



Designation: D7282 – 21<sup>ε1</sup>

# Standard Practice for Setup, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements<sup>1</sup>

This standard is issued under the fixed designation D7282; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

<sup>ε1</sup> NOTE—Editorially corrected Fig. X1.1, Fig. X3.1, and Fig. X4.1 in December 2022.

## 1. Scope

1.1 This practice covers consensus criteria for the setup, calibration, and quality control of nuclear instruments. Setup establishes the operating parameters of the instrument—for example, voltage or discriminator settings. Calibrations determine the instrument's response characteristics—for example, its counting efficiency or gain. Quality control ensures that the performance of the instrument remains acceptable for its intended use and consistent with the performance at the time of calibration.

1.2 This practice addresses four of the most commonly used types of nuclear counting instruments: alpha-particle spectrometer, gamma-ray spectrometer, gas proportional counter, and liquid scintillation counter.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions that are provided for information only and are not considered standard.

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

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

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.04 on Methods of Radiochemical Analysis.

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## 2. Referenced Documents

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

- D1129 Terminology Relating to Water
- D3648 Practices for the Measurement of Radioactivity
- D7283 Test Method for Alpha and Beta Activity in Water By Liquid Scintillation Counting
- D7902 Terminology for Radiochemical Analyses
- E2586 Practice for Calculating and Using Basic Statistics

### 2.2 Other Standards:

- ANSI N42.22 Traceability of Radioactive Sources to the National Institute of Standards and Technology (NIST) and Associated Instrument Quality Control<sup>3</sup>
- ANSI N42.23 Measurement and Associated Instrumentation Quality Assurance for Radioassay Laboratories<sup>3</sup>
- ANSI/HPS N13.30 Performance Criteria for Radiobioassay<sup>3</sup>
- ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories<sup>4</sup>
- JCGM 100:2008 Evaluation of Measurement Data – Guide to the Expression of Uncertainty in Measurement<sup>5</sup>

## 3. Terminology

### 3.1 Definitions:

3.1.1 For definitions of terms used in this standard, refer to Terminologies D1129 and D7902 and Practice E2586.

### 3.2 Definitions of Terms Specific to This Standard:

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

<sup>4</sup> Available from International Organization for Standardization (ISO), 1 rue de Varembe, Case postale 56, CH-1211, Geneva 20, Switzerland, <http://www.iso.ch>.

<sup>5</sup> Available from Bureau International des Poids et Mesures (BIPM), Pavillon de Breteuil F-92312 Sèvres Cedex France, <http://www.bipm.org/en/publications/guides/gum.html>.

3.2.1 *acceptable verification ratio (AVR)*, *n*—ratio of the absolute difference between the measured value of the verification sample and the known value added to the verification sample to the square root of the sum of the squares of their associated combined standard uncertainties.

3.2.1.1 *Discussion*—See [Eq 15](#) in [16.2.15](#).

3.2.2 *background subtraction count (BSC)*, *n*—a source count used to determine the background to be subtracted from the sample test source count.

3.2.3 *calibration*, *n*—determination of an instrument's response to a known amount of radioactive material.

3.2.3.1 *Discussion*—Instrument calibrations may include calibrations for counting efficiency, gain, and resolution.

3.2.4 *calibration source*, *n*—a known quantity of radioactive material, prepared and configured for calibrating nuclear instruments.

3.2.4.1 *Discussion*—A calibration source used for efficiency calibration must have quantity values and uncertainties with documented traceability to the SI.

3.2.5 *certified calibration source (CCS)*, *n*—a calibration source (see [3.2.4](#)) accompanied by a certificate that provides the values, uncertainties, and reference date of the source's primary radioactive constituents, with documentation of metrological traceability to the SI.

3.2.5.1 *Discussion*—ANSI N42.22 describes the required content of the certificate and presents criteria for ensuring traceability of radionuclide sources to NIST.

3.2.6 *continuing instrument quality control*, *n*—activities conducted to ensure that an instrument continues to respond in the same manner after its calibration.

3.2.7 *instrument check*, *n*—a test of the response of a nuclear counting instrument, typically using an instrument check source (see [3.2.8](#)) and including some combination of tests of efficiency, energy calibration, and peak resolution as appropriate for the instrument type.

3.2.8 *instrument check source (ICS)*, *n*—a radioactive source, not necessarily traceable to any standard, that is used to test the response of a nuclear instrument.

3.2.9 *instrument contamination check (ICC)*, *n*—a measurement to determine if a nuclear instrument is contaminated with radioactive material.

3.2.10 *instrument control chart*, *n*—a chart used to monitor and evaluate the performance of an instrument using predetermined statistically based limits.

3.2.11 *instrument tolerance chart*, *n*—a chart used to monitor and evaluate the performance of an instrument using tolerance limits appropriate to the method, scope of work, and data quality requirements.

3.2.12 *known value (KV)*, *n*—accepted true value of the analyte activity added to a verification sample.

3.2.12.1 *Discussion*—See [Eq 13](#) in [16.2.13](#).

3.2.13 *measured value (MV)*, *n*—result of a measurement performed on a verification sample.

3.2.13.1 *Discussion*—See [Eq 11](#) in [16.2.11](#).

3.2.14 *measurement quality objective (MQO)*, *n*—quantitative or qualitative statement of a performance objective or requirement for a particular method performance characteristic ([1](#)).<sup>6</sup>

3.2.15 *normalized residual*,  $\zeta_i$ , *n*—quotient of a *residual*,  $e_i$ , and its combined standard uncertainty,  $u_c(e_i)$

3.2.15.1 *Discussion*—See [Appendix X5](#) for the calculation and use of  $\zeta_i$ .

3.2.16 *operating parameter*, *n*—any of the configurable settings of a nuclear counting instrument, such as a detector operating voltage, amplifier gain, or energy discriminator setting.

3.2.17 *quality manual (QM)*, *n*—a document stating the management policies, objectives, principles, organizational structure and authorities, accountability, and implementation of a laboratory's quality system, to assure the quality of its data.

3.2.17.1 *Discussion*—The quality manual shall document the process by which appropriate analytical methods are selected, their capability is evaluated, and their performance is documented. The analytical methods manual and standard operating procedure manuals shall be part of but not necessarily included in the quality manual. The quality manual or standard operating procedures, or both, shall also include instructions that prescribe corrective action, for example, in the event of a failure of an instrument check source (ICS), instrument contamination check (ICC), or background subtraction count (BSC).

3.2.18 *relative residual*,  $\% \Delta_i$ , *n*—quotient of a *residual*,  $e_i$ , and the corresponding predicted value,  $\hat{e}_i$ , typically expressed as a percentage.

3.2.19 *relative standard deviation (RSD)*, *n*—ratio of the standard deviation to the mean (also known as *coefficient of variation*).

3.2.19.1 *Discussion*—See Practice [E2586](#).

