



Designation: E 181 – 98

## Standard Test Methods for Detector Calibration and Analysis of Radionuclides<sup>1</sup>

This standard is issued under the fixed designation E 181; 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 These 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 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:

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Spectroscopy Methods:	
Calibration and Usage of Germanium Detectors	3-12
Calibration and Usage of Scintillation Detector Systems:	13-20

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Calibration and Usage of Scintillation Detectors for Simple Spectra	16
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Counting Methods:	
Beta Particle Counting	25-26
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1.4 *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.*

### 2. Referenced Document

#### 2.1 ASTM Standards:

E 170 Terminology Relating to Radiation Measurements and Dosimetry<sup>2</sup>

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 12.02.

<sup>1</sup> These methods are under the jurisdiction of ASTM Committee E-10 on Nuclear Technology and Applications.

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### 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).<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 *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: 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 E 170.

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.

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

## 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,  $U = \sqrt{\sum \sigma_i^2 + 1/3 \sum \delta_i^2}$  where  $\delta_i$  are the estimated maximum systematic uncertainties, and  $\sigma_i$  are the random uncertainties at the 68 % confidence level (2). Other methods of error analysis are in use (3, 4).

### 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.

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 multipeaked 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

$N_\gamma$  = 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:

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

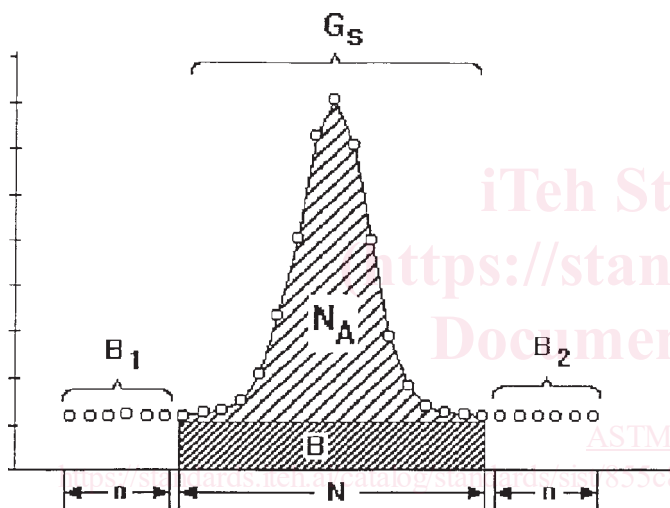
where  $T_s$  = spectrum live time.

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,<sup>4</sup>
- $B_{1s}$  = 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:

<sup>4</sup> 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.

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

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

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

where:

- $A$  = number of nuclear decays per second, and
- $P_\gamma$  = 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:

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

where:

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

and

- $S_{NA}$  = net peak area uncertainty (at 1 $\sigma$  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,  $S_{lb} = 0$ .

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.

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<sup>-1</sup> and the amplifier time constant is less than 5  $\mu$ s, this test need not be performed. Otherwise, accumulate a <sup>60</sup>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 <sup>60</sup>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 <sup>60</sup>Co source, introduce a <sup>57</sup>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 <sup>60</sup>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  $^{137}\text{Cs}$ ,  $^{60}\text{Co}$ , 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 %  $1\sigma$  (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:

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

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

$\epsilon_i$  = total detection efficiency of  $i$ th correlated photon.

Correlated summing correction factors for the primary gamma rays of radionuclides  $^{60}\text{Co}$ ,  $^{88}\text{Y}$ ,  $^{46}\text{Sc}$  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  $\epsilon_i$ , 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  $^{75}\text{Se}$  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.

### 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,

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

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.

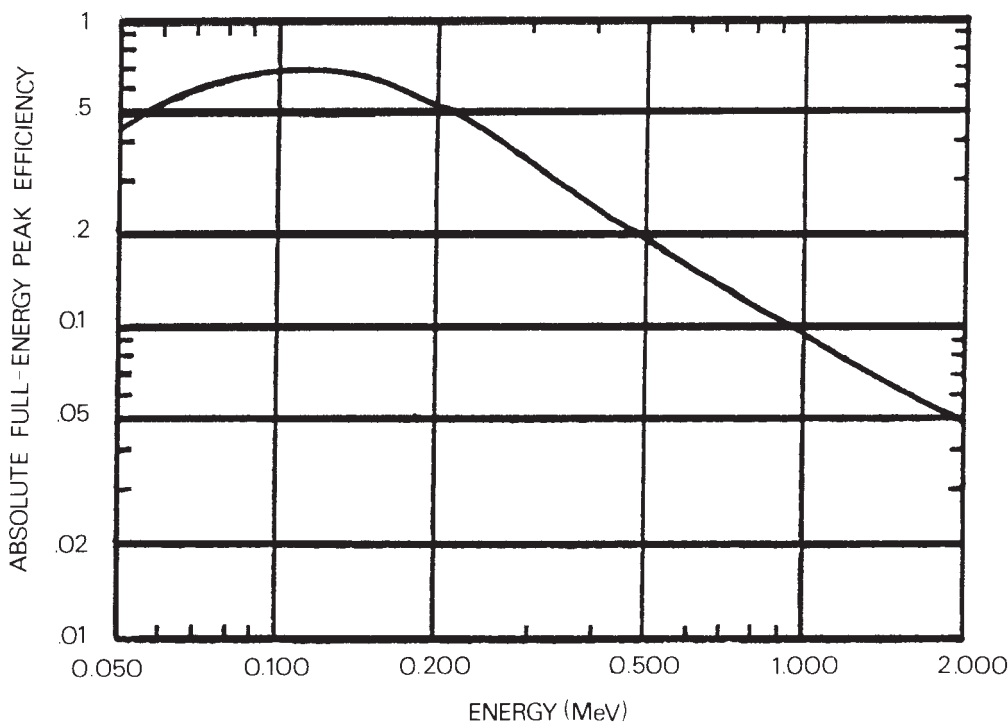


FIG. 2 Typical Efficiency Versus Energy for a Germanium Detector

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:

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

where:

$A_c$  = corrected number of nuclear decays per second,

$A_o$  = observed number of nuclear decays per second,

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

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

12.6.2 If the source shape, composition, and detector distance remain constant, the attenuation of an interspersed sorber 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).

## 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, multi-channel 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

$N_\gamma$  = 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

$P_\gamma$  = 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:

$$A = \frac{C}{R} \quad (22)$$

where:

$C$  = the net sample count rate (16.6.2), and

$R$  = the net standard count rate (16.6.2) divided by the time corrected (16.3.4) standard activity (see 19.1, 19.2, and 19.3).

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

### 17.1 Summary of Method:

17.1.1 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.

17.1.2 Matrix inversion (48) 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.

17.1.3 Linear least squares resolution of gamma spectra can only be performed with the aid of a computer (49, 50). Single nuclide standard spectra are acquired. Linear least squares fitting of selected standard spectra to the sample spectrum is performed to minimize residuals.

17.1.4 Neither the matrix inversion nor the linear least square methods utilize 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 16.2 and 16.3 (see 19.12).

17.2 *Energy Calibration* (same as 16.2).

17.3 *Matrix Inversion Method—Activity Calibration:*

17.3.1 Accumulate gamma-ray spectra using single 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 19.15).

17.3.2 Record the live time counting interval (see 19.6 and 19.9).

17.3.3 Determine the ambient background spectrum for each detector/geometry (see 19.7) 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 17.3.1) using the ratio of live time counting intervals as the normalization factor.

17.3.4 Correct the radioactivity standard source activity to the time at which the standard spectrum is acquired (see 19.10).



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

17.3.6 To calibrate, divide the peak areas of the radioactivity standard source spectra desired in 17.3.5 by the decay corrected standard activities derived in 17.3.4, for example, counts/second· Becquerel.

17.3.7 Calculate contribution ratios for a coefficient matrix  $a_{ij}$  (12)

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:

$$a_{33} = 1,$$

$$a_{36} = \sim 0.4,$$

$$a_{66} = 1, \text{ and}$$

$$a_{63} = 0.$$

17.4 *Matrix Inversion Method—Sample Activity Determination:*

17.4.1 Place the sample to be measured at the source-to-detector distance used for activity calibration (see 17.3.1).

17.4.2 Accumulate the gamma-ray spectrum for sufficient time to obtain the desired statistical level of confidence (see 19.6 and 19.8).

17.4.3 Record the live time counting interval (see 19.9).

17.4.4 If  $C_i$  equals the total area sum of the components in counts per second present in the representative photopeak areas (see 17.3.5) in a sample spectrum, and if  $X_j$  equals the photopeak area of the nuclide component to be determined, then:

$$C_i = \sum_{j=1}^k a_{ij} X_j \quad (23)$$

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:

$$a_{11} X_1 + a_{12} X_2 + a_{13} X_3 + \dots + a_{1k} X_k = C_1 \quad (24)$$

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

where:

$A$  = the  $a_{ij}$  coefficient matrix (see 17.3.7),

$X^t$  = 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.

17.4.5 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 17.3.3).

17.4.6 The sample nuclide activity equals  $X^t$  divided by the calibration factor (see 17.3.6).

$$\text{Bq}_j = \frac{(c/s)_j}{(c/s \cdot \text{Bq})_j} \quad (25)$$

17.5 *Linear Least Square Method—Activity Calibration:*

17.5.1 Accumulate gamma-ray spectra using single radioactivity standard sources in a desired and reproducible counting geometry (see 19.7). At least 10 000 net counts should be accumulated in the full-energy gamma-ray peaks of interest (see 19.6 and 19.15).

17.5.2 Record the live time counting interval (see 19.6 and 19.9).

17.5.3 Determine the ambient background spectrum for each detector/geometry (see 19.7) 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 section 19.5.1) using the ratio of live time counting intervals as the normalization factor.

17.5.4 Correct the radioactivity standard source activity to the time at which the standard spectrum is acquired (see 19.10).

17.5.5 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 (47-50). 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 19.15). This method requires standard spectra representing the response of the detector to gamma rays of the nuclides of interest (for comparison, see 17.5.1, 17.5.2 and 17.5.3). 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,

$$X_i = (C_{i1} + C_{i2} + C_{i3} + \dots) = \sum_{j=1}^n C_{ij} \quad (26)$$

accounts for all contributions to channel  $i$ .

17.5.5.1 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 17.5.4):

$$X_i = \sum_{j=1}^n M_j S_{ij} + R_i \quad (27)$$

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.

**17.6 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 section 18.15 and 16.5.3), 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:

$$\left( \sum_{j=1}^n M_j S_{ij} \right) \quad (28)$$

This translates to:

$$\text{Minimize } (X_i - \sum_{j=1}^n M_j S_{ij})^2 W_i \quad (29)$$

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.

**17.6.1** The sample nuclide activity equals the derived sample count rate divided by the standard calibration factor

$$\text{Bq} = \frac{(c/s)}{(c/s \cdot \text{Bq})_j} \quad (30)$$

## 18. Performance Testing

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

**18.2** 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.

**18.3** 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.

**18.4** 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.

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

**18.6** 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.

## 19. Precautions

**19.1 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.

**19.2 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.

**19.3 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.

**19.4 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.

**19.5 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.

**19.6 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.

**19.7 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.

**19.8 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 (51 and 52).

**19.9 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:

$$N_o = \frac{N}{1 - Nt_d} \quad (31)$$

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: