



Designation: E181 – 23

# Standard Guide for Detector Calibration and Analysis of Radionuclides in Radiation Metrology for Reactor Dosimetry<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This guide covers general procedures for the calibration of radiation detectors and measurement for radiation metrology for reactor dosimetry. For any particular radionuclide, one or more of these methods may apply.

1.2 These techniques 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 E3376, Standard Practice for Calibration and Usage of Germanium Detectors in Radiation Metrology for Reactor Dosimetry, was previously in Guide E181 and is now found in Volume 12.02 of the *Annual Book of ASTM Standards*. The discussion herein is not a sufficient substitute for the full standard. This guide is specifically NOT to be used as a direct reference to Practice E3376. Only the standard listed provides sufficient information to serve as a reference.

1.4 Additional information on the setup, 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 concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental 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.*

<sup>1</sup> This guide is under the jurisdiction of ASTM Committee E10 on Nuclear Technology and Applications and is the direct responsibility of Subcommittee E10.05 on Nuclear Radiation Metrology.

Current edition approved Feb. 15, 2023. Published April 2023. Originally approved in 1961. Last previous edition approved in 2017 as E181 – 17. DOI: 10.1520/E0181-23.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

C1402 Guide for High-Resolution Gamma-Ray Spectrometry of Soil Samples

D7282 Practice for Setup, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements

E170 Terminology Relating to Radiation Measurements and Dosimetry

E3376 Standard Practice for Calibration and Usage of Germanium Detectors in Radiation Metrology for Reactor Dosimetry

## 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 (NIST).<sup>3</sup>

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

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

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

3.1.6 *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.

<sup>2</sup> 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.

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

3.1.7 *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**.

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. **E3376** Standard Practice for Calibration and Usage of Germanium Detectors in Radiation Metrology for Reactor Dosimetry

4.1 *Scope*—This standard establishes techniques for calibration, usage, and performance testing of germanium detectors for the measurement of gamma-ray emission rates of radionuclides in radiation metrology for reactor dosimetry. The practice is applicable only to samples of small size, approximating to point sources. It covers the energy and full-energy peak efficiency calibration as well as the determination of gamma-ray energies in the 0.06 MeV to 2 MeV energy region and is designed to yield gamma-ray emission rates with an uncertainty of  $\pm 3\%$ . This technique applies to measurements that do not involve overlapping peaks, and in which peak-to-continuum considerations are not important.

4.2 *Use*—This practice replaces content in E181–17 relevant to the use of germanium detectors in gamma spectroscopy.

4.3 *Summary*—This practice describes the calibration and usage of germanium detectors for measurement of gamma-ray emission rates of radionuclides in radiation metrology for nuclear reactors. The practice is intended for use by knowledgeable persons who are responsible for the development of correct procedures for the calibration and usage of germanium detectors.

## CALIBRATION AND USAGE OF SCINTILLATION DETECTOR SYSTEMS

### 5. Scope

5.1 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:

5.1.1 *Multichannel Analyzer Counting for Simple Spectra* (see Section 8)—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 (2). This technique applies to total spectrum counting and single-channel analyzer counting.

5.1.2 *Multichannel Analysis Counting for Complex Spectra* (see Section 9)—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 (3).

5.2 The theory of operation of sodium iodide detectors is presented in numerous publications, including Refs (3–5).

## 6. Apparatus

6.1 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.

## 7. Preparation of Apparatus

7.1 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 11 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.

## 8. Multichannel Analyzer (MCA) Counting for Simple Spectra

### 8.1 Summary of Method: 19de71/astm-e181-23

8.1.1 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.

8.1.2 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.

8.2 *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 (3, 4). The energy

calibration shall be determined for each amplifier gain or photomultiplier high-voltage setting used.

### 8.3 Full-Energy Peak Efficiency Calibration:

8.3.1 Accumulate gamma-ray spectra using radioactivity standard sources in a desired and reproducible counting geometry (see 11.7). At least 10 000 net counts should be accumulated in full-energy gamma-ray peaks of interest (see 11.6 and 11.8).

8.3.2 Record the live time counting interval (see 11.6, 11.9, and 11.13).

8.3.3 For each radioactivity standard source, determine the net counts in the full-energy gamma-ray peaks of interest (see 11.14).

8.3.4 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 11.10).

8.3.5 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) (see 9.3.3), and

$N_\gamma$  = gamma-ray emission rate (gamma rays per second).