3.2.20 *residual*, *n*—difference between the observed value of the dependent variable,  $\varepsilon_i$ , and the corresponding predicted value,  $\hat{e}_i$ .

3.2.21 *sample test source (STS)*, *n*—a sample or sample aliquot prepared or configured for measurement of its emitted radiation.

3.2.22 *tolerance limit*, *n*—a limit established to evaluate the acceptability of a monitored process parameter.

3.2.23 *working calibration source (WCS)*, *n*—a calibration source (see [3.2.4](#)) diluted or prepared by the laboratory from radioactive reference materials.

### 3.3 Acronyms:

3.3.1 *ADC*—analog-to-digital converter

3.3.2 *AVR*—acceptable verification ratio

3.3.3 *BIPM*—Bureau International des Poids et Mesures (English: International Bureau of Weights and Measures)

3.3.4 *BSC*—background subtraction count

3.3.5 *CCS*—certified calibration source

<sup>6</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

- 3.3.6 *DF*—decay factor
- 3.3.7 *FWHM*—full width at half maximum
- 3.3.8 *ICC*—instrument contamination check
- 3.3.9 *ICS*—instrument check source
- 3.3.10 *KV*—known value
- 3.3.11 *LCS*—liquid scintillation counter
- 3.3.12 *MV*—measured value
- 3.3.13 *MQO*—measurement quality objective
- 3.3.14 *NIST*—National Institute of Standards and Technology
- 3.3.15 *NMI*—National Metrology Institute
- 3.3.16 *QC*—quality control
- 3.3.17 *QM*—quality manual
- 3.3.18 *RSD*—relative standard deviation
- 3.3.19 *STS*—sample test source
- 3.3.20 *WCS*—working calibration source

#### 4. Summary of Practice

4.1 This practice summarizes information and guidance for setup, calibration, and quality control for nuclear counting instruments. The procedure is divided into four main sections:

Introduction	Sections 1 through 6
Instrument set-up	Sections 7 through 9
Initial instrument quality control	Sections 10 through 13
Calibration	Sections 14 through 19
Continuing instrument quality control	Sections 20 through 25

4.2 Specific information about setup, calibration, and quality control for the four types of instruments is presented in the sections listed below.

Instrument Type	Setup	Calibration	Quality Control
Gas proportional counter	9.1	16	22
Gamma-ray spectrometer	9.2	17	23
Alpha-particle spectrometer	9.3	18	24
Liquid scintillation counter	9.4	19	25

#### 5. Significance and Use

5.1 This practice is consistent with a performance-based approach wherein the frequency of recalibration and instrument testing is linked to the results from continuing instrument quality control. Under the premise of this practice, a laboratory demonstrates that its instrument performance is acceptable for analyzing sample test sources.

5.2 When a laboratory demonstrates acceptable performance based on continuing instrument quality control data (that is, control charts and tolerance charts), batch QC samples (that is, blanks, laboratory control samples, replicates, matrix spikes, and other batch QC samples as may be applicable) and independent reference materials, traditional schedule-driven instrument recalibration is permissible but unnecessary.

5.3 When continuing instrument QC, batch QC, or independent reference material sample results indicate that instrument response has exceeded established control or tolerance limits, instrument calibration is required. Other actions related to sample analyses on the affected instruments may be required by the laboratory QM.

5.4 The data obtained while following this practice will likely be stored electronically. The data remain in electronic storage, where they are readily available to produce plots, graphs, spreadsheets, and other types of displays and reports. The laboratory QM should specify the frequency and performance of data storage backup.

#### 6. Hazards

6.1 The vendor-supplied safety instructions and laboratory safety regulations should be consulted before using electronic and electrical equipment.

6.2 Corrosive, flammable, reactive, and toxic materials may be used when performing some steps in this practice. Be aware of hazards involved with all materials and processes employed, and comply with any and all applicable health and safety procedures, plans, and regulations. Safety data sheets are a source of information.

### INSTRUMENT SETUP

#### 7. Scope

7.1 Instructions are provided for initial setup of instruments used for activity measurements. These instructions may also be applied when the operating parameters of an instrument are being reestablished.

#### 8. Significance and Use

8.1 Successful setup of an instrument and its subsequent routine use depend, at least in part, on how well the manufacturer's instructions are written and followed. Thus, the manufacturer's recommendations are an integral part of this process. Success also depends on how well the laboratory has planned, developed, and documented its own protocol for instrument use and how well personnel are trained.

#### 9. Instrument Setup Procedures

##### 9.1 Gas Proportional Counter Setup:

9.1.1 Upon initial setup, after major repair or service, or when QC results indicate the need to adjust operating parameters for an instrument, measure a suitable radioactive source as specified in the laboratory QM or manufacturer's protocol to confirm that the instrument responds according to QM or manufacturer's specifications. The instrument setup and calibration records should be maintained per applicable record requirements. ISO/IEC 17025 includes information regarding the type of records to retain.

9.1.2 If the instrument being configured has previously been used to generate sample test source results, the "as-found" instrument settings (that is, operating voltage and discriminator settings) should be recorded and compared to previous "as-left" parameters to ensure that instrument configuration has been maintained. If the instrument configuration has changed, an investigation into the potential impact of the changes shall be conducted and appropriate corrective action taken.

9.1.3 Set the appropriate instrument operating parameters for the intended measurements. For example, acquire voltage plateaus and establish the alpha or beta, or both, plateau operating voltages, and alpha or beta, or both, discriminator



settings (that is, adjust for crosstalk). The instrument configuration should be optimized for the intended applications. For example, when measuring evaporated sample solids deposited in a 50.8 mm (2-inch) diameter planchet, it may be desirable to perform voltage plateaus and optimize discriminator settings using a geometry and radionuclide similar to those that will be used for subsequent measurements (for example, a 50.8 mm diameter <sup>230</sup>Th source rather than a point source containing <sup>210</sup>Po). If setup procedures deviate from those recommended by the manufacturer, the procedures shall be specified in detail in the laboratory QM. Operating parameters should be set to produce consistency in performance across multiple detectors used for a common application. When the instrument operating parameters have been established, record the “as-left” instrument settings for future reference.

### 9.2 Gamma-Ray Spectrometer Setup:

9.2.1 Upon initial setup, after major repair or service, or when QC results indicate the need to adjust operating parameters for an instrument, measure a suitable radioactive source as specified in the laboratory QM or manufacturer’s protocol to confirm that the instrument responds according to QM or manufacturer specifications (for example, full-width-at-half-maximum resolution, peak-to-Compton ratio, and detector efficiency). The instrument setup and calibration records should be maintained per applicable record requirements. ISO/IEC 17025 includes information regarding the types of records to retain.

