Designation: E181 − 17

Standard Test Methods for Detector Calibration and Analysis of Radionuclides[1]

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
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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

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[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 ±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 δ_i are the estimated maximum systematic uncertainties, and σ_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

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

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:

$$E_f = \frac{N_p}{N_\gamma} \tag{1}$$

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:

$$N_A = G_s - B - I \tag{2}$$

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:

$$N_p = \frac{N_A}{T_s} \tag{3}$$

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):

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

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.

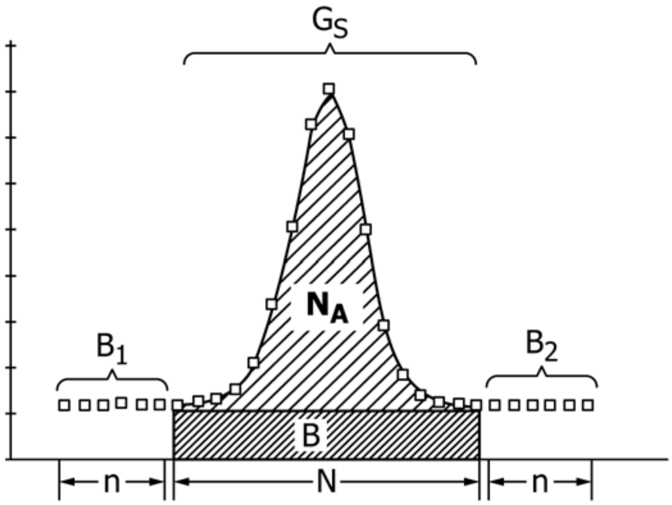

FIG. 1 Typical Spectral Peak With Parameters Used in the Peak Area Determination Indicated

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:

$$I = \frac{T_s}{T_b} I_b \tag{5}$$

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:

$$I_b = G_b - B_b \tag{6}$$

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:

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

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

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[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.

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:

$$N_\gamma = AP_\gamma \tag{8}$$

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:

$$N_\gamma = \frac{N_p}{E_f} \tag{9}$$

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,

$$A = \frac{N_\gamma}{P_\gamma} \tag{10}$$

9.1.6 Calculate the net peak area uncertainty as follows:

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

where:

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

and

S_{NA} = net peak area uncertainty (at 1σ 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 ±3 %).

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

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

11.1.5 The accuracy of the live-time determinations and pile-up corrections (typically ±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 µs, 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 % 1σ (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:

$$C = \frac{1}{\Pi_i^n \left(1 - q_i \varepsilon_i\right)} \tag{13}$$

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-cm^3 detector at 1 cm and at 4-cm sample-to-detector distances, respectively, and approximately 1.01 for a 100-cm^3 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-cm^3 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-cm^3 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,

$$F_b = \frac{\lambda t}{1 - e^{-\lambda t}} \tag{14}$$

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:

$$F_m = \frac{\lambda t}{1 - e^{-\lambda t}} e^{-\frac{\lambda t}{2}} \tag{15}$$

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:

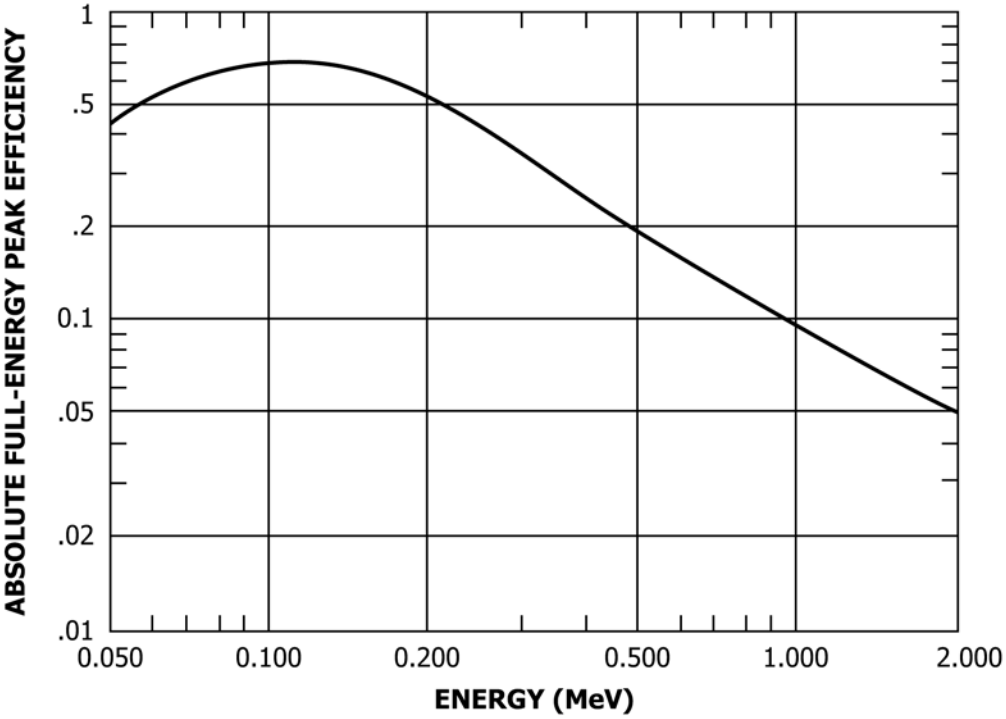


FIG. 2 Typical Efficiency Versus Energy for a Germanium Detector

$$A_c = A_o \frac{\mu x}{1 - e^{-\mu x}} \tag{16}$$

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 = $\text{g} \cdot \frac{1}{\text{cm}^3} \cdot cm$ = 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:

$$A_c = A_o \cdot e^{\mu x} \tag{17}$$

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, 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:

$$E_f = \frac{N_p}{N_\gamma} \tag{18}$$

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:

$$N_\gamma = AP_\gamma \tag{19}$$

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 amd 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:

$$N_\gamma = \frac{N_p}{E_f} \tag{20}$$

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,

$$A = \frac{N_\gamma}{P_\gamma} \tag{21}$$

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:

$$A = \frac{C}{R} \tag{22}$$

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} = $\dfrac{(\text{counts per second})\ \text{area}\ i}{(\text{counts per second})\ \text{area}\ j}$.