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

$$N_\gamma = AP_\gamma \quad (2)$$

where:

$A$  = number of nuclear decays per second, and  
 $P_\gamma$  = probability per nuclear decay for the gamma ray (6-13).

8.3.6 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 8.3.5) versus gamma-ray energy (2-4) (see 11.12).

### 8.4 Activity Determination:

8.4.1 Using the instrument settings of 8.3, place the sample to be measured in the same counting geometry that was used for the efficiency calibration (see 11.7 and 11.11).

8.4.2 Accumulate enough counts in the gamma-ray spectrum to obtain the desired statistical level of confidence (see 11.6 and 11.8).

8.4.3 Record the live time counting interval (see 11.9 and 11.13).

8.4.4 Determine the energy of the gamma rays present by the use of the energy calibration data obtained according to 8.2.

8.4.5 Obtain the net count rate in each full-energy gamma-ray peak of interest (see 11.10 and 11.14).

8.4.6 Determine the full-energy peak efficiency for each energy of interest from 8.3.5 or from the curve or function derived in 8.3.6 (see 11.12 and 11.13).

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

$$N_\gamma = \frac{N_p}{E_f} \quad (3)$$

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 (6-13), that is,

$$A = \frac{N_\gamma}{P_\gamma} \quad (4)$$

8.5 *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 8.2, 8.3, and 8.4) with the following variations:

8.5.1 *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.

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

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

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

8.5.3 *Activity Determination* (see 11.1, 11.2, and 11.3).

Using the instrument setting of 8.5.2, place the sample to be measured in the same counting geometry that was used for the efficiency calibration (see 11.7 and 11.11).

8.6 Total spectrum counting is valid only for single nuclide sample activity determinations (see 11.1, 11.2, and 11.3). Calibration and assay with a total spectrum counting system is the same as for SCA counting (see 8.5) except that the entire standard or sample spectrum is the peak of interest. No full-energy peak efficiency calibration (see 8.3) is performed. Standard total spectrum counts are ratioed directly to sample spectrum counts acquired with the same gain and low-level discriminator settings.

8.6.1 All Section 11 precautions apply.

8.6.2 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 11.10).

8.6.3 Calculate the activity of the sample by:

$$A = \frac{C}{R} \quad (5)$$

where:

$C$  = the net sample count rate (8.6.2), and

$R$  = the net standard count rate (8.6.2) divided by the time-corrected (8.3.4) standard activity (see 11.1, 11.2, and 11.3).

## 9. Multichannel Analyzer (MCA) Counting for Complex Spectra

### 9.1 Summary of Method:

9.1.1 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 emissions rates of mixtures of nuclides. This method is intended for use by knowledgeable persons who are responsible for the development of correct procedures for the calibration and usage of scintillation detectors.

9.1.2 Matrix inversion (14) of a matrix of full-energy peaks and their contribution to the energy range of other nuclide full-energy peaks can be performed on calculators, with or without memory storage. However, computer data reduction is easier and iterative solutions are possible. Single nuclide standard spectra are acquired and normalized to one standard unit of activity, for example, 1 Becquerel, Bq. Fixed whole channel ranges are assigned to represent each nuclide. A matrix of nuclide channel range count rate ratios is prepared and inverted. The representative nuclide channel range count rates are multiplied by the selected inverted matrix vectors to determine nuclide activities in the sample.

9.1.3 Linear least-squares resolution of gamma spectra can only be performed with the aid of a computer (15, 16). Single nuclide standard spectra are acquired. Linear least-squares fitting of selected standard spectra to the sample spectrum is performed to minimize residuals.

9.1.4 Neither the matrix inversion nor the linear least-squares method utilizes an efficiency curve or function. However, an efficiency curve or function is useful in determining the activity of an uncalibrated standard nuclide spectrum. To perform a full-energy peak efficiency calibration, perform 8.2 and 8.3 (see 11.12).

9.2 Energy Calibration (same as 8.2).

### 9.3 Matrix Inversion Method—Activity Calibration:

9.3.1 Accumulate gamma-ray spectra using single radioactivity standard sources in a desired and reproducible counting geometry (see 11.7). At least 10 000 net counts should be accumulated in full-energy gamma-ray peaks of interest (see 11.6 and 11.15).

9.3.2 Record the live time counting interval (see 11.6 and 11.9).

9.3.3 Determine the ambient background spectrum for each detector/geometry (see 11.7) using a blank if appropriate. The

ambient background may be used as a single-nuclide radioactivity standard in the determination of sample activity or stripped from radioactivity standard source spectra (see 9.3.1) using the ratio of live time counting intervals as the normalization factor.