9.2.2 If the instrument being configured has previously been used to generate sample test source results, the “as-found” instrument settings (that is, detector bias, amplifier gain, analog-to-digital converter (ADC) range, or equivalent digital spectrometer settings) should be recorded and compared to previous “as-left” parameters to ensure that instrument configuration has been maintained. If the instrument configuration has changed, an investigation into the potential impact of the changes shall be conducted and appropriate corrective action taken.

9.2.3 Set the energy range for the spectrometer to include all gamma emission energies of interest to the laboratory. Adjust the amplifier gain, ADC range, or equivalent digital spectrometer settings to produce the desired energy per channel relationship. When the instrument operating parameters are satisfactorily established, record the instrument settings for future reference.

### 9.3 Alpha-Particle Spectrometer Setup:

9.3.1 Upon initial setup, after major repair or service, or when QC results indicate the need to adjust operating parameters for an instrument, measure a suitable radioactive source as specified in the laboratory QM or manufacturer’s protocol to confirm that the instrument responds according to QM or manufacturer’s specifications (for example, bias voltage setting, full-width-at-half-maximum resolution, detector efficiency and background). The instrument setup and calibration records should be maintained per applicable record requirements. ISO/IEC 17025 includes information regarding the type of records to retain.

9.3.2 If the instrument being configured has previously been used to generate sample test source results, the “as-found”

instrument settings (for example, detector bias) should be recorded and compared to previous “as-left” parameters to ensure that instrument configuration has been maintained. If the instrument configuration has changed, an investigation into the potential impact of the changes shall be conducted and appropriate corrective action taken.

9.3.3 Establish the energy range for the spectrometer to include all alpha emission energies of interest to the laboratory. Adjust the amplifier gain and ADC range, or equivalent digital spectrometer settings, to establish the desired energy per channel relationship. When the instrument operating parameters are satisfactorily established, record the instrument settings for future reference.

### 9.4 Liquid Scintillation Counter Setup:

9.4.1 Upon initial setup, after major repair or service, or when QC results indicate the need to adjust operating parameters for an instrument, measure a suitable radioactive source as specified in the laboratory QM or manufacturer’s protocol to confirm that the instrument responds according to QM or manufacturer’s specifications (for example, detector efficiency, background for region of interest for beta or alpha applications). The instrument setup and calibration records should be maintained per applicable record requirements. ISO/IEC 17025 includes information regarding the type of records to retain.

9.4.2 If the instrument being configured has previously been used to generate sample test source results, the “as-found” instrument settings (for example, counting channels or energy windows) should be recorded and compared to previous “as-left” parameters to ensure that instrument configuration has been maintained. If the instrument configuration has changed, an investigation into the potential impact of the changes shall be conducted and appropriate corrective action taken.

9.4.3 Set the instrument operating parameters for the intended measurements according to the manufacturer’s recommendations. For example, establish the photomultiplier operating voltage, discriminator settings, and energy-range windows as applicable to the measurements to be performed. When the instrument operating parameters are satisfactorily established, record the instrument settings for future reference.

## INITIAL INSTRUMENT QUALITY CONTROL

### 10. Scope

10.1 Quality control should be initiated before or during instrument calibration to confirm the instrument’s operability and stability and to establish the continuing quality control parameters. The purpose of the instrument quality control is to verify that the instrument’s metrological characteristics are (1) acceptable for analysis of sample test sources and (2) equivalent to those that existed during calibration. Continuing instrument quality control results are compared to control limits or tolerance limits or are evaluated by other statistical tests to establish acceptability. Instrument quality control uses performance checks that include, but are not limited to, background stability, detector response (count rate) reproducibility with a known ICS, gain stability, and peak resolution stability, as appropriate to each type of instrument.

## 11. Significance and Use

11.1 Guidance is provided in this section for establishing the manner in which instrument performance parameters shall be monitored. These performance parameters should be established prior to or concurrent with counting calibration samples and shall be established before counting sample test sources. Two primary tools for monitoring instrument performance are the control chart and the tolerance chart. Instrument quality control protocols shall be clearly defined in the laboratory QM.

11.2 Instrument control charts are used to monitor those continuing instrument performance parameters where statistical control is necessary to ensure the quality of the reported sample test source result. For those performance parameters where statistical control is not necessary but where exceeding a threshold value may impact the quality or usability of sample test source results, a tolerance chart may be used. The laboratory QM shall indicate the appropriate tool, control chart or tolerance chart, for monitoring each performance parameter.

11.3 The limits for any chart that is used to test for changes in a calibrated parameter, such as counting efficiency or gain, should be established at the time of calibration. The limits should not be changed afterwards except for decay correction when appropriate, or as described in 12.1.5, 12.2, and 12.3, unless the calibrations are repeated.

11.4 Instrument QC is linked to measurement uncertainty. (1) Any assumptions made about the instrument's performance for QC purposes, such as assumptions about counting statistics, variability of backgrounds, efficiencies, or reproducibility of source placement, should be consistent with those made when evaluating measurement uncertainties. (2) The rigor of the QC regimen should be appropriate for the required uncertainty of sample measurements or other measurement quality objectives (MQOs). For example, the choice of control charts or tolerance charts may be based partly on the uncertainty requirements. (3) Instrument QC provides a large body of data that may often be used to evaluate uncertainty components that might otherwise be difficult to estimate—for example, variability of backgrounds or efficiencies.

## 12. Establishing the Control Chart

12.1 Using the appropriate ICS or ICC, perform at least 7 measurements of the performance parameter to be monitored, ensuring that the measurement conditions are reproducible and match the sample analysis conditions as closely as possible. These measurements may be performed sequentially over a short period of time but should span at least a 24 h period. In each case, the ICS or ICC should be removed from the instrument between measurements and re-inserted so that the control chart reflects variability in sample positioning.

12.1.1 For each instrument performance parameter that uses a radioactive source, accumulate sufficient net counts to obtain a relative standard counting uncertainty < 1 % (10 000 net counts minimum). Since a single instrument can be used for many different tests, the ICS used to measure detector response may be dissimilar to calibration sources (for example, <sup>99</sup>Tc source for gas proportional counting units, unquenched tritium for a liquid scintillation counter, or a multi-nuclide point source for gamma spectrometry systems).

12.1.2 Like the ICS, the ICC does not reflect every counting configuration on an instrument used for different tests. It should be configured, however, to ensure effective identification of gross contamination of the instrument.

12.1.3 The BSC must be closely matched to its associated sample test source configuration to ensure that the measurements used for background subtraction accurately reflect conditions when counting sample test sources. The BSC is counted to determine the value to use for subtraction from the sample. The BSC should be counted as long as or longer than the longest sample test source count. Although the BSC and ICC may be counted in the same test source configuration for the same length of time, the ICC is a holder for the sample test source that is free of the analyte (that is, empty planchet for gas proportional counting or a sample holder with a filter for alpha spectrometry or an empty chamber or Marinelli beaker for gamma spectrometry), which is counted for a shorter time than the BSC. The laboratory's QM shall specify the frequency and protocol for the ICC and BSC.