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

a_{33} = 1,
a_{36} = ~0.4,
a_{66} = 1, 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:

$$C_i = \sum_{j=1}^{k} a_{ij} X_j \tag{23}$$

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:

$$a_{11} X_1 + a_{12} X_2 + a_{13} X_3 + \ldots + a_{1k} X_k = C_1 \tag{24}$$

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 ... 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).

$$\mathrm{Bq}_j = \frac{(\mathrm{c/s})_j}{(\mathrm{c\ /\ s \cdot Bq})_j} \tag{25}$$

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 ($j = 1 \ldots n$) in channel i ($i = 1 \ldots k$) will be C_{ij} and the total count rate in channel i will be X_i. The expression,

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

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):

$$X_i = \sum_{j=1}^{n} M_j\, S_{ij} + R_i \tag{27}$$

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:

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

This translates to:

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

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

$$\mathrm{Bq} = \frac{(\mathrm{c/s})}{(\mathrm{c/s \cdot Bq})_j} \tag{30}$$

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 µs.

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:

$$N_o = \frac{N}{1 - Nt_d} \tag{31}$$

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 µs 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:

$$N_{1.0} = \frac{N_1}{1 - N_1 t_d} \tag{32}$$

$$N_{1.0} + N_{2.0} = \frac{N_{12}}{1 - N_{12} t_d} \tag{33}$$

$$N_{2.0} = \frac{N_2}{1 - N_2 t_d} \tag{34}$$

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 3 and Eq 5, is equal to that obtained from Eq 4:

$$t_d = \frac{1 - \left[1 - \frac{N_{12}}{N_1 N_2} (N_1 + N_2 - N_{12}) \right]^{1/2}}{N_{12}} \tag{35}$$

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.

19.10 *Correction for Decay During the Counting Period:*

19.10.1 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,

$$F_b = \frac{\lambda t}{1 - e^{-\lambda t}} \tag{36}$$

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

19.10.2 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

$$F_m = \frac{\lambda t}{1 - e^{-\lambda t}} e^{-\frac{\lambda t}{2}} \tag{37}$$

19.11 *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).

19.12 *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 16.3). 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.

19.13 *Correlated Photon Summing Correction:*

19.13.1 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:

$$C = \frac{1}{\Pi_i^n \left(1 - q_i \, \varepsilon_i\right)} \tag{38}$$

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 ith correlated photon.

Correlated summing correction factors for the primary gamma rays of radionuclides ^{60}Co, ^{88}Y, and ^{46}Sc are approximately 1.09 and 1.03 for a 65-cm^3 detector at 1 cm and at 4-cm sample-to-detector distances, respectively, and approximately 1.01 for a 100-cm^3 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)**.

19.13.2 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 (**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-cm^3 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-cm^3 detector.

19.14 *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.

19.15 *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 18.1) 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.

20. Sources of Uncertainty

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

20.1.1 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 ±3 %),

20.1.2 The reproducibility in determination of net full-energy peak counts (typically ±2 %),

20.1.3 The reproducibility of the positioning of the source relative to the detector and the source geometry (typically ±3 %),

20.1.4 The accuracy with which the full-energy peak efficiency at a given energy can be determined from the calibration curve or function (typically ±3 %—applies to single nuclide samples only), and

20.1.5 Ill-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.

20.2 Possible sources of systematic uncertainty in scintillation detector measurements are listed below (see 19.11) (**8**):

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

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

20.2.3 Iodine K X-ray escape for low-energy photon sources (**38, 46**).

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

20.2.5 The presence of radionuclide impurities.

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

20.2.7 Nonuniformity of the radioactivity distribution in the sample.

20.2.8 Timing, including errors in dead-time corrections.

20.2.9 Equipment malfunctions.

20.2.10 The counting of beta particles, conversion electrons, and bremsstrahlung which are energetic enough to enter the NaI(Tl) crystal and add, in an unpredictable way, to the gamma-ray pulse-height spectrum.

20.2.11 Random photon summing at high count rates (see 19.6).

20.2.12 Photomultiplier tube gain drift as a function of time or count rate, and

20.2.13 Gain shift caused by a changing magnetic field or change in the orientation of the detector in a fixed magnetic field.

COUNTING METHODS

BETA PARTICLE COUNTING

21. Scope

21.1 This method establishes methods for the counting of beta particles having a maximum energy of 0.220 MeV or greater. It is used primarily for the assay of radionuclide products mounted on counting media. The residual solids are expected to be less than 1 mg/cm.^{2}

22. Summary of Method

22.1 After evaporation or plating of the radionuclide solution on a commercially available beta counting planchet or on a rigidly mounted thin plastic film, total beta count rate is measured with the beta counter. The absolute beta activity is obtained by comparing the sample result with that obtained from a known standard of the same nuclide prepared and counted in the identical manner.