9.3.4 Correct the radioactivity standard source activity to the time at which the standard spectrum is acquired (see 11.10).

9.3.5 Assign identification codes or numbers to all photopeaks of interest. Assign integer numbers of channels to represent photopeak areas.

9.3.6 To calibrate, divide the peak areas of the radioactivity standard source spectra desired in 9.3.5 by the decay-corrected standard activities derived in 9.3.4, for example, counts/second·Becquerel.

9.3.7 Calculate contribution ratios for a coefficient matrix  $a_{ij}$  (11).

where:

$i$  = representative photopeak code of the photopeak area receiving the contribution,

$j$  = representative photopeak code of the radionuclide providing the contribution. This radionuclide spectrum is the spectrum from which contribution ratios are calculated, and

$$a_{ij} = \frac{(\text{counts per second}) \text{ area } i}{(\text{counts per second}) \text{ area } j}$$

For example, if the code for the 1.332 MeV peak of  $^{60}\text{Co}$  is 3, and the code for the 0.662 MeV peak of  $^{137}\text{Cs}$  is 6, then the matrix elements will be as follows:

$$\begin{aligned} a_{33} &= 1, \\ a_{36} &= \sim 0.4, \\ a_{66} &= 1, \text{ and} \\ a_{63} &= 0. \end{aligned}$$

### 9.4 Matrix Inversion Method—Sample Activity Determination:

9.4.1 Place the sample to be measured at the source-to-detector distance used for activity calibration (see 9.3.1).

9.4.2 Accumulate the gamma-ray spectrum for sufficient time to obtain the desired statistical level of confidence (see 11.6 and 11.8).

9.4.3 Record the live time counting interval (see 11.9).

9.4.4 If  $C_i$  equals the total area sum of the components in counts per second present in the representative photopeak areas (see 9.3.5) in a sample spectrum, and if  $X_j$  equals the photopeak area of the nuclide component to be determined, then:

$$C_i = \sum_{j=1}^k a_{ij} X_j \quad (6)$$

The total photopeak area  $C_i$  is the sum of the contributing parts having  $k$  components. The system of linear equations representing  $k$  nuclides is as follows:

$$a_{11} X_1 + a_{12} X_2 + a_{13} X_3 + \dots + a_{1k} X_k = C_1 \quad (7)$$

The series of linear equations may be written in the matrix form:  $AX^t = C^t$

where:

$A$  = the  $a_{ij}$  coefficient matrix (see 9.3.7),

$X^t$  = the transposed vector of unknown representative photopeak areas due to photopeaks  $j = 1, 2, 3 \dots k$ , and  
 $C^t$  = the transposed vector of total representative areas from the sample spectrum.

9.4.5 The solution to the equation  $AX^t = C^t$  is  $X^t = A^{-1}C^t$  where  $A^{-1}$  is the inverse of matrix  $A$ . Note that  $i$  and  $j$  vectors representing photopeaks of nuclides not present in the sample spectrum are eliminated from the larger matrices  $A$  and  $A^{-1}$  (see 9.3.3).

9.4.6 The sample nuclide activity equals  $X^t$  divided by the calibration factor (see 9.3.6).

$$\text{Bq}_j = \frac{(c/s)_j}{(c/s \cdot \text{Bq})_j} \quad (8)$$

### 9.5 Linear Least-Squares Method—Activity Calibration:

9.5.1 Accumulate gamma-ray spectra using single radioactivity standard sources in a desired and reproducible counting geometry (see 11.7). At least 10 000 net counts should be accumulated in the full-energy gamma-ray peaks of interest (see 11.6 and 11.15).

9.5.2 Record the live time counting interval (see 11.6 and 11.9).

9.5.3 Determine the ambient background spectrum for each detector/geometry (see 11.7) using a blank, if appropriate. The ambient background spectrum shall be treated as a single nuclide, radioactivity standard in the determination of sample activity and shall be stripped from all single radioactivity standard source spectra (see 9.5.1) using the ratio of live time counting intervals as the normalization factor.

9.5.4 Correct the radioactivity standard source activity to the time at which the standard spectrum is acquired (see 11.10).