12.1.4 Radioactive isotopes in the container or sample mounting materials may contribute to the overall method background and must be accounted for to ensure accurate background correction.

12.1.5 The false-alarm rates for control charts can vary significantly if the control limits are based on small data sets. If the laboratory has a large number of such control charts, even if all the instruments are equally stable, it will likely seem that some charts remain consistently in control while others go out of control frequently. For this reason, if the initial data set is small, the limits should be updated when more data points are available. Such an update should be performed at most once per chart and as soon as practical after the required number of points are obtained.

12.1.6 Although some QC software systems provide options for continually updating control limits, these options should not be used when monitoring calibrated parameters, since doing so could allow instrument performance to drift after calibration without ever triggering an alarm.

12.2 Calculate the mean and standard deviation of the measured parameter using equations appropriate for the expected type of distribution. For example, if the counting statistics are believed to be approximately Poisson and the parameter is based on a radionuclide that will decay measurably during the life of the chart, calculate a mean decay-corrected count,  $\hat{C}$ , and estimate the mean,  $\hat{\mu}_C$ , and standard deviation,  $\hat{\sigma}_C$ , for a future measurement of the same source as follows.

$$\hat{C} = \frac{\sum_{i=1}^n C_i}{\sum_{i=1}^n DF_i} \quad (1)$$

$$\hat{\mu}_C = \hat{C} \cdot DF \quad (2)$$

$$\hat{\sigma}_C = \sqrt{\hat{\mu}_C + (\xi_r \hat{\mu}_C)^2} \quad (3)$$

where:

$\hat{C}$  = estimated mean decay-corrected count,

- $n$  = number of measurements used to set up the chart,
- $C_i$  = observed count during the  $i$ th measurement,
- $DF_i$  = decay factor for the  $i$ th measurement,
- $\hat{\mu}_C$  = estimated mean count for the future measurement,
- $DF$  = decay factor for the future measurement,
- $\hat{\sigma}_C$  = estimated standard deviation for the future measurement, and
- $\zeta_r$  = tolerable additional non-Poisson relative standard deviation (consistent with the uncertainty model for sample measurements—may be zero).

12.2.1 If the initial limits are based on fewer than 15 (preferably 20) measurements, update the limits when 15 (or 20) data points have been obtained.

12.3 If Poisson statistics cannot be assumed, one may estimate the mean and standard deviation as follows.

$$\hat{\mu}_C = \bar{C} = \frac{1}{n} \sum_{i=1}^n C_i \quad (4)$$

$$\hat{\sigma}_C = \frac{n - 0.75}{n - 1} \sqrt{\frac{1}{n - 1} \sum_{i=1}^n (C_i - \bar{C})^2} \quad (5)$$

12.3.1 If the initial limits are based on fewer than 20 (preferably 30) measurements, update the limits when 20 (or 30) data points have been obtained.

12.4 An alternative estimator for the standard deviation is given by:

$$\hat{\sigma}_C = \frac{\sqrt{\pi}}{2(n - 1)} \sum_{i=1}^{n-1} |C_{i+1} - C_i| \quad (6)$$

12.4.1 The estimator given by Eq 6 is somewhat less sensitive to outliers than the one given by Eq 5. For normally distributed data without outliers, Eq 5 tends to outperform Eq 6.

12.5 Create a control chart with the observed result on the vertical axis and the observation number or date on the horizontal axis. Draw a horizontal line or a sloping (decay-corrected) curve on the chart to represent the predicted mean of the observed values. Draw lines or curves for the control limits at three standard deviations above and below the mean. Additional lines or curves for “warning limits” should also be drawn, typically at two standard deviations above and below the mean. The quality control data should be evaluated to check that they follow the expected distribution—for example, Poisson or normal—and that there are no outliers. Appendix X6 describes procedures that may be used to test the assumption of Poisson counting statistics. Reference (1) includes a discussion for pursuing root-cause analysis of excursions (departures from the expected condition). Practices D3648 and Reference (1), Chapter 18, present information on the preparation and interpretation of control charts.

12.6 Many instruments are provided with operation and analysis software that may include performance check and QC charting capabilities. Standalone charting software may also be used. It is not necessary that the software use exactly the same terminology or graphical features. However, if software is to be used for continuing instrument quality control, it must support the statistical evaluation of the necessary performance param-

eters and be able to compare individual observations with the established warning and control limits and advise the operator of performance warnings and failures. The software must be documented as specified in the laboratory QM.

### 13. Instrument Tolerance Charts

13.1 The purpose of tolerance charts is to compare observed instrument performance to acceptable performance limits. A tolerance may be expressed as a percent (%) deviation of an observed parameter from a nominal value, which might be an estimated mean, calibrated value, or other assumed target value. There may be different tolerances for values above and below the nominal value. The basis for the tolerances may also be taken from the MQOs associated with a project or statement of work.

13.2 Tolerance limits differ from control limits in that they are not based on statistical measures, but instead are based on acceptance criteria appropriate to the method and scope of work. (The QM shall define the basis and manner by which tolerance limits are established for each performance parameter). A tolerance chart, similar to a control chart, is a graphical tool that can be used to evaluate instrument performance and trending of instrument parameters. In Reference (1), Chapter 18, several examples are given for the use of tolerance limits, one of which is monitoring the resolution of a high-purity germanium detector. In addition, it may be appropriate to establish “warning limits” when using a tolerance chart to ensure appropriate actions are taken before a tolerance limit is crossed.

13.3 For each performance parameter to be charted, establish the tolerance limits. The tolerance limits should be selected so that operation of the instrument just within the limits will not adversely affect the performance of the test or method. Account for radioactive decay as appropriate when evaluating parameters based on short-lived radionuclides.

13.4 Perform a statistical analysis of a series of observations of the parameter to ensure that the tolerance limits are achievable. If the standard deviation of the observed values exceeds one-third of the required tolerance, either improve the measurement precision to an acceptable level, or reconsider the size of the tolerance itself. The consequence of not doing so would be an excessive frequency of out-of-tolerance situations.

13.5 Create a tolerance chart with the observed result on the vertical axis and the observation number or date on the horizontal axis. Draw a horizontal line on the chart to represent the nominal value of the observed parameter, and draw horizontal lines for the tolerance limits above and below the nominal value. It can also be informative to draw horizontal lines for the 3-sigma statistical control limits, although these 3-sigma limits will not be used to accept or reject observed parameter values. The 3-sigma limits may be used instead to provide early warnings of trends that might eventually impact data quality.

13.6 Many instruments are provided with operation and analysis software which may include performance check capabilities. It is not necessary that the software use exactly the same terminology or graphical features. However, if the



software is to be used for continuing instrument tolerance checks, it must be able to compare individual observations to the established tolerance limits and indicate out-of-tolerance conditions. Standalone charting software can also be used for this purpose. The software must be documented as specified in the laboratory QM.