23. Apparatus

23.1 *Beta Particle Detector*—Any of the following may be used. Each has areas and counting applications where it may be more appropriate than an alternate choice. All detectors have associated electronics:

23.1.1 Thin window ($\leq$3 mg/cm^2) Geiger-Muller tube,

23.1.2 Organic scintillation phosphor with photomultiplier tube,

23.1.3 Gas flow proportional detector,

23.1.4 Inorganic scintillation phosphor (or phosphor sandwich) with photomultiplier tube, and

23.1.5 Silicon semiconductor detector.

23.2 Beta-counting planchets or rigidly-mounted thin plastic film. Plastic film minimizes backscatter that is not critical to beta counting but is to the determination of absorption curves (see Section 26).

23.3 Planchet holder capable of accurately reproducing the vertical and lateral position of a planchet.

23.4 Rigid sample positioning device that accepts either planchet holders or plastic film mounts and accurately positions them in a reproducible geometry relative to the detector.

23.5 Shielding to reduce ambient background.

23.6 Scaler.

23.7 Timer.

24. Preparation of Apparatus

24.1 Follow the manufacturer's recommendations for preparing the detector for beta particle counting.

24.1.1 Devices such as proportional counters and Geiger-Müller tubes operate on a voltage counting plateau. The purchased detector is usually accompanied by a calibration curve indicating the operating voltage and threshold voltages. Using these voltage values as a guide, recalibrate the detector prior to each series of tests. Repeat the calibration periodically and every time a gas cylinder is changed (gas flow proportional detector).

24.1.2 Set the detector voltage to a value about 100 V below the threshold voltage indicated by the manufacturer.

24.1.3 Place a beta source in a counting position. If no counts are obtained on the scaler, increase the voltage in 50-V steps until counts are obtained.

24.1.4 Plot the counts per minute obtained on the scaler as a function of the voltage. A curve should be obtained that has a flat portion or plateau with a slope corresponding to the manufacturer's specifications. Set the operating voltage at one third the length or 75 V above the low voltage end of the plateau, whichever is less.

24.2 Prepare planchets to receive samples or standards, or both.

24.2.1 New planchets are preferred but even these must be degreased with a solvent such as acetone. The planchets may also have to be passivated to reduce the chemical reaction with the sample media and to promote planchet surface wetting to achieve a speed source with minimum solids per unit area (see 21.1).

24.2.2 If previously used planchets have been cleaned, count each one prior to reuse to ensure all previous radioactivity has been removed.

24.2.3 If film mounts are to be used, cement the film over a rigid frame. The frame must lay flat in the sample mount holder. Paper and most plastics are not suitable frame materials.

25. Beta Counting Procedure

25.1 Choose an aliquot of sample that meets the following maximum count rate criteria:

Detector type	MeV/minute
Organic	5 000
Geiger-Müller	10 000
Proportional	80 000
Phoswich	80 000
Silicon semiconductor	18 000 000

where MeV/min is defined as the beta energy maxima in MeV detected per minute.

25.2 Pipet an aliquot of sample solution onto the planchet or film mount.

25.3 Dry the sample with an infrared lamp or other device that will evaporate the sample liquid without spattering or running off the flat portion of the sample mount.

25.4 When the sample mount has dried and cooled, place it in the counter at a position that is reproducible and that does not exceed the count rate maxima in 25.1.

25.5 Collect a total of at least 10 000 counts per mount. Record the count time and calculate the sample count rate in counts per minute. Duplicate sample mount count rates should agree within ±2 %.

25.6 Prepare a standard mount(s) from a standard solution of the same radionuclide having a known rate of disintegrations per minute. Standards are available from the National Institute of Standards and Technology and commercial suppliers. Mount the standard on the same material as the sample, count in the same geometry, and determine the standard count rate.

25.7 Determine the ambient background of the counter by counting with a blank sample planchet or film in the same geometry as the sample/standard. If planchets are reused (see 24.2.2), the background that should be used is that obtained when counting the cleaned planchet prior to sample mounting. Calculate the background count rate in counts per minute.

26. Calculations

26.1 Correct the observed count rates of both standard and sample for background and coincidence losses as follows:

$$C = R + TR^2 - B \tag{39}$$

where:

C = corrected count rate of the sample/standard, cpm (Note 6),
R = observed count rate of the source, cpm,
T = resolving time of the counter, mpc, and
B = ambient background rate, cpm.

NOTE 6—Any other unit of time is acceptable provided it is used consistently throughout.

26.2 Correct the standard disintegrations per minute for radioactive decay as follows:

$$D = D_s e^{-\lambda T} \tag{40}$$

where:

D = disintegrations per minute of the standard at the time the standard was counted,
D_s = disintegrations per minute of the standard at the time certified by the standard supplier,
λ = ln 2 divided by half-life (**7-14**), and
T = the difference in time between the date and time the standard was counted and the time at which the standard activity was certified in the same units as the half-life.

No correction is required if the current activity or disintegration rate is known within 1 %.

26.3 Calculate the absolute activity of the sample as follows:

$$A = CD/E \tag{41}$$

where:

A = distintegrations per minute of the sample, and
E = corrected count rate of the standard, cpm.

26.4 Calculate the counting uncertainty (**52**) as follows:

$$\sigma_c = \frac{\sqrt{G+B}}{N} \quad \text{(see Note 2)} \tag{42}$$

where:

σ_c = counting uncertainty,
G = gross counts (not rate),
B = background counts, and
N = net counts.

26.5 For other sources of uncertainties, see Section 39.

ALUMINUM ABSORPTION CURVE

27. Scope

27.1 This method establishes a method for determining the approximate energies and intensities of beta particle spectra in radioactive sources. The method is intended to be used in the qualitative and quantitative determination of beta emitting impurities in such preparations as radiopharmaceuticals. It may also be used to determine beta energy and intensity levels of single radionuclide sources.