9.5.5 The resolution of a gamma spectrum into the concentrations of its component radionuclides can be treated as a curve-fitting problem by using least-squares techniques. The basic assumption is that the sample spectrum can be described by a linear combination of the gamma spectra of each component obtained separately. This discussion is intended to present the least-squares approach in nonmathematical terms (5-16). The linear least-squares method assumes that the pulse-height spectrum to be analyzed consists of the summed contributions of  $n$  nuclides, each of which is represented as a pulse-height spectrum of  $k$  channels (see 11.15). This method requires standard spectra representing the response of the detector to gamma rays of the nuclides of interest (for comparison, see 9.5.1, 9.5.2, and 9.5.3). The count rate in a sample spectrum due to standard  $j$  ( $j = 1 \dots n$ ) in channel  $i$  ( $i = 1 \dots k$ ) will be  $C_{ij}$  and the total count rate in channel  $i$  will be  $X_i$ . The expression:

$$X_i = (C_{i1} + C_{i2} + C_{i3} + \dots) = \sum_{j=1}^n C_{ij} \quad (9)$$

accounts for all contributions to channel  $i$ .

9.5.5.1 To obtain quantitative results from resolving a spectrum, the quantity of nuclide  $j$  must be expressed in terms of the standard for nuclide  $j$ . Therefore, a normalization factor  $M_j$ , the ratio of the activity of nuclide  $j$  in the unknown to the value of nuclide  $j$  in the standard, must be included (see 9.5.4):

$$X_i = \sum_{j=1}^n M_j S_{ij} + R_i \quad (10)$$

where  $R_i$  represents the random uncertainty in the channel  $i$  counts and  $S_{ij}$  is the count rate of the standard  $j$  in channel  $i$ .  $C_{ij}$  is simply the product of  $M_j$ , the normalization factor, and  $S_{ij}$ , the standard count rate.

9.6 Linear Least-Squares Method—Sample Activity Determination—If the only uncertainty in this calculation is the random uncertainty of the counts in a channel,  $R_i$  (see 8.5.3), then the least-squares technique can be used. This method estimates the parameters that minimize the weighted sum of the squared difference between two sets of values. The usual case has one set of values as observed data ( $X_i$ ) and another set of computed values:

$$\left( \sum_{j=1}^n M_j S_{ij} \right) \quad (11)$$

This translates to:

$$\text{Minimize} \left( X_i - \sum_{j=1}^n M_j S_{ij} \right)^2 W_i \quad (12)$$

where  $W_i$  is the weighing factor chosen to estimate the variance of the counts in a channel. If the variance is estimated for each channel, the result is a set of linear simultaneous equations (one for each nuclide of interest) that may be solved for the values of  $M_j$ . This solution is most easily derived by using matrix techniques on a computer.

9.6.1 The sample nuclide activity equals the derived sample count rate divided by the standard calibration factor:

$$\text{Bq} = \frac{(c/s)}{(c/s \cdot \text{Bq})_j} \quad (13)$$

## 10. Performance Testing

10.1 The system energy calibration shall be checked on each day of use with one or more check sources in the energy region of interest.

10.2 The system count rate reproducibility for at least one long-lived radionuclide check source shall be checked on each day of use. Correction for radioactive decay of the source since the original measurement shall be applied if more than 1 % of a half-life has expired.

10.3 The efficiency calibration shall be checked at least semi-annually by using radioactivity standard sources of radionuclides with energies that span the energy region of interest.

10.4 The ambient background of the system shall be measured at least once a week. The ambient background should be checked at the beginning and ending of each day's counting. For best results the ambient background should be measured before and after each batch of samples.

10.5 The resolution of the system shall be determined at the time of initial installation and should be checked at least monthly.

10.6 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.

## 11. Precautions

11.1 *Assay for a Radionuclide for Which No Radioactivity Standard Is Commercially Available*—A total-spectrum counting system or a single-channel analyzer counting system shall not be used for quantitative determinations of radionuclides for which radioactivity standards are not commercially available. Multichannel counting systems shall be used in such cases.

11.2 *Determination of Gross Gamma Activity*—The usage of the gross gamma activity of a sample containing more than one gamma-emitting radionuclide as a quantitative tool is not an acceptable practice. Relating of the gross gamma activity of a sample containing more than one gamma-emitting radionuclide to absolute quantities of specific radionuclides has no validity.

11.3 *Assay of Mixtures of Radionuclides*—A total-spectrum counting system or a single-channel analyzer counting system shall not be used for attempted quantification of the radionuclides contained within a mixture.