## CALIBRATION

### 14. Scope

14.1 The calibration process establishes the response of an instrument to calibration sources. The calibration sources shall have values (with uncertainties) that are traceable to the SI via a national metrology institute. When working calibration sources are used, they shall be prepared from certified SI-traceable radionuclide standards.

### 15. Significance and Use

15.1 Calibration of a gas proportional counter, gamma spectrometer, alpha spectrometer, and liquid scintillation counter is addressed in the following sections.

15.2 Consult Practices **D3648** for information regarding the use of instruments for performing radioanalytical measurements.

15.3 Efficiency calibration acceptance criteria are provided in this practice for gas proportional counting, gamma spectrometry, alpha spectrometry, and liquid scintillation counting instruments. Achievement of performance like that specified in standards such as ANSI N42.23, ANSI/HPS N13.30, and References **(1)** and **(2)** is more likely when the calibration acceptance criteria in this practice are met or exceeded.

### 16. Gas Proportional Counter Instrument Calibrations

16.1 Refer to the guidance in Sections **7** to **13** for counting the ICS and ICC at instrument setup in preparation for calibration. For those instruments already in use, count the ICS and ICC samples as prescribed in Section **22**.

16.2 *Single-Point Efficiency or Constant Test Mass for a Specific Radionuclide:*

16.2.1 Instructions for a single-point efficiency calibration of a gas proportional counter are provided below. A single-point efficiency is used when the efficiency change is negligible over the expected mass range for the test.

16.2.2 The guidance below assumes the use of working calibration sources (WCSs). To control possible bias due to non-representative calibration sources, the preparation method of the WCSs should produce sources that are as equivalent as practicable to the sample test sources. Since the preparation typically involves chemical procedures, with opportunities for loss of analyte, it is essential that the procedure be designed and performed carefully to ensure its quantitative nature and to preserve traceability to the SI. WCSs shall be prepared from certified SI-traceable radionuclide standards.

16.2.3 A minimum of three WCSs (or one CCS) shall be used.

16.2.4 A blank sample shall also be processed in association with the WCSs. The blank sample result should be compared to the performance criteria stated in the laboratory's QM.

16.2.5 The activity of each WCS should be selected to produce a count rate not exceeding 5000 counts per second ( $s^{-1}$ ). It is essential that the count rate of the WCS be low enough to avoid instrument dead time that would result in lost counts.

NOTE 1—The limitation of 5000 counts per second ( $s^{-1}$ ) was based on typical usage and may vary according to instrument type and manufacturer. Users should consult the manufacturer's specifications.

16.2.6 The laboratory QM shall state the uncertainty requirements for the measurement. The WCS should be counted long enough to obtain a relative standard counting uncertainty < 1 % (10 000 net counts minimum).

16.2.7 Correct the WCS activity for radioactive decay (from the reference time to the time of the measurement). Calculate the counting efficiency,  $\epsilon_{WCS}$ , using the equation defined in the laboratory QM or with example **Eq 7**.

$$\epsilon_{WCS} = \frac{R_a - R_b}{A_{WCS} \cdot Y_{WCS} \cdot DF} \quad (7)$$

where:

- $\epsilon_{WCS}$  = single point efficiency of WCS (counts per second per becquerel ( $s^{-1} \text{ Bq}^{-1}$ ),
- $R_a$  = count rate ( $s^{-1}$ ) of WCS,
- $R_b$  = count rate ( $s^{-1}$ ) of instrument background,
- $A_{WCS}$  = activity (Bq) of the WCS at the reference date and time of the calibration source,
- $Y_{WCS}$  = chemical yield of the WCS, if applicable,
- $DF$  = decay factor for the calibrating radionuclide  $e^{-\lambda(t_1 - t_0)}$ ,
- $\lambda$  =  $(\ln 2) / T_{1/2}$ , where  $T_{1/2}$  denotes the half-life of calibrating radionuclide (half-life units must match those used for the difference  $t_1 - t_0$ ),
- $t_0$  = reference date and time of the calibrating radionuclide activity value, and
- $t_1$  = start of WCS count (date and time).

16.2.7.1 **Eq 7** accounts for the total efficiency of the radionuclide even when the probability of alpha or beta emission per decay is less than 1.0 (less than 100 %).

16.2.7.2 Calculate the combined standard uncertainty  $u_c(\epsilon_{WCS})$ , using the equation defined in the laboratory QM or with example **Eq 9**.

$$u_c(\epsilon_{WCS}) = \left[ \frac{R_a / t_a + R_b / t_b}{(A_{WCS} \cdot Y_{WCS} \cdot DF)^2} + \epsilon_{WCS}^2 \left( \frac{u^2(A_{WCS})}{A_{WCS}^2} + \frac{u^2(Y_{WCS})}{Y_{WCS}^2} + \varphi_G^2 \right) \right]^{1/2} \quad (8)$$

where:

- $u_c(\epsilon_{WCS})$  = the combined standard uncertainty of the single point efficiency  $\epsilon_{WCS}$ ,
- $t_a$  = duration of count for WCS,
- $t_b$  = duration of count for the background,
- $u(A_{WCS})$  = the standard uncertainty of  $A_{WCS}$ ,
- $u(Y_{WCS})$  = the standard uncertainty of  $Y_{WCS}$ , and
- $\varphi_G$  = relative standard deviation of the efficiency due to source-to-source variability.

NOTE 2—The other symbols are as defined for Eq 7.

16.2.7.3 Correction for decay during counting may be made by multiplying  $DF$  by the value,  $DF_a$ , obtained using Eq 10.

$$DF_a = \frac{1 - e^{-\lambda t_a}}{\lambda t_a} = e^{-\lambda t_a/2} \frac{\sinh(\lambda t_a/2)}{\lambda t_a/2} \quad (9)$$

where:

$\lambda$  = decay constant of the radionuclide, and  
 $t_a$  = duration of count.

NOTE 3—The two expressions above for  $DF_a$  are theoretically equivalent; however, the second expression involving the hyperbolic sine function,  $\sinh$ , should give more accurate floating-point results when  $\lambda t_a$  is very small, in which case  $DF_a$  is also approximated very well by the simpler factor  $e^{-\lambda t_a/2}$ .

16.2.8 See Appendix X5 for guidance on the calculation of a weighted average and its uncertainty, and for assessing the fit of the calibration data. The total calibration uncertainty shall be included in the combined standard uncertainty of each sample result.

16.2.9 Verify the single-point efficiency calibration before use by analyzing one sample that contains the same radionuclide prepared from a second certified SI-traceable standard. If obtaining a second certified standard is impractical, a separate dilution of the original radionuclide standard shall be used, and this fact shall be documented appropriately. The laboratory QM shall state the uncertainty requirements for the verification measurement. See 16.2.5 and 16.2.6 for additional limits on count rate and counting uncertainty.

