method and the corrected activity shall differ from those in 12.1.2.2 by no more than $2\% 1\sigma$ (67% confidence level).

12.2 *Peak Evaluation:*

12.2.1 *Precaution*—Many methods (34-39) exist for specifying the full-energy peak area and removing the contribution of any continuum under the peak. Within the scope of this standard, various methods give equivalent results if they are applied consistently to the calibration standards and the sources to be measured, and if they are not sensitive to moderate amounts of underlying continuum. A test of the latter point is a required part of this method.

12.2.2 *Test:*

12.2.2.1 Accumulate a spectrum from a mixed isotopic point source until at least 20 000 net counts are recorded in the peaks of interest lower in energy than 0.662 MeV. The source may be placed at any convenient distance from the detector.

12.2.2.2 Determine the net peak areas of the peaks chosen in 12.2.2.1 with the method to be tested. Include any calculations employed by the analysis method to be tested to correct for dead time losses, pile-up, and background contributions.

12.2.2.3 Without moving the mixed isotopic point source, introduce a ^{137}Cs , ^{60}Co , or any other source with no full energy photons emitted with energies in the range 0.060 to 0.600 MeV so the continuum level of the spectrum in this range is increased 20 times.

12.2.2.4 Erase the first spectrum and accumulate another spectrum for the same live time as in 12.2.2.1, if the use of live time constitutes at least a part of the correction method.

12.2.2.5 Determine the net peak areas of the same peaks chosen in 12.2.2.1 with the method to be tested. Include any calculations employed by the analysis method to be tested to correct for dead time losses, pileup, and background contributions.

12.2.2.6 The deviations of the 12.2.2.5 net peak areas from the 12.2.2.2 values shall be no more than 2 % 1σ (67 % confidence level) for the evaluation method to be acceptable.

12.3 Correlated Photon Summing Correction:

12.3.1 When another gamma ray or X ray is emitted in cascade with the gamma ray being measured, in many cases a multiplicative correlated summing correction, C , must be applied to the net full-energy-peak count rate if the sample-to-detector distance is 10 cm or less. The correction factor is expressed as:

$$C = \frac{1}{\prod_i^n (1 - q_i \varepsilon_i)} \quad (13)$$

where:

- C = correlated summing correction to be applied to the measured count rate,
- n = number of gamma or X rays in correlation with gamma ray of interest,
- i = identification of correlated photon,
- q_i = fraction of the gamma ray of interest in correlation with the i th photon, and
- ε_i = total detection efficiency of i th correlated photon.

Correlated summing correction factors for the primary gamma rays of radionuclides ^{60}Co , ^{88}Y , ^{46}Sc are approximately 1.09 and 1.03 for a 65-cm³ detector at 1 cm and at 4-cm sample-to-detector distances, respectively, and approximately 1.01 for a 100-cm³ detector at a 10-cm sample-to-detector distance. The q_i must be obtained from the nuclear decay scheme, while the ε_i , which are slowly-varying functions of the energy, can be measured or calculated (40-42).

12.3.2 A similar correction must be applied when a weak gamma ray occurs in a decay scheme as an alternate decay mode to two strong cascade gamma rays with energies that total to that of the weak gamma ray (43). The correction is over 5 % for the 0.40-MeV gamma ray of ^{75}Se when a source is counted 10 cm from a 65-cm³ detector. Other common radionuclides with similar-type decay schemes, however, do not require a correction of this magnitude. For example, ^{47}Ca (1.297 MeV), ^{59}Fe (1.292 MeV), ^{144}Pr (2.186 MeV), ^{187}W (0.686 MeV), and ^{175}Yb (0.396 MeV) require corrections between 0.990 and 0.998 when counted at 4 cm from a 65-cm³ detector.

12.4 Correction for Decay During the Counting Period:

12.4.1 If the value of a full-energy peak counting rate is determined by a measurement that spans a significant fraction of a half-life, and the value is assigned to the beginning of the counting period, a multiplicative correction, F_b , must be applied,

$$F_b = \frac{\lambda t}{1 - e^{-\lambda t}} \quad (14)$$

where:

- F_b = decay during count correction (count rate referenced to beginning of counting period),
- t = elapsed counting time,
- λ = radionuclide decay constant $\left(\frac{\ln 2}{T_{1/2}}\right)$, and,
- $T_{1/2}$ = radionuclide half-life.

t and $T_{1/2}$ must be in the same units of time ($F_b = 1.01$ for $t/T_{1/2} = 0.03$).