28. Summary of Method

28.1 When a radioactive source decays by beta emission, the beta particles (electrons) are emitted with a distribution of energies. The maximum energy of the electrons is the "beta energy" as tabulated in nuclear-data tables (**7, 12**). When beta particles are absorbed by interposing varying thicknesses of material between the source and a detector, the attenuation follows to an approximation the equation

$$l = l_o e^{[(-\ln 2/T_{1/2})T]} \tag{43}$$

where:

l = corrected intensity (activity),
l_o = observed intensity,
T = thickness of absorber, mg/cm^2, and
$T_{1/2}$ = observed experimental half-thickness, that is, the thickness necessary to reduce l_o to 1/2 l_o.

Although the absorption of beta radiation is not necessarily exponential, in many nuclides it closely approximates it. Therefore, by obtaining beta absorption data, the energy (and thus the identity) of a radioactive species may be found. By properly calibrating the detecting device, usually a Geiger-Müller counter, the absolute amounts of several beta groups may be obtained by extrapolating each group to zero added absorber and dividing by the previously determined efficiency (or geometry) of the counter.

29. Apparatus

29.1 Same as Sections 23 and 24.

29.2 Plus aluminum absorbers, of a size to fit between sample and detector.

30. Aluminum Absorption Curve Procedure

30.1 Use the beta particle sample mount prepared in Sections 25.1 – 25.4. Because beta particles backscatter and interfere with the aluminum absorption curve measurements, a film mounted source is preferred. Low atomic weight planchets will cause low levels of interference. The activity of the source mount should be close to the maximum allowed in 25.1.

30.2 Collect a total of at least 10 000 counts for the source mount with no absorber. Calculate the corrected count rate, 26.1.

30.3 Without moving the sample source, place between the source and the detector and as close to the detector as possible, an aluminum absorber of a thickness approximately equal to 3 mg/cm^2. Acquire at least 10 000 counts and calculate the corrected count rate, 26.1.

30.4 Repeat the procedure described in 30.3 with each absorber, increasing the counting time as the observed counts per minute become smaller. When the observed counts per minute reach a constant, or background, the absorption data are complete.

30.5 Subtract from all points the constant value, if any, obtained in the last few data points.

30.6 Plot the data obtained on semi-log graph paper. Use as the ordinate the corrected rate of counts per minute. Use as the abscissa the thickness of the aluminum absorber in milligrams per square centimetre.

30.7 The plotted data may be compared to a known standard absorption curve for the particular nuclide to check for impurities (as evidenced by deviations from the known curve). Should no standard curve be available, or should there be a marked difference, the curve may be analyzed by subtraction, as follows:

30.7.1 Beginning at the high-energy end of the curve (thickest aluminum absorber), draw the best straight line possible through the largest number of last points. This line should intersect the zero axis and extend to the left of it. In Fig. 3 this is the component with half-thickness 28 mg/cm^2.

30.7.2 Subtract from selected counts-per-minute ordinate points the contribution of the most energetic component as given by the corresponding ordinate value from the extrapolated straight line. Plot each difference at the abscissa point at which it was found.

30.7.3 By use of these data again draw the best straight line starting at the high-energy end of the plot. Should there be data points above this line at the low-energy end of the plot, this is evidence for an even lower-energy beta group and the procedure given in 30.5 must be repeated. (In general, after two subtractions, the remaining data become highly uncertain.) Fig. 3 represents a two-component curve.

30.8 The approximate beta energy of each group may be obtained by observing the half-thickness of each straight-line component and relating this to the experimentally determined graph shown in Fig. 4.

30.9 Obtain the number of disintegrations of each beta ray group by extrapolating each straight line component to "true zero" as described in Section 31. Correct for efficiency as in 26.3.

30.10 While many absorption curve resolution codes are available for 30.6 – 30.9, it remains the responsibility of the user of the code to demonstrate the adequacy of the code on prepared standard mixtures of known activity similar to those occurring in sample analyses. Materials to prepare such standards are available from the National Institute of Standards and Technology and commercial suppliers.

31. Calculation

31.1 The observed counts per minute without additional aluminum absorber actually was reduced by air and counter window absorption. The" true zero" rate in counts per minute

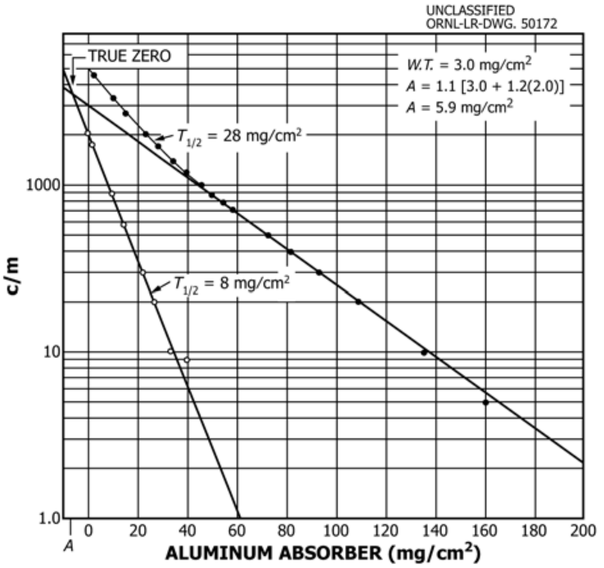
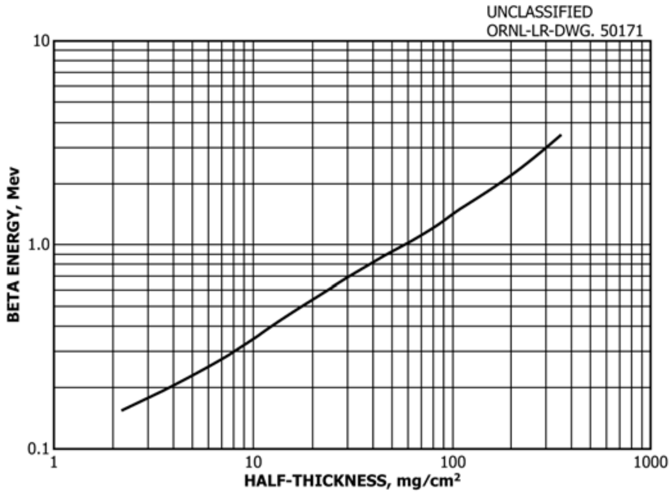