16.2.10 A blank sample should be analyzed with the verification sample. Compare the blank sample result to the performance criteria stated in the laboratory QM.

16.2.11 Calculate the verification sample activity,  $MV$ , using the equation defined in the laboratory QM or with example Eq 11.

$$MV = \frac{R_a - R_b}{\varepsilon \cdot Y \cdot DF} \quad (10)$$

where:

$MV$  = measured value (Bq) of the verification sample,  
 $R_a$  = count rate ( $s^{-1}$ ) of verification sample,  
 $R_b$  = count rate ( $s^{-1}$ ) of instrument background, (the net count rate of the blank sample should be subtracted also if it is significant when evaluated according to the laboratory's performance criteria),  
 $\varepsilon$  = detection efficiency (see Eq 7 and 16.2.7),  
 $Y$  = chemical yield of the verification sample, if applicable,  
 $DF$  = decay factor for the calibrating radionuclide  $e^{-\lambda(t_1 - t_0)}$ ,  
 $\lambda$  =  $(\ln 2)/T_{1/2}$ , where  $T_{1/2}$  denotes the half-life of calibrating radionuclide (half-life units must match those used for the difference  $t_1 - t_0$ ),  
 $t_0$  = reference date and time of the calibrating radionuclide activity value, and  
 $t_1$  = start of verification sample count (date and time).

16.2.11.1 To correct for decay during counting, refer to Eq 10.

16.2.12 Calculate the combined standard uncertainty  $u_c(MV)$ , using the equation defined in the laboratory QM or with example Eq 12.

$$u_c(MV) = \sqrt{\frac{R_a/t_a + R_b/t_b}{(\varepsilon \cdot Y \cdot DF)^2} + MV^2 \times \left( \frac{u^2(\varepsilon)}{\varepsilon^2} + \frac{u^2(Y)}{Y^2} \right)} \quad (11)$$

where:

$u_c(MV)$  = the combined standard uncertainty of the measured value, in Bq,  
 $t_a$  = duration of count for the verification sample,  
 $t_b$  = duration of count for the background,  
 $u(\varepsilon)$  = the standard uncertainty of  $\varepsilon$ , and  
 $u(Y)$  = the standard uncertainty of  $Y$ .

NOTE 4—The other symbols are as defined for Eq 11.

16.2.12.1 The uncertainty components included in Eq 12 are expected to be potentially significant. Other components such as those due to WCS preparation, reagent preparation, and radionuclide half-life should be included whenever they are considered significant. Furthermore, it is recommended that the user evaluate the possibility that there may be correlations between some input estimates, which affect the combined standard uncertainty. For additional information on the evaluation and expression of measurement uncertainty refer to JCGM 100:2008 or Reference (3).

16.2.13 Calculate the known value,  $KV$ , using the equation defined in the laboratory QM or with Eq 13.

$$KV = AC \cdot V \quad (12)$$

where:

$KV$  = known value of the activity added to the verification sample,  
 $AC$  = activity concentration in becquerels per litre (Bq/L) of the radioactive reference material used to prepare the verification sample (or massic activity in becquerels per gram), and  
 $V$  = volume (or mass) of the reference material used.

16.2.14 Calculate the combined standard uncertainty,  $u_c(KV)$  using the equation defined in the laboratory QM or with Eq 14.

$$u_c(KV) = \sqrt{V^2 u^2(AC) + AC^2 u^2(V)} \quad (13)$$

where:

$u_c(KV)$  = combined standard uncertainty of the activity (Bq) added to the verification sample ( $KV$ ),  
 $u(AC)$  = standard uncertainty of the activity concentration of the radioactive reference material used to prepare the verification sample, in becquerels per litre (Bq/L), and  
 $u(V)$  = standard uncertainty of the volume of the reference material used.

NOTE 5—The other symbols are as defined in Eq 13.

16.2.14.1 Refer to the statement on uncertainty in 16.2.12.1 after Eq 12.

16.2.15 The calculated (measured) value of this sample should agree with the known value of the sample within the uncertainty of the known and the uncertainty of the sample (including the calibration uncertainty) using Eq 15, the acceptable verification ratio ( $AVR$ ):

$$AVR = \frac{|KV - MV|}{\sqrt{u_c^2(KV) + u_c^2(MV)}} \leq 2.0 \quad (14)$$



where:

- $KV$  = known value of the activity added to the verification sample,  
 $MV$  = measured value of the verification sample as calculated with Eq 11,  
 $u_c(KV)$  = combined standard uncertainty of the known value, and  
 $u_c(MV)$  = combined standard uncertainty of the measured value.

NOTE 6—This equation is similar to the one used in Reference (1), Chapter 18, to assess results from laboratory control samples. A “z” value of 2 is typical; however other “z” values may be used.

16.2.15.1 Refer to the statement on uncertainty in 16.2.12.1 after Eq 12. If the value of AVR exceeds 2.0, the system should be recalibrated and the test repeated.

16.2.16 When the criteria defined in 16.2.8 and 16.2.15 are met, the calibration is acceptable. The efficiency ( $\epsilon_{WCS}$ ) determined in 16.2.7 is used for calculating sample test source results.

### 16.3 Calibration for Varying Sample Test Source Residue Masses:

16.3.1 To control bias due to non-representative calibration sources, the preparation method should produce WCSs that are as equivalent as practicable to the sample test sources. Since chemical processes are nearly always used to prepare calibration sources, it is essential that the process be carried out very carefully to ensure its quantitative nature and that measurements preserve traceability to the appropriate national standard.

16.3.2 If alpha and beta measurements are to be performed simultaneously, prepare separate sets of calibration sources for the alpha and beta calibrations.

16.3.3 Use a set of at least seven replicate pairs of working calibration sources prepared with targeted net residue (or precipitate) masses that bracket the expected range of masses for sample test sources. One pair should be near each end of the mass range, but not zero.

16.3.4 Select the activity of each WCS to produce a count rate not to exceed 5000  $s^{-1}$ .

NOTE 7—The count rate of the WCS should be low enough to avoid instrument dead time that would result in lost counts. The limitation of 5000 counts per second is a typical value and may vary according to instrument type and manufacturer. Users should consult the manufacturer’s specifications.

16.3.5 Consult the laboratory QM for chemical yield requirements. If the yield for a WCS fails to meet the requirements, remove that WCS from the set. If both members of a replicate pair are removed, restart the calibration with new WCSs. Otherwise, the calibration may continue with the remaining WCSs.

16.3.6 The laboratory QM shall state the uncertainty requirements for each WCS measurement. Typically, the relative standard counting uncertainty should not exceed 1 % (10 000 net counts minimum). Count each WCS long enough to meet the stated uncertainty requirements.