12.4.2 If under the same conditions the counting rate is assigned to the midpoint of the counting period, the multiplicative correction F_m will be essentially 1 for $t/T_{1/2} = 0.03$ and 0.995 for $t/T_{1/2} = 0.5$. If it need be applied, the correction to be used is:

$$F_m = \frac{\lambda t}{1 - e^{-\lambda t}} e^{-\frac{\lambda t}{2}} \quad (15)$$

12.5 *Efficiency Versus Energy Function or Curve*—The expression or curve showing the variation of efficiency with energy (see Fig. 2 for an example) must be determined for a particular detector (15-23), and must be checked for changes with time as specified in the standard. If the full energy range covered by this standard is to be used, calibrations should be made at least every 0.1 MeV from 0.06 to 0.30 MeV, about every 0.2 MeV from 0.3 MeV to 1.4 MeV, and at least at one energy between 1.4 MeV and 2 MeV. Radionuclides emitting two or more gamma rays with well-established relative gamma-ray probabilities may be used to better define the form of the calibration curve or function. A calibration with the same radionuclides that are to be measured should be made whenever possible and may provide the only reliable calibration when a radionuclide with cascade gamma rays is measured very close to the detector.

12.6 *Source Geometry*—A gamma ray undergoing even small-angle scattering is lost from the narrow full-energy peak, making the full-energy peak efficiency sensitive to the source or container thickness and composition. For most accurate results, the source to be measured must duplicate, as closely as possible, the calibration standards in all aspects (for example, shape, physical, and chemical characteristics, etc.). If this is not practicable, appropriate corrections must be determined and applied.

12.6.1 If the source shape and detector distance remain constant, changes in composition are corrected as follows:

$$A_c = A_o \frac{\mu x}{1 - e^{-\mu x}} \quad (16)$$

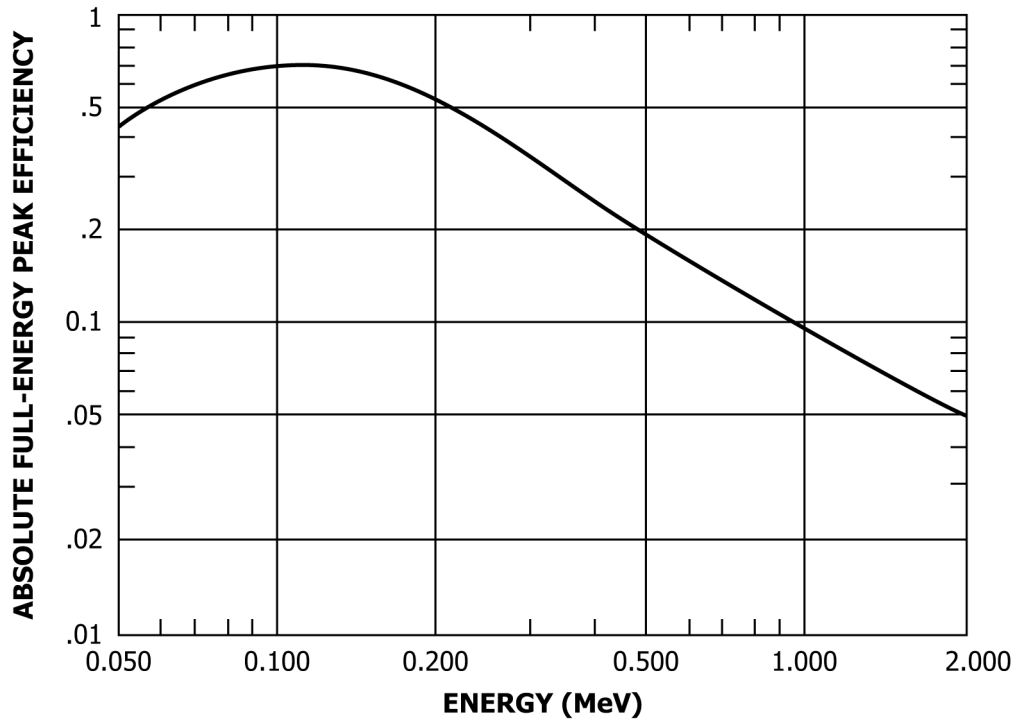


FIG. 2 Typical Efficiency Versus Energy for a Germanium Detector

where:

A_c = corrected number of nuclear decays per second,

A_o = observed number of nuclear decays per second,

μ = $\frac{\text{cm}^2}{\text{g}}$ = mass attenuation coefficient (44), and

x = $\frac{1}{\text{cm}^3} \cdot \text{cm}$ = mass times path length divided by volume.