FIG. 3 Aluminum Absorption Curve Showing Activity as a Function of the Aluminum Absorber Placed Before the Detector



BETA RADIATION HALF-THICKNESS IN ALUMINUM vs MAXIMUM ENERGY

FIG. 4 Beta Energy as a Function of Half-Thickness in Aluminum

the rate corrected for this absorber. The negative abscissa distance, *A*, in milligrams per square centimetre may be found as follows:

$$A = 1.1\,(T + 1.2\,d) \qquad (44)$$

where:

T = window thickness of the detector, mg/cm^2,
1.1 = correction for oblique passage through the window of the beta counter,
1.2 = density of air, mg/cm^3, and
d = distance from sample to window, cm.

31.2 The activity read from the ordinate at the negative abscissa "true zero" must be corrected for geometry. Obtain this geometry by taking aluminum absorption data using a standard material of known disintegration rate, extrapolating to true zero, and dividing the observed counts per minute by the known disintegrations per minute. The counts per minute observed divided by the geometry of the counter yields the disintegration rate of the beta group.

$$A = CD/E \qquad (45)$$

where:

A = disintegrations per minute of the beta group,
C = corrected count rate of the sample beta group, corrected to "true zero" absorption, cpm (see 26.1 and 31.1),
D = disintegrations per minute of the standard beta group at the time the standard was counted (see 26.2), and
E = corrected count rate of the standard beta group, corrected to "true zero" absorption, cpm (see 26.1 and 31.1).

For sources of error see Section 38.

ALPHA PARTICLE COUNT

32. Scope

32.1 This method establishes methods for the counting of alpha particles. It is used primarily for the assay of radionuclide products mounted on counting media. The residual solids are expected to be less than 1 mg/cm^2 and uniformly distributed.

33. Summary of Method

33.1 After evaporation or electrodeposition of the radionuclide solution on a commercially available alpha counting disk or planchet or on a rigidly mounted thin plastic film, total alpha count rate is measured with the alpha counter. The absolute alpha activity is obtained by comparing the sample result with that obtained from any known alpha standard of the same energy range prepared and counted in the identical manner.

34. Apparatus

34.1 *Alpha Particle Detector*—Any of the following may be used. Each has areas and counting applications where it may be more appropriate than an alternate choice. All detectors have associated electronics:

34.1.1 Windowless gas flow proportional detector, for example, an Alpha Simpson Proportional counter,

34.1.2 Thin window (≤100 µg/cm^2) gas flow proportional detector,

34.1.3 Windowless inorganic scintillation phosphor (or phosphor sandwich) with photomultiplier tube, and

34.1.4 Silicon semiconductor detector.

34.2 Alpha counting disk, planchet, or rigidly mounted thin plastic film. Disks are preferentially used for alpha energy analysis.

34.3 Planchet holder capable of accurately reproducing the vertical and lateral position of a planchet.

34.4 Rigid sample positioning device that accepts planchet holders, disks or thin plastic film mounts and accurately positions them in a reproducible geometry relative to the detector.

34.5 A vacuum chamber for silicon semiconductor detectors.

34.6 Scaler and timer, or an MCA (particularly for semiconductor detectors).

35. Preparation of Apparatus

35.1 Follow the manufacturer's recommendations for preparing the detector for alpha particle counting.

35.1.1 Devices such as proportional counters operate on a voltage counting plateau. The purchased detector is usually accompanied by a calibration curve indicating the operating voltage and threshold voltages. Using these voltage values as a guide, recalibrate the detector prior to each series of tests. Repeat the calibration periodically and every time a gas cylinder is changed (gas flow proportional detector).

35.1.2 Set the detector voltage to a value about 100 V below the threshold voltage indicated by the manufacturer. Note that the alpha plateau is usually lower in voltage and flatter than the beta plateau.

35.1.3 Place an alpha source in a counting position. If no counts are obtained on the scaler, increase the voltage in 50-V steps until counts are obtained.

35.1.4 Plot the counts per minute obtained on the scaler as a function of the voltage. A curve should be obtained that has a flat portion of plateau with a slope corresponding to the manufacturer's specifications. Set the operating voltage at one third the length or 75 V above the low voltage end of the plateau, whichever is less. Note that the operating voltage must be below the beta threshold.

35.2 Prepare disks or planchets to receive samples or standards, or both.

35.2.1 New disks or planchets are preferred but even these must be degreased with a solvent such as acetone. The disks or planchets may also have to be passivated to reduce the chemical reaction with the sample media and to promote surface wetting to achieve a spead source with minimum solids per unit area (see 32.1). Disks must also be very clean to achieve uniform electrodeposition or plating.

35.2.2 If previously used disks or planchets have been cleaned, count each one prior to reuse to ensure all previous radioactivity has been removed.

35.2.3 If film mounts are to be used, cement the film over a rigid frame. The frame must lay flat in the sample mount holder. Paper and most plastics are not suitable.