16.3.7 Correct the WCS activity value for radioactive decay (from the reference time to the time of the measurement).

Calculate the individual WCS efficiencies ( $\epsilon_{WCS}$ ) using the equation defined in the laboratory QM or with example Eq 16.

$$\epsilon_{WCS} = \frac{R_a - R_b}{A_{WCS} \cdot Y_{WCS} \cdot DF} \quad (15)$$

where:

- $\epsilon_{WCS}$  = single-point measured efficiency of the WCS ( $s^{-1} Bq^{-1}$ ),  
 $R_a$  = count rate ( $s^{-1}$ ) of WCS,  
 $R_b$  = count rate ( $s^{-1}$ ) of instrument background,  
 $A_{WCS}$  = activity (Bq) of the WCS at the reference date and time of the calibration source,  
 $Y_{WCS}$  = chemical yield of the WCS, if appropriate,  
 $DF$  = decay factor for the calibrating radionuclide,  $e^{-\lambda(t_1 - t_0)}$ ,  
 $\lambda$  =  $(\ln 2) / T_{1/2}$ , where  $T_{1/2}$  denotes the half-life of calibrating radionuclide (half-life units must match those used for the difference  $t_1 - t_0$ ),  
 $t_0$  = reference date and time of the calibrating radionuclide activity value, and  
 $t_1$  = start of WCS count (date and time).

16.3.7.1 To correct for decay during counting, refer to Eq 10.

16.3.7.2 Eq 16 accounts for the total efficiency of the radionuclide even when the probability of alpha or beta emission per decay is less than 1.0 (less than 100 %).

16.3.8 Plot the measured WCS efficiencies against their residue masses.

NOTE 8—The calibration curve, (also referred to as an attenuation or absorption curve) should have a general downward trend when the dependent variable  $\epsilon$  (the efficiency) is plotted against the independent variable  $X$  (the test source residue mass). Thus, the detector efficiency should decrease as the test source residue mass increases. The slope of the curve will depend on the alpha or beta energy, and for a high-energy beta such as that from  $^{90}Y$ , it may be flat or nearly so but still non-increasing. The efficiency in the alternate channel (that is, crosstalk) should also have a general downward trend with increasing residue mass. Several physical factors may be involved as a radioactive particle passes through the test source mass and into the detector. See References (4) and (5) for additional information.

16.3.9 Several mathematical models or equations might be used for the efficiency curve, including polynomials and power functions such as  $am^b$ ,  $a/(b + m)^c$ ,  $ae^{-bm}$  and  $(\ln m)^a$ , where  $m$  is the numerical value of the sample mass, and  $a$ ,  $b$ , and  $c$  are calibration parameters. In the case of the polynomial model, the degree of the polynomial should not exceed three and the number of discrete masses (not data pairs) used to generate the curve must be at least two more than the degree of the polynomial.

16.3.10 The laboratory should assess and document any calibration model before use. Typically, at most a few models are provided as options by off-the-shelf analysis software. If the laboratory implements its own models, significant effort may be required to implement a new model fully, including the uncertainty analysis of the parameters and assessment of the data for outliers and lack of fit as described in Appendix X5.

16.3.11 Choose a mathematical model of the calibration from the available options. Use the appropriate fitting technique (such as ordinary or weighted least squares) with the model and the measured efficiencies to solve for the values of

the curve parameters and for the associated covariance matrix, which provides the squared uncertainties of the parameters and their covariances.

16.3.12 After fitting the curve, evaluate the residual for each data point. The residual,  $e_i$ , equals the difference between the measured efficiency,  $\varepsilon_i$ , and the predicted efficiency,  $\hat{\varepsilon}_i$ , calculated from the curve at the same residue mass. A variety of software packages, including basic spreadsheet applications, can determine the calibration parameters that optimize the residuals according to some criteria. These parameter values may or may not minimize the overall uncertainty associated with the calibration. A fitting procedure using generalized linear or nonlinear least squares should provide optimal results provided that the uncertainties and correlation coefficients of the measured efficiencies are estimated properly.

16.3.13 Evaluate the agreement of the fitted curve with the measured values and ensure that it is appropriate to the residue mass range of the sample test sources. See [Appendix X5](#) for guidance on assessing the fit. A single data point identified as an outlier may be excluded from the data set, although removal of a high or low mass point will restrict the usable range of the curve to that bracketed by the retained working calibration standards. Decisions to exclude outliers must be documented. After removing an outlier, the analyst must repeat the fitting procedure using the remaining data points and test the new fit against the same acceptance criteria as described above. If multiple data points are identified as outliers, the calibration should be stopped and the protocol evaluated before the process is resumed or restarted.

16.3.13.1 The loss of one replicate of a pair should not be cause for rejecting the calibration.

16.3.14 Assess the overall form of the curve in terms of height, slope, and curvature. Efficiencies should decrease with increasing mass, although the decrease may be so slight that the curve appears flat. The presence of multiple inflection points (points where the curvature changes between upward and downward) may indicate problems with either the data or the model.

**NOTE 9**—Multiple inflection points cannot occur with some models, including simple exponential or power functions and polynomials of degree 3 or less.

16.3.15 If acceptable results are not obtained using the chosen calibration model, a different model may be selected, starting at [16.3.11](#). One may also try all the available models and select the one that provides the best results by the criteria described in [Appendix X5](#).

16.3.16 If all data points meet the documented acceptance criteria, the analyst may proceed to [16.3.17](#).

16.3.17 Whatever fitting technique is used to solve for the curve parameters, it should provide an estimated covariance matrix for the solution. The parameter uncertainties and covariances from this covariance matrix are then combined with the uncertainties of any additional fixed factors that are common to all the measured efficiencies and that are not accounted for in the covariance matrix (for example, the massic or volumic activity of the calibration reference material) to obtain the total combined standard uncertainties and covariances of the parameters. These total uncertainties and

covariances shall be propagated and included in the combined standard uncertainty of each sample test source result.

16.3.18 Verify the calibration curve by analyzing three verification samples that contain the same radionuclide from a second certified SI-traceable standard. Include a blank sample with the verification samples. If obtaining a second certified standard is impractical, the calibration curve shall be verified by analyzing three verification samples that contain the same radionuclide from another dilution of the original standard. The residue masses of the verification samples should be distributed across the mass range of the efficiency curve. The laboratory QM shall state the uncertainty requirements for each verification measurement.

16.3.19 Measure the verification samples according to [16.3.6](#). The measured value should fall within the quality control limits as established in the laboratory QM. The AVR test, [Eq 15](#) in [16.2.15](#), may also be used.

16.3.20 After the verification requirements described in [16.3.18](#) and [16.3.19](#) are met, the calibration may be approved and made available for calculation of sample test source results.

**NOTE 10**—An example of a gas proportional counter calibration is provided in [Appendix X1](#).

16.4 A discussion of crosstalk is found in [Appendix X2](#).

## 17. Gamma-Ray Spectrometry Instrument Calibrations

17.1 Refer to the guidance in Sections [7](#) to [13](#) for counting the ICS and ICC at instrument setup in preparation for calibration. For instruments already in use, count the ICS and ICC as prescribed in Section [23](#).

17.2 Calibration sources are commercially available with radionuclides that typically provide at least 10 calibration data points. The gamma-ray energy range in a CCS is typically 50 keV to 2000 keV. Select the CCS activity to produce a count rate not to exceed  $2000\text{ s}^{-1}$ . To control bias due to non-representative calibration sources, the CCS geometry should be as equivalent as practicable to that of sample test sources.

17.2.1 The density and average Z number of a CCS can have an effect on the detection efficiency, especially at energies  $< \sim 100\text{ keV}$ .

**NOTE 11**—The count rate of the CCS should be low enough to avoid instrument dead time (typically  $< 5\%$ ) that would result in lost counts. The limitation of 2000 counts per second is a typical value and may vary according to instrument type and manufacturer. Users should consult the manufacturer's specifications.

**NOTE 12**—A WCS may also be used instead of a CCS.

17.3 Accumulate an energy spectrum using sealed, calibrated activity sources (that is, CCS or WCS) traceable to the SI, in an appropriate and reproducible counting geometry. Accumulate sufficient net counts (total counts minus the Compton) in each full-energy gamma-ray peak of interest to obtain a relative standard counting uncertainty  $< 1\%$  (10 000 net counts minimum). The gamma spectrometry calibration includes energy, resolution and efficiency calibration. It assumes the detection and reporting of several gamma-ray-emitting radionuclides. The calibration procedure may be modified for single radionuclide calibration and measurement.

17.4 Using the gamma emission data from the CCS and the peak location data from the calibration spectrum, establish the energy-per-channel relationship (energy calibration) as:

$$En = Offset + Ch \times Slope \quad (16)$$

where:

- $En$  = peak energy (keV),
- $Offset$  = energy offset for the energy calibration equation (keV),
- $Ch$  = peak location channel number, and
- $Slope$  = energy calibration equation slope (keV/channel).

17.4.1 Most modern spectrometry software packages perform this calculation, sometimes including higher-order terms to account for minor nonlinearity in the energy calibration.

17.5 Using the gamma emission data from the CCS and the peak resolution data from the calibration spectrum, establish the resolution-versus-energy relationship (resolution calibration) as:

$$FWHM = Offset + En \times Slope \quad (17)$$

where:

- $FWHM$  = full width of the peak at one-half the maximum counts in the centroid channel (keV),
- $Offset$  = width offset for the resolution calibration equation (keV),
- $En$  = peak energy (keV), and
- $Slope$  = resolution calibration equation slope (keV/keV).

17.5.1 Most modern spectrometry software packages perform this calculation, sometimes including higher-order terms to account for minor nonlinearity in the resolution calibration.

17.6 Calculate the full-energy peak efficiency,  $\epsilon_f$ , using the equation defined in the laboratory QM or with example Eq 20:

$$\epsilon_f = \frac{R_n}{A_{CS} \cdot I_\gamma \cdot DF} \quad (18)$$

where:

- $\epsilon_f$  = full-energy peak efficiency (counts per gamma ray emitted),
- $R_n$  = net gamma-ray count rate ( $s^{-1}$ ) in the full-energy peak of interest,
- $A_{CS}$  = activity (Bq) of the calibration source at its reference date and time,
- $I_\gamma$  = probability per nuclear decay for the gamma ray,
- $DF$  = decay factor for the calibrating radionuclide,  $e^{-\lambda(t_1 - t_0)}$ ,
- $\lambda$  = half-life of calibrating radionuclide (half-life units must match those used for the difference  $t_1 - t_0$ ),
- $t_0$  = reference date and time of the calibrating radionuclide activity value, and
- $t_1$  = start of CCS count (date and time).

17.6.1 To correct for decay during counting refer to Eq 10.

17.6.2 *Summing:*

17.6.2.1 The calibration and subsequent measurements may be subject to small errors as a consequence of events referred to as “summing.” Summing occurs when the energy from two or more gamma rays is collected within the resolving time of the detector system, and thus the apparent resulting full-energy peak(s) are in fact the sum of the combined energies of the

gamma rays. Summing effects are typically addressed in two separate categories: “random” and “true coincidence” or “cascade.”

17.6.2.2 The random summing effects are dependent only on the total gamma emission rate and proximity to the detector; at higher detector system observation rates (count rates), the random summing is more pronounced. The effect is due to the probability that any two or more gamma rays emitted randomly from the sample test source may be collected simultaneously. Random summing effects can be calibrated for and corrected by making empirical measurements of sources with differing source strengths in the same counting geometry. However, it is generally preferable to reduce random summing by positioning the source or sample to lower the count rate (for example, to less than 2000  $s^{-1}$ ).

17.6.2.3 True coincidence or cascade summing is a consequence of the decay scheme of specific radionuclides. There are numerous radionuclides whose decay schemes provide for near-simultaneous emission of two or more gamma rays, which have some probability of being collected together. A common example of this is  $^{134}\text{Cs}$ , with simultaneous emissions of gamma rays at approximately 605 and 796 keV. When both of these gamma rays are collected by the detection system at the same time, the apparent resulting full-energy peak is seen at 1401 keV. This effect is not count-rate dependent; rather, it is geometry dependent since the proximity to the detector and solid angle from the source to the detector crystal affect the simple geometric probability of two or more gamma rays interacting with the detector volume.

17.6.2.4 True coincidence or cascade summing can be calibrated for and corrected empirically on a nuclide-by-nuclide basis by making direct observations of all of the full-energy peaks and combinations of possible sum peaks, then applying a manual correction to each full-energy peak in the source or sample. Alternatively, some nuclear instrument manufacturers offer gamma-ray analysis software with cascade-summing calibration and correction features.

17.6.2.5 It should be noted that summing has both a “summing-in” and “summing-out” effect, whereby summing-in (simply called “sum”) peaks are created by the simultaneous collection of two or more gamma-ray energies, while the summing-out effect accounts for the observations lost from the individual full-energy peaks. Using the  $^{134}\text{Cs}$  example above, each count in the 1401 keV (summing-in or sum) peak would mean one less count in each of the 605 and 796 keV peaks (summing-out).

17.7 Most modern spectrometry systems are computerized, and the determination of the gamma-ray efficiencies is performed automatically at the end of an appropriate counting interval. Refer to the manufacturer’s manuals for specific instructions.

17.8 Mathematical modeling may be used for efficiency determinations and for estimating changes to established efficiency curves when there are slight changes in sample test source geometry. The user is responsible for validating the model. The total uncertainty of the efficiency shall include any uncertainty due to modeling.