12.6.2 If the source shape, composition, and detector distance remain constant, the attenuation of an interspersed absorber are corrected as follows:

$$A_c = A_o \cdot e^{\mu x} \quad (17)$$

12.6.3 Distribution of the radioactive constituents in the sample must be the same as in the calibration standard. Care shall be taken to avoid deposition of source material on the surfaces of the sample container. For multiphase samples, care shall be taken to control the distribution of radiation among the phases (for example, by shaking just prior to counting). For liquid solutions containing suspended material, filtration of the sample and separate counting of the filtrate and suspended activities may be necessary. The use of liquid calibration standards is discouraged. If their use is necessary, they should be used immediately after preparation and disposed to waste.

CALIBRATION AND USAGE OF SCINTILLATION DETECTOR SYSTEMS

13. Scope

13.1 This method establishes methods for calibration, usage, and performance testing of scintillation detector systems, for example, sodium iodide (thallium activated) [NaI(Tl)]. Scintillation detector systems are used for the measurement of gamma-ray emission rates of radionuclides, the assay for radioactivity, and the determination of gamma-ray energies. The method covers both energy calibration and efficiency calibration. The following two techniques are considered:

13.2 *Multichannel Analyzer Counting for Simple Spectra* (see Section 16) This technique applies to measurements that do not involve overlapping peaks and those for which the continuum under the full-energy peak can be subtracted without introducing unacceptable error (38). This technique applies to total spectrum counting and single-channel analyzer counting.

13.3 *Multichannel Analysis Counting for Complex Spectra* (see Section 15) This technique applies to measurements that involve multiple nuclides, overlapping peaks, and those for which the continuum under the full-energy peak cannot be subtracted without introducing unacceptable error (45).

13.4 The theory of operation of sodium iodide detectors is presented in numerous publications, including Refs (45-47).

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14. Apparatus

14.1 A typical spectrometry system consists of a scintillating medium; for example, NaI(Tl), one or more photomultipliers, optically coupled to the scintillator, a photomultiplier power supply, detector preamplifier, linear amplifier, multichannel analyzer, and data readout device, for example, a printer, plotter, oscilloscope, or computer. Ionizing radiation interacts with the detector to produce a flash of light, the photomultipliers convert the light flash to an amplified electrical impulse, and the supportive electronics analyze and count the pulses.

15. Preparation of Apparatus

15.1 Follow the manufacturer's instructions for setting up and preliminary testing of the equipment. Observe all the manufacturer's limitations and cautions. All preparations in Section 19 should be observed during calibration and sample analysis, and all corrections shall be made when required. A check source should be used to check the stability of the system at least before and after calibration.

16. Multichannel Analyzer (MCA) Counting for Simple Spectra

16.1 Summary of Method:

16.1.1 The purpose of this method is to provide a standardized basis for the calibration, usage, and performance testing of scintillation detector systems for measurement of gamma-ray emission rates of single nuclides or from simple mixtures of nuclides that do not involve overlapping peaks.

16.1.2 The source emission rate for a gamma ray of a selected energy is determined from the counting rate in a full-energy peak of a spectrum, together with the measured efficiency of the spectrometry system for that energy and source location. It is usually not possible to measure the efficiency directly with emission rate standards at all desired energies. Therefore, a curve or function is constructed to permit interpolation between available calibration points.

16.2 *Energy Calibration*—Establish the energy calibration of the system over the desired energy region at fixed gain. Using known sources, record a spectrum containing full-energy peaks which span the gamma-ray energy region of interest. Determine the channel numbers which correspond to two gamma-ray energies that are near the extremes of the energy region of interest. From these data determine the slope and the intercept of the energy calibration curve. For most applications such a linear energy calibration curve will be adequate. Determine nonlinearity correction factors if necessary (45, 46). The energy calibration shall be determined for each amplifier gain or photomultiplier high-voltage setting used.

16.3 Full-Energy-Peak Efficiency Calibration (see section 16.12):

16.3.1 Accumulate gamma-ray spectra using radioactivity standard sources in a desired and reproducible counting geometry (see 19.7). At least 10 000 net counts should be accumulated in full-energy gamma-ray peaks of interest (see 19.6 and 19.8).

16.3.2 Record the live time counting interval (see 19.6, 19.9, and 19.13).

16.3.3 For each radioactivity standard source determine the net counts in the full-energy gamma-ray peaks of interest (see 19.14).

16.3.4 Correct the radioactivity standard source gamma-ray emission rate for decay from the time of standardization to the time at which the count rate is measured (see 19.10).