11.4 *Thin-Window Detectors*—When working with a thin-window detector, one must be cautious about radionuclides emitting conversion electrons which have energies close to that of the gamma ray of interest. To avoid counting the conversion electrons in such detectors, insert a sufficient amount of absorbing material between the source and the detector.

11.5 *Simulated Sources*—Simulated sources shall not be used for energy calibration or efficiency calibration of scintillation detector systems. Such sources may be used for checking the system count-rate reproducibility.

11.6 *High Count Rates*—It is recommended that count rates be limited to less than 5000 counts per second at the amplifier output. Random photon summing correction should not be necessary if this recommendation is employed with amplifier time constants of less than 5  $\mu$ s.

11.7 *Geometrical Positioning*—The dependence of the measurement on the geometrical configuration and composition of the sample container shall be taken into consideration in the calibration procedure. Positioning of sample containers within detector wells usually provides good positional reproducibility. Positioning of sample containers on or above the surface of detectors requires a method for reproducing the position. New calibrations shall be obtained for assaying for radionuclides in containers of different sizes or shapes.

11.8 *Counting Statistics*—The recommendation of 10 000 net counts is made for measurements of activities which are not near the lower limit of detectability (LLD). For measurements of activities near LLD see Refs (17, 18).

11.9 *Dead-Time Corrections*—For a number of systems there is internal dead-time compensation. However, for those systems that have no such compensation, the dead-time-corrected count rate  $N_o$  is given by:

$$N_o = \frac{N}{1 - Nt_d} \quad (14)$$

where  $N$  is the observed count rate and  $t_d$  is the dead time which can be experimentally determined, as described below, using the so-called “two-source method.” This gives, for example, a correction of 1 % for a dead time of 10  $\mu$ s and a count rate of 1000  $s^{-1}$ . In this method three measurements are taken, first of a source (say, source 1), second of source 1 and another source (source 2), and finally of source 2 alone. From these measurements, the respective count rates  $N_1$ ,  $N_{12}$ , and  $N_2$  are obtained, and one is able to write three equations:

$$N_{1,0} = \frac{N_1}{1 - N_1 t_d} \quad (15)$$

$$N_{1,0} + N_{2,0} = \frac{N_{12}}{1 - N_{12} t_d} \quad (16)$$

$$N_{2,0} = \frac{N_2}{1 - N_2 t_d} \quad (17)$$

where  $N_{1,0}$  and  $N_{2,0}$  are the respective dead-time-corrected count rates for sources 1 and 2. The dead time  $t_d$  is determined using the condition that the sum of  $N_{1,0}$  and  $N_{2,0}$ , obtained from Eq 15 and Eq 17, is equal to that obtained from Eq 16:

$$t_d = \frac{1 - \left[ 1 - \frac{N_{12}}{N_1 N_2} (N_1 + N_2 - N_{12}) \right]^{1/2}}{N_{12}} \quad (18)$$

When making the measurements, it is important not to disturb source 1 when introducing source 2, and similarly, when removing source 1, not to disturb source 2. For multichannel analyzer systems, the “live-time” feature is designed to compensate for counting time lost during pulse processing, and a further correction for dead-time losses is usually not required.

### 11.10 Correction for Decay During the Counting Period:

11.10.1 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:

$$F_b = \frac{\lambda t}{1 - e^{-\lambda t}} \quad (19)$$

where:

$F_b$  = decay during count correction (count rate referenced to beginning of counting period),

$t$  = elapsed counting time,

$\lambda$  = 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  $t/T_{1/2} = 0.03$ ).

11.10.2 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:

$$F_m = \frac{\lambda t}{1 - e^{-\lambda t}} e^{-\frac{\lambda t}{2}} \quad (20)$$

11.11 *Counting Geometry*—The source to be measured shall duplicate, as closely as possible, the calibration standards in all aspects (such as shape, physical and chemical characteristics,

homogeneity, etc). The source-to-detector relationship shall be the same for source and standard. Care shall be taken to avoid deposition of source material on the surfaces of the sample container. For multiphase samples, such as radon in radium solution and krypton in saline solution, care shall be taken to carefully control the partitioning of the radioactivity between the gaseous and liquid phases (for example, by shaking just prior to counting).

**11.12 Full-Energy Peak Efficiency versus Energy Function or Curve**—The expression or curve showing the variation of the full-energy peak efficiency with energy shall be determined for a particular detector and shall be checked for changes with time as specified in this standard (see 8.3). There shall be a minimum of three calibration points, approximately evenly spaced, spanning the energy region of interest below 0.300 MeV. Above 0.300 MeV, calibration points shall be obtained approximately every 0.250 MeV, spanning the energy region of interest. Full-energy peak efficiency calibrations below 0.100 MeV should be determined using a radioactivity standard of the radionuclide to be measured. A full-energy peak efficiency calibration using the same radionuclides that are to be measured should be made whenever possible and may provide the only reliable full-energy peak efficiency calibration when a radionuclide with cascade gamma rays is measured.

**11.13 Correlated Photon Summing Correction:**

**11.13.1** 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:

$$C = \frac{1}{\prod_i^n (1 - q_i \varepsilon_i)} \quad (21)$$

where: [s://standards.iteh.ai/catalog/standards/sist/1abfd308-1ec0-4560-8151-181-23](https://standards.iteh.ai/catalog/standards/sist/1abfd308-1ec0-4560-8151-181-23)

- $C$  = 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
- $\varepsilon_i$  = total detection efficiency of  $i$ th correlated photon.

Correlated summing correction factors for the primary gamma rays of radionuclides  $^{60}\text{Co}$ ,  $^{88}\text{Y}$ , and  $^{46}\text{Sc}$  are approximately 1.09 and 1.03 for a 65 cm<sup>3</sup> detector at 1 cm and at 4 cm sample-to-detector distances, respectively, and approximately 1.01 for a 100 cm<sup>3</sup> detector at a 10 cm sample-to-detector distance. The  $q_i$  must be obtained from the nuclear decay scheme, while the  $\varepsilon_i$ , which are slowly varying functions of the energy, can be measured or calculated (19-21).

**11.13.2** 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 (22). The correction is over 5 % for the 0.40 MeV gamma ray of  $^{75}\text{Se}$  when a source is counted 10 cm from a 65 cm<sup>3</sup> detector. Other common radionuclides with similar-type decay schemes, however, do

not require a correction of this magnitude. For example,  $^{47}\text{Ca}$  (1.297 MeV),  $^{59}\text{Fe}$  (1.292 MeV),  $^{144}\text{Pr}$  (2.186 MeV),  $^{187}\text{W}$  (0.686 MeV), and  $^{175}\text{Yb}$  (0.396 MeV) require corrections between 0.990 and 0.998 when counted at 4 cm from a 65 cm<sup>3</sup> detector.

**11.14 Net Count Rate**—When using multichannel analyzer systems, the appropriate continuum in the region of interest shall be subtracted from the ambient background spectrum and from the sample spectrum. The difference of those two results is the net sample count in the full-energy peak.

**11.15 Comparative Standard Spectra**—When using multichannel analyzer counting methods for complex spectra, the standard spectra all must have identical energy gains and intercepts in order to be additive. Use of a check source (see 10.1) shall be used before and after the acquisition of every standard spectrum and those spectra exhibiting shifts shall be discarded. Unless the computer program performs gain and intercept shifts, the same restrictions will apply to sample spectra.

## 12. Sources of Uncertainty

**12.1** Other than Poisson distribution uncertainties, the principal sources of random uncertainty (and typical magnitudes) in scintillation detector measurements are:

**12.1.1** 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  $\pm 3$  %);

**12.1.2** The reproducibility in determination of net full-energy peak counts (typically  $\pm 2$  %);

**12.1.3** The reproducibility of the positioning of the source relative to the detector and the source geometry (typically  $\pm 3$  %);

**12.1.4** The accuracy with which the full-energy peak efficiency at a given energy can be determined from the calibration curve or function (typically  $\pm 3$  %—applies to single nuclide samples only); and

**12.1.5** III-conditioned equations, those equations whose solutions are sensitive to very small alterations in coefficient values, can cause the program to produce invalid results. Certain combinations of nuclides having similar spectral shapes or overlapping peaks can cause such a problem. These instances are outside the context of this method.

**12.2** Possible sources of systematic uncertainty in scintillation detector measurements are listed below (see 11.11) (7):

**12.2.1** Scattering from the surroundings, including induced X-ray emissions from lead shielding.

**12.2.2** Summing of coincident Compton events to give spurious pulses in the region of a full-energy peak of interest.

**12.2.3** Iodine K X-ray escape for low-energy photon sources (2, 4).

**12.2.4** Variations in ambient radiation background (particularly for low-activity measurements).

**12.2.5** The presence of radionuclide impurities.

**12.2.6** Differences in attenuation due to differences in container wall thickness or material.