36. Alpha Counting Procedure

36.1 Choose an aliquot of sample that is within the counting capabilities of the detector as indicated by the manufacturer. Note that alpha particles can cause particles of the sample to recoil and imbed themselves in a detector, particularly when close to the detector and in a vacuum. This will gradually increase the detector background, making it unsuitable for low rate counting.

36.2 Pipet an aliquot of sample solution onto the disk, planchet, or film mount. Sample preparation methods using electrodeposition are beyond the scope of this method.

36.3 Dry the sample with an infrared lamp or other device that will evaporate the sample liquid without spattering or running off the flat portion of the sample mount. For alpha contamination control of aqueous samples on metal, it may be advisable to flame to red heat, cool, and seal with a drop of a 1 % solution of Collodion in ether.

36.4 When the sample mount has dried and cooled, place it in the counter at a position that is reproducible and that does not exceed the count rate maxima in 36.1.

36.5 Collect a total of at least 10 000 counts per mount. Record the count time and calculate the sample count rate in counts per minute. Duplicate sample mount count rates should agree within ±2 %.

36.6 Prepare a standard mount(s) from a standard solution having a known rate of disintegrations per minute. Standards are available from the National Institute of Standards and Technology and commercial suppliers. Mount the standard on the same material as the sample, count in the same geometry, and determine the standard count rate. Most detectors detect all alpha particles with one hundred percent efficiency; therefore, it is not necessary to duplicate the radionuclide(s) being counted when preparing standards, but it should be of similar energies.

36.7 Determine the ambient background of the counter by counting with a blank sample mount in the same geometry as the sample/standard. If disks or planchets are reused (see 35.2.2), the background that should be used is that obtained when counting the cleaned planchet prior to sample mounting. Calculate the background count rate in counts per minute.

37. Calculations for SCA Methods

37.1 Correct the observed count rates of both standard and sample for background and coincidence losses as in 26.1.

37.2 Correct the standard disintegrations per minute for radioactive decay as in 26.2.

37.3 Calculate the absolute activity of the sample as in 26.3.

37.4 Calculate the counting uncertainty **(52)** as in 26.4.

37.5 For other sources of uncertainty, see Section 38.

38. Sources of Uncertainty: SCA Counting Methods

38.1 Gross beta particle and alpha particle counting accuracy is usually limited by counting uncertainty. Total uncertainties of± 4 % are expected. The aluminum absorption method is limited by the multiplicative uncertainties of multiple beta groups. An accuracy of± 5 % is expected for the first component, ±20 % for the second, and ±50 % for the third.

38.2 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 ±3 %).

38.3 The reproducibility of the positioning of the source relative to the detector and the source geometry (typically ±3 %).

38.4 Alpha-beta cross talk or beta detector sensitivity for gamma, or both, emissions are sample and detector dependent.

39. Calculations for MCA Methods

39.1 This discussion addresses the calculations involved in determining nuclide activities when samples are prepared with an alpha emitting tracer and each isotope group is prepared in separate samples.

39.2 It is assumed that samples can be prepared in such a manner that the peaks from the nuclides to be analyzed do not appreciably overlap, or that such overlap is negligible.

39.3 Define a region-of-interest (ROI) for the tracer. Define an ROI for each nuclide to be analyzed. Make sure the ROIs do not overlap.

39.4 Calculate a net peak area for each peak, including the tracer.

39.5 The net peak area is calculated as:

$$N_p = G - B - I \tag{46}$$

where:

N_p = net peak area,
G = sum of all counts within the ROI,
B = background under the ROI (if applicable), and
I = contamination from the tracer (if applicable).

39.6 The background B is calculated as follows:

39.6.1 The first channel of the ROI and the last channel of the ROI are both converted into an energy value using the energy calibration of the current sample spectrum. These energy values are then converted back into channel values using the energy calibration of the background spectrum as specified in the background spectrum.

39.6.2 The background counts, B, are calculated as the sum of the counts in the background spectrum in the ROI thus established, divided by the live time of the background spectrum and multiplied by the live time of the sample spectrum, as follows:

$$B + \frac{T_s}{T_b} \cdot B_r \tag{47}$$

where:

B_r = counts in the equivalent ROI in the background spectrum,
T_b = live time of the background spectrum, and
T_s = live time of the sample spectrum.

If no background subtraction is needed, $B = 0$.

39.7 The interference I from the tracer is calculated as follows: Assuming that information exists on fractions of contamination in the tracer for each nuclide to be analyzed, the interference I for nuclide x can be calculated as:

$$I_x = f_x \cdot N_t \tag{48}$$

where:

f_x = fraction of nuclide x present in the tracer, and
N_t = net area of the tracer peak, that is, $N_t = G_t - B_t$.

39.8 The activities are calculated from the peak counts as follows: First calculate an effective efficiency factor using the tracer peak as follows (see Fig. 5 for a clarification on the time intervals):

$$F = \frac{N_t}{A_t \cdot y_t \cdot K_{t_3} \cdot K_{t_2} \cdot T_s} \tag{49}$$

where:

F = effective efficiency factor,
N_t = net area of the tracer peak,
A_t = known activity of the tracer nuclide, assumed to be valid for the tracer on the tracer reference date/time. This value must be disintegrations per second (Bq) for this equation to be valid,
y_t = branching ratio of the energy of the tracer nuclide used as the tracer peak, and
T_s = sample spectrum collect live time, s.

39.8.1 $K_{t3}$is the decay correction of the tracer for any decay between the tracer reference date and the start of the spectrum collect as follows:

$$K_{t_3} = e\left(-\frac{ln(2)\cdot t_3}{T_{1/2}}\right) \tag{50}$$

where:

t_3 = elapsed time between the tracer reference time and the start of spectrum collect and
$T_{1/2}$ = half-life of the tracer nuclide.

39.8.2 K_{t2} is the decay correction of the tracer during the spectrum collect as follows:

$$K_{t_2} = \frac{T_{1/2}}{1n(2)\cdot t_2}\cdot\left[1 - e\left(-\frac{ln(2)\cdot t_2}{T_{1/2}}\right)\right] \tag{51}$$

where:

t_2 = spectrum collect true time and
$T_{1/2}$ = half-life of the tracer nuclide.

The time units of t_2, t_3, and $T_{1/2}$ must be the same.

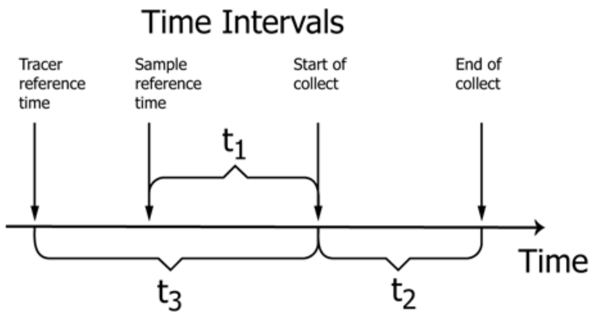


FIG. 5 Clarification of Time Intervals

39.9 The percent uncertainty of the effective efficiency factor, S_F, is calculated as follows:

$$S_F = \sqrt{S_{A_t}{}^2 + S_{y_t}{}^2 + S_{N_t}{}^2} \tag{52}$$

where:

S_{A_t} = percent uncertainty of the known tracer activity,
S_{y_t} = percent uncertainty of the branching ratio of the tracer energy used (if applicable), and
S_{N_t} = percent uncertainty of the tracer net peak area.

39.10 The percent uncertainty of the net peak area of the tracer peak, S_{Nt}, is calculated as follows:

$$S_{N_t} = \frac{100}{N_t}\cdot\sqrt{G_t + \left(\frac{T_s}{T_b}\right)^2 \cdot B_r} \tag{53}$$

where:

G_t = gross counts in the tracer peak.

The other symbols are in accordance with the aforementioned equations.

39.11 For the non-tracer nuclides, the activity per unit mass (or per unit volume) is calculated as follows:

$$A_x = \frac{N_x}{F \cdot y_x \cdot K_{t_1} \cdot K_{t_2} \cdot m \cdot f} \tag{54}$$

where:

A_x = activity per unit mass (or volume) for nuclide x,
N_x = net counts in the peak ROI for nuclide x, that is,

$$N_x = G_x - B_x - I_x \tag{55}$$

F = effective efficiency factor calculated from the tracer nuclide (a single value for all nuclides),
y_x = branching ratio of the observed alpha energy nuclide x,
K_{t_2} = ecay correction factor for decay during spectrum collect (if applicable). The equation is the same as used in calculating the effective efficiency factor F, except that the nuclide x half-life must be used,
K_{t_1} = decay correction for any decay between the sample reference date/time and the start of the spectrum collect, that is,

$$K_{t_1} = e\left(-\frac{1n(2)\cdot t_1}{T_{1/2}}\right) \tag{56}$$

f = conversion factor from disintegrations per second (Bq) to the activity units desired for reporting (if applicable), and
m = mass (or volume) of the sample.

39.12 The percent uncertainty of A_x is calculated as follows:

$$S_{A_x} = \sqrt{S_F{}^2 + S_{y_x}{}^2 + S_{N_x}{}^2} \tag{57}$$

where:

S_F = percent uncertainty of the effective efficiency factor, F, calculated from the tracer peak,
S_{y_x} = percent uncertainty of the branching ratio of the nuclide energy used (if known), and

S_{N_x} = percent uncertainty of the net peak area of the nuclide peak.

39.13 The percent uncertainty of the net peak area of the nuclide peak is calculated as follows:

$$S_{N_x} = \frac{100}{N_x} \cdot \sqrt{G_x + \left(\frac{T_s}{T_b}\right)^2 \cdot B_r + \left(\frac{I_x \cdot S_{I_x}}{100}\right)^2} \tag{58}$$

where:

G_x = gross counts in the nuclide peak,
N_x = net counts in the nuclide peak, and
S_{I_x} = percent uncertainty of the interference produced into the peak area of the nuclide as a result of the fact that the tracer also contains small amounts of this nuclide.

39.14 The percent uncertainty of I_x is calculated as follows:

$$S_{I_x} = \sqrt{S_{f_x}^{\ 2} + S_{N_t}^{\ 2}} \tag{59}$$

where:

S_{f_x} = percent uncertainty of the fraction of the nuclide present in the tracer (if applicable), and
S_{N_t} = percent uncertainty of the net peak area of the tracer peak.

39.15 Note that all calculations explained in this section assume that there is no sample mass uncertainty or that it is sufficiently small to be negligible. Furthermore, the equations do not include any time uncertainties.

LIQUID SCINTILLATION COUNTING

40. Scope

40.1 This method covers the liquid scintillation measurement of radioactivity. Liquid scintillation counting is the most sensitive and widely used technique for the detection and quantification of radioactivity. This measurement technique is applicable to all forms of nuclear decay emissions (alpha and beta particle, and gamma and X-ray emitting radionuclides). Liquid scintillation counting is an analytical technique which measures activity of radionuclides from the rate of light photons emitted by a scintillating matrix.

40.2 This method can be used for either absolute or relative determinations. For radioassay, data may be expressed in terms of disintegrations rates after calibration with an appropriate standard. General information on liquid scintillation counting has been published (**51-56**).

40.3 *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.*

41. Summary of Method

41.1 The scintillator solution (cocktail), mixed with the radioactive sample, is excited by nuclear decay emissions and emits light pulses by a molecular de-excitation process. The number of pulses per unit time is proportional to the quantity of activity present. Solutes and emulsifier agents are used in the scintillator to provide the best combination of wavelength, pulse height, and solubility for this application. The pulses are detected by two multiplier phototubes connected in coincidence and converted to electric signals. The amplified pulses are recorded and the count rate is measured. The efficiency of the system can be determined by use of prepared standards having the same volume, density, and color as the sample.

42. Interferences

42.1 Quenching, the reduction in emitted light, and hence attenuation of pulse heights, can cause significant reduction in the absolute counting efficiency. Quenching may be caused by impurities in the scintillator solution which inhibits the transfer of energy or by color in the sample which absorbs the emitted light. Many of the newer instruments determine a quench correction factor.

42.2 Another phenomenon which influences counting is the background produced by environmental radiation. Several sources of radiation can affect the sensitive scintillation solution and the detection process: (*a*) cosmic radiation, (*b*) building materials of the laboratory which contain activity, (*c*) natural activity in the sample vial and the walls of the photomultiplier tubes, and (*d*) stray radiation from sources of activity in the laboratory.

42.3 Any foreign ionizing radiation in the sample may interfere in the analysis of the radionuclide being measured.

42.4 Some types of samples or scintillator stock solutions exposed to daylight or fluorescent lighting must be dark-adapted (several minutes to several hours) or erratic results will occur.

43. Apparatus

43.1 *Liquid Scintillation Counter:*

43.2 *Sample Vials,* glass or polyethylene, of suitable size to fit the counting chamber. The conventional size has a total capacity of approximately 20 mL. Smaller vials can also be successfully used and reduce the measurement cost.

43.3 *Scintillator Stock Solution*—Choose an appropriate scintillation cocktail that is compatible (attains homogeneity) with the sample to be measured.

44. Standardization and Calibration

44.1 There are several methods of counting efficiency calibration in use (for example, internal standardization, external standardization, channels ratio).

44.1.1 Internal standardization consists of adding a known amount of standard to the sample after it has been counted, and then to recount. The activity of the added internal standard should generally be much greater than that of the sample in order to give good counting statistics, but not so great that the combined activities of standard and sample will introduce coincidence losses. Also, the volume of added standard must be small so that no additional quenching is introduced.

44.1.2 Generally, the counting efficiency is determined by an indirect comparison of the unknown sample with a set of standard samples of the same radionuclide. This set consists of several samples containing known amounts of the standard radionuclide to be measured and varying in degree of quenching over the range to be encountered in actual sample preparation. An alternative method is to add the quenching agent in small increments to a single sample, calculating a counting efficiency for each increment added. In either case, the overall result is a calibration curve consisting of either efficiency versus channels ratio (two counting channels chosen so that together they include most of the pulse height distribution of the radionuclide) or efficiency versus external standard ratio (counting channels determined by the instrument after exposing the sample to a gamma source placed nearby). This chief advantage of external standardization over the channel ratios method is the reduction of total counting time for low-activity samples.

44.1.3 Count a blank and standard and calculate the efficiency factor(s) for the scintillator mixture:

$$Eff = (C/t - B)/D \tag{60}$$

where:

Eff = counting efficiency,
C = number of counts accumulated,
t = time of counting, s,
B = background of blank, counts/s, and
D = disintegration rate of standard, Bq.

45. Procedure

45.1 Pipet an aliquot of sample solution to a vial containing the same scintillator solution as the calibration standard. Cap the vial and mix.

45.2 Prepare a blank by pipetting an aliquot of a solution that has the same volume, density, and color as the sample. Cap the vial and mix.

45.3 Place the sample and blank in the counter and dark-adapt if necessary.

45.4 Count the sample for the length of time to give the desired reliability. Use the same energy settings as those of the calibration standard.

NOTE 7—The same sample blank may be used for a number of samples provided the same scintillator solution is used for each and the blank has the same volume, density, and color as the samples.

46. Calculation

46.1 Calculate the radioactivity, A, in disintegrations per second (Bq) as follows:

$$A = (C/t - B)/Eff \tag{61}$$

where:

C = number of counts accumulated,
t = time of counting, s,
B = background of blank, counts/s, and
Eff = efficiency factor determined in 44.1.3.

47. Precision and Bias

NOTE 8—Measurement uncertainty is described by a precision and bias statement in this standard. Another acceptable approach is to use Type A and Type B uncertainty components **(57, 58)**. The Type A/B uncertainty specification is now used in the International Organization for Standardization (ISO) standards and this approach can be expected to play a more prominent role in future uncertainty analyses.

47.1 General practice indicates that disintegration rates can be determined with a bias of ±3 % (1S %) and with a precision of ±1 % (1S %).

48. Performance Testing

48.1 *General*—Operate the instrument in conformance with the manufacturer's recommendations.

48.2 *Frequency of Testing*—Monitor the instrument performance following installation, service, replacement of sealed check sources, or any other circumstance that may affect the accuracy of the data obtained. Establish a quality-control program to monitor the performance of the instrument. Count a background and check source appropriate for the radionuclide selected on a daily basis during periods in which the counter is in use. Record the data. The check-source data should also be recorded on a control chart. If any instrument operating parameter is changed, a new control chart should be started.

49. Keywords

49.1 calibration; chemical composition; dosimetry; isotope analysis; radiation; radiation and particle detectors; radiation detectors; radioactive isotopes; radiochemical analysis; radionuclide

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