16.3.5 Calculate the full-energy peak efficiency, E_f , as follows:

$$E_f = \frac{N_p}{N_\gamma} \quad (18)$$

where:

E_f = full-energy peak efficiency (counts per gamma ray emitted),

N_p = net gamma-ray count in the full-energy peak (counts per second live time) (see 17.3.3), and

N_γ = gamma-ray emission rate (gamma rays per second) (see **Note 3**).

If the standard source is calibrated in units of becquerels, the gamma-ray emission rate is given as follows:

$$N_\gamma = AP_\gamma \quad (19)$$

where:

A = number of nuclear decays per second, and

P_γ = probability per nuclear decay for the gamma ray (**7-14**).

16.3.6 To obtain full-energy peak efficiency calibration data at energies for which radioactivity standards are not available, plot or fit to an appropriate mathematical function the values for the full-energy peak efficiency (from **16.3.5**) versus gamma-ray energy (**38, 45, 46**) (see **19.12**). (See **Fig. 1** for an example.)

16.4 Activity Determination:

16.4.1 Using the instrument settings of **16.3**, place the sample to be measured in the same counting geometry that was used for the efficiency calibration (see **20.7** and **20.11**).

16.4.2 Accumulate enough counts in the gamma-ray spectrum to obtain the desired statistical level of confidence (see **19.6** and **19.8**).

16.4.3 Record the live time counting interval (see **19.9** and **19.13**).

16.4.4 Determine the energy of the gamma rays present by the use of the energy calibration data obtained according to **16.2**.

16.4.5 Obtain the net count rate in each full-energy gamma-ray peak of interest (see **19.10** and **19.14**).

16.4.6 Determine the full energy peak efficiency for each energy of interest from **16.3.5** or from the curve or function derived in **16.3.6** (see **19.12** and **19.13**).

16.4.7 Calculate the number of gamma rays emitted per unit live time for each full-energy peak as follows:

$$N_\gamma = \frac{N_p}{E_f} \quad (20)$$

When calculating a nuclear transmutation rate from a gamma-ray emission rate determined for a specific radionuclide, a knowledge of the gamma-ray probability per decay is required (**7-14**), that is,

$$A = \frac{N_\gamma}{P_\gamma} \quad (21)$$

16.5 *Single-Channel Analyzer (SCA) Counting System*—Calibration and assay with an SCA counting system are the same as for MCA counting for simple spectra (see **16.2, 16.3** and **16.4**) with the following variations:

16.5.1 *Energy Calibration*—Following the manufacturer's directions, or using a multichannel analyzer to observe the gamma-ray spectrum, or using an oscilloscope to observe the pulse height at the amplifier output, establish the approximate desired output range of the system. This may be done using either a pulse generator or gamma-ray sources. Establish the energy calibration of the system over the desired energy region at a fixed gain. Using known sources, determine the relationship between the gamma-ray energies and the corresponding settings of the upper level and lower level discriminators. Measure the count rate as a function of the lower level discriminator setting at gamma-ray energy increments of not more than 0.025 MeV, spanning the energy range of interest. (Window widths of less than the 0.025 MeV, for example, 2 % of full range, might be more appropriate when radionuclides emitting low-energy gamma rays are to be assayed.) For practical purposes, the center of the window position corresponding to the highest count rate may be assumed to be the center of the full-energy peak. The energy calibration shall be determined for each amplifier gain or photomultiplier high-voltage setting used. For best results, radionuclides for which assays will be performed should be used for the energy calibration. If not practical, radionuclides with gamma rays that span the energy region of interest shall be used (see **20.5** and **20.6**).

16.5.2 *Full-Energy-Peak Efficiency Calibration*—Set the lower level and upper level discriminators such that:

16.5.2.1 The window width corresponds to approximately three times the FWHM.

16.5.2.2 The lower level discriminator is set at the minimum just lower in energy than the photopeak of interest.

16.5.3 *Activity Determination* (see **19.1, 19.2**, and **20.3**). Using the instrument setting of **16.5.2**, place the sample to be measured in the same counting geometry that was used for the efficiency calibration (see **19.7** and **19.11**).

16.6 Total spectrum counting is valid only for single nuclide sample activity determinations (see **19.1, 19.2**, and **20.3**). Calibration and assay with a total spectrum counting system is the same as for SCA counting (see **16.5**) except that the entire standard or sample spectrum is the peak of interest. No full-energy peak efficiency calibration (see **16.3**) is performed. Standard total spectrum counts are ratioed directly to sample spectrum counts acquired with the same gain and low-level discriminator settings.

16.6.1 All Section **19** precautions apply.

16.6.2 Obtain the net count rate for the standard and for the sample by subtracting the ambient background count rate from the total count rates (see **19.10**).

16.6.3 Calculate the activity of the sample by: